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fax: +48 61 822 43 72
e-mail: publication@itd.poznan.pl

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Dear Readers,

Forest management and wood management, which is closely related to it, are extremely complex areas and are continually developing. Therefore there is a need for multidisciplinary research to be conducted, which considers issues from the point of view of the whole wood chain. In addition, it is essential that there is a speedy exchange of knowledge in this field.

Striving to fulfil these expectations, we are publishing a special issue of “DREWNO” containing scientific articles prepared on the basis of research papers presented at the international scientific conference entitled “WOOD – Science – Economy”, organised on 5th and 6th October 2015 in Poznan (Poland). The aim of the conference was to create a platform for the exchange of knowledge (not only scientific) in the field of forestry and the wood industry, as well as to facilitate the identification of research areas with the potential for commercialisation, since the financing nowadays of scientific research is considered justified as long as the results can be utilised in economic practice.

The papers presented touch upon a broad spectrum of issues: from socio-economic and environmental aspects of the wood market, to, the subject of wood mobilisation and the properties of this raw material, and from technological and product innovations in various wood applications (industrial and those connected with energy generation) to the zero waste economy and recycling.

The conference “WOOD – Science – Economy” was organised by the Wood Technology Institute in Poznan in cooperation with the Directorate General of the State Forests. Further information on the conference and summaries of the research papers are available at: www.wood-science-economy.

Prof. Ewa Ratajczak, PhD
Editor-in-chief

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RESEARCH PAPERS

Jim L. Bowyer

THE U.S. FOREST PRODUCTS INDUSTRY – PAST, PRESENT, AND FUTURE

Long the dominant producer and consumer of wood products globally, the U.S. has nonetheless gone through several cycles of forest sector decline and renewal. Now, as the sector begins to emerge from an historic economic reversal, it is clear that the forest products industry of the future will look different from the industry of the past. New product lines and entirely new markets will increasingly bolster the financial bottom lines of forest sector companies that will also continue to serve long-established markets.

Keywords: forest history, forest trends, forest products, industrial productivity, globalization

Introduction

The United States has long been both the largest volume producer and consumer of wood globally. Wood has played a prominent role in the nation's basic materials picture since the beginning of European settlement, with its use more than doubling over the last half of the 20th century. However, a significant downward shift in domestic wood consumption that began around the beginning of the 21st century, and that was accentuated by a pronounced economic recession, has created major challenges for the U.S. forest sector. In what ways and how significantly the sector is likely to change over the long term in response to these factors is an open question, though current developments provide clues to the future.

This article is intended to provide an historical context for understanding the recent dynamics of the U.S. forest industry and how the industry is changing to adapt to new realities. Forest trends from the time of European settlement to the present are reviewed, as is the history of wood use and the influences of

Jim L. Bowyer (jimbowyer@comcast.net), Dovetail Partners, Inc., Minneapolis, MN USA
industrialization, competing materials development, economic disruption and social upheaval, and prolonged economic growth. Recent technological advances are also examined in the context of opportunities for wood-based industrial expansion.

**Research Methodology**

This article is based on a review of literature regarding historical forest trends and wood uses and consumption in the United States, and contemporary articles, reports, and data sources related to the U.S. forest sector. Information as to relatively recent developments is supplemented by the personal experience and knowledge of the author.

**Forests and Wood in U.S. History**

Heavy reliance on wood traces back to the beginnings of U.S. history. Confronted with vast forests, early settlers who arrived on wooden ships began clearing land and used wood for virtually everything. In colonial America wood was the foundation on which society was built. Buildings and furniture, spinning wheels and looms, dishes and pails, wagons and carriages, boats and ships, bridges and sidewalks, ploughs and hay rakes, milling machinery and sawmills, and products of every kind and shape were made of wood. Wood was also a major fuel source, used for heating and cooking and as the principal fuel of industry [Youngquist 1977]. It all added up to substantial growth in wood consumption (fig. 1a).

![Graph showing U.S. consumption of wood and wood products, 1800-2006](image)

*Source: Frederick and Sedjo [1991]; Howard and Westby [2013].

**Fig. 1.** U.S. consumption of wood and wood products, 1800-2006 (thousand cubic meters, roundwood equivalent)
Clearing of land for agriculture to feed a growing population and to provide wood for building of towns and cities and the rail lines that carried people westward took a toll on the new nation’s forests. In just 50 years, from 1800 to 1850, the area of cropland grew from about eight million hectares to thirty-one million not counting pasture land, estimated to be as much as double the area of cropland; much of this expansion was at the expense of forests. The clearing of forests, primarily to agricultural conversion, accelerated following 1850 with another seventy-seven million ha of forests cleared in the succeeding sixty years (fig. 2). This brought the area of forest lost since the initial year of settlement (1607) to about 117 million hectares, a development that paralleled growth in the population from a few thousand in 1607 to about seventy-six million in 1900 [Fedkiw 1989]. Agricultural practices used at the time required the establishment of 1.2-1.6 ha of farm and pasture land for every new resident [MacCleery 1992].


**Fig. 2. U.S. crop and forest land area, 1850-2013 (million ha)**

Then, in the early 20th century, five developments fundamentally changed the relationship between those living in the U.S. and their forests:

1. Mechanized farming equipment largely replaced horses and mules used as draft animals, which in turn greatly reduced the need for pasture land.
2. Modern agricultural practices were adopted that led to far greater crop yields per hectare.
3. A new conservation ethic led to the establishment of the U.S. Forest Service, and soon thereafter to the Park Service.
4. Forestry was introduced as a profession, with several forestry colleges established.
5. Research led to the development of effective wood preservatives.
The combined effect of these changes was dramatic. Despite further growth of the population, from seventy-six million in 1900 to 317 million in 2012, the area of agricultural land remained nearly constant. So too did the area of forest land [Fedkiw 1989; MacCleery 1992]. The need for replacement of wood due to decay was also substantially reduced.

The reversal, from a period of rapid forest loss to forest stabilization is consistent with the experience of a number of other developed countries. Described by the term “forest transition”, stemming of forest losses followed by reforestation of at least a portion of previously converted land areas has been extensively documented [Rudel et al. 2010].

Wood consumption continued to grow through the early 20th century, after which the great economic depression combined with a number of other factors dramatically reduced wood use. First, lumber consumption declined almost as fast as it had increased. There were many causes of the decline, including substitution of non-wood materials for many applications, increased efficiency of wood use, and development of new technologies. The development of wood preservatives and preservative treatments alone resulted in a substantial reduction in the quantity of wood needed for replacement of ties, poles, fencing, and similar products. The invention of barbed wire meant that as the 3.2 million miles of wooden fencing estimated to have existed in the mid-1800s began to deteriorate, far smaller quantities of wood were needed for replacement [MacCleery 1992]. In addition to declining lumber consumption, growth in the use of wood as a source of energy leveled off at the turn of the century and then began to decline as fossil fuels became increasingly more important. Wood energy rebounded during the great depression of the 1930s, but then began a steep decline that continued through the early 1970s. By 1945, overall consumption of wood in the United States had fallen to a level similar to that of 1880 despite an almost 3-fold increase in population during that period (fig. 1b). At the beginning of the mid-20th century, however, the U.S. forest sector experienced a major resurgence, driven by an extended period of economic prosperity. New homes were built at a rapid pace. More than thirty-one million housing units were constructed between 1940 and 1970 [US Census Bureau 2011], resulting in a near doubling of the nation's housing stock. The production of durable and non-durable goods of all kinds, including wood furniture and cabinets, grew rapidly as well, as did production of a wide range of paper and paperboard products. As the economy grew, wood use rebounded, reaching record levels by the late 1960s and with new records set almost every year thereafter. The oil shocks of the 1970s triggered new interest in wood as a fuel, and wood use for energy rose rapidly through the 1980s, helping to push wood use to ever higher levels (fig. 1c).

The dominant uses of wood in the U.S. are as building materials, production of paper and paperboard, and energy. More than half of solid wood products are used for building construction and remodeling. In 1950, forty-nine percent of
wood was used for this purpose, a figure that increased to over sixty-three percent in 2006 (fig. 3). Other uses include furniture and packaging (fig. 3).

![Diagram of wood uses in 1950 and 2006]

Source: McKeever [2009].

**Fig. 3. Primary uses of wood in the United States, 1950 and 2006**

The growth of wood use in the 1960s and ’70s closely matched growth in population, meaning that wood use per capita remained relatively constant during this period. However, in the economic boom years from the late 1970s through the mid-1980s wood use grew more quickly than population numbers, and wood use on a per capita basis rose substantially [Howard and Westby 2013].

The unprecedented expansion of wood use did not bring about further loss of forests as many feared. Not only were increasing harvest levels more than matched by rising forest growth rates (fig. 4) – leading to steady increases in standing timber volume (fig. 5) – but, as noted earlier, the forested area remained stable (fig. 2).
Now there are concerns about the possibility of a new period of forest loss. A recent assessment of the future of forest and rangelands projected losses of 6-14 million ha of forest land by 2060 due to urban expansion and low density housing development in forested areas [USDA – Forest Service 2012].

The U.S. Forest Industry

19th and 20th Century

The early 19th and 20th century forest industry was largely dedicated to production of lumber, timbers, and railroad ties; and poles and ship masts. Products such as pitch and tar were also produced. Fuel-wood was commercially harvested for distribution in many cities. Wood-based paper came into the
picture in the early 1900s. These products defined the U.S. forest products industry through the first half of the 20th century.

Mid-century not only marked the beginning of wood industry resurgence, but, (not coincidentally) a period of process and product innovation as well. As rapidly as wood consumption rose in the post war years, the rise would have been far more spectacular were it not for innovations relative to both process and new products. For instance, in the twenty-five years between 1948 and 1973 the yield of lumber from a given quantity of logs doubled, while the quantity of useful products obtained quadrupled. New products brought into production during this period include softwood plywood, particleboard (using technology developed in Germany), hardboard, and waferboard. Significant increases in paper making efficiency were also achieved during this period.

The new family of products made of fibers, particles, and flakes served to greatly expand the options of wood products manufacturing and to increase the yield of final products. Subsequently, innovation brought to the market more new composite products. Structural composites such as oriented strand board, laminated veneer lumber, parallel strand lumber, and wood composite I-beams allowed the use of less wood for a given application. At the same time, improvements in recycling technology greatly increased waste paper recovery and reuse rates, with these numbers up from fifty to sixty-five percent in the last fifteen years alone [American Forest and Paper Association 2015b]. These developments are reflected in a long history of rising wood productivity (fig. 6).

![Graph showing U.S. industrial wood productivity from 1965 to 2010](image)

**Fig. 6.** U.S. industrial wood productivity, 1965-2010 (industrial wood product output per unit of roundwood input (tons/ton), expressed in percent)
By the end of the century the U.S. forest products industry served largely the same markets as fifty years earlier, but with a wider array of products. Although markets were strong, however, and manufacturing performance at unprecedented levels, there were several areas of concern.

A troubling trend that first appeared in the 1970s and significantly accelerated in the 1980s was loss of domestic wood furniture markets to imported goods – largely from China. By 2000 what had begun as a small increase in imports turned into a forty percent capture of domestic markets, a number that would continue to climb in subsequent years with devastating effects on the nation’s furniture manufacturers [Schuler and Lawser 2007; Luppold and Bumgardner 2011]. Then, in the late 1990s, domestic newsprint and printing and writing paper markets began to show signs of weakness.

**Early 21st Century**

The decline in domestic paper production that began in the late ‘90s has continued into the new millennium, with production in 2014 down about thirteen percent from 1995 (fig. 7). The decline has not been uniform across industry sectors, however. While the production of pulp, and printing and writing papers declined sharply from 2000 to 2014, due to a combination of increasing reliance on electronic communication and competition from overseas paper producers, domestic production of paperboard, packaging, tissue, towels, and specialty papers expanded during this period. Nonetheless, capital spending in the U.S. paper and paperboard industry in recent years has been less than a third of that in the mid-1990s, suggesting further contraction in the coming years.

![Bar chart showing U.S. paper and paperboard production, 1995-2014](source)

**Fig. 7. U.S. paper and paperboard production, 1995-2014**
Meanwhile, imports of wood furniture continued to grow following 2000, and by 2011 imports accounted for seventy percent of the U.S. market [Koenig 2013]. Over a forty year period, a major portion of the U.S. wood furniture industry was shuttered, as foreign competitors and offshore subsidiaries of U.S. companies increased the volume of exports to the U.S. (Luppol and Bumgardner 2011). Office furniture manufacturers are now experiencing similar pressures.

The forest products industry overall shed 220,000 jobs (fifteen percent) from 1997-2006. The vast majority of the job losses during this period were in the non-upholstered wood products and pulp and paper sectors [USDA – Forest Service 2014]. Caution is in order when interpreting the causes and significance of reductions in employment. As in many industries, investments aimed at improving manufacturing efficiency have been ongoing in the forest products industry throughout its history, and particularly in the 1980s. The result has been a steady reduction in employment per unit of wood products output (fig. 8). Consequently, the majority of the reduction in forest products industry employment up through 2006 was attributable to gains in labor efficiency.


![Graph showing labor intensity in the U.S. Forest Products Industry, 1961-2013](image)

**Fig. 8. Labor intensity in the U.S. Forest Products Industry, 1961-2013 (persons employed per 1,000 m³ of product output)**

*Expressed in industrial roundwood equivalent.*

An indication of the effect of the 2007-2009 economic recession on the U.S. forest products industry is indicated by the fact that housing construction – the primary market for wood products – literally collapsed, falling by more than seventy-five percent between 2005 and 2010. Predictably, the impact on most sectors of the industry was severe. Particularly hard hit was the southern U.S., a region that contains only two percent of global forests, but which annually provides nineteen percent of the world harvest of pulpwood and twelve percent
of the global production of industrial timber and over half of U.S. wood products production. This region experienced a reduction of softwood and hardwood lumber production of thirty-six percent and fifty-five percent, respectively, and of plywood and engineered wood by more than forty-eight percent. Job losses in the non-upholstered wood furniture industry, already substantial prior to the recession, declined by another sixty-two percent. The paper industry lost an additional eighteen percent of its workforce, and non-furniture wood products manufacturing employment, fell by more than thirty-six percent [Hodges et al. 2012]. In short, it was a devastating period for the U.S. forest products industry.

Now, as the U.S. economy rebounds from recession, there are early signs of a rebound in the forest sector as well. As of late 2015 annual housing starts were more than double those of 2009 and construction activity in most categories of commercial buildings was up sharply [Gavin 2015; Trading Economics 2015]. Currently, production and consumption of lumber and other building products is increasing, and paper and paperboard production and consumption is now above the 2006 low point. In addition, a wood pellet export industry that emerged through the course of the recession continues to grow, providing a market for some of the wood that had previously been used in paper and paperboard manufacturing.

Results and Discussion

U.S. forest sector recovery from recent events depends in large part on the extent to which the residential housing market recovers. Though increasing, housing starts for 2015 are forecast at only about one-half of pre-recession highs, and there is some concern that the next generation may be less interested in, or able to afford, home ownership than previous generations [Gavin 2015]. Thus there is uncertainty as to the likelihood of fully regaining previous housing-related markets [Hodges et al. 2012; Prestemon et al. 2015]. There is similar uncertainty regarding future paper markets. Continued decline in demand for newsprint and printing and writing papers is viewed as likely, and the future of a currently strong sector – containers and containerboard – is seen as closely linked to domestic manufacturing activity overall, which has been declining for some time [Prestemon et al. 2015]. Current low pulpwood prices provide some optimism for improved competitiveness in export markets [Hodges et al. 2012].

With regard to hardwood markets, there is some indication that domestic manufacturers that shifted manufacturing to offshore locations in the past, may be reconsidering location decisions. Hidden costs of offshoring, such as shipping and inventory costs, long lead times, delayed returns, negative impacts on innovation caused by reduced interaction between engineers and factory workers, and rising labor costs in current producing regions are cited as factors driving re-examination of mill location [North Carolina in the Global Economy
2014, Neil 2013]. This is creating optimism in some quarters that at least a portion of lost domestic furniture production may be restored.

In view of these challenges and trends it would be easy to conclude that the U.S. forest products industry is on brink of extinction. The reality is, however, that the U.S. still consumes and produces more forest products than any other country [Prestemon et al. 2015], employs three-quarters of a million people [US Department of Labor 2015a, b], and accounts for about four percent of US manufacturing GDP – US$210 billion [American Forest and Paper Association 2015a].

A key question is how the industry will adapt going forward to changing global realities. Another is what the impact on the nation’s forests would be if the industry were to substantially down-size over the long-term.

Among the areas viewed as presenting opportunity for the U.S. forest sector are the following:

- New, innovative uses of wood in creating commercial structures, including tall buildings.
- Use of wood as a feedstock in industrial chemicals production.
- Involvement in the emerging nanotechnology industry.
- Wood energy, including wood pellets for export and domestic use, and wood-derived biofuels.

The development of engineered wood products over the past three decades, coupled with the recent development of cross-laminated timber in Austria, has created new opportunities for use of wood in construction. Based on European experience, interest in tall wood buildings is growing in the United States, assisted by initial adoption in western Canada and the potential for reducing the carbon footprint of buildings through greater wood use. Current regulations limit the use of wood as a structural material in buildings to no more than five to six stories. Extending that limit to ten to twelve stories or more would significantly expand opportunities for wood use.

The Department of Energy forecast in 1999 that some ten percent of industrial chemicals and materials would be produced from renewable resources (including wood and agricultural fiber) by as early as 2020, with this number approaching fifty percent by 2050 (fig. 9). Even at a ten percent share for wood, such chemicals would have an annual value of about $400 billion (1999 dollars), or about twice the value of all forest products currently produced in the U.S. In addition to this, great potential is seen in nanocellulose for applications in paper and packaging, construction, automotive manufacture, textiles, and personal care products; a recent estimate found that if nanocellulose were to account for just three percent of U.S. nanotechnology potential in 2020, it would amount to a US$100 billion industry [Goergen et al. 2013]. The opportunities would appear to be substantial.

U.S. production of wood fuel pellets has increased substantially in recent years. Production grew from near zero in 2003 to an estimated six to seven
million dry tons in 2014 (fig. 10), about half of which was exported to the E.U. Significant growth is expected in the future, with the magnitude of growth highly dependent upon government policies in the U.S. and E.U.

**Fig. 9. An estimate of U.S. biochemicals potential**

**Fig. 10. Growth in U.S. fuel pellet production capacity, 2000-2014**
In addition to fuel pellets, there is considerable research activity directed towards the development of liquid fuels from biomass, including woody biomass, motivated in large part by government mandates that call for biofuels production of $61 \times 10^9$ liters in 2015, an amount that increases to $133 \times 10^9$ liters in 2022. With a focus on advanced biofuels, this represents a potential new market for wood.

Successful development in all of these areas will require ongoing research and development as well as attention to government policies regarding forests and their management. Global trends and competition will continue to shape the industry.

With respect to forests, in view of the fact that over eighty-eight percent of commercial timber harvests in the U.S. occur on privately owned forest land [Oswalt et al. 2014 – table 35], the long-term fate of much of this land is somewhat dependent upon the health and vitality of the domestic forest products industry. There are growing concerns that a loss of markets over an extended period could lead to the conversion of forest land to urban development, agricultural energy crops, or traditional agriculture.

Surveys of non-industrial private forest owners across the United States have found that they are primarily interested in such amenities as aesthetics and privacy that their forests provide. Although timber production is not a primary objective of most such owners, timber harvesting is, however, a common activity [Butler 2008], and financial considerations have been found to be an important factor in land management decisions. A survey of private forest landowners in the southern and dominant timber producing region in the U.S. found that owners primarily interested in timber production controlled over one-third of forestland in the region, whereas those who indicated that they would never harvest timber from their land controlled only twelve percent of the total private timberland acreage [Wicker 2002].

Consequently, forest land is under constant pressure from agricultural interests – often in the form of current owners who own both farm and forest land [McCraw 2014]. In addition there is also considerable and mounting interest in forest conversion to urban development or for subdivision to vacation homes. In fact, the U.S. Forest Service has forecast forest losses of seven to thirteen percent of forested area in the southern region by 2060, primarily due to urbanization trends. The greatest losses are projected in an environment of high economic growth and low timber prices. Conversely, increasing timber prices (i.e. strong timber markets) and low economic growth lead to the lowest extent of forest loss. Other assessments of drivers of forest retention have similarly found that increased wood demand can slow the loss of forest or even lead to expansion of forest area [Miner et al. 2014].
Conclusions

The forest products industry of the United States is the world’s largest, having grown substantially since the mid-1960s. Wood is dominant in residential housing, engineered wood products are increasingly specified, and wood use in commercial/industrial construction is growing. Moreover, annual net forest growth on land available for periodic harvest is more than double removals, the standing timber inventory is increasing, and wood that is harvested is converted to products with essentially zero waste.

Despite all these positive indicators, the U.S. industry has suffered severe losses over the past two decades, with substantial loss of markets in the printing and writing papers and wooden household furniture sectors. The severe U.S. economic recession of 2007-2009 hit the forest products industry particularly hard because of the impact on home-building and remodeling, the primary domestic markets for wood products.

Nonetheless, emerging from the trials of recent decades, the industry is highly efficient, diverse, and still large by any measure. A number of new potential markets also offer significant opportunities in the relatively near term.

An ongoing challenge will be the retention of forest land in an era of increasing urbanization and pressures from other competing land uses. Success will depend in part on the existence of a stable, robust, and profitable forest sector.

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Niels JANZEN, Holger WEIMAR

MARKET COVERAGE OF THE EUTR – WHAT SHARE OF WOOD IMPORTS INTO THE EU IS COVERED BY THE EUTR?

Illegal Logging is one of the major global causes of deforestation and degradation of forests. To combat the negative effects of illegal logging, the European Union (EU) introduced various forest related policies and measures. Among them is the EU Timber Regulation (EUTR). The objective of the presented analysis is to identify the percentage share that the EUTR applies to wood and wood-based products. We analysed the imports into the EU by using different reference units: the import value in Euro, the roundwood equivalent and the wood fibre equivalent. Our results show that about 90% of the imported quantities and 74% of the imported values are covered by the EUTR. This means, that in 2013 the EU imported a total wood quantity of 6 million m³ wood fibre equivalents (or 17 million m³ roundwood equivalents, respectively) which is not covered by the EUTR. This amount is almost equally distributed between wood products and paper products. Coverage ratios for further differentiated product groups differ. Typically, raw materials have a higher coverage ratio, and finished products have a lower coverage ratio. The wood quantities that are not covered by the EUTR are highly concentrated between a few commodities like wood charcoal, other articles of wood, recovered paper, printed books and brochures.

Keywords: EUTR, coverage ratio, wood-based products, illegal logging, international trade, reference units

Introduction

Illegal logging is one of the major global causes for deforestation and the degradation of forests. The manufacturing of this illegally logged wood and the ensuing products, as well as the associated trade, negatively affects social and economic interests. To combat the severe effects of illegal logging, the European Union (EU) introduced various forest related policies and measures. The adoption of the EU Action Plan on Forest Law Enforcement, Governance and Trade (FLEGT) in 2003 was a major milestone in this respect. The FLEGT Action Plan proposes various measures for the support of international efforts to
combat illegal logging and its associate trade in relation to the general efforts of the Union to achieve sustainable forest management [EC 2003]. Since then, two main mechanisms have been implemented in order to achieve the goals of the FLEGT Action Plan: Voluntary Partnership Agreements (VPAs) and the EU Timber Regulation (EUTR). The VPA mechanism is based on agreements between partner countries and the EU, to ensure that only wood products based on legal logging will be exported to the EU Member States. The EU Timber Regulation deals with a different matter: its objective is to ban products made of illegally logged wood from the European market. The EUTR prohibits the placing of “illegally harvested timber or timber products derived from such timber” on the EU internal market. Typically, this placement can either be done by selling removals from European forests or by importing wood and wood-based products into the EU. Other countries also use the ban of imports of products made of illegally logged wood as a measure to combat illegal logging. E.g. in 2008 the United States (U.S.) adopted an amendment to the Lacey Act (LAA). In Australia the Illegal Logging Prohibition Act came into effect in 2012. It also aims at a prohibition policy.

The EUTR came into effect on March 1, 2013. The regulation specifically applies to timber and timber products (EUTR 995/2010). Detailed specifications for the regulation refer to its Annex which has a list of respective commodities. The products are structured according to the trade classification of the Combined Nomenclature (CN). The main focus is on wood and articles of wood (chapter 44 of the CN), on pulp of wood (chapter 47) and on paper and paperboard and articles made thereof (chapter 48). Additionally, some commodity codes for furniture and one code for prefabricated buildings is listed. Certain wood based products are not included in the EUTR, which raises a number of questions: how many wood based products are not covered by the EUTR? To what extent does the regulation apply? And what is the coverage ratio of the EUTR if we are looking at all wood-based products? Several studies have been published regarding illegal logging and the effects on wood markets [e.g., Seneca Creek Associates and Wood Resources International 2004; Li et al. 2008; Dieter 2009; Lawson and MacFaul 2010; Dieter et al. 2012]. Among others, in Iben et al. [2014] publications with a focus on VPAs and timber legality verification can be found. The number of scientific publications on the topic of EUTR, however, is comparatively small.

Geraets and Natens [2013], Fishman and Obidzinski [2014], Jakel [2015] cover legal issues in the context of international trade law and analyse whether the EUTR constitutes an illegal trade barrier.

Some publications want to gain information about the effectiveness of the EUTR. Levashova [2011] analyses the text of the regulation itself. She concludes that the regulation lacks elements that are essential in the fight against illegal logging. Among them, she points out that the product scope of the regulation is too narrow as it omits printed matter. In their analysis of statements
by stakeholders in 15 secondary sources Giurca and Jonsson [2015] reveal and classify the heterogeneity of the roles and their different perception of the EUTR. For example, they identify bureaucracy as an overall issue and evaluate that the EUTR creates a strong market advantage for low-risk countries of illegally logged timber as stakeholders in these countries see the regulation as beneficial for their business. Information about stakeholder opinions is of importance to avoid unwanted side effects of the regulation like the ones pointed out earlier by Giurca et al. [2013]: General uncertainty around the EUTR, interpretation and the cost of compliance with the EUTR may lead to substitution in the EU of tropical timber with timber from low risk countries within Europe and North America or to trade diversion to less regulated markets. Overdevest and Zeitlin [2014] argue that the EUTR might undermine the FLEGT VPA development and the significant improvements (identified by informed observers) in forest governance in signatory countries. This is due to the incentive that FLEGT export licences might still be some years off, and consequently exporters in these countries and European importers pursue private solutions (like certification) to meet the due diligence requirements of the EUTR. This may allow coalitions in the countries behind the VPA to collapse and may let the governments abandon their commitment to difficult reforms in the forest sector. Prestemon [2015] uses a statistical model to analyse the effect of the U.S. LAA on prices and quantities of imported wood products hardwood lumber and hardwood plywood to the U.S. For the analysed products he is able to empirically validate and quantify expected effects from economic theory: higher prices and lower quantities are found in the market equilibrium of the U.S. due to the LAA for the imported commodities. For the EUTR similar effects can be expected, but due to the shorter time span of the EUTR data they are not proven yet. In any case, an impact on wood markets in general and not only on import markets of special tropical hardwood lumber commodities would be desirable.

Jonsson et al. [2015] state in their review of scientific and expert studies which deal with the effectiveness and impacts of EUTR, VPAs and LAA: “It is still too early to be able to draw strong conclusions, in particular quantifiable ones, regarding the impacts of FLEGT and EUTR on reducing illegal logging”.

Research methodology

The objective of this analysis is to provide basic data about the EUTR. To the best of our knowledge, there is no coherent information available about how much wood in wood and wood-based products are imported to the EU or how much of this quantity is covered by the EUTR. Hence, our objective is to identify to which share the EUTR applies for wood and wood-based products, and to provide knowledge of trade flows and markets in this regard. The analysis will compare twelve different product groups of wood and wood-based products
in the categories wood products, paper products and other wood-based products. Based on the objective the outline of the report is as follows: This chapter focuses on the research methodology, then the results of the analysis are presented and finally, conclusions are provided.

The analysis of the market coverage of imports of wood and wood-based products to the EU requires the clarification of two main aspects: (1) the definition and scope of wood products and (2) the respective reference units for the calculation of the trade flows in a physical unit.

(1) Wood and wood-based products cover a wide variety of different goods and commodities. These include, for example, wood products such as firewood, sawnwood, pellets or window frames, paper products such as newsprint, sanitary paper, paper for packaging or printed articles. Other products beyond the scope of these traditional goods are also manufactured and have to be considered. Regenerated cellulose or cellulose nitrate can be listed as examples. Additionally, there are a lot of products which also can be made of or contain wood (e.g., toys, chewing gum). Hence, it is essential to define the scope of wood-based products for our analysis. Most of the wood-based products can be attributed to the forest-based sector, which has been described by the European Union in 1999 [EC 1999]. In this definition the forest-based sector consists of the wood working industry, the wood processing industry, the construction industry, the pulp and paper industry and the printing and publishing industry. Based on this, the scope of wood and wood-based products of our analysis can be derived. Table 1 gives an overview and structures the wood-based products in categories and product groups.

The wood-based products can be categorized into raw materials, semi-finished products and finished products. Additionally, they can be subdivided into the following categories: wood products, paper products and other wood-based products. Based on this structure we aggregated the products into twelve product groups for further analysis. The matrix contains all products for which the EUTR applies (EUTR 995/2010). As already mentioned, however, the EUTR does not cover all commodities which have been defined as wood-based products. It can be seen that e.g. products such as wood charcoal, wood marquetry, printed matter or regenerated cellulose are not listed in the annex of the EUTR.

As can be seen in the annex of the regulation, especially further processed wood-based products are not included in the EUTR. Also, another aspect that has to be taken into account is that the EUTR only applies to products as classified in the CN. Hence, the EUTR does not apply for wood and paper products which are already in use as packaging material. Also commodity codes of the CN which are not explicitly defined as goods made of wood, products which only partly consist of wood or contain wood and products which are manufactured in an industry branch outside of the forest based sector are not in
the scope of our analysis. Examples of these products are wooden toys, caravans, chewing gum and musical instruments.

Table 1. Scope of wood and wood-based products

<table>
<thead>
<tr>
<th>Specification</th>
<th>Wood products</th>
<th>Paper products</th>
<th>Other wood-based products</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw material</td>
<td>(1) Roundwood</td>
<td>(8) Wood pulp and recovered paper</td>
<td></td>
</tr>
<tr>
<td></td>
<td>(2) Wood processing residues</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Semi-finished products</td>
<td>(3) Sawnwood</td>
<td>(9) Paper and paperboard</td>
<td>(12) Regenerated cellulose, artificial fibres a.o.</td>
</tr>
<tr>
<td></td>
<td>(4) Wood-based panels</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>(5) Other semi-finished wood products</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Finished products</td>
<td>(6) Finished wood products (excl. furniture)</td>
<td>(10) Articles of paper or paperboard</td>
<td></td>
</tr>
<tr>
<td></td>
<td>(7) Furniture</td>
<td>(11) Printed matter</td>
<td></td>
</tr>
</tbody>
</table>

(2) The different industries of the forest based sector manufacture a broad variety of different wood-based products. In trade statistics, these products are basically measured in the net mass of the traded volumes and in monetary values. For some products a supplementary unit of the traded quantity is also provided. In the case of some wood-based products this traded volume is also recorded in the supplementary unit of cubic meters. For our analysis we used the trade data of Eurostat [Eurostat 2015]. The analysis is mainly conducted for the year 2013, the year when the EUTR came into force. We also analysed the market coverage for the period 2010 to 2013 in order to recognize a trend in these years. The focus, however, is on the year 2013.

In the Eurostat trade database the bilateral trade of all countries of the EU is recorded. The trade data is provided on the 8-digit level of the Combined Nomenclature. For comparison of the different trade flows, however, neither the given trade volume in tons or in cubic meters is sufficient, as many wood-based products also consist of other materials such as adhesives in panels or minerals and additives in paper and paperboard. We therefore converted all trade data into physical reference units.

In the scope of this analysis, we used three different reference units: the mandatory trade values as a monetary value denoted in Euros, and two physical reference units, the roundwood equivalent and the wood fibre equivalent. Both physical reference units are units of volume. The roundwood equivalent is measured in cubic meters (m³ (r)). It has been used in various studies for balancing and analysing material flows of wood-based products [e.g. Ollmann 2001; UN 2005; WWF 2008; Dieter 2009; UN 2011; Dieter et al. 2012; Weimar 2014]. The roundwood equivalent expresses the amount of roundwood, which is
needed for the production of one unit of a product. As such, it indicates the required resource input of roundwood for the manufacturing of a product.

The wood fibre equivalent as the second physical reference unit is also measured in cubic meters (m$^3$ (f)). In contrast, however, to the roundwood equivalent it does not focus on the raw material input, but describes the wood fibres which are effectively in the product. It is defined as the equivalent volume of the wood fibres or wood-based fibres that are contained in the product [Weimar 2011]. Hence, the calculated volumes in m$^3$ (f) indicate how much wood fibres have effectively been traded within a given product. The wood fibre equivalent has been used for material flow analysis [Weimar 2011; Bösch et al. 2015]. The volume of the wood fibres is calculated above the fibre saturation point to take into account the swelling and shrinking of the wood fibres below this threshold. For the purpose of this study, we firstly calculated the mass of the wood fibres of the different commodities based on the study by Diestel and Weimar [2014]. We then calculated the volume by using the density by volume of the respective commodity code. In this regard, we had to take into account that most commodity codes of the CN do refer to more than a single wood species. In these cases we had to estimate an average density by volume of the wood species indicated in the description of the commodity code. We calculated the simple arithmetic average as there is no information on the specific share of the individual wood species of traded volumes of a commodity code. Information on the density by volume is taken from Sell [1989], Anon. [1995], Koch and Richter [2009], Koch and Sieburg-Rockel [2011].

For commodity codes where no specific wood species are explicitly mentioned, we also used the arithmetic average of the wood species to which the description applies. This is the case for commodity codes which refer, for example to softwood, hardwood, tropical wood or also if there is no reference to a specific species group. As an example, for the commodity code 44032011 (sawlogs of spruce of the species *Picea abies* Karst. or silver fir *Abies alba* Mill.) we used the arithmetic average of the densities by volume of both wood species. For better illustration table 2 provides some examples of the resulting conversion factors for both physical reference units.

As can be seen in table 2, the two physical reference units represent the possible range of resource impact for the manufacture of a given commodity. The roundwood equivalent implicitly assumes that wood products are exclusively made of roundwood and can as such be interpreted as an input unit. The eventual use of wood by-products or residues in the manufacture of the product is not taken into account. The wood fibre equivalent describes the other side of the spectrum. It only accounts for the wood fibres contained in a given product. Hence, it can be interpreted as an output based unit. For example, for the manufacture of one ton of wood charcoal 6 m$^3$ of roundwood are needed. The mass, however, of one ton of the product only contains 2.5 m$^3$ of equivalent wood fibres. The other parts of the wood are emitted during the production
process, but are not part of the product. Since the use of wood processing residues for further material use is increasing in a lot of countries, the range covered by both physical reference units allows analyses of the full variety of resource demand for the manufacture of wood-based products.

Table 2. Samples of conversion factors for the two physical reference units wood fibre equivalent m³ (f) and roundwood equivalent m³ (r)

<table>
<thead>
<tr>
<th>CN Code</th>
<th>CN Description (short)</th>
<th>Conversion factor – m³ (f)</th>
<th>Base unit for m³ (f)</th>
<th>Conversion factor – m³ (r)</th>
<th>Base unit for m³ (r)</th>
</tr>
</thead>
<tbody>
<tr>
<td>44029000</td>
<td>Wood charcoal</td>
<td>0.0025</td>
<td>kg</td>
<td>0.0060</td>
<td>kg</td>
</tr>
<tr>
<td>44032011</td>
<td>Sawlogs of spruce or silver fir</td>
<td>1.0000</td>
<td>m³</td>
<td>1.0000</td>
<td>m³</td>
</tr>
<tr>
<td>44081015</td>
<td>Sheets for veneering</td>
<td>0.0022</td>
<td>kg</td>
<td>0.0045</td>
<td>kg</td>
</tr>
<tr>
<td>44101110</td>
<td>Particle board</td>
<td>0.0015</td>
<td>kg</td>
<td>0.0020</td>
<td>kg</td>
</tr>
<tr>
<td>44152020</td>
<td>Pallets and pallet collars</td>
<td>0.0015</td>
<td>kg</td>
<td>0.0038</td>
<td>kg</td>
</tr>
<tr>
<td>47041100</td>
<td>Unbleached coniferous chemical wood pulp</td>
<td>0.0019</td>
<td>kg 90% sdt*</td>
<td>0.0041</td>
<td>kg</td>
</tr>
<tr>
<td>48010000</td>
<td>Newsprint</td>
<td>0.0015</td>
<td>kg</td>
<td>0.0032</td>
<td>kg</td>
</tr>
<tr>
<td>48030090</td>
<td>Toilet or facial tissue stock, […]</td>
<td>0.0017</td>
<td>kg</td>
<td>0.0048</td>
<td>kg</td>
</tr>
<tr>
<td>48131000</td>
<td>Cigarette paper</td>
<td>0.0012</td>
<td>kg</td>
<td>0.0042</td>
<td>kg</td>
</tr>
<tr>
<td>49111010</td>
<td>Commercial catalogues</td>
<td>0.0017</td>
<td>kg</td>
<td>0.0035</td>
<td>kg</td>
</tr>
</tbody>
</table>

*kg 90% sdt: kilogram of substance 90% dry

Source: own calculation

By using the reference units we can aggregate the trade data of the commodities (8-digit-level of combined nomenclature) to product groups and we are able to calculate the EUTR coverage ratio accordingly to the formula below.

\[
CR^u = \frac{\sum_k EUTR_k \cdot cf^u_k \cdot X_k}{\sum_k cf^u_k \cdot X_k}
\]

(1)

where: \( CR \) – coverage ratio of EUTR; \( u \) – m³ (f), m³ (r) or Euro; \( k \) – commodity; \( EUTR \) – dummy variable (1 if commodity is covered by EUTR, 0 if commodity is not covered by EUTR); \( cf \) – conversion factor per import unit; \( X \) – import quantity (in kg, m³ or Euro)

Results

Analysis of global imports to the European Union

Table 3 presents the import quantities to the EU-28 for product groups and product categories expressed in the three reference units. Furthermore, the import quantities are differentiated by EUTR coverage. It should be noted, that trade within the EU-28 is not included in the import quantities.

In 2013, wood and wood-based products were imported to the EU-28 with an import value of 29 billion Euros (see table 3). This is almost equally distributed between wood and paper products. Imports of other wood-based products amount to 1.2 billion Euros. In terms of import value, finished wood
products and furniture are the most important product groups in the wood product category. Roundwood imports contribute only 3% of the total value of imports. In the paper product category all product groups contribute between 10% and 15% to the total import value. About one quarter of the imports, measured by their import value, are not covered by the EUTR. The most important product group in this respect is printed matter. Commodities in the product groups furniture and finished wood products also account for a high share of the import value not covered by EUTR. The products of wood pulp & recovered paper, which are not covered by EUTR, account for 0.2 billion Euros, which equals less than 1% of total import value.

### Table 3. Imports of the EU by product groups and by EUTR coverage for import value, roundwood equivalent and wood fibre equivalent

<table>
<thead>
<tr>
<th>imports, absolute</th>
<th>IMPORT VALUE</th>
<th>ROUNWDWOOD EQ.</th>
<th>WOOD FIBRE EQ.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>covered by EUTR</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td>1 roundwood</td>
<td>0.0 1.0 1.0 100</td>
<td>0.0</td>
<td>16.6</td>
</tr>
<tr>
<td>2 wood processing residues</td>
<td>0.0 0.3 0.3 100</td>
<td>0.0</td>
<td>8.0 8.0 100</td>
</tr>
<tr>
<td>3 sawnwood</td>
<td>0.0 2.0 2.0 100</td>
<td>0.0</td>
<td>10.8 10.8 100</td>
</tr>
<tr>
<td>4 wood-based panels</td>
<td>0.0 1.6 1.6 100</td>
<td>0.0</td>
<td>8.7 8.7 100</td>
</tr>
<tr>
<td>5 other semi-finished wood p.</td>
<td>0.0 0.8 0.8 98</td>
<td>0.1</td>
<td>2.3 2.3 95</td>
</tr>
<tr>
<td>6 finished wood p.</td>
<td>1.5 2.1 3.6 59</td>
<td>6.7</td>
<td>13.4 20.1 67</td>
</tr>
<tr>
<td>7 furniture</td>
<td>1.7 2.9 4.6 64</td>
<td>1.4</td>
<td>4.9 6.3 77</td>
</tr>
<tr>
<td>8 wood pulp &amp; recov. paper</td>
<td>0.2 4.3 4.5 95</td>
<td>4.7</td>
<td>30.0 34.7 86</td>
</tr>
<tr>
<td>9 paper and paperboard</td>
<td>0.0 3.7 3.7 100</td>
<td>0.0</td>
<td>20.1 20.1 100</td>
</tr>
<tr>
<td>10 articles of paper &amp; paperb.</td>
<td>0.0 3.0 3.0 100</td>
<td>0.0</td>
<td>4.4 4.4 100</td>
</tr>
<tr>
<td>11 printed matter</td>
<td>3.0 0.0 3.0 0</td>
<td>2.0</td>
<td>2.0 2.0 0</td>
</tr>
</tbody>
</table>

Source: own calculation

Looking at physical import quantities of the EU-28, we calculated the actual wood imports by using the reference units roundwood equivalents and wood fibre equivalents. Naturally, by using completely different reference units the structure of import, as well as the coverage ratios will be different. Still, it is useful to compare these structures to get a better understanding of the different reference units and hence the resulting product structures of imports as well as coverage ratios.

Measured in cubic meters of roundwood equivalents (m³ (r)) the EU-28 imported a total of 136 million m³ (r) in 2013 of which 73 million m³ (r) are wood products and 61 million m³ (r) are paper products. Further processed products like furniture or printed matter, which typically have a higher price per m³ (r), contribute less to total import volumes than in the case of import value.
Vice versa product groups that can be classified as raw material like roundwood or wood processing residues as well as wood pulp and recovered paper are more important when using roundwood (or wood fibre) equivalents. These product groups contribute with 60 million m³ (r) (= 44%) to total imports of roundwood equivalents, which is much more than its share of 20% of the total import value.

Also, by using the reference unit of wood fibre equivalents (m³ (f)) the product structure of imports changes compared to the structure when using import value. This is also true for a comparison between m³ (f) and m³ (r) structures. In 2013, the EU-28 imported in total 77 million m³ (f) – of which 53 million m³ (f) were wood products (= 68%) – and 24 million m³ (f) were classified as paper products. Roundwood and wood processing residues account for about 30% of the total wood fibre imports for an even higher share, than measured in roundwood equivalents (18%).

Coverage ratios of the EUTR for all wood-based products are about 90% based on m³ (r) and m³ (f) (88% and 92%). Calculations based on the import value are considerably lower at 74%.

For the category wood products, 94% of imported m³ (f) are covered by the EUTR. Coverage ratios based on m³ (r) and import value amount to 89%, and 77% respectively in this category. Coincidently the same coverage ratios as for wood products were calculated for paper products based on m³ (r) (89%) and import value data (77%). Measured in m³ (f) 88% of all imported wood fibres in paper products are covered by the EUTR. Regenerated cellulose and artificial fibres are not listed in the annex of EUTR. As this is the only product group in our category of “other wood-based products”, the coverage ratios are zero for both the product group and the product category.

**Analysis of commodities not listed in the EUTR**

The wood volume not covered by EUTR amounts to 6 million m³ (f). It is highly concentrated between a few commodities. About 80% of this volume can be accounted for within ten commodities. Wood charcoal (CN code 44029000) can be identified as the main product which is not covered by the EUTR. Its import volume equates to 1.4 million m³ (f). This accounts for 23% of all imported wood quantities that are not covered by EUTR. In second place are “Articles of wood, not elsewhere specified” (CN code 44219098) which account for another 0.7 million m³ (f). Additionally, there are three other commodities or commodity groups of importance in the top 10. Firstly, six commodities related to recovered (waste and scrap) paper and paperboard (a total of 1.8 million m³ (f) or a 31% share of total imports not covered by EUTR). Secondly, a commodity code related to printed books, brochures, etc. (0.5 million m³ (f)). And thirdly, “upholstered seats with wooden frames” (0.3 million m³ (f)).
Regional analysis

In this section we focus on the geographical origin of imports of wood and wood-based products. For this purpose, we defined nine regions by aggregating the sub-regions which are given by the UN regional classification\(^1\).

In table 4 the regions are listed and it presents the trade flows of wood and wood-based products in wood fibre equivalents into the EU-28 in the year 2013\(^2\). The largest exporter of wood and wood-based products into the EU-28 in the year 2013 was Russia and Eastern Europe (Non-EU28). The EU-28 imported 25 million \(\text{m}^3\) wood fibre equivalents from this region, which equals a third of the total imports of wood and wood-based products into the EU-28. The second largest exporter to the EU-28 is North America, being followed by WNS Europe (Non-EU28). These three regions together, account for about 70% of total \(\text{m}^3\) (f) imports. The least important in terms of total wood fibre imports are the regions of WCS Asia, Africa and Oceania. Imports into the EU-28 from these regions add up to only 4 million \(\text{m}^3\) (f), which is less than 5% of the total wood fibre imports.

Table 4. Wood and wood-based products: Imports to EU-28 by export region and by EUTR coverage, measured in \(\text{m}^3\) (f)

<table>
<thead>
<tr>
<th>Region</th>
<th>WNS Europe (Non-EU28)</th>
<th>Russia &amp; EE (Non-EU28)</th>
<th>North America</th>
<th>Latin America</th>
<th>WCS Asia</th>
<th>ESE Asia</th>
<th>Africa</th>
<th>Oceania</th>
<th>All Regions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Imports in 1,000 (\text{m}^3) (f)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>not covered by EUTR</td>
<td>1,817</td>
<td>579</td>
<td>571</td>
<td>515</td>
<td>155</td>
<td>1,825</td>
<td>542</td>
<td>3</td>
<td>6,017</td>
</tr>
<tr>
<td>covered by EUTR</td>
<td>12,019</td>
<td>24,628</td>
<td>15,696</td>
<td>10,590</td>
<td>683</td>
<td>5,119</td>
<td>2,017</td>
<td>263</td>
<td>71,138</td>
</tr>
<tr>
<td>total imports</td>
<td>13,837</td>
<td>25,207</td>
<td>16,267</td>
<td>11,105</td>
<td>838</td>
<td>6,944</td>
<td>2,559</td>
<td>265</td>
<td>77,155</td>
</tr>
<tr>
<td>Import share by region</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>not covered by EUTR</td>
<td>30%</td>
<td>10%</td>
<td>9%</td>
<td>9%</td>
<td>3%</td>
<td>30%</td>
<td>9%</td>
<td>0.0%</td>
<td>100%</td>
</tr>
<tr>
<td>covered by EUTR</td>
<td>17%</td>
<td>35%</td>
<td>22%</td>
<td>15%</td>
<td>1%</td>
<td>7%</td>
<td>3%</td>
<td>0.4%</td>
<td>100%</td>
</tr>
<tr>
<td>total imports</td>
<td>18%</td>
<td>33%</td>
<td>21%</td>
<td>14%</td>
<td>1%</td>
<td>9%</td>
<td>3%</td>
<td>0.3%</td>
<td>100%</td>
</tr>
<tr>
<td>Regional coverage ratio</td>
<td>87%</td>
<td>98%</td>
<td>96%</td>
<td>95%</td>
<td>81%</td>
<td>74%</td>
<td>79%</td>
<td>99%</td>
<td>92%</td>
</tr>
</tbody>
</table>

Source: own calculation

Coverage ratios vary between regions according to export composition of commodities and product groups. Imports from Oceania and Russia & EE (Non-EU28) are almost completely covered by EUTR (99% and 98%). But one has to be mindful of the associated volumes. In the case of Russia & EE (Non-EU28) this high coverage ratio is associated with a third of the total imports in wood fibre equivalent. The high coverage ratio for Oceania is less important, because

\(^1\)EU28: European Union with 28 member states; WNS Europe (Non-EU28): “Western, Northern and Southern Europe, excluding EU28 member states”; Russia & EE (Non-EU28): “Russia and Eastern Europe, excluding EU28 member states”; WCS Asia: “Western, Central and Southern Asia”; ESE Asia: “Eastern and South-Eastern Asia”; North Am.: North America; Latin Am.: Latin America.

\(^2\)For further interest, see more detailed results in our working paper Weimar et al. [2015].
the associated trade flows are negligible (0.3%). The region Eastern and South-Eastern Asia has the lowest coverage ratio with 74%. Imports from ESE Asia account for 9% of total wood-fibre imports (7 million m\(^3\) (f)).

The regional structure of the products not covered by the EUTR is very different to the regional structure for total wood and wood-based product imports based on m\(^3\) (f). The major export regions Russia & EE (Non-EU28) and North America each contribute significantly less (=10%). On the other side, WNS Europe is almost twice as important for not covered imports as it is for the total imports. It is the source of 30% of all imported wood fibre equivalents which are not covered by EUTR. This is due to high imports of recovered paper commodities. Three other regions triple their shares: WCS Asia with 3%, ESE Asia with 30% and Africa 9% (shares of total imports: 1%, 9% and 3%, respectively). The reason for this increase in ESE Asia is on the one hand the relatively high amount of printed matter that is exported from ESE Asia. In fact, 69% of all imported printed matter into the EU-28 is exported from ESE Asia. On the other hand, relatively high shares of furniture and finished wood products can be seen in their export product structure combined with a low coverage ratio in the finished wood products group. The imports of the EU-28 from Africa which are not covered by the EUTR amount to 9% of all imports not covered by the EUTR. This is mainly due to the import of wood charcoal.

**Conclusions**

Our results show that about 90% of the imported quantities are covered by the EUTR. This means, the EU-28 imported in 2013 a wood quantity of 6 million m\(^3\) wood fibre equivalents (17 million m\(^3\) roundwood equivalents) that is not covered by the EUTR. This quantity is almost equally distributed between wood products and paper products. For the twelve wood and wood-based product groups we quantified the wood imports and coverage ratios of EUTR. Coverage ratios for product groups differ. Typically, raw materials have a higher coverage ratio and finished products have a lower coverage ratio. The wood quantities that are not covered by EUTR are highly concentrated on a few commodities like wood charcoal, articles of wood, n.e.s. and printed books and brochures. The regional import structure of EU-28 for all wood and wood-based imports is very different to the structure of imports not covered by EUTR.

When looking at the regional import structure of products that are not covered by EUTR, Russia and Eastern Europe (Non-EU28) are less important, while Eastern and South-Eastern Asia is now the most important region.

If measured in monetary terms, the overall coverage ratio only accounts for 74% of all wood-based imports. This significant drop is mainly because further processed wood-based products typically show an increasing value per unit. As the coverage ratio of the EUTR decreases with increasing stages of processing, this leads to a discrepancy in physical and monetary reference units.
Having a detailed look at the coverage rate of the physical reference units, the ratio of wood products based on m³ (f) are slightly higher than the coverage ratio based on m³ (r). This is due to the combination of two facts: On the one hand, furniture, finished wood products and semi-finished wood products account in m³ (r) for much higher quantities than in m³ (f) as the other product groups in the wood product category. On the other hand, not all commodities in the product groups of furniture, finished wood products and semi-finished wood products are covered by the EUTR. For paper products the coverage ratios are the same between m³ (r) and m³ (f) because the spread of m³ (f) to m³ (r) ratios for these product groups are rather small (between 0.37 and 0.46).

It is obvious that the coverage ratio we calculate in this study mainly depends on the definition and scope of the wood-based products in total. A lot of other commodity codes, however, contain wood or products made of wood. Other products with a minor share in the given commodity code (e.g., caravans) or products with a high content of wood are commingled in a commodity code with products made of other materials (e.g., toys). In this respect wood-based packaging material can also be mentioned for imported products (e.g., cardboard boxes). For these kinds of products the EUTR might not serve as an appropriate measure for combating illegal logging due to limits of practical implementation. Costs for acquisition of necessary information, legal uncertainty and administrative workload come to mind. In this regard, exports of VPA countries might also cover these possible material flows.

According to the regional analysis, an extension of the commodities listed in the EUTR would affect imports from regions differently. For example, (1) an inclusion of printed matter would affect trade relations of the EU-28 and ESE Asia the most, as 69% of imported printed matter originates here. (2) A complete expansion of the EUTR to the same product scope as in this analysis would only marginally affect the trade with Russia & EE (Non-EU28). (3) An inclusion of recovered paper commodities or generally, a complete coverage in the product groups of wood pulp & recovered paper would affect WNS Europe (Non-EU28) the most as it has a very low coverage ratio (22%) in this product group and a significant percentage of exports to EU-28 are in this product group. (4) Finally, an entry of wood charcoal in the Annex of the EUTR would affect Africa and Latin America the most. They are the largest exporters to the EU-28 with around 500 million m³ (f) each.

A deeper analysis of the consequences (if any) on trade relations and import structures, due to an expansion of the commodity list in the EUTR, is not in the scope of this study. It is worth mentioning, however, that at least importers of covered commodities have to engage with their trade partners, so they will provide the necessary information for due diligence actions according to the EUTR. This raises awareness and might positively influence behaviour along the production chain as the continuation of trade relations gives financial incentives. The EUTR can basically be a suitable measure for all wood-based products as
classified in this paper, even if the proposed positive effects of the EUTR (in combination with VPAs) on the forest sector in the wood producing and the wood manufacturing countries have just begun to evolve [Jonsson et al. 2015].

In contrast, measures like the EUTR can have ambiguous effects: For example Prestemon [2015] described in his analyses of the U.S. LAA negative results for consumers and further processing industries as import quantities decrease and prices increase. Also in this context, Giurca et al. [2013] described two possible consequences, especially for tropical timber: substitution by temperate hardwood species and trade diversion from more strictly regulated regions to less regulated markets. Generally, if the EUTR acts as a non-tariff-barrier, a higher level of domestic wood processing and/or consumption in exporting countries or shifts in regional structure of imports in EU-28 are possible effects.

As these examples as well as the results of our analysis show, there is still the necessity for improvement. For example, following the concept of the EUTR it seems logical to include more products and especially more further processed wood-based products in the annex of the EUTR in order to avoid possible leakages in trade.

Typically exports are rather small compared to domestic consumption. Hence, the EUTR alone is unlikely to solve the problem of illegal logging. Also other measures, such as VPAs, by Europe (and other countries) with partners in the producing regions have to be further developed and applied in order to achieve the ambitious goals of the fight against illegal logging in the international community.

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Jan-Henning Blohm, Robert Evans, Gerald Koch, Uwe Schmitt

Identification and Characterisation of Douglas-fir (Pseudotsuga menziesii (Mirb.) Franco) Juvenile and Adult Wood Grown in Southern Germany

More than one-third of Germany’s Douglas-fir resources, stock in age-classes from twenty-one to fifty-nine years. As such timber increasingly enters markets, detailed knowledge of the anatomy and properties of its wood is of importance to forest managers and wood processors. Anatomical and mechanical wood analyses in this study were carried out on twenty trees from four scientifically managed plantations in Southern Germany. The age of the trees selected was forty-two years whereby varying growth conditions were considered. Juvenile and adult woods were identified by segmented linear regression of radial profiles of anatomical characteristics, such as latewood percentage, tracheid wall thickness microfibril angle and density. Additionally, the width of earlywood, latewood and growth rings as well as bending modulus of elasticity were determined. Variance was dependent on the trait used for differentiation, juvenile wood comprised of an eleven to thirty-one growth rings resp. radial amounts of fifteen to sixty-five percent. When compared to adult wood, juvenile wood showed corresponding features of approximately thirty percent wider growth rings, thirty four percent lower latewood percentage, fourteen percent thinner tracheid walls, and eighty percent larger microfibril angles, eleven percent lighter wood and fifty-seven percent lower bending modulus of elasticity. As the assortment features fast grown trees, adult heartwood characteristics were slightly inferior to the characteristics of European Douglas-fir.

Keywords: Douglas-fir, plantation trees, juvenile/adult wood identification, histometry

Introduction

At the end of the 19th century Douglas-fir (Pseudotsuga menziesii (Mirb.) Franco) was re-introduced to Europe, as a fast growing species extending the

Jan-Henning Blohm (jan.blohm@thuemen.de), Gerald Koch (gerald.koch@thuemen.de), Uwe Schmitt (uwe.schmitt@thuemen.de), Thünen Institute of Wood Research, Hamburg, Germany; Robert Evans (robert.evans@silviscan.com), Silviscan Pty Ltd., Victoria, Australia
spectrum of conifers. Forestry nowadays regards the rather summer drought resistant coastal form (var. menziesii/viridis) as a promising alternative to Norway Spruce, which might in the future suffer due to extended periods of summer droughts, and therefore, is intended to enlarge the amount of plantations. The majority of German plantations can be found in the Southwest, where Douglas-fir stocks, on three to six percent of the federal countries forest area [Thünen Institute 2014]. On commercial plantations, tree ages vary mostly between twenty-one and fifty-nine years, while rotation periods range from eighty and 120 years for achieving high value timber. Consequently, the amount, characteristics and quality of juvenile wood (JW) have to be considered. JW can be defined as wood that is formed under the hormonal influence of the apical meristem by young cambium initials while trees are exposed to lateral forces like winds or passing animals [Lichtenegger et al. 1999; Barnet and Bonham 2004]. The cylinder of JW formed from the base to the top of a tree, is interpreted as a mechanic optimisation providing flexibility to young tissues prone to breakage. When gravitation forces exceed lateral forces, adult wood (AW) formation starts at the stem base where leverage is maximal and hormonal influence of the apical meristem is minimal. Depending on genetic and external influences, the amount of JW in Douglas-fir varies by seventeen to thirty growth rings [Abdel-Gadir and Krahmer 1993b].

As compared to the relatively constant characteristics of AW, the anatomical traits of JW show significant variations. In detail, conifer JW is characterised by bigger microfibril angles, shorter and thinner walled tracheids and lower latewood percentages. From these, physical and mechanical properties like lower density, transverse shrinkage and strength can be derived [Bendtsen 1978]. Because of its lower wood quality, JW is rejected for many applications. The aim of this study [Blohm 2015] is to identify JW, determine its properties and to quantify differences compared to adult wood. Accordingly, the obtained results are valuable for both foresters and wood processors in order to optimise silvicultural regimes as well as to quantify JW.

**Materials and methods**

Investigations were carried out on twenty coastal Douglas-fir trees of the seed origin ‘Südbaden’ from scientifically managed sites of a growing space experiment. Site elevations and average annual precipitations ranged from 410-780 m and 740-1100 mm. Selected trees were harvested in the summer of 2012 at an average age of 42 ±1 years representing the widest span of tenable planting/growth conditions.

From each of the trees, at breast height a disc was obtained out of which the diameter marking the angle bisector of maximum and minimum radius was cut avoiding reaction wood or opposite wood. The radius containing less fibre deviations was chosen for the anatomical investigations.
Prior to the measurements, the radii were cut to the dimensions 2 mm × 7 mm (tangential × longitudinal), extracted with acetone, reconditioned at forty percent relative humidity and 20°C, resulting in a moisture content of about seven percent. One transverse surface was polished with sandpaper down to 1500 grit size. Methods include growth ring measurements as described by Aniol [1983, 1987] regarding both earlywood (EW) and latewood (LW) width, which result in the growth ring width (GRW) and the latewood percentage (LW%). By means of SilviScan-3® radial profiles of tracheid wall thickness (TW), density (D), microfibril angle (MFA) and bending modulus of elasticity (MOE) were determined [Lundqvist and Evans 2004]. The SilviScan-3® system is an automated tool for rapid determination of wood microstructure. It consists of an image analysis unit, an x-ray diffractometer and an x-ray densitometer, which combine to give a range of primary and secondary data. Data were exported both as radial profiles at 25 micron intervals and as growth ring statistical parameters (average, median, percentiles and standard deviation). In order to differentiate JW and AW, growth ring average LW%, TW, MFA and D were plotted against cambial age. Following the segmented linear regression model suggested by Abdel-Gadir and Krahmer [1993a], each cambial age was presumed to mark the demarcation of JW and AW. The best segmentation of JW and AW was identified by the method of least squares while both connected, but also separated segments were assumed (see fig. 2).

For statistical comparison of JW and AW characteristics, the H-test of Kruskal and Wallis [1952] was applied with significance levels as proposed by Miller [1966].

Results and discussion

Segmented linear regression applied on LW%, TW, MFA and D identifies different cambial ages for the beginning of AW formation [Bendtsen 1978]. As shown in figure 1 exemplarily for one of the trees, using LW% results in a cambial age of twelve years to mark the beginning of AW formation, while MFA values result in cambial age of twenty-two years. Table 1 gives average cambial ages of initial AW formation and the average amounts of JW of the radii. The cambial age of initial AW formation, determined by usage of the D agrees quite well with the cambial age of twenty-six determined by Abdel-Gadir and Krahmer [1993a], while another study using D and MFA resulted in mostly younger cambial ages [Bawcombe 2012]. Wood traits, however, LW% and TW were not yet used for JW and AW demarcation of Douglas-fir.

In figure 2, radial profiles of average MFA, LW%, MOE, TW, GRW and D with cambial age are depicted, regarding segmentations of JW and AW for wood traits used for identification. It is obvious, that LW% (fig. 2b) and TW (fig. 2e) influence D (fig. 2f) resulting in the D check pattern described in Douglas-fir
Fig. 1. Segmented linear regression results for the identification of the beginning of adult wood formation by average microfibril angle [°] (left, \( R^2 = 0.95 \)) and latewood percentage [%] (right, \( R^2 = 0.77 \)) with cambial age [y]

Table 1. Average onset of adult wood (AW) formation [cambial age] and amount of juvenile wood (JW) along radii [%] as identified by latewood percentage (LW%), tracheid wall thickness (TW), microfibril angle (MFA) and density (D)

<table>
<thead>
<tr>
<th></th>
<th>LW%</th>
<th>TW</th>
<th>MFA</th>
<th>D</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average cambial age of initial AW formation [y]</td>
<td>18 ±6</td>
<td>20 ±4</td>
<td>23 ±4</td>
<td>25 ±7</td>
</tr>
<tr>
<td>Average amount of JW [%]</td>
<td>38 ±23</td>
<td>45 ±16</td>
<td>53 ±15</td>
<td>51 ±14</td>
</tr>
</tbody>
</table>

[Di Lucca 1989; Abdel-Gadir and Krahmer 1993a; Fabris 2000; Gartner et al. 2002; Peterson et al. 2007; Bawcombe 2012]. The check pattern is characterised by an initial D maximum in early formed growth rings, followed by a local minimum in cambial ages coincident with maximal GRW (fig. 2e) and a subsequent increase.

As TW also shows the check pattern, this trait is considered as the key predictor of D shown by the strong correlation (\( R^2 = 0.94 \)) of traits [Rathgeber et al. 2006]. Contrary to former studies, LW% does not follow the check pattern, which explains the weak correlation (\( R^2 = 0.23 \)) calculated for LW% and D of 730 growth rings. LW% instead increases continually until the cambial age of eighteen, after which rather constant percentages were recorded. The average MFA (fig. 2a) and MOE (fig. 2c) reveal reverse trends and by means of high-resolution correlation, MFA is one of the traits predicting MOE (\( R^2 = 0.63 \)). Even stronger, positive correlations were calculated for MOE and D (\( R^2 = 0.79 \)) as well as for MOE and TW (\( R^2 = 0.82 \)).
Fig. 2. Radial profiles of properties with cambial age [y]: a – average microfibril angle (MFA) [°] differentiated for juvenile and adult wood, b – average latewood percentage (LW%) [%] differentiated for juvenile and adult wood, c – average bending modulus of elasticity (MOE) [kN/mm²], d – average tracheid wall thickness (TW) [µm] differentiated for juvenile and adult wood, e – average width of growth ring (GR), earlywood (EW) and latewood (LW) [mm], f – average density (D) [g/cm³] differentiated for juvenile and adult wood.

Once JW and AW are differentiated, the average values of anatomical characteristics can be calculated. Of all the characteristics investigated, AW values differ conclusively (p ≤ 0.001) from JW values as shown in table 2. In comparison with earlier studies, AW average values indicate rather inferior quality, as the assortment features of fast grown trees. The only exceptions are
LW% of AW, which is in line with both average values of Douglas-fir grown in Northwest America and Europe [Krigge 1958; Lachenbruch et al. 2010; Bawcombe 2012] and D of both JW and AW which agrees with average values of Douglas-fir grown in Wallonia [Pollet et al. 2013].

**Table 2. Characteristics of juvenile wood (JW) and adult wood (AW) and JW/AW ratio**

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>Juvenile wood</th>
<th>Adult wood</th>
<th>JW/AW ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Growth ring width</td>
<td>[mm]</td>
<td>6.2 ± 38 %</td>
<td>4.8 ± 44 %</td>
</tr>
<tr>
<td>Latewood percentage</td>
<td>[%]</td>
<td>27 ± 44 %</td>
<td>41 ± 22 %</td>
</tr>
<tr>
<td>Tracheid wall thickness</td>
<td>[µm]</td>
<td>2.4 ± 38 %</td>
<td>2.8 ± 43 %</td>
</tr>
<tr>
<td>Microfibril angle</td>
<td>['']</td>
<td>27 ± 33 %</td>
<td>15 ± 53 %</td>
</tr>
<tr>
<td>Density</td>
<td>[g/cm³]</td>
<td>0.470 ± 43 %</td>
<td>0.526 ± 52 %</td>
</tr>
<tr>
<td>Bending modulus of elasticity</td>
<td>[N/mm²]</td>
<td>9077 ± 20 %</td>
<td>13193 ± 10 %</td>
</tr>
</tbody>
</table>

**Conclusion**

The studies revealed that besides JW and AW differentiation by means of segmented linear regression of radial D and MFA profiles, also LW% and TW enable distinction. Characteristics of the identified juvenile wood are of conclusive inferior quality when compared to adult wood. Average characteristics of the investigated adult wood are slightly lower than reported for European Douglas-fir due to the involvement of fast grown trees in the assortments selection.

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Ewa BAKINOWSKA, Piotr S. MEDERSKI, Anna SZCZEPANSKA-ÁLVAREZ, Zbigniew KARASZEWSKI, Mariusz BEMBENEK

THE PARALLEL APPLICATION OF TWO PROBABILITY MODELS, LOGIT AND PROBIT, FOR THE ACCURATE ANALYSIS OF SPRUCE TIMBER DAMAGE DUE TO THINNING OPERATIONS

Logit and probit models belong to the class of generalised linear models. A few applications of both models have been documented in the field of forestry. The objective of this paper was to test the parallel use of these models to discover the differences in damage to a spruce stand after thinning using the full tree system, the long wood system and the short wood system. In particular the aim was to ascertain the general damage probability caused by the harvesting systems (HS) and the particular damage class probability in each HS. When the general damage probability was calculated the logit model was used. When nine damage classes were taken into account, however, the probit model was found to fit the data better. In this case, the results obtained gave accurate information on the probability of the appearance of a particular damage class for each HS. It was concluded that the probit and logit models should be considered in parallel in order to obtain the best possible goodness of fit and to get accurate information on the distribution of damage classes.

Keywords: generalised linear models, categorical data, goodness of fit, Akaike criterion

Introduction

Thinning operations in Polish forests are performed once per decade. Zasady Hodowli Lasu (Principles of Silviculture) [2012] for spruce stands recommend early and late thinning, before and after 40 years of age, respectively. During this
process, damage occurs to some of the remaining trees. Aside from the human factor, the level of damage can depend on, among other things, thinning intensity, the method of thinning, the operation and machine applied and the harvesting system (HS) [Bembrnek et al. 2013a, b; Karaszewski et al. 2013]. The differences in the level of damage can be analysed with simple data comparison using the Kruskal-Wallis or U Mann-Whitney tests [Stańczykiewicz et al. 2015a]. There are also studies based on the $\chi^2$ Pearson test [Lageson 1997], T-tests [Modig et al. 2012] and Student’s $t$ distribution [Glöde and Sikström 2001; Stańczykiewicz et al. 2015b]. Sirén et al. [2013] used mixed-model analysis for nested data structures, including both fixed and random effects.

Tree damage in lowland spruce stands caused by early and late thinning was also analysed by Bembrnek et al. [2013a, b]. These authors used the $\chi^2$ Pearson test and the Fisher test to study the relationship between the system of wood harvesting and percentage of trees with damage.

The above-mentioned experiment by Bembrnek et al. [2013a, b] only gave three sets of information: 1) the percentage of trees with damage in each damage class, but without division into HSs, 2) the percentage of trees with damage in each HS, but without division into damage classes, and 3) the statistical differences in the frequency of trees with damage (without division into classes) in each HS. It was hypothesised, however, that the use of a model could give more exact results, such as the probability of the appearance of a particular damage class in each HS separately.

The general objective of this paper was to find out:

1) the probability of damage (without division into classes) for each HS:
   full tree system (FTS), long wood system (LWS) and short wood system (SWS)
2) the probability of the appearance of a particular damage class (from one to nine) separately in each HS
3) if there are (statistically significant) differences for 1) and 2)
4) if there are differences between the results obtained and previous work based on simple statistics.

In order to achieve this, two generalised linear models were used: logit and probit. Two models were used mainly to find out if they would be characterised by different goodness of fit, and if so, the one (model) with a better goodness of fit would be used for the final analysis.

The use of both generalised linear models in this work aims to extend the results published by Bembrnek et al. [2013a, b]. The parallel use of two models has also been applied in medicine [Zhou and Wong 2008], animal breeding [Röhe and Kalm 2000] and in economic studies [Layton and Katsuura 2001].

In certain publications, for example, there are applications of, the logit model, to compare varieties of seed pea in respect to lodging [Bakinowska and Kala 2007] and to analyse downy mildew infection of field pea varieties [Bakinowska et al. 2015]. In forestry, the logit model has been used to analyse
tree damage [Sirén 2001]. Taking into account the above-mentioned studies, however, in which only the logit model was used, it was assumed that there was a risk that it would not give the best (possible) goodness of fit to describe a particular phenomenon.

**Material and methods**

**Experiment**

In the experiment, the number of damaged trees and the damage class formed during early and late thinning were studied. Trees with damage were divided according to nine classes (tab. 1).

<table>
<thead>
<tr>
<th>Table 1. Tree damage classes</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Damage class</strong></td>
</tr>
<tr>
<td>1</td>
</tr>
<tr>
<td>2</td>
</tr>
<tr>
<td>3</td>
</tr>
<tr>
<td>4</td>
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<tr>
<td>5</td>
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<td>6</td>
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<tr>
<td>7</td>
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<tr>
<td>8</td>
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<tr>
<td>9</td>
</tr>
</tbody>
</table>

The damage data was analysed from sample plots selected in North and North-West Poland. Early thinning was carried out on lowland spruce stands of second age class (AC2, 21-40 years old) in the forest districts of Zaporowo and Różańsko, on eight sample plots, each measuring 0.25 ha. On all the sample plots, the total number of observations amounted to 2381 trees, 2163 and 218, respectively, with and without damage (tab. 2).

<table>
<thead>
<tr>
<th>Table 2. Number of observations (trees) evaluated statistically</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Trees</strong></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>FTS</td>
</tr>
<tr>
<td>LWS</td>
</tr>
<tr>
<td>SWS</td>
</tr>
<tr>
<td>Total</td>
</tr>
</tbody>
</table>

Et – early thinning, Lt – late thinning, FTS – full tree system, LWS – long wood system, SWS – short wood system.
Late thinning was provided on lowland spruce stands in AC4 (61-80 y.o.) in the forest districts of Dretyń, Wejherowo, Mieszkowice and Różańsko, on ten sample plots, each with an area 0.25 ha. On all the sample plots, the total number of observations amounted to 1235 trees, 1102 and 133, respectively, with and without damage (tab. 2).

During the thinning process, three different systems of wood harvesting were applied: FTS, on two plots in both early and late thinning, LWS, on two plots in early thinning and on four plots in late thinning, as well as SWS, on four plots in both early and late thinning.

**Generalised linear model**

For analysis of the experiment, both the logit and probit models were used. These models belong to a wider class of models called generalised linear models [Agresti 1984; McCullagh and Nelder 1989], which in matrix form can be written as:

$$\eta = X\beta$$  \hspace{1cm} (1)

where the vector \( \eta \) is called the link function, \( X \) is the matrix of covariates, and \( \beta \) is the vector of unknown parameters.

If the link function \( \eta \) in model (1) is the logit function, then the model is called the generalised linear model with logit link function, or, in short, the logit model or the logistic regression model [Rao and Toutenburg 1999]. This model is often used in the analysis of experiments in which the observed units are assigned to separate categories. The scalar form of the logit model [McCullagh and Nelder 1989; Miller et al. 1993; Rao and Toutenburg 1999; Bakinowska and Kala 2007] is as follows:

$$\eta_{ji} = \log \frac{\pi_{1j} + \pi_{2j} + \ldots + \pi_{ij}}{1-(\pi_{1j} + \pi_{2j} + \ldots + \pi_{ij})} = \theta_j + \tau_i, \hspace{0.5cm} j=1,2, \ldots, k-1, \hspace{0.5cm} i=1,2, \ldots, s$$  \hspace{1cm} (2)

where \( \theta_j \) is the border (cut point) of the \( j \)th category, \( \tau_i \) is the effect of the \( i \)th treatment (so \( \theta_j + \tau_i \) represents the cut point of the \( j \)th category for the \( i \)th treatment), \( \pi_{ji} \) are the probability of success of the \( j \)th category, \( j=1,2, \ldots, k \) for the fixed \( i \)th treatment, which equal to one: \( \sum_{j=1}^{k} \pi_{ji} = 1 \). If the link function \( \eta \) in model (1) is the vector of inverses \( \Phi^{-1} \) of the standard normal cumulative distribution functions, then the model is called the generalised linear model with a probit link function or, in short, the probit model [Rao and Toutenburg 1999]. For a fixed \( i \)th treatment, this model can be written in the following scalar form:

$$\eta_{ji} = \Phi^{-1}(\pi_{1j} + \pi_{2j} + \ldots + \pi_{ij}) = \theta_j + \tau_i, \hspace{0.5cm} j=1,2, \ldots, k-1, \hspace{0.5cm} i=1,2, \ldots, s.$$  \hspace{1cm} (3)
For this research, it was assumed that the vector of unknown parameters \( \mathbf{\beta} \) in model (1) is of the form \( \mathbf{\beta}^T = (\mathbf{\theta}^T, \mathbf{\tau}^T) \), where \( \mathbf{\theta} \) denotes the vector of cut points and \( \mathbf{\tau}^T = (\tau_1, \tau_2, \ldots, \tau_s) \) is the vector of effects. The system of hypotheses was tested:

\[
H_0: \mathbf{\beta} = \mathbf{\beta}_* \quad \text{against} \quad H_1: \mathbf{\beta} \neq \mathbf{\beta}_*
\]

using the Wald statistic, which has the form:

\[
(\hat{\mathbf{\beta}} - \mathbf{\beta}_*)^T \hat{\mathbf{F}}(\mathbf{\beta}_*)(\hat{\mathbf{\beta}} - \mathbf{\beta}_*)
\]

and has approximately \( \chi^2_p \) distribution (\( p \) being the order of \( \mathbf{\beta}_* \)), where \( \hat{\mathbf{\beta}}_* \) is an estimate of \( \mathbf{\beta}_* \), and \( \hat{\mathbf{F}}(\mathbf{\beta}) \) is the Fisher information matrix of \( \hat{\mathbf{\beta}} \) [Agresti 1984; McCulloch and Searle 2001].

The aim of the analysis was to estimate the unknown probabilities in model (2) which can be expressed as:

\[
\mathbf{\pi}_{1i} + \mathbf{\pi}_{2i} + \ldots + \mathbf{\pi}_{ji} = \frac{\exp(\mathbf{\theta}_j + \mathbf{\tau}_i)}{1 + \exp(\mathbf{\theta}_j + \mathbf{\tau}_i)}, \quad j = 1, 2, \ldots, k - 1, \quad i = 1, 2, \ldots, s,
\]

or in model (3) which can be written as:

\[
\mathbf{\pi}_{1i} + \mathbf{\pi}_{2i} + \ldots + \mathbf{\pi}_{ji} = \Phi(\mathbf{\theta}_j + \mathbf{\tau}_i), \quad j = 1, 2, \ldots, k - 1, \quad i = 1, 2, \ldots, s,
\]

based on the experimental data. In order to select the model which fitted the experimental data better, one of the goodness of fit statistics, namely the Akaike information criterion was used:

\[
\text{AIC} = -2I(\hat{\mathbf{\pi}}) + 2p,
\]

where \( I(\hat{\mathbf{\pi}}) \) is the log-likelihood function obtained in the current model, and \( p \) is the number of parameters in the model. The smaller the Akaike information criterion, the better the model fits the observed data [Lindsey 1997].

The Akaike criterion was considered the most suitable for the number of observations in the analysis. Other statistics were also tested in order to measure the goodness of fit, e.g. the Schwarz Information Criterion (SIC), -2logL and Deviance, however they produced the same results as the Akaike criterion.

Analysis

In the experiment, observations were made of the trees (experimental units), which were assigned to fixed disjoint categories. As the data were categorised, the analysis was carried out using the generalised linear models: logit and probit. All the calculations were performed in SAS [SAS Institute 1997] using the logistic glm procedure (with logit or probit link function). Based on the results of the analysis, estimates of the unknown parameters in models (2) and (3) were
obtained. A comparison was then made using Akaike’s goodness of fit for both models – the lower the value, the better the goodness of fit.

Finally, the model with the better goodness of fit was selected. Based on the estimates of the parameters of the selected model, the unknown probability of success for each category was evaluated.

It transpired that when the values of the AIC statistics were identical for both the models, the calculated probability also had the same values. In this case, the results of the logit model were selected (as they proved easier to interpret) for presentation in tables 2 and 3. In these tables, the number in brackets ‘(n)’ denotes the standard error of the estimate, asterisk ‘*’ denotes significance at level 0.05, and two asterisks ‘**’ denote significance at level 0.01. In all the cases considered, the unknown probabilities were estimated under the restriction that the effect \( \tau_s = 0 \), and the system of hypotheses \( H_0 : \tau_i = 0 \), against \( H_1 : \tau_i \neq 0 \), for \( i = 1,2,\ldots,s-1 \), was tested.

Results and discussion

Early thinning

Initially, a comparison of the various timber HSs was made to estimate the probability of tree damage during early thinning. The trees remaining after thinning were divided into two categories: damaged trees (category one) and undamaged trees (category two). In this case, it was observed that both models fit the data with the same goodness of fit statistics \( AIC_{\text{logit}} = AIC_{\text{probit}} = 1314.018 \). The results showed that the FTS and LWS differed significantly (\( \alpha=0.01 \)) from the SWS with respect to the level of trees with damage (the estimates of the parameters and values of the Wald statistic for the logit model (2) (tab. 3)).

Additionally, using the logit model to compare the FTS with LWS and based on the value of the Wald statistics, it was found that these systems did not differ significantly. These results were not different to those presented by Bembenek et al. [2013a], where simple statistical analysis (\( \chi^2 \) Pearson test and the Fisher test) also gave the same differences.

Based on the estimates of the parameters (tab. 3), the probabilities of tree damage for each timber HS were calculated using formula (5). The probabilities of tree damage were similar for the FTS and LWS, and were close to 0.16, while the SWS was the method causing the lowest level of damage (fig. 1).

Further analysis was carried out for the damage class to obtain the probabilities of different classes of damage caused to the trees due to the various timber HSs. In this case, based on the AIC fit statistics, the probit model fitted the experimental data better (\( AIC_{\text{logit}} = 617.818, AIC_{\text{probit}} = 617.561 \)), therefore the analysis was based on model (3) with the use of estimate(s) for early thinning.
Table 3. Significance of estimates of parameters and Wald statistics for trees with and without damage (two categories) of three analysed HSs

<table>
<thead>
<tr>
<th>Thinning</th>
<th>Parameter</th>
<th>Estimate</th>
<th>Wald statistic</th>
</tr>
</thead>
<tbody>
<tr>
<td>Early</td>
<td>Intercept</td>
<td>3.8128 (0.20)</td>
<td>355.61**</td>
</tr>
<tr>
<td></td>
<td>$\tau_1$</td>
<td>2.1255 (0.23)</td>
<td>82.16**</td>
</tr>
<tr>
<td></td>
<td>$\tau_2$</td>
<td>2.1463 (0.23)</td>
<td>88.95**</td>
</tr>
<tr>
<td></td>
<td>$\tau_3$</td>
<td>0</td>
<td>–</td>
</tr>
<tr>
<td>Late</td>
<td>Intercept</td>
<td>3.8673 (0.29)</td>
<td>175.87**</td>
</tr>
<tr>
<td></td>
<td>$\tau_1$</td>
<td>2.9712 (0.33)</td>
<td>81.36**</td>
</tr>
<tr>
<td></td>
<td>$\tau_2$</td>
<td>2.0354 (0.32)</td>
<td>39.82**</td>
</tr>
<tr>
<td></td>
<td>$\tau_3$</td>
<td>0</td>
<td>–</td>
</tr>
</tbody>
</table>

Significance level: (*) $\alpha = 0.05$, (**) $\alpha = 0.01$.

Fig. 1. Probability of tree damage

It transpired that the differences between the FTS and LWS methods were not statistically significant from the SWS in respect of each damage class considered separately (tab. 4).

An analysis comparing the FTS and LWS was also conducted and the differences between these systems were also not significant. Based on the formula (6), the probability of the appearance of a particular tree damage class was calculated. The damage class most likely to appear in all the HSs was type 1 (damage class 1, fig. 2). The values of the probabilities were between 0.45 and 0.58 (fig. 2). The next most probable was damage class 3 at about 0.25. The trend for the distribution of the probabilities of tree damage type, however, was similar for all the systems: the FTS, LWS and SWS, and there were no statistically significant differences (fig. 2).
Table 4. Significance of estimates of parameters and Wald statistics for all considered damage classes (nine categories) of three analysed HSs

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Early thinning</th>
<th>Late thinning</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>estimate</td>
<td>Wald statistic</td>
</tr>
<tr>
<td>Intercept</td>
<td>( \theta_1 )</td>
<td>0.2073 (0.23)</td>
</tr>
<tr>
<td>Intercept</td>
<td>( \theta_2 )</td>
<td>0.3003 (0.23)</td>
</tr>
<tr>
<td>Intercept</td>
<td>( \theta_3 )</td>
<td>1.0514 (0.24)</td>
</tr>
<tr>
<td>Intercept</td>
<td>( \theta_4 )</td>
<td>1.2683 (0.25)</td>
</tr>
<tr>
<td>Intercept</td>
<td>( \theta_5 )</td>
<td>1.6969 (0.26)</td>
</tr>
<tr>
<td>Intercept</td>
<td>( \theta_6 )</td>
<td>1.9905 (0.28)</td>
</tr>
<tr>
<td>Intercept</td>
<td>( \theta_7 )</td>
<td>2.0581 (0.28)</td>
</tr>
<tr>
<td>Intercept</td>
<td>( \theta_8 )</td>
<td>2.7488 (0.41)</td>
</tr>
<tr>
<td>FTS</td>
<td>( \tau_1 )</td>
<td>-0.0421 (0.26)</td>
</tr>
<tr>
<td>LWS</td>
<td>( \tau_2 )</td>
<td>-0.2238 (0.25)</td>
</tr>
<tr>
<td>SWS</td>
<td>( \tau_3 )</td>
<td>0</td>
</tr>
</tbody>
</table>

Significance level: (*) \( \alpha = 0.05 \), (**) \( \alpha = 0.01 \).

The results based on formula (6) were different to those in the work of Bembenek et al. [2013a]. The application of the model(s) makes it possible to find out the particular probability of the appearance of a damage class in each analysed HS.

**Late thinning**

As in the case of early thinning, the trees remaining in the forest after late thinning were divided into two categories: damaged trees (category one) and undamaged trees (category two), and the analysis was performed based on models (2) and (3). In this case, the goodness of fit statistics were equal, \( AIC_{\text{logi}} = AIC_{\text{probi}} = 727.090 \) and the logit model was used (due to ease of interpretation). The estimates obtained by the logit model (2) were selected for the analysis, and showed that the FTS and LWS differed significantly from the SWS with respect to tree damage (tab. 3, late thinning). In late thinning, further analysis also showed that differences between the FTS and LWS were significant. In contrast to early thinning, in all the studied systems, the differences in damage were statistically significant at \( \alpha = 0.01 \). These results were similar to earlier work done by Bembenek et al. [2013b]. In late thinning, the heaviest damage was caused by the FTS, with the probability of tree damage close to 0.3, while the least harmful was the SWS, with a probability of tree damage close to 0 (fig. 1). Not all of the tree damage classes that appeared in early thinning were present in late thinning: only the first six damage classes were observed (fig. 2).
Based on the goodness of fit statistics, $AIC_{\text{logit}} = 463.684$ and $AIC_{\text{probit}} = 462.871$, the probit model (3) was selected for the analysis. It was observed that, taking each damage class into account separately, all three HSs did not differ significantly (tab. 4). These results are different to those of Bembenek et al. [2013b], where statistically significant differences were observed between the damage class groups (joined), such as bark (class 1 and 2), cambium (class 3 and 4) and wood (class 5 and 6). The cited work, however, does not explain precisely what the probability was of the appearance of a particular damage class in each HS. The results obtained with the use of the model(s) are more accurate and, ultimately, more useful for the forester, who can precisely select less invasive HS with regards to a particular damage class. It is important when decisions are made concerning particular species. Spruce is particularly vulnerable to fungi infections when the bark is removed, therefore, focus on the probability of the appearance of this particular damage is important and possible when the model is used. Furthermore, the application of both models, logit and probit, gives an even higher level of accuracy in the prediction of the appearance of damage.

**Conclusion**

The parallel use of the logit and probit models made it possible to accurately distinguish the probability of damage in early and late thinning. When only the damaged trees were analysed, the differences between the SWS, and both the FTS and LWS, were statistically significant in early thinning. In contrast, in late thinning, those differences were statistically significant between all the HSs. The use of logit and probit models in this case did not give different results from earlier findings based on the $\chi^2$ Pearson test and the Fisher test.

Further analysis of the probability of the appearance of a particular damage class in each HS was possible only when the models were used, but there were no differences observed which were statistically significant. In previous work,
based on the $\chi^2$ Pearson test and the Fisher test, only damage classes in groups (bark, cambium and wood) were analysed, although with statistically significant differences in some cases.

The new approach using the application of models gives useful results indicating an accurate level of a particular damage class probability in each HS. This can be useful when decisions are made concerning which HS to apply in spruce stands vulnerable to fungi disease.

Additionally the parallel use of the two models, logit and probit, made it possible to choose the one with the better goodness of fit when calculating the probability of the remaining stand damage after thinning. When two categories (damaged trees and undamaged trees) were considered, it was observed (based on the $AIC$ goodness of fit statistics) that both models fit equally well, although the logit model was eventually used (as it was easier to interpret). When nine categories (damage classes) were taken into account, however, the probit model was found to fit the data better. Based on the above results, therefore, it can be concluded that to achieve the best and most accurate results, two models, probit and logit, should be considered in parallel to describe such phenomenon.

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Tomasz BORECKI, Edward STĘPIEŃ, Roman WÓJCIC, Michał ORZECHOWSKI

VERIFICATION OF THE PRINCIPLES OF ACCOUNTING FOR THE SIZE OF THE ALLOWABLE FELLINGS IN FOREST MANAGEMENT PLANNING

The timber market in Poland is dominated by raw materials from the State Forests that conduct planned and sustainable forest management based on forest management plans. Previous rules regulating the planning of final felling are mainly based on the maturity of stands expressed in dominant tree species age and on categories of protection. Attention is paid to the shortage of raw timber material compared to demand and the processing capacity of the industry. There is a danger that unevenness in timber supplies could occur because of the age structure of the forests in the coming decades. Due to the unfavourable age structure of forests in Poland, a not fully satisfactory condition of the resources as well as the high demand for timber in Poland, there is a need for increased usage and regeneration the forests. The amount of harvested timber should be a compromise of silvicultural, protective and productive purposes with the local role of forestry as a stimulator of economic development. Implementation of forest management should take into account the continuity and the relative uniformity of timber supplies in the long term (e.g. fifty years), and a quantitative and qualitative improvement in resources. This requires effective forecasting of forestry resources. The paper presents a methodology for classification of forest stands for felling and the concept of a reassessment of existing accounting rules for the size of the fellings used in the practice of forest management and any related necessary amendments to legal provisions.

Keywords: forest management in Poland, planning of allowable fellings size

Introduction

The current situation

Throughout history the forest was and remains of great interest to mankind. Perceptions about the multi-functionality of the forests have changed over the
centuries in accordance with new priorities. The delivery of timber material was the main function of forests in the days of dynamic industrial development. Currently it is expected that forestry will fulfil its productive function as well as its ecological, protective and social functions. The growth of the range and the changes in the ranking of the forestry tasks bring an inevitable conflict of interest. Different ideas about range and intensity of the utilisation of forest resources are the basis of these conflicts. This is because of how ecology and economy are understood in relation to the function of the forest and the factor of time. Society's increased interest in the protection of nature, the forest environment and its aesthetic beauty has ensured that this subject is widely discussed by forest users. The conducting of a balanced forest economy requires the readiness to compromises and the integration of the positions, of all parties interested in the particular services of the forests. These circumstances conclude that the implementation of these assumptions does not depend only on foresters. The concept of a balanced and sustainable development of forestry should be respected in all areas of the economy and by society as a whole.

In this context the significance of the interdisciplinary approach of the forest management planning process increases [Stepień 1995; Bachmann et al. 2002; Luescher et al. 2005; Przybylska 2005b; Sheppard and Meitner 2005]. This process becomes more complex and requires the co-operation between experts from various fields. The representatives of, among others, the timber industry, agriculture, urbanisation, transport, water management, health care, nature preservation, hunting, tourism and recreation, they all usually act in their own interests with regards to the range and the intensity of utilisation of the forest and its resources. This relates to a need to appease the conflicts arising from the determination of the range of the tasks, and the means of their implementation. This correlates to the conduct of the forest economy, relating especially to the intensity of the use of the forests and the widely understood expectations and limitations concerning nature preservation. [Szucecki 2010; Olaczek 2014a, b]. The role of forest management, therefore, in the planning of temporal and spatial organisation of the forests is increased. Current expectations relating to the multi-functionality of the forests, the flexibility and choice of planning are obligatory in forest management [Mś 2004; Poznański 2004].

It should, however, be noted that good plans and programs do not constitute a guarantee of success. There exists a need for change in the way foresters think, acknowledging the need for self-criticism, curiosity and a lack of prejudice. Such undertakings should contribute to the attainment and enforcement of a suitable synchronisation in the realisation of works in the range of forest management and the protection of forest resources. It is also important to retain the feeling of responsibility for the future and the recognition that the forests are not only objects of management but also permanent fixtures of the landscape.
Premise of the verification of the principles of accounting for the size of the fellings

The increase of the involvement of the forests fulfilling a non-productive function, especially the protective ones, will limit the usage intensity and will reduce the significance of the clear cutting system of management, thus enhancing the complex and modified cutting systems (e.g. the increase in surface of actually regenerating stands, called KO-KDO classes participate in the woods – fig. 1.).

![Bar chart showing forest stand age structure change between 1967 and 2014](image)

**Fig. 1. Change of the forest stand age structure in State Forests between year 1967 and 2014**

This will result in the need to consider new conditions relating to the silvicultural and management planning. The need for certain re-evaluation in this instance results from the three following facts.

1 – Unfavourable from an economic point of view, especially in relation to the possible maintenance of the relative constancy of the supply of raw timber material in the future, is the age structure of the Polish forests (fig. 1).

According to data supplied by the Bureau for Forest Management and Forest Geodesy [GDSF 2015] this structure from 1967-2014 showed approximately a twofold drop in participation of the youngest stands (1-20 years old), manifested by the shortage of age class II stands (21-40 years old) and the tendency of an increase in the proportion of 41-100 year old stands.

2 – The unsatisfactory condition of forest resources in many parts of the country. There are a number of factors, which support this opinion: the mismatch of the species composition with the site (continually still large area coverage...
with pine and spruce monocultures), low nursery and technical quality as well as
the bad health condition of many forest stands, incomplete utilisation of the
productive area due to low stocking and reforestation of the stands, the disorder
in the forest spatial order, including large total areas of stands with little
diversity of age and composition.

Circumstances resulting from the first two facts indicate the urgent need to
increase the intensity of the utilisation and regeneration of the forests, while the
extent of the utilisation should not be treated as the main purpose, but as a tool
of the implementation of the strategic objectives of management. Thus, the
quantity of felled raw material is not merely an internal issue of forestry
management itself. It should be the result of compromises concerning the
principles of forest management that take into consideration silvicultural,
conservation and productive objectives, as well as the widely understood role of
forestry as a stimulus of the economic growth of our country and its regions. The
economic effects of such implementations of usage will result in better supplies
of raw timber material to the market. The outcomes of the nature preservation
character should allow increases in the diversity of the species as well as the
improvement of the age structure of the forest stands, their stability and health
condition.

3 – Lastly, the great demand for wood in Poland, and also the expected
growth of demand for this material in the future [Ratajczak 2013]. This fact
concerns, both, the usable timber, as well as the wood used as an energy source.
The change in the concept in the field of regulations concerning the extent of
forest utilisation, should satisfy growing demand. The new concept relating to
utilisation is based on the conviction that the existence of real premises justifies
the possibility of periodical enlargement of forest utilisation integrated with the
improvement of the condition of resources.

The possibility of shaping the relationship between the forest functions
and timber supply

In the declaration of the third Pan-European Ministerial Conference on the
Protection of Forests in Europe, it states that “the task of European forestry
sector in the XXI century will be the optimisation of its contribution in the forms
of the social, economic, environmental and cultural functions of forests in the
on-going development of society” [Klocek 2001]. The declaration is very
general in its character and it needs to be more specific regarding which of the
functions and at what level they should be implemented, considering the
priorities resulting from the expectations of society. Making a judgment and
choosing which of these functions from the social, natural, and technical-
economical point of view should be the priority, is a complex task and requires
the observance of the requirements of balanced forest management [Kurtilla
2001; Szujecki 2002; Przybyska 2005a; Przybyska and Zięba 2009; Zięba
2012].
The advancement of balanced forest management is determined by internal and external conditions. In the first instance, the difficulty of credible assessment of the influence of the current economic decisions on the future forest development is evident, especially in the context of increasing limitations in the intensity of forest management. The consequences of an external character, result from a dynamically increasing influence originating from the surroundings that render the change prognosis difficult. The doubts concerning the possibilities of implementing the concept of a balanced development of forestry are also a concern, the financial consequences of which have yet to be fully recognised. These undoubtedly, will be caused by the increase in demand for non-productive and protective functions of the forests, especially in the areas under the protective care of Natura 2000 [Olaczek 2014a].

The current understanding of the matter relating to the usage of natural resources should be based on present realities. In a situation where natural resources diminish while the human population grows, increasing the expectations, which are only achievable by the responsible use of such resources by man. This calls for the moderation of the usage of large forested areas. Rational usage of forests, therefore, seals the guarantee of the retention of the versatile character of the forests ecosystem. Mankind has the right to satisfy its material and non-material needs, but not at the expense of nature [Benedict XVI 2009]. This approach is a warrant for the economic and civil development of society. For mankind is a part of nature and should, for its own welfare, manage natural assets rationally not provoking a conflict of objectives arising from economic and protective grounds. The formulation of a strategy of forest usage that fulfills the requirements of the supply of timber requires acknowledgment of historic assumptions relating to the way of conducting forest management and the consequences thereof. For many decades man has influenced the forests which, often, contributed to negative changes to the internal structure of stands (species composition, age) and the disturbances of the spatial build of forest complexes. Such changes, to a considerable degree, were caused by the afforestation of large areas after the Second World War. This fact significantly determines the shaping of the present possibilities and exerts an influence on the form and intensity of the acquisition of usable land. In such situations the specifics of forestry demand monitoring of changes and predicting threats in order to limit economic risk and its consequences in the context of retaining the relative stability in the field of timber supply in the long term.

Controlling the timber supply is a complex problem and should result from the multi-topic analyses (at national level) as well as specifying conditions taken into consideration at the stage of formation of regional forest management plans relating to the felling regulation. Two ideologically different concepts concerning the issue of the regulation are feasible [Borecki and Stepień 2012]. The essence of the first concept is the preference given to the "forest way" principle. It signifies the acceptance of the extent of utilisation resulting from
present realities (age structure, state of resources). One may expect that the
realisation of this concept will duplicate the present age structure and that it will
not ensure the equableness of the usage. Neither will it cause the desirable
acceleration of the improvement of the state of resources. The disadvantage of
this concept is also the fact that it does not take into account the regional
diversification in the encumbrance of non-productive functions which, in turn,
does not influence the satisfaction of conflicting functions fulfilled by forests.

The second concept prefers the desirable direction of the development of
resources (among others: age and species structure, the condition of the supply,
vertical structure, increment) through assignation of the suitable size of the
forest utilisation. The essence of this concept is the observance of the relative
balance between the utilisation and the improvement of the state of resources
simultaneously. Such a task requires the formulation of a prognosis and
recommendations concerning the size of felling on a long term scale, i.e. fifty
years, with regard to the extent of utilisation and the expectations concerning the
minimisation of the contradictions between the principles of equableness and
intensification should be fulfilled. The realisation of the postulate of
equableness, mainly understood as the improvement of age structure and the
state of resources, requires the establishment of the average size of felling yield
($U_{avr.}$) on the basis of the size prognosis ($U_{pr.i}$) for the set timescale of prognosis
and divergence between the average size and prognoses for individual
management periods. The average yield of felling for a given time scale of the
prognosis may be found with the use of the following equation:

$$U_{avr.} = \frac{U_{pr.1} + U_{pr.2} + U_{pr.3} + \ldots + U_{pr.i}}{n}$$  (1)

where: $U_{avr.}$ – average felling yield,
$U_{pr.1, 2, 3, \ldots, i}$ – size of cutting prognosis for individual periods,
$1, 2, 3, \ldots, i$ – index of periods of a given time scale of prognosis,
n – the number of management periods of prognosis.

The divergence between the average and forecast size of felling ($R_i$)
enumerated for individual management periods (1, 2, 3, \ldots, i) of the given time
scale of prognosis may be defined with the use of the following equation:

$$R_i = U_{avr.} - U_{pr.i}$$  (2)

where: $i$ – index as per previous equation 1, 2.

The positive sign of the found divergence, that is, if the average yield is
larger than prognosis it means that, for the observance of relative equability of
felling, there exists a possibility of increase of the utilisation in that period. The
negative value, however, points to the supremacy of the prognosis size over the
average, which shows the possibility of decrease in felling. The size of increase
or decrease of the utilisation depends on a given time scale of prognosis. This
creates the possibility of variant planning of the realisation of the principles of equableness and intensification, as well as flexibility of adjustment to the changes of the situation of the timber market.

In each instance it is necessary to define the principles of determined divergences within the earlier usage (conversion) of the younger stands, as well as selecting the suitable quantity of stands destined for the extension of the time they are retained on the stock. The realisation of the introduced concept requires the formulation of the principles of the detailed classification of stands demonstrative of need and the diligence of interference, as well as the definition of criteria justifying longer retention of some stands. The simultaneous combination of both methods of the conduct for the needs of balancing the divergence may be allowed.

The burden of non-productive functions, especially limitations resulting from protective functions, does influence the intensity of the utilisation [Szulacki 2010; Olaczek 2014a]. It is necessary, therefore, to recognise the regional differentiation of the burden associated with these functions. This should become possible through a methodically uniform system of evaluation and classification of the forest stands within a given managerial-planning body. It therefore indicates the need for the formulation of the principles of forest stand classification that will encompass individual categories of preservation as well as the location of the Nature 2000 areas (the necessity of their “assignment” to forest unit addresses). This should facilitate the development of regional forest management strategies [Szulacki 2002; Zięba 2012; Gieburowski and Janas 2013].

**Materials and methods**

This research is conceptual in nature. It was based on an analysis of existing legislation and instructions regulating the use of forests in Poland. In analyses, the proposed solutions were tested for their use in the forecast state timber resources based on data from the State Forests IT System (as in 2010), and of large-scale National Forest Inventory (for about 20% of all forests, mostly private, that are not covered by these updated IT systems). The State Forests IT System is the best source of regularly updated information about every forest stand in State Forests in Poland.
Results and discussion

Verification of the principles of accounting for the size of cuttings: current practices

The decisions concerning the methods, timing and intensity of conducted fellings do cause long term effects, thus influencing the age and species structure of the forest, as well as the dynamics of the change in resources [Rosa and Smykała 1985; Borecki et al. 2012]. The main utilisation, especially in the forest stands of clear felling units, is strictly connected with the harvesting of the mature crop. In this instance the principles of defining the size of main utilisation are dependent on the maturity criteria [GDSF 2012]. The problem of monitoring becomes more complex in the case of intermediate felling and is connected with the implementation of nurture fellings in younger stands. The size of intermediate utilisation results from subjective assessment of the silvicultural needs defined at the stage of forest stands inspection during the evaluation of the forest. The actual size of intermediate fellings is known only at the time of the execution of these interventions. Hence, the merchantable volume of those fellings has only a general significance and is treated as a prognosis. This size would lose such nature at the moment when, together with the main allowable felled volume it became a component of complete allowable felling cut.

In the Forestry Act and in the decision that sanctions the forests’ management plans the total allowable cut, that is final and intermediate, was seen as the maximum volume of timber estimated for felling over a ten year time period [GDSF 2012]. This meant, that in the case of greater than planned intermediate utilisation (through natural causes), the need for compensation arose, that is, a reduction of planned final utilisation.

Independent of environmental consequences of such conduct – this relates particularly to the units under the shelterwood management system – withholding the implementation of planned positions of the final utilisation considerably disturbed the relationships between the intensity of the process of aging and the process of the felling of stands, as well as the possibility of formation of the desirable age structure of stands. Despite the general acceptance of the need for verification of the method of accounting for the total allowable cut, in the premise of the legal draft to change the Forestry Act (draft dated 14 of August 2013) one may find an entry that does not solve the problem in a satisfactory manner. This entry foresees that the total amount of timber felling yield will be defined in the forest management plan as the final allowable cut expressed in volume and intermediate felling size expressed in area [Forestry Act as stated in 2014].

The incidental removals will not increase the implementation of merchantable volume allowable cut. The above mentioned entry is not entirely clear and requires corrections in the matters relating to area based approach to
the intermediate felling as well as the method of accounting for incidental removals. For the needs of managerial planning this matter is regulated by the entries in the General Director of the State Forests Instruction number thirty that treats the allowable volume of final cuts as “the maximum volume of felling in the duration of the forest management plan”, while the allowable volume of intermediate cuts is determined as “the minimal obligatory area of the intermediate cuts estimated for execution in the duration of the forest management plan and expressed in cubic metres”. There is a noteworthy entry concerning the need for separate accounting for the realisation of both allowable cut volumes “without the possibility of volume compensation” and also allowance for the possibility of exceeding the sizes of intermediate cut usage with regards to the appearance of natural disasters in the forest.

The realisation of forest utilisation during the period between 1984 and 2003 demonstrated [GDSF 2006] that the final allowable cut volume was fulfilled in approximately 86%, while in the intermediate cut - it was approximately 127% (tab. 1). The differences found between the planned and carried out amounts over such a long time period certainly had a negative effect at a national level, as well as at the level of individual units. So far the employed method of defining the allowable volumes of intermediate cut usage did not fulfil expectations.

<table>
<thead>
<tr>
<th>Table 1. Planned and carried out amounts of timber harvest in State Forests during 1984-2003 period (in thousands m³ of net merchantable volume)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Years</strong></td>
</tr>
<tr>
<td>-----------</td>
</tr>
<tr>
<td>1984-1988</td>
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<tr>
<td>1989-1993</td>
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<td>1994-1998</td>
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<td>1999-2003</td>
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<td><strong>Average</strong></td>
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The main cause of the large divergences among planned and performed values in the realisation of intermediate usage appears to be the current definition of the incidental usage. According to the last forest management instructions [GDSF 2003, 2012], the incidental usage is credited to the intermediate or final cut usage. These cuts relate to the removal of, dying trees (deadwood), windthrows and windsnaps. The necessity of logging in the context of these cuts is regionally diverse and occurs virtually every year [Orzechowski and Wójcik 2014]. The participation of incidental cuts in yearly allowable usage volume in State Forests in the years 1990-2009 is illustrated in figure 2. These cuts apply to the removal of, among others, suppressed (dead) trees, broken and wind thrown trees. In the years between 1990 and 2009 the necessity of timber harvesting within those cuts arose every year showing a considerable quantitative differentiation. This participation varies in ranges of between
approximately 15% (in the years 1998, 1999) to approx. 40-45% (in the years 1993, 2002). It is noted that, in order to maintain good health and the sanitary condition of the forest, execution of these cuts should be regarded as a priority.

![Graph showing forest participation]

**Fig. 2. The participation of the accidental cuts in yearly allowable usage volume in State Forests during years 1990 to 2011 (GDSF 2012)**

More often than not, it is interpreted that all incidental usage carried out, outside the areas planned for the final cuts is treated as intermediate usage. Defining the incidental usage in this way originates from the forest management instruction [MLiPD 1957], where the incidental usage is treated as timber utilisation that results as a consequence of sanitary cuts conducted singly or en masse and should not result in the necessity to regenerate.

The definition of the incidental usage was rather different in earlier publications. Jedliński [as per Hausbrandt 1922] claimed that incidental usage includes all un-foreseen uses that occur as an after-effect of circumstances beyond ones control (windthrows, windsnaps, trees damaged by snow). The regulation of the intermediate cut should, therefore, take into account the fact of random occurrences and enable such planning, so that the negative after-effects of the forces of nature in one stand do not necessitate the change in the conduct of the others. The inclusion of the incidental removals of the intermediate or final cut usage and exchangeable compensation of these values brings negative effects to the entire forest unit.

The intermediate cut usage has two functions: the silvicultural and the sanitary, hence, it necessarily follows that, in the intermediate cut usage, one may distinguish two types of cuts: silvicultural and incidental. Rutkowski [1988] defined incidental cuts as an elimination of the sick, dying and dead trees, meanwhile the intensity of these cuts depended on the sanitary condition of the forest stands. He also claimed, that if one wishes to harvest the crop from the
forest (final cut usage) then, the surplus of increment over the size of this usage must be present, originating from the intermediate cuts as well as from the incidental cuts. The entire size must be subject to regulation. The lack of common features between the intermediate and incidental cuts demands that one must not treat them together, instead they should be perceived as two independent types of utilisation.

The conditions of accounting of the allowable usage volume in the forest management plan - legal premises

The changes to the existing legal entry relating to the definition of the total allowable volume cut \( (E_c) \) and the interpretation of the components of this volume are necessary. The proposal for the calculation of the total allowable volume cut is calculated with the use of the following equation:

\[
E_c = E_{UR} + E_{UPR} + U_{PRZ}
\]  

(3)

where: \( E_{UR} \) – set for realisation of optimum final allowable volume cut that constitutes the maximum value and may not be exceeded in the given economic period,
\( E_{UPR} \) – estimated allowable volume of intermediate cut determined on the basis of State Forests Information System (SILP) data concerning the utilisation carried out in the previous period (without the incidental removals); the proposed calculation of the volume size of this usage with the use of the following equation:

\[
E_{UOR(V)} = \sum E_{UOR(P)} \times W_{i, UPR}
\]  

(4)

where: \( E_{UOR(P)} \) – the obligatory allowable intermediate cut area set in the management planning works for individual age sub-classes (ha),
\( W_{i, UPR} \) – the ratio of the intensity of the intermediate usage calculated for individual age sub-classes based on the SILP data concerning executed removals (m³/ha); taking into account the method of calculating this particular usage, at the stage of its realisation there may be divergences between obligatory allowable volume and the actual size of the usage that should, however, not exceed 75% of the expected increase of the group of forest stands destined for intermediate cuts,
\( U_{PRZ} \) – the incidental removals estimated based on data from SILP from the previous management period; if the size of those cuts did not cause the necessity of preparing an appendix; this usage concerns only the volume of the removals that could not be covered under the intermediate usage. In the realization of the plan, the incidental usage may be exceeded in relation to the amount set in the total allowable volume, but by no more than 20% of the total allowable volume that constituted the necessity of preparing the appendix.
The actual size of the utilisation in the management period in individual forest districts as well as in relation to the set allowable volume cut may be larger or smaller. It should be noted, however, that the total allowable volume cut for the regional directorate of State Forests (being the sum of allowable volumes of individual forest districts) has remained an approximation to the realised size of the utilisation.

Accepting the proposed concept of integration of the principle of the equability of usage and the improvement of the condition of resources, the limited size of the usage for State Forests should be the average size of the main utilisation calculated every year for the period of prognosis set in the strategy of SF.

Conclusions

To solve the problems of the age structure of forests in Poland, changes are required in the rules of planning the allowable cuttings size. At the stage of creation of the forest management plan the following modifications have been proposed.

1. In planning the final cut the indexation of stands is needed for the urgency of the required cuts and retention capabilities of stands in the forest. The introduction of a special code is proposed for stand registers, based on the characteristics of individual stands according to the proposed methodology. The final size of the planned use should also take into account the volume of the stands requiring urgent intervention.

2. The approximate allowable volume of intermediate cuts should be calculated on the basis of the obligatory area-based allowable cut in accordance with the formulae proposed earlier.

3. The incidental cuts should be estimated on the basis of the State Forests IT System data from the previous management period.

4. It is important not to connect the planned size of the allowable final, intermediate and incidental cuts, in the forest management plan approved by the Minister. The incidental increase of forest use should not restrict implementation of the remaining tasks.

5. Forest management planning must create, according to a previous proposal, a guarantee of security that limits excessive usage. This guarantee should be calculated using the average size gradually, on the basis of the forecast for the respective periods of the size of the cuts. Adoption of the proposition based on forecasts of the average as the maximum size of the cuts, at present divergences between the average and actual use, creates the possibility of a flexible approach to the needs of the raw wood market.
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Zbigniew Karaszewski, Agnieszka Lacka, Piotr S. Mederski, Andrzej Noskowiak, Mariusz Bembenek

DAMAGE CAUSED BY HARVESTER HEAD FEED ROLLERS TO ALDER, PINE AND SPRUCE

The harvester head causes damage to the bark and wood on the lateral surface of the processed assortment. The severity of the damage may be influenced by the construction of the harvester head and tree species characteristics, such as bark thickness and wood hardness. The study aimed to recognize and compare damage caused to hardwood and softwood. Wood from alder, pine and spruce was harvested using a Valmet 911.4 harvester equipped with a 360.2 head. Logs measuring 2.40 and 2.50 m in length were used in this study. The depth of the damage caused by the feed roller spikes was measured using a digital caliper. Comparison of these defects across the three species revealed that as bark thickness increased so the depth of damage to the timber decreased. Damage to the alder logs in the form of dents and gouged timber fibres was shallow: from 1.7 to 3.7 mm, and significantly less than that to the softwood logs: in the pine from 5.9 to 7.8 mm, and in the spruce from 3.9 to 5.6 mm. Damage to the middle and top logs for each species was similar and depth varied little along the entire length of the processed assortment. Such damage only slightly lowered the quality of the alder wood, which had the thickest bark. Application of the Valmet 911.4 harvester caused less damage to the alder wood than to the softwood (pine and spruce).

Keywords: Wood damage, forest operations, mechanised harvesting, bark thickness, wood quality, black alder, Scots pine, Norway spruce

Introduction

The bark and wood of processed logs are damaged by the harvester head. Certain sections of the harvester head, to a varying degree and extent, can lower the quality of the roundwood. The harvester head is equipped with a set of
2-4 feed rollers which determine the feed speed of the wood and can significantly affect the quality of the delimming process [Sowa et al. 2013]. The rolls should control the transfer of logs at a speed of several metres per second, without slipping or causing excessive mechanical damage [Węgrzyn and Leszczyński 2014]. As an alternative to the steel spikes on feed rollers, rubber spikes were proposed, but although rubber spikes caused less damage to the wood, their traction properties were much poorer [Mäkelä 1993, as cited by Nuutinen et al. 2010].

Researchers have also raised the issue of blue-stain – a secondary defect, which affects wood damaged along the lateral plane. This is related to the loss of bark during transfer through the harvester head [Lee and Gibbs 1996]. A further problem which frequently arises in harvester-processed roundwood is assortments that have not been properly delimbed [Gerasimov et al. 2012]. Hatton et al. [2015] set out to determine the significance of branch cutting speed during processing, and indicated, amongst other things, the need to investigate differences in this parameter according to tree species. Likewise, bucking accuracy can vary depending on the species of the tree being processed, as well as the diameter of the logs cut [Bembenek et al. 2015].

Damage to roundwood caused by feed rollers, the subject of this study, is not well recognised. The work of Spinelli et al. [2011] concerning poplars focused on surface damage to logs, while Mederski [2013] studied the use of a harvester in a mixed pine and birch stand. Connell [2003] investigated eucalyptus wood damage, highlighting the frequency of splitting during the felling and bucking of the logs by harvester.

In this study, the extent of the damage to three species of wood was investigated: one broadleaved – black alder (Alnus glutinosa Gærtn.) and two conifer – Scots pine (Pinus sylvestris L.) and Norway spruce (Picea abies (L.) H. Karst). Pine and spruce are species commonly cut and processed by harvester. Alder, due to morphological features of the trunk which are similar to coniferous species, is also successfully cut by harvester. The analysed species exhibit different physical and mechanical properties. The density of dry wood varies from 450-600, 300-860 and 300-640 kg m⁻³, for alder, pine and spruce, respectively [Wagenführ 2006]. Additionally, these species are characterised by different bark thickness. Taking the above-mentioned characteristics into account, it was hypothesised that the penetration of the feed roller spikes would be shallower when the bark was thicker. Therefore, the objective of the research was mainly to recognise and compare the depth of damage of the broadleaved species – alder, and the two softwood species: pine and spruce. Special attention was paid to the mean and maximum depth of damage and its distribution along the stem.
Materials and methods

Logs from the three species of tree were harvested using a Valmet 911.4 harvester equipped with a 360.2 head. The harvester was produced in 2008 and the feed rollers – which determine the extent of the damage – had been replaced approximately six months prior to the study. There were no signs of wear on the rollers. Roller width was 25 cm and height 45 cm, and it was equipped with 6 steel spikes of 15 mm in height. The roller pressure was 75-120 bars.

Logs measuring 2.50 m (hardwood) and 2.40 m (softwood) in length were cut from the middle and top sections of the stem. Timber from the butt section was not included in this study, as it was processed into long logs and examined according to different methodological assumptions [Karaszewski et al. 2016].

The extent of the damage to the timber was assessed 3 weeks after harvesting in all cases. After this time the cuts had opened slightly allowing more accurate measurements to be taken. The average monthly temperature during the study was 17.2°C. The logs were cut from the following trees: alder from location I (alder I) – 84 years old, and from location II (alder II) – 91 years old, pine – 80 years old, and spruce – 75 years old.

The depth of the damage on the logs was measured using the depth gauge on a Mitutoyo digital caliper (30 cm jaw length) connected to a portable computer. In order to achieve the most accurate results possible, the chip of wood damaged by the feed roller spikes was removed with a chisel. As the trunk moved through the harvester head, one spike cut three ‘walls’ of a wood chip. The fourth ‘wall’ of the chip was cut manually with a chisel and the wood chip was removed using a small screwdriver. Measurements were taken at the deepest point of damage next to the semi-circular wall cut by the steel spikes (fig. 1). The depth of the damage was measured in three sections of each log: at the bottom (the biggest diameter), in the central section and at the top (the smallest diameter). In order to get a full picture of the damage, six measurements were taken within a given section of the roundwood, in four rows created by the spikes along the log (fig. 1). The measurements were taken on both sides of the log, thereby assessing both sides of the feed rollers. The thickness of the bark (together with the phloem), detached from near the point of the depth measurement, was also measured using the Mitutoyo digital caliper. Three measurements of bark thickness were made at each point where depth of damage was measured.

The results of the depth of damage study were analysed according to two criteria: 1) mean depth for a log, where all the measurements in a given set (e.g. the results from a top log) were used to calculate the mean, and 2) maximum depth, where the mean of the maximum depth was taken from three measurement points along the log – top, middle and bottom. Three variables were taken into account: 1) tree species, 2) trunk section – logs cut from the middle or top section of the stem: middle logs and top logs, and 3) in the case of the alder, trees from two stands/locations were analysed - alder I and alder II.
Two alder stands were selected for research in order to have a better representation of hardwood samples. However, in the initial analysis, both the alder group samples were considered separately in case there were any differences.

![Diagram of log damage measurement points](image)

* 2.50 m for alder logs; 2.40 m for pine and spruce logs

**Fig. 1. Log damage measurement points**

Prior to the one-way ANOVA analysis, the Bartlett's test for equality of variance and the Cramér–von Mises criterion to verify the null-hypothesis that the population is normally distributed were performed. The significance level for the analysis was \( \alpha = 0.05 \).

A detailed comparative analysis based on orthogonal contrasts was performed (tab. 1).

<table>
<thead>
<tr>
<th>Tree groups</th>
<th>Contrast 1 alder logs in comparison to the pine and spruce logs</th>
<th>Contrast 2 comparison between the alder logs from both stands</th>
<th>Contrast 3 pine logs in comparison to the spruce logs</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alder I</td>
<td>-1</td>
<td>-1</td>
<td>0</td>
</tr>
<tr>
<td>Alder II</td>
<td>-1</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>Pine</td>
<td>1</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>Spruce</td>
<td>1</td>
<td>0</td>
<td>-1</td>
</tr>
</tbody>
</table>

The following basic contrasts were investigated: Contrast 1 – mean depth of damage recorded in the alder logs (from both stands) in comparison to the pine
and spruce logs; Contrast 2 – comparison of mean depth of damage between the alder logs from both stands; Contrast 3 – mean depth of damage to the pine in comparison to the spruce logs. In addition, the above-mentioned contrasts were investigated for bark thickness. Statistical data was processed using R software.

**Results and Discussion**

In total, 32 trees (16 alder, 8 pine and 8 spruce) and 64 logs were measured (32 alder, 16 pine and 16 spruce), and 2304 depth of damage measurements were taken. Due to the lack of variation between the mean and maximum depth of damage on the middle and top logs for each species, this division was not considered in the models and in further analysis (figs. 2a, 2b).

![Box-plots](image.png)

**Fig. 2.** Mean (a) and maximum (b) depth of damage to middle and top logs

Elements in box-plots: • – single observations; lower whisker is the smallest observation greater than or equal to lower hinge – 1.5 * IQR (the inter-quartile range); lower edge of notch is a median – 1.58 * IQR / sqrt(n); middle is a median; upper edge of notch is a median + 1.58 * IQR / sqrt(n); upper whisker is the largest observation less than or equal to upper hinge + 1.5 * IQR

Likewise, there was no significant difference in the mean (Pr (> F) = 0.064) and maximum (Pr (> F) = 0.053) bark thickness between the middle and top logs (figs. 3a, 3b). There was a statistically significant difference between the bark thickness of the analysed species (Pr (> F) < 2e-16***). The analysis of contrasts revealed that the alder bark was the thickest of all the species (Pr (> |t|) < 2e-16***), and that the pine bark was thinner than the spruce bark (Pr (> |t|) = 2.07e-09***).
Fig. 3. Mean (a) and maximum (b) bark thickness of middle and top logs

Elements in box-plots: • – single observations; lower whisker is the smallest observation greater than or equal to lower hinge – 1.5 * IQR (the inter-quartile range); lower edge of notch is a median – 1.58 * IQR / sqrt(n); middle is a median; upper edge of notch is a median + 1.58 * IQR / sqrt(n); upper whisker is the largest observation less than or equal to upper hinge + 1.5 * IQR

The results of the variance analysis regarding the differences in damage to the wood of the studied species revealed highly significant differences between the mean size of damage as well as the maximum size of damage (tab. 2).

Table 2. Results of variance analysis: mean and maximum depth of damage between species

<table>
<thead>
<tr>
<th>Feature</th>
<th>Intercept</th>
<th>Degrees of freedom</th>
<th>Sum of squares</th>
<th>Mean square</th>
<th>Value F</th>
<th>Pr (&gt; F)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean depth of log damage</td>
<td>species</td>
<td>3</td>
<td>174.26</td>
<td>58.087</td>
<td>87.501</td>
<td>&lt; 2.2 e-16***</td>
</tr>
<tr>
<td></td>
<td>error</td>
<td>60</td>
<td>39.83</td>
<td>0.664</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>total</td>
<td>63</td>
<td>214.09</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Maximum depth of log damage</td>
<td>species</td>
<td>3</td>
<td>197.85</td>
<td>65.950</td>
<td>60.951</td>
<td>&lt; 2.2 e-16***</td>
</tr>
<tr>
<td></td>
<td>error</td>
<td>60</td>
<td>64.92</td>
<td>1.082</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>total</td>
<td>63</td>
<td>262.77</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
</tbody>
</table>

Standard significance code: 0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ‘ 1
The analysis of damage caused to particular species revealed less damage in the alder than the pine and spruce. This was confirmed by both the mean and maximum values (tab. 3).

**Table 3. Mean and maximum depth of damage in the tested species; 95% confidence interval**

<table>
<thead>
<tr>
<th>Tree groups</th>
<th>Mean depth of log damage</th>
<th>Maximum depth of log damage</th>
<th>Mean bark thickness</th>
<th>Mean bark thickness + mean depth of damage</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>mm</td>
<td>mm</td>
<td>mm</td>
<td>mm</td>
</tr>
<tr>
<td>Alder I</td>
<td>±0.4</td>
<td>±0.5</td>
<td>±0.6</td>
<td>±0.6</td>
</tr>
<tr>
<td>Alder II</td>
<td>2.2</td>
<td>3.7</td>
<td>10.6</td>
<td>12.8</td>
</tr>
<tr>
<td>Pine</td>
<td>1.7</td>
<td>3.3</td>
<td>11.3</td>
<td>13.0</td>
</tr>
<tr>
<td>Spruce</td>
<td>5.9</td>
<td>7.8</td>
<td>2.2</td>
<td>8.1</td>
</tr>
</tbody>
</table>

The mean depth of damage ranged between 1.7 mm for alder II and up to 5.9 for the pine, while the maximum depth of damage ranged from 3.3 mm for alder II to 7.8 mm for the pine (tab. 3). Regarding bark thickness, the relation was precisely the opposite: the lowest mean thickness was recorded for the pine bark (2.2 mm), while the spruce bark was thicker (5.0 mm). The alder bark from both stands was the thickest (10.6 and 11.3 mm for alder I and II, respectively; tab. 3).

Highly significant differences in the mean depth of damage to the alder compared to the coniferous species were confirmed, Pr (>|t|) < 2e-16*** (tab. 4).

**Table 4. Basic contrast values**

| Contrasts                                                      | Estimator | Standard error | Value t  | Pr (>|t|)          |
|---------------------------------------------------------------|-----------|----------------|----------|-------------------|
| Contrast 1 alder logs in comparison to the pine and spruce logs | 1.582     | 0.130          | 12.166   | < 2e-16***        |
| Contrast 2 comparison between the alder logs from both stands | -0.210    | 0.184          | -1.143   | 0.258             |
| Contrast 3 pine logs in comparison to the spruce logs        | -1.065    | 0.184          | -5.791   | 2.74e-07***       |

Standard significance code: 0 '****' 0.001 '***' 0.01 '**' 0.05 '.' 0.1 1.

No difference was recorded in the mean depth of damage to the alder wood regarding location, Pr (>|t|) = 0.258 (tab. 4). Statistical analysis revealed highly
significant differences in the mean depth of damage to the pine logs in comparison to the spruce, Pr (> |t|) = 2.74e-07*** (tab. 4).

The deepest total penetration (understood as penetration through the bark and wood tissue) caused by the feed roller spikes was measured in the alder stems. The total depth of spike penetration for this species was 12.8-13.0 mm, while the results for the two coniferous species were significantly lower (tab. 3).

The comparison of all the species measured revealed a clear linear relation between the depth of damage and bark thickness, in both mean and maximum damage results (fig. 4).

![Fig. 4. Relation between mean and maximum depth of damage and mean thickness of bark for all the analysed species](image)

Regression analysis of the dependent variables reflected the linear models for depth of damage:

\[
\text{Mean depth of damage} = 6.386 - 0.406 \times \text{ToB}_{\text{mean}} \quad (1)
\]

\[
\text{Maximum depth of damage} = 8.315 - 0.440 \times \text{ToB}_{\text{mean}} \quad (2)
\]

where: ToB – thickness of bark.

For the depth of damage models, the coefficients of determination $R^2$ were 0.776 and 0.742 for the mean and maximum, respectively. The significance of
the coefficient estimators was statistically confirmed for both the models with \( \Pr (|t| < 2e^{-16}) < 0.0001 \).

The bark of the black alder, especially in older trees, is quite thick and split into scales [Surmiński 1980]. In the research presented, an age difference of 7 years between both the alder stands (locations) did not reveal any variation in the depth of damage. Apart from the effects of the harvester head on stem transfer, it might be expected that thicker bark would provide better protection for the wood beneath. The 15 mm-long spikes of the feed rollers punctured the bark, additionally compressing it and then damaging the wood. As the bark of pine and spruce is thinner, the spikes penetrated the outer wood layers more deeply. However, wood tissue cannot be ignored, and possibly the harder wood of the alder could have limited the spike penetration in comparison with the softwood of the pine and spruce. The fact that the alder wood suffered the least feed roller damage is as hypothesised, although this has not been confirmed in any literature to date. The correlation of the degree of damage – both mean and maximum values – on the mean thickness of bark provides a starting point for further discussion on log harvesting in broadleaved and mixed tree stands. An important methodological element of this paper was the investigation into the three timber species harvested using the same machine and harvester head. A different machine equipped with a head with a different configuration of spike shape and size would result in damage on a different scale. A comparison of five types of feed rollers performed by Nuutinen et al. [2010] revealed different damage depths in birch, pine and spruce. The depth of damage in the tested species exhibited broad dispersion: birch 1.8-6.0, pine 4.2-8.7 and spruce 4.3-8.7 mm, with mean values of 3.7, 5.5 and 5.8 mm, respectively. The results presented in this paper revealed that the alder suffered the least damage, with a mean value of 1.7-2.2 mm. The mean damage to the pine and spruce was 5.9 and 3.9 mm, respectively.

The processing of unbarked wood was mentioned for a reason. During harvesting in spring at the start of the growing season, logs stripped of their bark after harvesting are a common sight, especially in broadleaved species. The delimbing knives can easily strip the logs which means the harvester head may, to some extent, be in contact with the stripped wood and affect the timber directly, thereby increasing the depth of the damage.

Generally speaking, damage caused by the Valmet 360.2 harvester head did not exceed a depth of 1 cm. This information is beneficial to practitioners from both the forestry and timber industries. Worth noting is that the resulting depth of damage should in fact be doubled, considering that it occurs on both sides of the outer surface of the stem. For this reason, such damage to the wood should perhaps be recognised as a significant factor in wood processing. The applied regulations, standardisation or technical framework conditions should take these defects into consideration.
In assessing damage to the outer layer, one aspect should be emphasized. When timber is left in the forest, such defects, which are comparable to shallow wounds, allow access to pathogens which affect the quality of the wood to a greater extent than the original damage. Thus far, there has been no data in the literature on disease progression in hardwood timber after machine harvesting. In conifers, Lee and Gibbs [1996] noted a maximum development of blue-stain on up to 10% of the surface at 10 weeks after harvesting. Due to how the head functions, harvesters can spread fungi that cause discolouration in softwood. The feed roller spikes carry fungal spores which are transferred as the spikes penetrate the wood [Uzunovic et al. 2014]. Fungal disease may not only affect industrial wood, but may also be a reason for the lowering of the quality of wood chips for energy purposes [Kropacz and Fojutowski 2014]. It should also be remembered that damage to the outer surface of the stem increases the risk of accidents during processing, especially where lines are not fully automated [Connell 2003].

The mean and maximum values presented show the problem of wood quality deterioration from a slightly different perspective. The mean values provide a better picture of all the resulting damage, and also include information about the absence of damage in accordance with the methodology used. During the study, there were points where 3 to 4 measurements out of 6 equalled 0 mm. On the one hand, these values reduced the value of the mean, making it possible to marginalize the problem of timber damage. On the other hand, they illustrated the depth of damage over a larger area of wood. Zero values and values less than the maximum were present in the outer rows measured (the damage created in four rows on the timber by the feed rollers was measured). Additionally, the size of the trees felled by the harvester should be taken into consideration. For thinner assortments, the feed roller spikes were only able to cut into the bark or wood of a processed log to a limited extent (fewer rows). Thick assortments, where the feed rollers are in contact with more of the surface, were affected differently.

A maximum depth of timber damage is to be expected from the spikes which create the middle row of damage. This is the point where the feed rollers first make contact with the wood. The maximum values indicate how deeply the timber is damaged, where the incision or indentation of wood fibres end. In the case of timber production for garden components, wood processing cuts to the maximum depth of damage. During processing, shallower damage is removed after surface cutting, while the deepest damage requires a greater depth of log to be removed and causes a major loss of wood. It therefore seems important to make data on both aspects available to interested parties, who will then be able to exploit this information in relation to the timber processing methods they apply.
Conclusions

The quality of the alder wood processed using the Valmet 911.4 harvester equipped with the 360.2 head was rated as good as regards the damage caused by the feed rollers. This assessment was based on a comparison of the quality of the black alder wood with the Scots pine and Norway spruce. This machine can be used for the harvesting of alder timber with the expectation that little damage will appear on the logs.

Damage to the alder timber in the form of indentations and incisions in the wood fibres was shallow, up to 0.5 mm, and was significantly less deep than damage on the softwood logs. The main reason was a difference in bark thickness. The comparison of the three species confirmed that as the thickness of the bark increased, the depth of damage decreased. However, the harder wood could have also caused the shallower spike damage to the alder. The hardwood defects did not lower its quality significantly. The depth of the damage was similar along the whole length of the processed log.

Damage to the softwood did not exceed 1 cm. The pine timber was affected to a greater extent than the spruce.

There was no difference in the damage level between the top and middle logs.

There is a risk of secondary defects such as discolouration, which can cause a lower efficiency in plywood production from valuable assortments.

The findings within this paper suggest that further research into this field is recommended, taking into account the following variables: other broadleaved forest species of different wood hardness, high quality assortments (mainly veneer and plywood timber), the harvesting season, and varying harvester heads.

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Acknowledgments

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Agnieszka JANKOWSKA, Paweł KOZAKIEWICZ

DETERMINATION OF FIBRE SATURATION POINT OF SELECTED TROPICAL WOOD SPECIES USING DIFFERENT METHODS

The main objective of this research was to determine the fibre saturation point of tropical wood. Two different methods were used to achieve this aim: the logarithm of strength properties versus moisture content and volumetric shrinkage-moisture content plot to zero shrinkage. The test included selected wood species from Africa: Opepe, Iroko, African padouk, and Wenge, and South America: American mahogany and Ipe. For comparison, selected domestic wood species of a similar structure – European beech (Fagus sylvatica L.) – were also tested. Determination of the fibre saturation point of the selected wood species using two methods delivered similar results (the small differences were not significant). The results showed that, generally, the fibre saturation point of the tropical wood species was lower than in the case of the European wood species. The lowest values of the fibre saturation point were shown by the African padouk and Ipe (approx. 17 %). Moreover, it was found that in the case of the tropical wood, the basic density had a significant influence on the sorption properties of the tested wood species.

Keywords: tropical wood, European beech, fibre saturation point, compressive strength, volumetric shrinkage

Introduction

The demand for tropical wood products has led to increased trade on markets, as well as further knowledge of the characteristics of exotic wood, such as its physical, mechanical and technological properties. Knowledge in this field is still incomplete and often limited to a presentation of the names and appearance of the wood. For an evaluation of the sorption behaviour of domestic wood species the sorption properties of fir are usually used [Popper et al. 2007, 2009; Adampoulos and Voulgaridis 2012]. Due to differences in wood structure and chemical composition, it seems clear that differences in the sorption behaviour will appear [Popper et al. 2009]. A determination of the fibre saturation point, as

Agnieszka JANKOWSKA (agnieszka.jankowska@sggw.pl), Paweł KOZAKIEWICZ (pawel.kozakiewicz@sggw.pl), Warsaw University of Life Sciences, Warsaw, Poland
well as the physical and mechanical behaviour of the wood as a hygroscopic material are very useful in the drying, conversion and utilization of timber [Hamami et al. 1998].

The term fibre saturation point (FSP) was introduced by Tiemann [1906] early in the 20th century, in connection with his work on strength-moisture relations. Since then, the fibre saturation point has been the subject of numerous investigations. In spite of this fact, discussion concerning how it should be defined and measured is ongoing [Babiak and Kudela 1995]. The concept of FSP is defined in terms of the theoretical condition of the wood when its cell cavities are completely devoid of water, while at the same time the cell walls are saturated with water.

This value can be determined using many methods. Some of them require extrapolation, e.g.: moisture content adsorption isotherms to unit relative vapour pressure, the differential heat of wetting (moisture content plot to zero heat evolved), volumetric shrinkage-moisture content plot to zero shrinkage or shrinkage involved, determining the ratio of the total volumetric shrinkage to the green volume specific gravity and a correction for the average density of the absorbed water. Two methods are involved in determining the transition point using the relationship: determination of the logarithm of electrical conductivity versus the moisture content, and determination of the logarithm of the strength properties versus the moisture content [Stamm 1964]. The described methods of determining the fibre saturation point of wood are characterized by some limitations. Therefore, it is advisable to determine the fibre saturation point using more than one method. The sorption behaviour of wood has been described in detail in the literature [Themelni 1998; Popper et al. 2006, 2007]. However, knowledge in this area is still incomplete due to the number of new wood species on the European market [Popper et al. 2009; Adampoulos and Voulgarridis 2012].

Determining the properties of the tropical wood commercially available on the European market is important and the results of research should be taken into account during the stage of wooden product design. A knowledge in this area would help manufacturers avoid many problems during the exploitation of wooden products such as floors. The main objective of this research was to determine the fibre saturation point in wood of various morphologies and to improve the knowledge of the sorptive properties of tropical wood. Sorption tests were combined with measurements of the physical and mechanical properties providing to obtain the value of the fibre saturation point of the wood species selected for the tests. This group included tropical wood from Africa and South America.
Materials and methods

The wood species used in this study are summarized in table 1.

<table>
<thead>
<tr>
<th>Trade name according to PN-EN 13356:2005</th>
<th>Latin name</th>
<th>Plant family</th>
<th>Origin</th>
<th>Special features</th>
</tr>
</thead>
<tbody>
<tr>
<td>African padouk</td>
<td><em>Pterocarpus soyauxii</em> Toub.</td>
<td><em>Fabaceae</em></td>
<td>West Central Africa</td>
<td>axial parenchyma in narrow bands</td>
</tr>
<tr>
<td>American mahogany</td>
<td><em>Swietenia macrophylla</em> King.</td>
<td><em>Meliaceae</em></td>
<td>South America</td>
<td>irregular fibres arrangement, axial parenchyma in narrow bands</td>
</tr>
<tr>
<td>European beech</td>
<td><em>Fagus sylvatica</em> L.</td>
<td><em>Fagaceae</em></td>
<td>Europe (Poland)</td>
<td>wide wooden rays</td>
</tr>
<tr>
<td>Ipe</td>
<td><em>Tabebuia</em> sp.</td>
<td><em>Bignoniaceae</em></td>
<td>South America</td>
<td>irregular fibres arrangement</td>
</tr>
<tr>
<td>Iroko</td>
<td><em>Milicia excelsa</em> (Welw.) C.C.Berg</td>
<td><em>Moraceae</em></td>
<td>West Central Africa</td>
<td>irregular fibres arrangement, axial parenchyma in bands</td>
</tr>
<tr>
<td>Opepe</td>
<td><em>Nauclea diderrichii</em> Merrill.</td>
<td><em>Rubiaceae</em></td>
<td></td>
<td>irregular fibres arrangement</td>
</tr>
<tr>
<td>Wenge</td>
<td><em>Millettia laurentii</em> De Wild.</td>
<td><em>Fabaceae</em></td>
<td></td>
<td>axial parenchyma in wide bands</td>
</tr>
</tbody>
</table>

Samples of each wood species were collected from one board to obtain “identical samples”. Thanks to this, the samples were similar and the structure was preserved in order to avoid differences in the tested properties caused by differences in wood anatomy. The samples, each measuring 30 mm (T) × 30 mm (R) × 5 mm (L), were prepared in order to find the dimensional changes at different levels of relative humidity. Furthermore, samples measuring 20 mm (T) × 20 mm (R) × 30 mm (L) were prepared in order to determine the compressive strength along the fibres at different levels of relative humidity. The specimens were exposed to a moisture sorption test (adsorption), as follows: oven-drying, conditioning at five different levels of relative humidity ranging from 9 to 97%, and water wetting. As soon as each point of sorption was reached, the mass of specimens was measured to the nearest 0.001 g and their dimensions were taken to the nearest 0.01 mm. The conditioning of the specimens to an appropriate moisture content was possible with the use of sealed enclosures in which prescribed saturated salt solutions were placed at a temperature close to 20°C. The relative humidity was monitored and recorded using a hygrometer. The salt solutions used to create various levels of the relative humidity of air at 20 ±2°C are listed in table 2. A criterion for equilibrium was established as three successive identical mass readings at 24 hour intervals.
The FPS was obtained at the intersection of the extrapolated sets of data - the results of testing the strength of the wood at different levels of moisture content ($R_{mc}$) and after wetting ($R_w$) in water, and in the second method, the results of determining partial wood shrinkage ($S_{mc}$) and total wood shrinkage ($S_v$). The procedure was described by Stamm [1971], and details of how the FSP was determined are shown figure 1.

Table 2. Relative humidity of air at a constant temperature $20 \pm 2^\circ C$ obtained in sealed enclosures with the use of saturated salt solutions

<table>
<thead>
<tr>
<th>Saturated salt solution</th>
<th>Relative humidity in [%] at $20 \pm 2^\circ C$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Potassium hydroxide KOH</td>
<td>9</td>
</tr>
<tr>
<td>Magnesium chloride MgCl</td>
<td>37</td>
</tr>
<tr>
<td>Sodium bromide NaBr</td>
<td>55</td>
</tr>
<tr>
<td>Sodium chloride NaCl</td>
<td>70</td>
</tr>
<tr>
<td>Potassium sulphate K$_2$SO$_4$</td>
<td>97</td>
</tr>
</tbody>
</table>

Fig. 1. Scheme of determination FSP: a – using volumetric shrinkage ($S_{mc}$ – volumetric shrinkage at different levels of moisture content, $S_v$ – total volumetric shrinkage), b – using compressive strength along fibres ($R_{mc}$ – compressive strength at different levels of moisture content, $R_w$ – compressive strength after water wetting)

The equilibrium moisture content of the samples was determined according to the standards PN-D-04100:1977 and ISO 3130:1975. The wood density of the samples was determined according to PN-D-04101:1977 and ISO 3131:1975. The volumetric shrinkage of the wood was determined according PN-D-04111:1982 and ISO 4858:1982, while determination of the compressive strength along the fibres was made based on PN-D-04102:1979 and ISO 3787:1976.
Results and discussion

The average values of the measured equilibrium moisture content for adsorption at 20°C are shown in table 3. For each wood species, 10 samples were used. In the case of every wood species tested, the equilibrium moisture content at different levels of humidity was diverse. The largest differences in the moisture content (MC) were visible when the air humidity was highest (approx. 97%).

<table>
<thead>
<tr>
<th>Wood species</th>
<th>Equilibrium moisture content at relative humidity</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>9%</td>
</tr>
<tr>
<td>African padouk</td>
<td>2.10</td>
</tr>
<tr>
<td>American mahogany</td>
<td>3.28</td>
</tr>
<tr>
<td>European beech</td>
<td>3.00</td>
</tr>
<tr>
<td>Ipe</td>
<td>2.11</td>
</tr>
<tr>
<td>Iroko</td>
<td>2.25</td>
</tr>
<tr>
<td>Opepe</td>
<td>3.01</td>
</tr>
<tr>
<td>Wenge</td>
<td>2.75</td>
</tr>
</tbody>
</table>

The effects of the determination of the FSP of the selected wood species using two different methods are given in table 4 and table 5. It may be said that irrespective of the wood tested, the results of FSP determination were almost identical and the small differences were not significant (falling within the margin of error). The results show that, generally, the fibre saturation point of the tropical wood species was lower than in the case of the European beech. The highest value of the FSP was observed for the beech wood. The lowest values of the fibre saturation point were shown by the African padouk and Ipe (approx. 17%).

Furthermore, the results showed that the basic density of the tropical wood had a significant influence on the sorption properties of the tested wood species (the European beech wood was not included in this analysis). The same conclusion was reached by Hernandez [2006] following research on nine tropical hardwoods from Peru and sugar maple wood from Quebec. According to Babiak and Kúdela [1995], it is not only wood density but also wood structure that can play an important role. The wood species tested revealed a similar structure. All of the species constituted diffuse-porous wood. In some cases, expanded axial parenchyma were observed (Wenge, Iroko), while in others they were absent.
Table 4. Parameters describing relationship between volumetric shrinkage of wood and relative humidity of air

<table>
<thead>
<tr>
<th>Wood species</th>
<th>Approximation of linear function in hygroscopic interval</th>
<th>Total volumetric shrinkage</th>
<th>Computed value of fibre saturation point</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>equal</td>
<td>$r$</td>
<td>[%]</td>
</tr>
<tr>
<td>African padouk</td>
<td>$S_v=0.4232\cdot MC$</td>
<td>0.998</td>
<td>7.45</td>
</tr>
<tr>
<td>American mahagony</td>
<td>$S_v=0.4520\cdot MC$</td>
<td>0.997</td>
<td>11.00</td>
</tr>
<tr>
<td>European beech</td>
<td>$S_v=0.6751\cdot MC$</td>
<td>0.996</td>
<td>21.56</td>
</tr>
<tr>
<td>Ipe</td>
<td>$S_v=0.6688\cdot MC$</td>
<td>1.000</td>
<td>12.53</td>
</tr>
<tr>
<td>Iroko</td>
<td>$S_v=0.3965\cdot MC$</td>
<td>0.998</td>
<td>9.00</td>
</tr>
<tr>
<td>Opepe</td>
<td>$S_v=0.5799\cdot MC$</td>
<td>1.000</td>
<td>13.34</td>
</tr>
<tr>
<td>Wenge</td>
<td>$S_v=0.8099\cdot MC$</td>
<td>0.998</td>
<td>16.73</td>
</tr>
</tbody>
</table>

$S_v$ – total volumetric shrinkage, MC – moisture content of wood, $r$ – coefficient of correlation.

Table 5. Parameters describing relationship between compressive strength along fibres of wood and relative humidity of air

<table>
<thead>
<tr>
<th>Wood species</th>
<th>Approximation of linear function in hygroscopic interval</th>
<th>Compressive strength along the fibres after soaking wood in water</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>equal</td>
<td>$r$</td>
</tr>
<tr>
<td>African padouk</td>
<td>$R_c=145.6 – 4.182\cdot MC$</td>
<td>-0.994</td>
</tr>
<tr>
<td>American mahagony</td>
<td>$R_c=75.5 – 1.953\cdot MC$</td>
<td>-0.997</td>
</tr>
<tr>
<td>European beech</td>
<td>$R_c=122.2 – 3.480\cdot MC$</td>
<td>-0.989</td>
</tr>
<tr>
<td>Ipe</td>
<td>$R_c=183.3 – 5.229\cdot MC$</td>
<td>-0.996</td>
</tr>
<tr>
<td>Iroko</td>
<td>$R_c=88.3 – 2.519\cdot MC$</td>
<td>-0.994</td>
</tr>
<tr>
<td>Opepe</td>
<td>$R_c=115.8 – 2.670\cdot MC$</td>
<td>-0.996</td>
</tr>
<tr>
<td>Wenge</td>
<td>$R_c=153.4 – 3.077\cdot MC$</td>
<td>-0.994</td>
</tr>
</tbody>
</table>

$R_c$ – compressive strength along fibres at different moisture content, MC – moisture content of wood, $r$ – coefficient of correlation

The difference between the fibre saturation point of the European beech wood and the tropical wood was probably caused by the number of extractives in the wood structure which may act hydrophobically. This supposition finds confirmation in the literature [Popper et al. 2009; Adampoulos and Voulgarridis 2012]. Previous research has indicated the influence of extractives on the sorptive properties of wood. However, studies in this area are incomplete and knowledge in this field of expertise should be expanded. European beech is a non-heartwood species and the other tested wood was derived from the
heartwood zone. The differences between the FSP of the European beech and tropical wood seem obvious, but differences between tropical wood species require verification.

![Graph showing the relationship between fibre saturation point and density of tested tropical wood species](image)

**Fig. 2. Relationship between fibre saturation point and density of tested tropical wood species**

Analysis of the obtained results is reason enough to consider further calculation of the FSP according to the formula given by Vorreiter [1949], Trendelenburg and Mayer-Wegelin [1955] and Krzysik [1957]:

\[ \text{FSP} = S_v \cdot \frac{G_{\text{H}_2\text{O}}}{G_0}, \]

where \( S_v \) – total volumetric shrinkage, \( G_{\text{H}_2\text{O}} \) – density of water and \( G_0 \) – density of absolutely dry wood. According to our calculation, this equation should not be used in the case of tropical wood. Only in the case of the European beech were the results similar. In the case of all the tested tropical wood species using the presented formula, the values of FSP were lower and the differences were significant, e.g. in the case of the African padouk, the FSP value was 11.86%. For this reason it may be said that information concerning the sorptive properties of wood should not be generalized and the knowledge in this area needs to be extended.

**Conclusions**

During the test it was confirmed that two methods of determining the fibre saturation point - the logarithm of the strength properties versus the moisture content and the volumetric shrinkage – moisture content plot to zero shrinkage delivered similar results. The findings showed that, generally, the fibre saturation
point of tropical wood species (heartwood zone) was lower than in the case of European wood species. The lowest values of the fibre saturation point were shown by the African padouk and Ipe (approx. 17%).

It was also found that in the case of the tropical wood, the basic density had a significant influence on the sorption properties of the tested wood species. The fibre saturation point of the tropical wood species was negatively correlated with the wood density.

The influence of extractives on sorptive properties will be considered in future research.

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List of standards

ISO 3130:1975. Wood – Determination of moisture content for physical and mechanical tests


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Agnieszka LASKOWSKA, Ewa DOBROWOLSKA, Piotr BORUSZEWSKI

THE IMPACT OF ULTRAVIOLET RADIATION ON THE COLOUR AND WETTABILITY OF WOOD USED FOR FACADES

The study examined the effect of UV radiation on the colour and wettability of cedar, gaboon, meranti and Scots pine wood. The tested species of exotic wood are mainly used in European countries for facades. For this kind of usage they act as a substitute for pine wood. In the study, the colour parameters were determined using the CIE L*a*b* colour space model. The tests showed that the most significant changes in the lightness of the wood occurred after 20 hours of UV radiation exposure. After that time, the L* value change was linear. The cedar wood turned the darkest and it also showed the greatest total change of colour ΔE. Comparable colour changes ΔE, and thus the greatest colour stability of all the tested wood species, were shown by the gaboon and meranti. In addition, it should be noted that all the tested wood species were characterized by a much smaller susceptibility to colour change under UV radiation than the pine sapwood and heartwood. The results also revealed that UV radiation significantly affects the contact angle, and therefore the surface free energy of the cedar and pine sapwood.

Keywords: cedar, gaboon, meranti, Scots pine, ultraviolet radiation, colour, surface energy

Introduction

Wood used for facades is exposed to a number of biotic and abiotic factors. An important factor that predetermines whether wood can be used as an exterior wall material is colour stability and water permeability, and, in the case of impregnation, permeability to chemicals. In several European countries, Scots pine is the wood species commonly used for facades. However, more and more often exotic species of wood, such as cedar, gaboon or meranti, are used. Its main advantages are: the absence of knots, a uniformly coloured heartwood and its original texture.
Colour is described as the phenomenon of the selective reflection and dissipation of light rays of a particular wavelength which reach the eye in the form of a spectrum determined for a given body. During the process of the natural ageing of wood, e.g. its use outdoors, the colour of wood changes. Colour changes are a good indication of the qualitative evaluation of wood surface resistance to the impact of environmental factors [Aydin and Colakoglu 2005; Tolvaj and Mitsui 2010]. The degree of colour change depends on many factors, including wood species [Pastore et al. 2004; Tolvaj et al. 2013], type of protective layer [Aydin and Colakoglu 2005; Deka et al. 2008], and conditions of use [Persze and Tolvaj 2012]. Colour change is also affected by the chemical composition of the wood [Hon and Glasser 1979; Yazaki et al. 1994; Gierlinger et al. 2004]. Temperature and UV radiation exposure time are the factors with the greatest effect on wood colour change [Sharratt et al. 2009, Huang et al. 2012a]. With an increase in temperature and the time of its impact, the colour of unprotected and protected wood surfaces at first darkens but, in time, it can also turn grey or fade [Feist 1983; Feist and Hon 1984].

Changes in wood surface properties can be reflected in changes in the free surface energy determined by the contact angle [Petrič and Oven 2015]. Wetting, which is regarded as a property of the wood surface, has a crucial impact on the processes of adhesion related to surface coating and the formation of protective layers [Cao and Kamdem 2007; Sandberg 1996]. Changes in wood moisture content have a direct impact on the changes in its structural properties and the physico-chemical properties of its surface. In the case of wood finished with varnish or paint, the contact angle determines the quality and the protective properties of the obtained layer. Wood with a low capacity of moisture absorption maintains its resistance much longer than wood species with a higher susceptibility to wetting. It should be noted that apart from the properties of the wetting substance, the parameters of the wood surface, i.e. the arrangement of annual growth rings, the ratio of late wood to early wood, wood type (sapwood, heartwood), type of anatomical section, also have an impact on surface wettability [Mantanis and Young 1997]. Gindl et al. [2006] studied the effects of ultraviolet light exposure on the wetting properties of wood. Nussbaum [1999] and Huang et al. [2012b] stated that the type of wood machining affects wood wetting. Moreover, the ratio of non-structural elements in wood is important [Nzoekou and Kamdem 2004]. The variety in the methodology of wood wettability tests is worth noting. In the case of tests for wood wettability, it is important to determine the time after which the contact angle is calculated, and thus the surface free energy for the wood-reference liquid system. The wetting angle can be determined after a drop of the liquid separates itself from a needle, i.e. when $t = 0$ s [Küdela 2014], several seconds after placing the drop of the reference liquid on the wood surface and before the total absorption of the drop into the wood structure [Santoni and Pizzo 2011].
The impact of ultraviolet radiation on the colour and wettability of wood used for facades. It was particularly important to determine the direction of the changes in the wood colour components, i.e. \( L^* \), \( a^* \) and \( b^* \), depending on the UV radiation time. This factor is crucial in the process of predicting the character of wood colour difference and choosing the wood species for facades more efficiently, resulting in the higher aesthetic value of buildings with such facades.

**Materials and methods**

The following species of wood were used for the analysis: cedar (**Thuja plicata** Donn. ex D. Don), gaboon (**Aucoumea klaineana** Pierre), meranti (**Shorea** spp.) and Scots pine (**Pinus sylvestris** L.). The heartwood of the cedar was used for testing. The Scots pine (sapwood and heartwood) was used as a comparative material. The sample used for analysis had a tangential-radial section on the broad surface. Selection of this wood section for testing resulted from the fact that wooden elements used for facades have most often tangential-radial sections. Basic data concerning the tested wood species is presented in table 1. Planing was used to finish the surface of the wood samples. The wood moisture content was determined in accordance with ISO 13061-1:2014. The density of the wood was determined in accordance with ISO 13061-2:2014. The wood moisture ranged from 6.38\% (±0.11\%) to 6.73\% (±0.25\%) in all the tested wood species. The density of the cedar wood amounted to 361 kg/m\(^3\) (±10 kg/m\(^3\)), the gaboon wood to 412 kg/m\(^3\) (±12 kg/m\(^3\)), and the meranti wood to 539 kg/m\(^3\) (±24 kg/m\(^3\)), while the density of the sapwood and the pine heartwood totalled 466 kg/m\(^3\) (±17 kg/m\(^3\)) and 558 kg/m\(^3\) (±9 kg/m\(^3\)), respectively.

**Table 1. Selected information concerning analysed wood species**

<table>
<thead>
<tr>
<th>Latin name</th>
<th>English trade name of wood (and code) according to EN-13556:2003</th>
<th>Origin</th>
<th>Structure of wood</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Aucoumea klaineana</strong> Pierre</td>
<td>gaboon (AUKL)</td>
<td>Africa</td>
<td>diffuse-porous hardwood</td>
</tr>
<tr>
<td><strong>Pinus sylvestris</strong> L.</td>
<td>Scots pine (PNSY)</td>
<td>Europe</td>
<td>softwood</td>
</tr>
<tr>
<td><strong>Shorea</strong> spp.</td>
<td>dark red meranti (SHDR)</td>
<td>Asia</td>
<td>diffuse-porous hardwood</td>
</tr>
<tr>
<td><strong>Thuja plicata</strong> Donn. ex D. Don.</td>
<td>western red cedar (THPL)</td>
<td>North America</td>
<td>softwood</td>
</tr>
</tbody>
</table>

The wood samples were subjected to ultraviolet radiation within a spectrum range of 340-360 nm. Four fluorescent lamps were used. Each of them had a capacity of 100W. The used source of radiation imitated solar radiation, in particular the UVA component. The UVA component causes the greatest changes
in the appearance and structure of organic materials exposed to the external environment. This results from the fact that UVA constitutes 90-95% of the solar radiation reaching Earth [Miller et al. 1998]. The wood samples were exposed to UV radiation for 300 hours, with the colour parameters determined every 20 hours. The tests were carried out indoors, in normal climatic conditions (a temperature of 20 ±2°C and relative humidity of 65 ±5%). The colour parameters were determined using the CIE L*a*b* colour space model. A 3nh NH300 colorimeter was used. The lightness (L*), the chromatic coordinate on the red-green axis (a*) and the chromatic coordinate on the yellow-blue axis (b*) were determined before and after radiation, as well as the ΔE colour difference, in accordance with ISO 7724-3:2003. In the case of the analysed parameters of wood colour, the trend lines were set and the parameters of the equation of curve (y) as well as the coefficient of determination \( r^2 \) were provided.

Table 2. Data for surface tension and components of the test liquids

<table>
<thead>
<tr>
<th>Liquid</th>
<th>Properties</th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>surface tension</td>
<td>dispersion</td>
<td>polar</td>
<td>acid</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[mN/m]</td>
<td>[mJ/m²]</td>
<td>[mJ/m²]</td>
<td>[mJ/m²]</td>
</tr>
<tr>
<td>Water ((H_2O))</td>
<td>72.80</td>
<td>21.90</td>
<td>51.00</td>
<td>25.5</td>
<td>25.50</td>
</tr>
<tr>
<td>Diiodomethane ((CH_2I_2))</td>
<td>50.80</td>
<td>50.80</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
</tbody>
</table>

Re-distilled water was used as a reference liquid for the wettability calculations. The surface free energy of the tested wood species was determined in accordance with the Owens-Wendt method [Owens and Wendt 1969], on the basis of the sessile drop method, measuring the contact angles of the re-distilled water and diiodomethane (tab. 2). A Phoenix 300 goniometer manufactured by Surface Electro Optics was used to determine the wettability parameters. Given the number of variables influencing the wettability of wood [Petrič and Oven 2015], the time after which the wetting angle should be marked has not been determined unambiguously. Liptáková and Kúdela [1994] and Kúdela [2014] determined the angle after a drop of liquid separated itself from the needle and in the condition of balance. Huang et al. [2012a] diversified the time the wetting angle of jack pine wood \((Pinus banksiana)\) depending on the type of reference liquid. Therefore, in this case it was assumed that the test of the values of the contact angles would be performed 3 seconds after placing the drop of the reference liquid on the samples’ surface. The statistical study of the test results was carried out at a significance level of 0.050.
Results and discussion

Substantial colour differences were noted in the case of the exotic wood species and pine (sapwood – S and heartwood – H) exposed to UV radiation. The most significant change was a darkening of the wood, in particular of the cedar and pine heartwood. The numerical values confirmed the results of the organoleptic observations.

![Graph](attachment:image.png)

**Fig. 1. Change of lightness ($L^*$) of wood under UV radiation exposure**

The changes in wood lightness under the influence of UV radiation are presented in figure 1. The study showed that the most substantial changes in wood lightness occurred after 20 hours of UV radiation exposure. After that time, the $L^*$ value change was linear. The research results obtained confirm the results of research conducted by other authors. Tolvaj and Mitsui [2010] showed that the greatest changes in the lightness of wood species such as black locust, beech, Japanese cedar and spruce occurred within the first 20 hours of sunlight irradiation time.

The lightness of the cedar, gaboon and meranti directly after planing were at a comparable level, i.e. 67.67 ($\pm$1.36), 63.90 ($\pm$1.50) and 65.14 ($\pm$2.65), respectively. The pine wood was characterized by a colour much lighter than the exotic wood tested. The $L^*$ values for the pine sapwood and heartwood were 81.43 ($\pm$3.48) and 80.76 ($\pm$0.81), respectively. It was observed that the greatest
difference in wood lightness was in the case of the cedar. After 20 and 300 hours of UV radiation exposure, a drop in the $L^*$ value by 12% and 18%, respectively, was found. The smallest changes in lightness were noticed in the case of the meranti wood. After 20 and 300 hours of UV radiation exposure, the $L^*$ value for the meranti wood amounted to 63.29 ($\pm5.31$) and 61.60 ($\pm1.74$) respectively, translating into a 3% and 5% decrease in the $L^*$ value.

Changes in the other colour component values were also observed. The tests showed that the most significant changes in the $a^*$ colour component for the gaboon, meranti and pine wood occurred after 20 hours of UV radiation exposure (fig. 2). After that time, the $a^*$ value change was linear.

![Graph showing changes in $a^*$ parameter of wood colour under UV radiation exposure](image)

**Fig. 2. Change in $a^*$ parameter of wood colour under UV radiation exposure**

Based on the analysis of the changes in the chromatic coordinate on the red-green colour axis, it was concluded that, after UV exposure, the meranti and pine wood (sapwood and heartwood) showed a tendency to become redder, while the cedar showed a tendency to become greener. The gaboon wood showed a colour change from red to green ($\Delta a^*$ after 20 and 300 hours of UV exposure was 0.18 ($\pm0.02$) and $-0.15$ ($\pm0.02$), respectively). The biggest changes in the $a^*$ parameter were observed in the case of the pine wood. After 300 hours of UV exposure, the $\Delta a^*$ values for the pine sapwood and heartwood were 4.66 ($\pm0.29$) and 6.44 ($\pm1.16$), respectively. The changes in the redness of the meranti...
were not as noticeable as those of the pine wood. In the case of the meranti, the \( \Delta a^* \) value after 300 hours of UV exposure totalled 0.90 (±0.07).

Changes in the chromatic coordinate value on the yellow-blue colour axis \( (b^*) \) were analysed and the results are presented in figure 3. It was concluded that after UV radiation exposure, the colour of the gaboon, meranti and pine (sapwood and heartwood) changed to yellow. In turn, the cedar wood showed a colour change from blue to yellow. The biggest changes in the \( b^* \) parameter were observed for the pine wood. After 300 hours of UV exposure, the \( \Delta b^* \) values for the pine sapwood and heartwood were 12.71 (±0.79) and 11.71 (±1.11), respectively. In the case of the meranti and gaboon, the \( \Delta b^* \) values after 300 hours of UV radiation exposure amounted to 7.58 (±1.93) and 5.05 (±1.60), respectively.

Lignin is a natural polymer which constitutes a kind of binder in wood structure. Softwood contains more cellulose (up to 60%) and lignin (up to 30%), but less hemicelluloses (ca. 10%) than hardwood. In hardwood, the cellulose content amounts to 50%, lignin ca. 20% and hemicelluloses ca. 30% [Pożgaj et al. 1993]. According to Persze and Tolvaj [2012], chromophoric groups in wood are mostly found in the lignin, extractives and their derivatives. Lignin derivatives are mainly responsible for the process of wood yellowing. Yellowing is the main wood colour change resulting from lignin photodegradation. Due to
the fact that softwood contains more lignin, it is more susceptible to the yellow oriented colour change, confirmed in the tests conducted. Persze and Tolvaj [2012] state that low extractive contents may be the reason for the low thermal effect on specimens exposed to light irradiation. This results in wood colour changes in the red colour space. Those dependencies were also confirmed by tests carried out by Mitsui et al. [2001]. Colour parameter changes may result from the differences in extractive contents in particular wood species. Pandey [2005] tested colour changes in natural wood, without extractives. Wood samples without extractives were characterised by moderate colour changes over a longer irradiation time.

![Graph showing the total colour difference (ΔE) of the tested wood species over time.](image)

**Fig. 4. Total colour difference (ΔE) of the tested wood species**

The most important parameter to describe the stability of the colour of the tested wood species is the total colour difference ΔE, which is the resultant of the changes in individual parameters, i.e. colour components. On the basis of the calculated ΔE values, it can be concluded that the greatest total colour difference in the gaboon, meranti and pine woods occurred at the beginning of UV radiation exposure (fig. 4). The colour changes are particularly noticeable within the first 100 hours of exposure. These relations were not so strong in the case of the cedar wood, whose ΔE remained within a narrow spectrum. The most significant colour changes, and thus the lowest colour stability, were characteristic of the pine wood. The ΔE value of the pine heartwood after 100
and 300 hours of UV radiation exposure amounted to 15.99 (±1.64) and 19.92 (±1.58), respectively. The ΔE value for the pine sapwood after 100 and 300 hours of UV radiation exposure totalled 11.29 (±1.18) and 15.70 (±0.91), respectively. In general, it can be stated that the gaboon and meranti were characterized by similar ΔE values, which were half the ΔE value of the pine sapwood.

Changes in the particular colour components and the total colour difference in the tested wood species under UV radiation exposure in time (t) were described using curves. Table 3 presents the relations between $L^*(t)$, $a^*(t)$, $b^*(t)$ and $\Delta E(t)$ determined for $t \geq 20$ h. Based on the presented comparisons, it is possible to forecast the changes in the wood colour components and the total colour difference depending on the UV irradiation time. It should be noted that the values of the coefficient of determination $r^2$ were from 0.84 to 0.99. This indicates the significant correlation between the colour components of the tested wood and the UV irradiation time.

<table>
<thead>
<tr>
<th>Wood species</th>
<th>Parameters</th>
<th>$a^*$</th>
<th>$b^*$</th>
<th>$r^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$y = at + b$</td>
<td>$r^2$</td>
<td>$y = aln(t) + b$</td>
<td>$r^2$</td>
</tr>
<tr>
<td>cedar</td>
<td>$y = -0.0053t + 15.47$</td>
<td>0.96</td>
<td>$y = 0.69ln(t) + 20.78$</td>
<td>0.98</td>
</tr>
<tr>
<td>gaboon</td>
<td>$y = -0.0043t + 14.06$</td>
<td>0.87</td>
<td>$y = 1.07ln(t) + 16.31$</td>
<td>0.97</td>
</tr>
<tr>
<td>meranti</td>
<td>$y = 0.0024t + 12.47$</td>
<td>0.85</td>
<td>$y = 2.27ln(t) + 11.90$</td>
<td>0.97</td>
</tr>
<tr>
<td>Scots pine (S)</td>
<td>$y = 0.0151t + 8.48$</td>
<td>0.93</td>
<td>$y = 3.45ln(t) + 15.47$</td>
<td>0.98</td>
</tr>
<tr>
<td>Scots pine (H)</td>
<td>$y = 0.0113t + 13.12$</td>
<td>0.88</td>
<td>$y = 1.42ln(t) + 26.68$</td>
<td>0.92</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Wood species</th>
<th>Parameters</th>
<th>$L^*$</th>
<th>$\Delta E$</th>
<th>$r^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$y = at + b$</td>
<td>$r^2$</td>
<td>$y = aln(t) + b$</td>
<td>$r^2$</td>
</tr>
<tr>
<td>cedar</td>
<td>$y = -0.0115t + 59.33$</td>
<td>0.96</td>
<td>$y = 1.04ln(t) + 5.33$</td>
<td>0.94</td>
</tr>
<tr>
<td>gaboon</td>
<td>$y = -0.0046t + 59.39$</td>
<td>0.88</td>
<td>$y = 0.91ln(t) + 2.39$</td>
<td>0.84</td>
</tr>
<tr>
<td>meranti</td>
<td>$y = -0.0069t + 63.34$</td>
<td>0.85</td>
<td>$y = 1.89ln(t) - 1.92$</td>
<td>0.89</td>
</tr>
<tr>
<td>Scots pine (S)</td>
<td>$y = -0.0159t + 77.54$</td>
<td>0.95</td>
<td>$y = 3.84ln(t) - 5.99$</td>
<td>0.97</td>
</tr>
<tr>
<td>Scots pine (H)</td>
<td>$y = -0.0258t + 72.83$</td>
<td>0.92</td>
<td>$y = 3.51ln(t) + 0.22$</td>
<td>0.99</td>
</tr>
</tbody>
</table>

Wood exposed to UV radiation should be characterized in terms of the surface properties determining further behaviour in an aggressive environment (water, impregnating agents) and, as a consequence, susceptibility to degradation. An analysis of the water contact angle makes it possible to determine the characteristics of the surface properties. Determining these characteristics makes it possible to predict interactions between the wood and the wetting materials. During analysis of the contact angles, the change in their
values in time constitutes an important factor [Gindl et al. 2001; Wolkenhauer et al. 2009].

![Fig. 5. Contact angle of water on wood (a) and surface free energy (b) of wood before and after 300 hours of UV radiation](image)

The tests showed that the UV radiation had a significant impact on the contact angle of the cedar and pine sapwood (fig. 5a). Those dependencies determined the value of the surface free energy (fig. 5b). This was due to the fact that the tested softwood had a lower density than the hardwood. The decisive factor was the openness of the wood structure for water permeability. Water not only penetrated the crevices of the cell walls, but it also went through the cellular pits and cell bore holes. After 300 hours of UV irradiation, the contact angle of the cedar and pine sapwood was, respectively, 75% and 30% greater than the contact angle of the cedar and pine sapwood after planing. The surface free energy of the cedar and pine sapwood was, respectively, 28% and 12% lower than the surface free energy of the cedar and pine sapwood after planing. These changes may have resulted from the extractive changes in the wood under the influence of UV irradiation [Teacă et al. 2013]. As a result of wood aging, the wood fibers rise, which leads to an increase in wood surface area and its greater susceptibility to wetting. Those dependencies have been confirmed by tests conducted by Nzokou et al. [2011].

**Conclusions**

The tests conducted showed that ultraviolet radiation significantly contributed to the change in colour and wettability of the tested wood species. After irradiation, the cedar, gaboon and meranti wood were darker than the pine heartwood or sapwood. All the tested wood species showed the greatest decrease in lightness...
after 20 hours of UV irradiation. After that time, the $L^*$ value change was linear. All the tested exotic wood species were characterised by less significant colour changes, as compared to the pine sapwood and heartwood. It was concluded that the greatest total colour changes in the case of the gaboon, meranti and pine wood occurred after 100 hours of irradiation. Such dependencies were not observed for the cedar wood. The significant influence of UV irradiation on the contact angle and the surface free energy of the cedar and pine sapwood was also observed.

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**List of standards**

**EN 13556:2003.** Round and sawn timber – Nomenclature of timbers used in Europe


**ISO 13061-1:2014.** Physical and mechanical properties of wood – Test methods for small clear wood specimens – Part 1: Determination of moisture content for physical and mechanical tests


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Tomasz ZIELENKIEWICZ, Jan SZADKOWSKI, Michał DROŻDŻEK, Anastazja ZIELENKIEWICZ, Teresa KŁOSIŃSKA, Andrzej ANTCZAK, Janusz ZAWADZKI, Jakub GAWRON

APPLICATION OF X-RAY FLUORESCENCE TECHNIQUE FOR DETERMINATION OF HEAVY METALS UPTAKE BY DIFFERENT SPECIES OF POPLAR

X-ray fluorescence (XRF) analysis of six metals: chromium, manganese, iron, nickel, copper and zinc (possible inhibitors of enzymatic hydrolysis of wood) in samples of two poplar species, Populus trichocarpa and Populus maximowiczii, was performed in order to check which of them collect more of the metallic inhibitors during tree growth and steam explosion pretreatment. The XRF point scan (for solid and ashed wood) and mapping (for stem cross-sections) options were used. Samples of the different parts – stem, branches, leaves and bark were studied. Steam explosion at 130°C, 160°C and 190°C was performed on both species and the influence of steam on the chosen metals content was analysed. On the basis of the results, P. trichocarpa is the species which accumulates a higher amount of the metals during tree growth and P. maximowiczii – during steam explosion.

Keywords: XRF, poplar, enzymatic hydrolysis, inhibitors, hot water pretreatment

Introduction

Poplar is a common fast growing tree which can be used for bio-fuel production. It may be useful as solid fuel [Barontini et al. 2014] as well as the raw material further processed to biogas [Galvagno et al. 2009] or liquid bio-fuels [Antczak et al. 2014]. This last application is probably the most complicated as processing contains many stages: preparation of raw material, hydrolysis, fermentation, and distillation are the most important of them. The hydrolysis stage may be
improved on, as shown in the results of the studies presented in this paper. Nowadays biomass hydrolysis is preferably performed via the enzymatic process, although acidic hydrolysis is better known. Acidic hydrolysis causes a higher negative impact on the natural environment and that is why the application using enzymes has been focused on. Enzymatic hydrolysis is basically a well known process and has been used many times by different authors [e.g., Studer et al. 2011], but the high cost of the enzymes to be used makes the process unprofitable. That is why the studies on new, more efficient and less expensive enzymes are continually performed. On the other hand, there are also some parameters of the ligno-cellulosic material that can be improved to decrease the quantity of enzyme needed. These are, among others, the material disintegration degree, its porous structure or the initial chemical composition. The content of possible enzymatic hydrolysis inhibitors in raw material is also a very important factor which should be taken into account.

Poplar, like all other trees, contain many different low-molecular mass non-structural components. These are compounds such as fatty acids, terpenes, resins and others. Many of them are believed to be the inhibitors of the enzymatic hydrolysis [Chandel et al. 2013; Jönsson et al. 2013]. There is also another type of enzyme poison – heavy metals, which may be up-taken from the soil before tree-cutting, or during further material pretreatment. Iron, copper, chromium and nickel are mentioned in the literature [Chandel et al. 2013]. As the new species of poplar are artificially cultivated, there is also a need to check their ability to absorb specified metals. The choice of the best species for further processing will be easier, when there is more knowledge about their properties.

Some types of material pretreatment may also cause the metals content to increase. Steam explosion is one of them. Recently, this has been an increasingly popular method of ligno-cellulosic materials pretreatment [Tutt et al. 2014]. It consists of material treatment with steam at a temperature exceeding 100°C. As the conditions in the reactor are very severe, many different organic compounds may form from the wood during this treatment. Another question arises: could the reactor material pollute the treated ligno-cellulosic material with the aforementioned metals?

The aim of this paper was to investigate the differences in metal content and distribution in two species of poplar before and after steam pretreatment, and to check the usefulness of the XRF technique for this purpose. This is a fast technique which may be successfully used for comparative measurements of wood elemental composition [Zielenkiewicz et al. 2012].

**Materials and Methods**

Two common species of poplar, *Populus trichocarpa* and *Populus maximowiczii*, were analysed. Two and a half year old trees were cut (three specimens of each species). Samples of trunk (breast height), branch, bark and
leaves were collected from each tree. A Spectro Midex M XRF spectrometer was used to analyse the content of iron, copper, chromium and nickel which inhibiting action was stated. Trunk and branch were measured as wood pieces (without disintegration), bark and leaves were in the form of milled powder. Each sample was measured three times with the “point scan” method (each “point” was a 2 mm × 2 mm surface), the time of the x-ray exposure was 300 s. The results, in ppm, should be treated qualitatively and comparatively, because the XRF spectrometer calibration was originally prepared with respect to metallic types of samples.

Disks from a breast height of 2.5 year old stems of _Populus trichocarpa_, _Populus maximowiczii_ and _Populus tremula_ (as the reference material) were collected. Additional disks from 4 year old stems of _P. trichocarpa_ and _P. maximowiczii_ were collected to check the influence of tree age on the metals uptake. In order to analyse the distribution of chosen metals on the stem cross-section, the “mapping” option of the XRF spectrometer was used. The surface of the disk was divided into 2 mm × 2 mm squares, the time of x-ray exposure (each square) was 30 s. The values of so called impulse counts for each analysed metal, were the results for comparison and metals distribution determination.

In order to check the influence of steam pretreatment on the chosen metals content, additional measurements were performed. Samples of 2.5 year old _P. trichocarpa_ and _P. maximowiczii_ (cca. 5 g of each) were treated in water boiled in a hermetic stainless steel reactor (0.5 litres ) up to 130°C, 160°C and 190°C using an oil bath. The time from the beginning of heating until the end of the experiment (rapid decompression) was 6 hours. Samples were rinsed with distilled water, dried at 105°C until a constant mass was reached and ashed for 6 hours at 600°C in a muffle furnace. The increase in temperature from 20°C to 600°C lasted an additional 2.5 hours. Ashing was performed in order to pre-concentrate samples (it was used for this purpose by Harju et al. 1997). The ash obtained was analyzed with the XRF “point scan” method, as above.

**Results and discussion**

The results of the content of particular elements in both poplar species are collected in figures 1-6. It was assumed that both poplar species had the same matrix for XRF analysis. Without this assumption comparison of the results obtained for _P. trichocarpa_ and _P. maximowiczii_ would be unjustified [Zielenkiewicz et al. 2012]. Results for wood samples, however, cannot be compared with bark and leaves, because these parts of the tree definitely form another type of matrix, especially after they were milled. In addition to the heavy metals mentioned earlier, described by Chandel et al. [2013] as hydrolysis inhibitors, manganese and zinc were analysed as additional elements. They are present in wood in high proportions and that is why their analysis is easier. Additionally these elements are important for the trees metabolism.
The comparison of chromium content in both poplar species is presented in figure 1. *P. trichocarpa* contains more chromium in the trunk and leaves while *P. maximowiczii* – in the branches and bark. The most significant difference between the species is observable in branch samples. Chromium content is the highest in the trunk in both cases.

Fig. 1. Chromium content in analysed samples of *P. trichocarpa* and *P. maximowiczii*

Fig. 2. Manganese content in analysed samples of *P. trichocarpa* and *P. maximowiczii*
The results of the manganese analysis are presented in figure 2. Manganese content in *P. trichocarpa* samples of trunk and leaves is higher (like in the case of chromium) in relation to *P. maximowiczii*. Visible differences are even more significant. The content of this metal is the highest in the leaves. Differences between values obtained for the trunk and branches are much more significant in the case of *P. trichocarpa* (like in the fig. 1).

Results of the iron analysis are presented in figure 3. Also, this time *P. trichocarpa* samples contain more iron in the trunk and leaves samples. The highest content of iron is found in leaves for *P. trichocarpa* and in the bark for *P. maximowiczii*. Again, the difference between values obtained for the trunk and branches is more significant in *P. trichocarpa* samples.

The results of nickel content in the analyzed samples are presented in figure 4. Its content in the trunk is higher in *P. trichocarpa*, as in previous cases, while values obtained for the leaves are similar in both species. The branches and bark of *P. maximowiczii* contain more nickel in relation to *P. trichocarpa*. There are similar relations between trunk and branches as in previous cases.

![Iron content in analysed samples of *P. trichocarpa* and *P. maximowiczii*](image)

Copper content (fig. 5) in the trunk of *P. trichocarpa* is higher in relation to *P. maximowiczii*. In other samples values are higher for *P. maximowiczii*. The highest value in *P. trichocarpa* was reported for the trunk again, while in *P. maximowiczii* branches contain the highest amount of copper.

Similar results were obtained for zinc (fig. 6). Relations between particular values are parallel to copper.
Fig. 4. Nickel content in analysed samples of *P. trichocarpa* and *P. maximowiczii*

Fig. 5. Copper content in analysed samples of *P. trichocarpa* and *P. maximowiczii*
Fig. 6. Zinc content in analysed samples of *P. trichocarpa* and *P. maximowiczii*

There are many different observations that can be made. First of all, the content of each analysed element in trunk wood is higher for *P. trichocarpa* samples. A difference of even 37.5% in the case of manganese. In contradiction, all values for branch wood are higher in the case of *P. maximowiczii* samples (up to 70% for chromium). This may mean that *P. maximowiczii* trees accumulate heavy metals evenly within the whole volume of the plant – values for branch and trunk are similar for most of the analysed elements (in branch: lower for chromium, manganese and iron, higher for copper and zinc, exactly the same for nickel). Results for *P. trichocarpa* are always higher in trunk samples and the differences are significant. It may also indicate a more effective transport of nutrients to branches in *P. maximowiczii*.  

The content of all analysed elements in the bark is higher in *P. maximowiczii* samples and for zinc it is almost three times higher than in *P. trichocarpa*. Results obtained for leaves are ambiguous. The contents of chromium, manganese and iron are higher in *P. trichocarpa* samples (even 47% higher in the case of manganese) but, contents of copper and zinc are clearly lower in this species while the content of nickel is almost the same in both of poplars. It does not correspond to differences observed in branches at all. Generally, leaves are the only part of the tree where no unequivocal tendency is observed.

An additional interesting observation is that the contents of nickel, copper and zinc in bark and leaves are much lower in relation to the contents of manganese and iron (in samples of *P. trichocarpa* bark contents of copper and zinc is even below the determination limit), while in trunk and branch, values of all elements are more or less on the same level. Values for chromium are of
a similar dependence, but, they are also significantly lower in relation to manganese and iron in branch samples.

The distribution of metals on a cross-section of both the studied species are presented in figures 7a-7f (2.5 and 4 year old) and P. tremula (2.5 year old) for comparison. Ranges of metal impulse counts correspond to different colours. Distribution of chromium is visible in figure 7a. A darker colour means a higher content of the element. It may be observed that the diameter of P. tremula is the lowest and of P. trichocarpa – the highest (each square dimension is 2 mm × 2 mm). The distribution of chromium is rather regular. P. tremula contains the highest amount of chromium on almost the whole surface of the cross-section, while 4 year old P. trichocarpa – the lowest amount (the darkest colour surface is much smaller than on other graphs).

![Fig. 7a. Distribution of chromium on the cross-sections of P. maximowiczii, (2.5 and 4 year old) P. trichocarpa (2.5 and 4 year old) and P. tremula (2.5 year old) poplar with XRF mapping option (values of impulse counts)](image)

Distribution of manganese is more regular in comparison to chromium (fig. 7b). Again, the P. tremula sample contains a higher amount of analysed metal than others. In the outer parts of other samples (especially 4 year olds), near the bark, darker surfaces may be observed. This is probably a phloem area, however, the difference in relation to the rest of the cross-section is insignificant.

Graphs with the distribution of iron are collected in figure 7c. The distribution is regular and the levels of impulse counts values are similar for all species. Distribution is more differentiated for only one sample, 4 year old P. trichocarpa. Increased iron content in the phloem section may be caused by the cutting saw.
Fig. 7b. Distribution of manganese on the cross-sections of P. maximowiczii, (2.5 and 4 year old) P. trichocarpa (2.5 and 4 year old) and P. tremula (2.5 year old) poplar with XRF mapping option (values of impulse counts)

Fig. 7c. Distribution of iron on the cross-sections of P. maximowiczii, (2.5 and 4 year old) P. trichocarpa (2.5 and 4 year old) and P. tremula (2.5 year old) poplar with XRF mapping option (values of impulse counts)
The distribution of nickel (fig. 7d) is rather regular. Some differences are observed (excluding the *P. tremula* sample) but no true pattern could be found. Similar observations may be denoted in the case of copper distribution (fig. 7e). Some increase in the copper content is visible in the phloem area (only for *P. maximowiczii*).

![Cross-sections of P. maximowiczii and P. trichocarpa](image)

**Fig. 7d. Distribution of nickel on the cross-sections of *P. maximowiczii*, (2.5 and 4 year old) *P. trichocarpa* (2.5 and 4 year old) and *P. tremula* (2.5 year old) poplar with XRF mapping option (values of impulse counts)**

An increase of zinc content (fig. 7f) in the phloem area is observed for all samples excluding *P. tremula*. The distribution of this element is regular in other parts of the samples.

In summary, mapping measurements of stem cross-sections collected from breast height does not give any information about the possible areas in stem, where metals are accumulated. Some increases in the phloem area are insignificant in character. Older samples seem to contain lower amounts of the analysed metals.

Table 1 shows the contents of the studied elements in the ashes is the ashes of samples. Both species were first submitted for steam explosion at three different temperatures: 130°C, 160°C and 190°C. Reference samples of ash without steam treatment were also measured. First of all, differences between data observed in table 1 and figures 1-6 should be explained. Solid wood and ash are definitely different matrices for XRF measurements. That is why there is no justification for any attempt at comparison of these two sets of results.
Fig. 7e. Distribution of copper on the cross-sections of *P. maximowiczii* (2.5 and 4 year old) *P. trichocarpa* (2.5 and 4 year old) and *P. tremula* (2.5 year old) poplar with XRF mapping option (values of impulse counts)

Fig. 7f. Distribution of zinc on the cross-sections of *P. maximowiczii* (2.5 and 4 year old) *P. trichocarpa* (2.5 and 4 year old) and *P. tremula* (2.5 year old) poplar with XRF mapping option (values of impulse counts)
Table 1. Average contents (in ppm) of chosen metals in ashed samples of *P. trichocarpa* and *P. maximowiczii* after steam treatment in 130, 160 and 190°C

<table>
<thead>
<tr>
<th>Element</th>
<th>Cr</th>
<th>Mn</th>
<th>Fe</th>
<th>Ni</th>
<th>Cu</th>
<th>Zn</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trich</td>
<td>11</td>
<td>120</td>
<td>56</td>
<td>–*</td>
<td>12</td>
<td>160</td>
</tr>
<tr>
<td>Trich 130</td>
<td>128</td>
<td>172</td>
<td>2383</td>
<td>14</td>
<td>74</td>
<td>319</td>
</tr>
<tr>
<td>Trich 160</td>
<td>17</td>
<td>120</td>
<td>1373</td>
<td>16</td>
<td>33</td>
<td>174</td>
</tr>
<tr>
<td>Trich 190</td>
<td>32</td>
<td>126</td>
<td>1464</td>
<td>40</td>
<td>63</td>
<td>208</td>
</tr>
<tr>
<td>Max</td>
<td>–</td>
<td>47</td>
<td>73</td>
<td>–</td>
<td>19</td>
<td>170</td>
</tr>
<tr>
<td>Max 130</td>
<td>–</td>
<td>41</td>
<td>503</td>
<td>–</td>
<td>58</td>
<td>271</td>
</tr>
<tr>
<td>Max 160</td>
<td>–</td>
<td>25</td>
<td>1500</td>
<td>–</td>
<td>46</td>
<td>172</td>
</tr>
<tr>
<td>Max 190</td>
<td>26</td>
<td>91</td>
<td>6002</td>
<td>19</td>
<td>40</td>
<td>1201</td>
</tr>
</tbody>
</table>

*Below determination level.

Comparing reference samples of *P. trichocarpa* with samples after steam explosion at 160°C and 190°C, it may be observed that the contents of all the analysed elements increase with temperature. The increase is insignificant for Mn and Zn but, the content of others raises fivefold (Cu) or even almost 30fold (Fe). The values for all metals, however, (excluding nickel) are the highest in the case of 130°C treatment. This is quite a surprising result and in the context of the rest of the positions should be probably acknowledged as an error.

Results for *P. maximowiczii* samples are not so unequivocal. Treatment at 190°C causes the highest increase in content of all metals excluding copper. Iron content is almost 100 times higher and zinc content is about sevenfold higher than in reference samples without treatment. The influence of lower treatment temperatures is also visible but the dependence is not obvious. Comparing the results for both species, it may be stated that the influence of 130°C and 160°C on studied elements content is quite similar. Treatment at 190°C causes significantly higher increases of iron and zinc content in the *P. maximowiczii* sample.

**Conclusion**

More heavy metals are accumulated in the trunk of *P. trichocarpa* compared to that of *P. maximowiczii*. As the trunk contains most of the poplar mass at this age, *P. trichocarpa* probably generally collects higher amounts of heavy metals, including hydrolysis inhibitors such as chromium, nickel, copper and iron. Thus, if other important parameters for material processing (to bio-fuels) of these species are similar, it can be concluded that *P. maximowiczii* is recommended for further processing.

Mapping studies suggest that older samples may contain lower amounts of metallic inhibitors.
Additional XRF analysis of all samples after ashing could be the verification of the presented comparison. Although laborious, ashing is a good method for the samples pre-concentration. It unifies the matrix and raises the certainty that more parallel results for both analysed species could be obtained. In addition, more comparable results will be obtained for different wood fractions (wood, bark and leaves).

The content of metals for ashed samples after steam explosion was found to be much higher than without treatment. This may lead to the conclusion that decomposition of the material at 190°C steam treatment in the case of *P. maximowiczii* caused a higher amount of aggressive compounds to formresulting in a much higher uptake of iron and zinc.

XRF is a good tool for comparative analysis because it is a fairly fast technique and does not demand laborious sample preparation. It enables complex analysis including comparative content and distribution measurements of the studied elements.

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Tomasz Krystofiac, Barbara Lis, Monika Muszyńska, Stanisław Proszyk

THE EFFECT OF AGING TESTS ON GLOSS AND ADHESION OF LACQUER COATINGS ON WINDOW ELEMENTS FROM PINE WOOD

The aim of this work was to investigate the formation of gloss and adhesion in accelerated thermal aging test selected lacquer systems formed on pine wood. Window elements were finished with lacquer systems, including impregnate, primer, and inter- and top lacquer layers in two colour versions white and cypress respectively. The range of investigations included gloss measurement and adhesion (pull-off method) of coatings to a substrate. Based on the contact angle, the values of surface free energy ($\gamma_s$), work of adhesion ($W_a$) and surface tension at the interface ($\gamma_{sl}$) were calculated, together with their dispersion and polar parts. On the basis of the experimental results it was stated among others, that these finishing’s were characterized by a semi-gloss effect, which was stable under thermal aging cycles. The lacquer coatings showed good adhesion to the substrate. The values of the $W_a$ parameter remained at a high level as well. In turn the $\gamma_s$ parameter indicated the occurrence of strong adhesion interactions in the substrate and particular layers of the coating systems. Aging processes had no significant effect on the manner of the obtained relations.

Keywords: pine wood, window element, coating, aging test, gloss, contact angle, parameter of adhesion

Introduction

Wooden window joinery is exploited under extremely unfavourable conditions, radically different from those in which the majority of other products operate. Their external part is exposed to changing weather conditions, while the other side is exposed to agents present in certain rooms [Hora 2003; Mateńko-Nożewnik and Proszyk 2004a, b; Grüll et al. 2005]. Functional properties and the durability of windows mainly depend on the applied wood species, accepted technical solutions, precision of their manufacturing, and above all, on the protection of the surface from the influence of biological and atmospheric
factors. It is done through the impregnation and application of lacquer coatings [Graystone 2003; Dawson et al. 2005; Ozgenc et al. 2012]. Apart from the destructive effect of biotic factors, abiotic factors also cause significant changes [Bulcke et al. 2008]. A decisive impact on the stability of windows is found in the following factors of corrosive atmospheric attack:

- variable humidity and direct contact with water due to rain and pollution
  e.g. acid rains
- interaction of solar radiation, especially UV
- significant changes in temperature [Williams et al. 2000; Custódio and Eusebio 2006].

These factors may be characterized by short or long-term, accidental or periodic fluctuations of different intensity [Roux et al. 1988; Creemers et al. 2002]. One of the most important factors determining the durability, cost of production and the aesthetic qualities of wooden door and window joinery is, as already mentioned by the type and quality of the coating forming a specified varnish system [Budakçı and Taşcioğlu 2013]. The most important in this respect are different combinations based on acrylic polymers and acrylic copolymers, because of their valuable properties, e.g. low toxicity, high resistance of UV-light and temperature and other aggressive factors as well as aging processes. The varied range of monomers and acrylic copolymers exhibit numerous properties which enable the production of a wide range of solvent and waterborne products with various film-forming substances intended for application by different methods [Prozyk 1999; Baumstark and Tiarks 2002; Hora 2004]. Wood surface finishing systems for window joinery usually consist of several layers. Manufacturers offer both covering systems and transparent sets, available in a wide range of colours. Impregnation formulations usually contain biocides, however, it is the topcoat that determines aesthetic-decorative and resistance properties of finishes [Ozgenc et al. 2012; Baysal et al. 2013].

Lacquer coatings on wood during the service life of building joinery are subjected to aging processes. The natural aging process of lacquer coatings is mainly caused by the above mentioned climatic factors. A significant role in the aging processes of coatings is played by chemical processes occurring in the film-forming substances, which are manifested in:

- brittleness due to progressive polymer decay, oxidation, or crosslinking
- migration of wood components through coatings, especially extractive substances and of components of lacquer products (chalking, bloom) [Pecina and Paprzycki 1995].

Those processes often lead to deterioration of the aesthetic-decorative and protective advantages of finishes, primarily gloss. Moreover, scratches and cracks of different types occur on the molecular level and one by one in the nano, micro and macro scale. All of them may cause reduced adhesion of coatings to the substrate [Ahola 1995; Williams et al. 2000; Custódio and Eusebio 2006]. This parameter is decisive to the functional characteristics and
durability of the finished surface [Bardage and Bjurman 1998]. In this context, it was decided to determine the formation of adhesion of coatings for wood, based on the normative pull-off methods and taking into account the assumptions of the adsorption theory of adhesion.

The aim of this work was to investigate the formation of gloss and adhesion selected lacquer systems formed on pine wood under an accelerated thermal aging test.

**Materials and methods**

Semi-finished products obtained under industrial conditions from Scots pine wood (*Pinus sylvestris* L.) were the experimental material. The products were connected longitudinally with finger joints and glued together into three-layer elements with a one-component PVAC adhesive (D3 durability class). The elements were improved with four layers of lacquer systems including the impregenate, primer product in interlayer and top layer in two colour versions, cypress and white respectively. Detailed information concerning the products may not be published as they are covered by a clause of total confidentiality.

Figure 1 is a simplified block diagram of the production of windows from which test samples were obtained.

Investigations were performed on the thermal aging of lacquer coatings under artificial conditions according to the standard [PN-88/F-06100/07:1988] in the function of the number of cycles of changing temperatures, after three, six and nine cycles respectively. The aesthetic-decorative values of sand adhesion relative to the control samples was evaluated.

The investigation of gloss of coatings using the photoelectric method with a PICO GLOSS apparatus, model 503, were based on ten measurements taken, along the grain at three angles of incidence 20°, 60° and 85°, respectively. Gloss degrees (expressed in gloss units GU) were determined for coatings at the angle of incidence of 60°.

Adhesion of coatings to the substrate was tested by the pull-off method, and was performed, based on the procedure described in the respective standard [PN-EN ISO 4624:2004], which allowed the determination of the minimum strength necessary to the tear off, of the coating perpendicular to the substrate surface. Aluminium dollies were bonded with the two-component silane-epoxy adhesive. After seven days of conditioning (20 ±2°C, RH 65 ±5%), adhesion was tested with a *PosiTest AT* apparatus. The surfaces of both the dolly and the sample after de-lamination were assessed.

The test of contact angle (θ) of coatings was performed according to the procedure described in the respective standard [PN-EN 828:2000]. A microscope (magnification ×56), equipped with a goniometric head was used. Ten drops of
redistilled water (3.5 μl) were applied to tested surfaces with a chromatographic syringe. The $\theta$ angle was measured statically, five seconds after the application of drops. Investigations were performed, versus the number of aging cycles. Based on the $\theta$ angle, the values of surface free energy ($\gamma_s$), work of adhesion [W_a] and surface tension at the interface [$\gamma_{SL}$] were calculated, together with their dispersion and polar shares, according to formulas given in literature [Kloubek 1974; Neumann et al. 1974; Nguyen and Johns 1978; Liptáková 1980].
Results and discussion

The assessment of the appearance of different coating colours described in the study revealed that they were characterised by high aesthetic-decorative values. Thermal aging of the coatings in cycles of fluctuating temperatures did not have a negative impact on their appearance. The tests proved that the lacquer coatings under study were characterised by high stability and resistance especially thermal stress, as well as the accompanying humidity and shrinkage stress in the wood-coating system.

![Gloss vs. Number of Cycles Graph](image)

**Fig. 2. Change of gloss as a function of the number of aging cycles**

The tested surfaces in the form of control samples, for both coating colours: white and cypress, had gloss values from 15 to 25 GU, classified in the descriptive assessment as semi-gloss. There was no significant effect on the number of cycles of temperature changes, on the level of gloss of tested lacquer finishes.

An elementary statistical estimation of the development of adhesion for coatings of various colours to the pine wood surface as a function of the number of cycles of thermal aging and evaluation of disconnection mechanisms at destructive loadings are shown in table 1.

The overall assessment of the research findings indicates very high repeatability of the results. The values of the variation coefficient $v$ for the options under consideration were low, and did not exceed 10%. The average adhesion values of the reference samples were in the range of 0.84-0.87 MPa for the considered colour variants.

The aging tests of all the test samples resulted in increasing adhesion to the substrate. Delaminations of the tested systems, with the destructive loadings...
Table 1. Adhesion of coatings to the substrate

<table>
<thead>
<tr>
<th>Colour of finishings</th>
<th>Number of cycles</th>
<th>Statistical data&lt;sup&gt;*)&lt;/sup&gt;</th>
<th>( \chi_{\text{min.}} )</th>
<th>( \chi_{\text{av.}} )</th>
<th>( \chi_{\text{max.}} )</th>
<th>( \nu )</th>
<th>disconnection mechanism&lt;sup&gt;**)&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>[MPa]</td>
<td>[%]</td>
<td>[%]</td>
<td>[MPa]</td>
<td>[%]</td>
</tr>
<tr>
<td>White pine</td>
<td>0</td>
<td>0.76</td>
<td>0.84</td>
<td>0.94</td>
<td>7.43</td>
<td>5A</td>
<td>85B/C, 10n/m</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>1.14</td>
<td>1.22</td>
<td>1.29</td>
<td>4.71</td>
<td>85B/C</td>
<td>10n/m, 5-/Y</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>1.12</td>
<td>1.23</td>
<td>1.41</td>
<td>4.04</td>
<td>90B/C</td>
<td>5n/m, 5-/Y</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>0.99</td>
<td>1.05</td>
<td>1.11</td>
<td>4.75</td>
<td>90B/C</td>
<td>5n/m, 5-/Y</td>
</tr>
<tr>
<td>Cypress pine</td>
<td>0</td>
<td>0.81</td>
<td>0.87</td>
<td>0.95</td>
<td>6.09</td>
<td>70A</td>
<td>30-/Y</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>1.02</td>
<td>1.12</td>
<td>1.25</td>
<td>9.21</td>
<td>60-/Y</td>
<td>30A, 10n/m</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>0.97</td>
<td>1.03</td>
<td>1.16</td>
<td>7.42</td>
<td>85A</td>
<td>10n/m, 5-/Y</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>1.09</td>
<td>1.20</td>
<td>1.34</td>
<td>8.09</td>
<td>90A</td>
<td>5n/m, 5-/Y</td>
</tr>
</tbody>
</table>

<sup>*)</sup> \( \chi_{\text{min.}} \) – minimum value, \( \chi_{\text{max.}} \) – maximum value, \( \chi_{\text{av.}} \) – arithmetic average, \( \nu \) – coefficient of variation.

<sup>**) </sup>A – cohesive in substrate, B/C – adhesive between first and second coating, n/m – cohesive between n-layer and m-layer of the coating system, -/Y – adhesive of last coating and adhesive.

were quite varied. For the version pine wood cypress, the cohesiveal mechanism was dominant in the substrate, whereas in case of white pine wood mainly an adhesive destruction between the first and second layers was recorded. An elementary statistical estimation of the contact angle formation the two lacquer systems as a function of the number of cycles of thermal aging are summarized in table 2.

Table 2. Contact angle of tested systems before and after thermal aging with elementary statistical estimation

<table>
<thead>
<tr>
<th>Colour of finishings</th>
<th>Number of cycles</th>
<th>Statistical data&lt;sup&gt;*)&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>( \chi_{\text{min.}} )</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[deg]</td>
</tr>
<tr>
<td>White pine</td>
<td>0</td>
<td>65.13</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>65.50</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>62.00</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>60.48</td>
</tr>
<tr>
<td>Cypress pine</td>
<td>0</td>
<td>66.01</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>66.01</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>64.00</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>65.27</td>
</tr>
</tbody>
</table>
The values of the coefficients of variation within the range of 1.15-9.77% indicate good repeatability of measurements. There was a slight decrease in the Θ angle values with an increasing number of aging cycles. More dynamic changes in the values were recorded for white pine wood finishing. The absolute value of this parameter decreased by 1.04-7.29 deg. As far as cypress-coloured coatings are concerned, these relations amounted to 1.01-2.17 deg. On the basis of the Θ angle and the theoretical formulas, which are based on the concept of adsorption theory of adhesion of polymers to the wood, values of γS, Wa and γSL, together with the dispersion and polar shares were calculated. Based on the literature data [Liptáková and Paprzycki 1983], γS for pine wood was adopted as 64.9 mJ/m², with the polar share (γSρ) of 38.9 mJ/m² respectively.

Figure 3 illustrates the formation of the γS parameter and γSd and γSp shares.

![Graph showing formation of γS, γSd, and γSp](image)

**Fig. 3.** Formation of surface free energy (γS) and dispersion (γSd) and polar (γSp) shares for surface of various coloured lacquer systems formed on the pine wood as a function of the number of cycles of thermal aging

The overall assessment of the γS parameter proved that they were similar for individual finishes, ranging within 40.76-42.15 mJ/m². In the function of the number of carried out aging tests generally, a slight increase in this parameter was found. The γSp share affected the volume of the observed changes in γS. In turn, the values of the γSd component in the tested samples were stable at 32 mJ/m². This indicates, therefore, that the conditions included in the thermal aging test experiments of coatings, the occurring changes influenced physico-chemical interactions of polar groups. Figures 4 and 5 present the results of Wa and γSL for the considered systems of substrate lacquer coating and their formation as a function of the number of thermal aging cycles.
Fig. 4. Formation of the work of adhesion (Wa) and dispersion (Wa^d) and polar (Wa^p) shares for surface of various coloureds lacquer systems formed on pine wood as a function of the number of thermal aging cycles.

Fig. 5. Formation of γSl and dispersion (γSl^d) and polar (γSl^p) share for surface of various coloureds lacquer systems formed on pine wood as a function of the number of thermal aging cycles.
It was found that the tested coating systems were characterized by high values of $W_a$, exceeding 90 mJ/m². According to the literature data, these were good relations. The values of $W_a^d$ ranked at a relatively similar level of 58 mJ/m², while $W_a^p$ values were varied. An interdependence was observed in its relations modifying the values of $W_a$ in the tested lacquer coatings and $\gamma_{SL}$. Higher values of $W_a$ were recorded at lower $\gamma_{SL}$. This finding confirms the theoretical assumptions included in the criterion of minimizing energy on the boundary interfaces. The formula assumes that the criterion claiming that maximum adhesion can be achieved if the system minimises $\gamma_{SL}$ at the interface, where the materials are in contact with each other. It is the principle of the minimisation of surface tension on the interface of contacting materials. Some authors report that this value should range from 1 to 3 mJ/m² [Hellwig et al. 1968; Potente and Krüger 1978; Pirmasens 1983; Pecina and Paprzycki 1995]. It should be noted, however, that none of these studies of lacquer systems met this criterion. It proves that the physicochemical interactions considered in reference to this criterion are not fully adequate to the theoretical possibilities.

**Conclusions**

1. Tested finishes including lacquer coating systems in the colour versions of white and cypress formed on pine wood, were characterised by a degree of gloss, determined in the descriptive evaluation as semi-gloss. This property was stable in thermal aging cycles in the version of changing temperature cycles.

2. The finishing coatings showed good adhesion to the substrate. The aging tests revealed an upward tendency for values of this parameter.

3. The tests on adhesion of lacquer coatings to pine wood revealed that the cypress colour system was characterised by better relations in the delamination mechanisms.

4. The value of surface free energy ($\gamma_S$) in the tested coatings was approx. 42 mJ/m², where the dispersion share was predominant. The $\gamma_S$ of coatings increased in function of the number of aging tests cycles. The $\gamma_S^p$ share influenced variation in the $\gamma_S$ value.

5. The value of the $\gamma_S$ parameter in the coating systems was lower than this value in pine wood. It indicates strong adhesive interactions between the substrate and lacquer coating systems.

6. The values of the adhesion work ($W_a$) remained high, exceeding 90 mJ/m². Values of this parameter were increasing in the function of the number of aging tests cycles.

7. The tested lacquer coating systems did not meet the principle of minimisation of surface tension at the interface of materials in contacting materials.
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Ozgenc O., Hiziroglu S., Yildiz U. C. [2012]: Weathering properties of wood species treated with different coating applications. Bio Resources 7 [4]: 4875-4888

List of standards
PN-EN 828:2000 Adhesives – Wettability – Determination by measurement of contact angle and critical surface tension of solid surface

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Roger ROWELL

DIMENSIONAL STABILITY AND FUNGAL DURABILITY OF ACETYLATED WOOD

The reaction of wood to acetic anhydride greatly reduces moisture sorption and improves the dimensional stability of the wood due to the esterification of the accessible hydroxyl groups in the cell wall, reducing hydrogen bonding with water and bulking the cell wall back to its green volume. The sorption of both primary and secondary water are reduced. Dimensional stability is not 100% since the water molecule is smaller than the acetyl group, therefore water can access hydroxyl sites even when the wood is fully acetylated. The equilibrium moisture content is reduced in a linear relationship to the level of acetyl content. This means that the reduction in moisture content is not dependent on where the acetylation reaction takes place in the cell wall. Resistance to fungal attack increases as the level of acetylation increases. Resistance to attack by white-rot fungus occurs at a much lower level of acetylation (7-10%) than that to brown-rot fungal attack (17-19%). The cell wall moisture content may be too low at high levels of acetylation to support fungal attack, therefore initial colonization does not take place.

Keywords: Acetylation, dimensional stability, fungal resistance, moisture sorption, equilibrium moisture content

Introduction

As fossil resources become increasingly expensive, alternatives are being sought that are not based on diminishing fossil resources. If a transition is to be made from a fossil-based economy to a bio-based economy, major changes have to take place in technology, codes and standards, and, perhaps more importantly, in the way society thinks and acts.

Wood has been used since the first humans walked the earth for fuel, shelter, weapons, tools and for decoration. It is considered easy to work, and is renewable, sustainable and widely available. For the most part, it has been used without modification. Solid timber and lumber were treated for decay and fire resistance as recorded in ancient accounts; however, most applications for wood today have little treatment other than a coating or finish. Humans have learned to

Roger ROWELL (rmrowell@wisc.edu), Biological Systems Engineering, University of Wisconsin, Madison, WI, USA
use wood accepting that it changes dimensions with changing moisture content, can be decomposed by a wide variety of organisms, burns and is degraded by ultraviolet energy.

With an increased awareness of the fragility of the environment and the need for durability in wood products, new technologies have been developed to increase the service life of wood materials without the use of toxic chemicals. Issues of sustainability, carbon sequestration and performance converge in this search for new technologies to improve stability and durability.

Chemical modification using acetic anhydride is an environmentally friendly method of wood stabilization and protection. This technology has been studied for many years and is now commercially available.

**Acetylation**

All woods contain acetyl groups: softwoods – 0.5-1.7%, and hardwoods – 2-4.5%, therefore adding more acetyl groups introduces chemical groups that already exist in the wood. The acetylation of wood was first performed in Germany by Fuchs [Fuchs 1928], using acetic anhydride and sulfuric acid as a catalyst. Fuchs found an acetyl weight gain of over 40%, which meant that in the process he decrystallized the cellulose. He used the reaction to isolate lignin from pine wood. In the same year, Horn acetylated beech wood to remove hemicelluloses in a similar lignin isolation procedure [Horn 1928]. Tarkow first demonstrated that acetylated balsa was resistant to decay [Tarkow 1945]. Tarkow was also the first to describe the use of acetylation to stabilize wood in order to prevent it from swelling in water [Tarkow 1946].

While laboratory acetylation of wood has been practised for nearly a century, the commercialization of acetylated wood has been met with several challenges. The Koppers Company may have made the first earnest, albeit short-lived, attempt at entry into the commercial acetylated wood market in the 1960s. This was followed by efforts in Russia and Japan (Diaken) in the 1970s and 1980s. In the late 1980s and early 1990s, A-Cell Acetyl Cellulosics AB, in Sweden, were granted a number of patents and built two pilot plants: one for solid wood, using microwave technology, and one for acetylating fibers. Accsys Technologies, which had acquired technologies developed earlier at Stichting Hout Research (the Netherlands) and Scion (New Zealand), launched trial quantities of Accoya®, an acetylated Pinus radiata, onto the market and began full commercial scale production in Arnhem, the Netherlands. This was followed in 2012 by Eastman Chemical Company introducing Perennial Wood™ using acetylated southern pine produced at its pilot facility in Kingsport, Tennessee, although production was stopped in 2014 [Rowell 2012].

Acetylation is a single-addition reaction, which means that one acetyl group is on one hydroxyl group with no polymerization:
WOOD–OH + CH₃C(=O)–O–C(=O)–CH₃ → WOOD–O–C(=O)–CH₃ + CH₃C(=O)–OH

Acetic anhydride  Acetylated wood  Acetic acid

Thus, all the weight gain in acetyl can be directly converted into the units of hydroxyl groups blocked. This is not true for a reaction where polymer chains are formed (epoxides and isocyanates, for example). In these cases, the weight gain cannot be converted into units of blocked hydroxyl groups.

Isolated lignin reacts faster with acetic anhydride than hemicelluloses and holocellulose [Kumar and Agarwal 1983, Rowell et al. 1994] Kumar and Agawal reported that at an acetyl weight percent gain of 13.5, 86.4% of the lignin was acetylated, 21.6% of the hemicelluloses and 9.3% of the cellulose. Reacting wood at 120°C with acetic anhydride and no catalyst, at an acetyl weight gain of 16 to 19%, theoretically approximately 90% of the lignin is esterified, and 25% of the holocellulose [Rowell 1982]. It is assumed that 100% of the hemicellulose hydroxyl groups are substituted and no cellulose hydroxyl substituted. There may be a small number of hydroxyls esterified on the surface hydroxyls in the amorphous regions of the cellulose. This conclusion is based on the observation that pure cotton cellulose cloth can be used to hold wood fiber and no weight gain is observed in the cotton cloth after several acetylation reactions.

**Moisture and dimensional stability**

It is important to understand how moisture enters the wood and how it moves within the wood. Although wood is a porous material (60-70% void volume), its permeability or flow of water is extremely variable. This is due to the highly anisotropic arrangement of the component cells and to the variable condition of the microscopic channels between cells. Wood is much more permeable in the longitudinal direction than in the radial or tangential directions. Due to this anisotropy, longitudinal flow paths are of major importance in the wetting of wood exposed to the weather [Miller and Boxall 1984]. Moisture enters wood in one of two ways: by capillary action as liquid water in the end grain or as moisture from the surrounding atmosphere. It is the end grain capillary uptake of liquid water that causes problems in the corners of windows and doors. Since wood is hygroscopic, it attracts moisture which bonds to the cell wall polymers through hydrogen bonding [Rowell 1984].

As moisture is added to the cell wall, wood volume increases nearly proportionally to the volume of water added [Tiemann 1944; Stamm 1964]. Swelling of the wood continues until the cell reaches the fiber saturation point (FSP) and water, beyond the FSP, is free water in the void structure and does not contribute to further swelling. This process is reversible, and wood shrinks as it loses moisture below the FSP.

According to the Dent sorption theory, water is added to the cell wall polymers in mono-layers [Dent 1977]. Figure 1 shows the mechanism of water molecules adding to the wood cell wall. In figure 1A, water molecules enter the
cell wall and start hydrogen bonding with accessible hydroxyl groups. Figure 1B shows the “unzipping” of hydrophilic polymer chains [Caulfield 1978]. Figure 1C shows the sorption of primary water ● molecules and secondary water ○ molecules, while figure 1D illustrates the fully hydrogen bonded water in the cell wall. Hydrogen bonds between hydroxyl groups on and between hemicellulosics, cellulose and lignin are constantly changing.

![Diagram A](image1)

![Diagram B](image2)

![Diagram C](image3)

![Diagram D](image4)

**Fig. 1. Models of water added to the wood cell wall: A – Water molecules entering the wood cell wall, B – water molecules unzipping hydrophyllic polymer chains, C – water bonding to the cell wall either as primary water ● or secondary water ○, D – fully hydrated cell wall at the fiber saturation point**

**Materials and methods**

Freshly cut Scotch Pine (*Pinus sylvestris* L.) sapwood was cut into boards measuring 2.5 cm thick and dried. The test samples were cut from this wood as follows: for the acetylation reaction: 2.5 × 5 × 25 cm (radial × tangential × longitudinal); to test equilibrium moisture content and water swelling: 1 × 3 × 0.5 cm (radial × tangential × longitudinal); for the ASTM soil block test: 2.5 × 2.5 × 2.5 cm (radial × tangential × longitudinal); for the fungal cellar: 1 × 2 × 5 cm (radial × tangential × longitudinal); and for the in-ground tests: 2.5 × 3 × 30 cm (radial × tangential × longitudinal).

Acetylation was carried out in a 1 liter glass reactor using 5% acetic acid in acetic anhydride at reflux for 4 hours. The volume and oven dry weight of each sample was recorded before the reaction. After the reaction was complete, the modified wood was placed in a vacuum desiccator in water, a vacuum was
drawn for 30 minutes, then released, and finally the water was discarded and fresh water was added. This process was repeated 3 times to remove excess acetic anhydride and the by-product, acetic acid. The samples were then oven dried overnight at 105°C. The weight and volume of each sample was recorded and the acetyl weight percent gain (WPG) and change in volume were calculated.

The oven-dried and weighed control and acetylated samples were placed in humidity rooms that were controlled at 30, 65 and 90% relative humidity at 27°C. After 30 days, the samples were weighed again and the equilibrium moisture content determined.

Swelling in liquid water was carried out by placing the oven-dried and measured control and acetylated samples in water at room temperature. The swelling was measured using a flatbed micrometer until no further swelling was observed. The swelling coefficients and antishrink efficiencies were then calculated.

The control and acetylated samples were placed for testing in a soil block according to the ASTM standard D-2017-71 using the brown-rot fungus *G. trabeum* or the white-rot fungus *T. versicolor*. The test was run for 12 weeks and the weight loss was recorded.

The control and acetylated samples were measured and placed in a fungal cellar where the soil contained brown-, white-, and soft-rot fungi and tunneling bacteria. The samples were pulled at 2, 3, 4, 5, 6, 12, 24 and 36 months and rated for decay.

The control and acetylated samples were placed in the ground in three different places in Sweden in three different soil types: compost, sandy and forest soils. The samples were pulled after 300 days and rated for decay.

**Results and discussion**

Table 1 shows the change in the volume of the wood from green to dry to acetylated. The elastic limit of the cell wall was not exceeded and the bulked acetylated wood returned to the wood’s original green volume. From green volume to dry resulted in a loss of approximately 10% in wood volume, while acetylation brought the wood volume back to that of the original green wood.

<table>
<thead>
<tr>
<th>Green Vol cm³</th>
<th>OD Vol cm³</th>
<th>Change %</th>
<th>Ac WPG</th>
<th>OD Vol cm³</th>
<th>Change %</th>
</tr>
</thead>
<tbody>
<tr>
<td>38.84</td>
<td>34.90</td>
<td>-10.1</td>
<td>22.8</td>
<td>38.84</td>
<td>+10.1</td>
</tr>
</tbody>
</table>

The equilibrium moisture content (EMC) reduced as the level of acetyl weight gain increased. Table 2 shows the reduction in EMC as a function of acetyl weight gain.
Table 2. Equilibrium moisture content of acetylated pine [Rowell 2012]

<table>
<thead>
<tr>
<th>Percent Gain</th>
<th>Weight EMC at 27°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>30%RH</td>
</tr>
<tr>
<td>0</td>
<td>5.8</td>
</tr>
<tr>
<td>6.0</td>
<td>4.1</td>
</tr>
<tr>
<td>10.4</td>
<td>3.3</td>
</tr>
<tr>
<td>14.8</td>
<td>2.8</td>
</tr>
<tr>
<td>18.4</td>
<td>2.3</td>
</tr>
<tr>
<td>20.4</td>
<td>2.4</td>
</tr>
</tbody>
</table>

Figure 2 shows the sorption/desorption isotherms for the control and two levels of acetylation. The curves for the acetylated samples were lower than the control but there was still a separation (hysteresis) in the adsorption/desorption curves. The differences in the acetylation curves were larger due to the slower adsorption than desorption.

![Figure 2. Sorption/desorption curves for control and two levels of acetylated spruce fiber [Rowell 2012]](image)

Table 3 shows the dimensional stability resulting from the acetylation of solid pine wood and for a fiberboard made from acetylated pine fiber.
Table 3. Dimensional stability of acetylated wood (solid wood, 21.6 WPG, fiber 22.7 WPG, 24 hour water-soak)

<table>
<thead>
<tr>
<th></th>
<th>EMC</th>
<th>S</th>
<th>ASE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solid Pine</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>21.7</td>
<td>13.8</td>
<td>–</td>
</tr>
<tr>
<td>Acetylated</td>
<td>8.4</td>
<td>4.2</td>
<td>81.3</td>
</tr>
<tr>
<td>Pine Fiberboard (5% phenolic resin)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>20.2</td>
<td>21.3</td>
<td>–</td>
</tr>
<tr>
<td>Acetylated</td>
<td>3.4</td>
<td>2.1</td>
<td>90.1</td>
</tr>
</tbody>
</table>

S – swelling coefficient, ASE – antishrink efficiency [Rowell 2012].

Over the years, several mechanisms have been put forward to explain the resistance provided by acetylated wood to brown-rot fungal attack. The earliest ideas centered around the modification of the conformation and configuration of the substrate such that the specific enzymatic attack could not take place [Stamm and Baechler 1960; Takahashi et al. 1989a, b]. Another theory is based on the bulking effect of the covalently bonded acetyl group [Foster 1988; Foster et al. 1997]. Another was advanced that the mechanism was based on the physical blocking of the cell wall micropores so that enzyme penetration cannot take place [Hill 2001, 2006; Papadopoulos and Hill 2002; Hill et al. 2005]. Highley et al. [1994] showed that the smallest enzyme of a brown-rot fungi is too large to penetrate the cell wall. The average size of a cellulitic enzyme is ca 5 nm and the smallest pore size in wood is < 3.8 nm. Mohebby speculated that there are very small regions in the cell wall that are not acetylated due to the size of the acetate group but which are accessible to free radicals produced by the fungus [Mohebby 2003].

As the level of bonded acetyl increased, resistance to decay increased to both brown- and white-rot fungi (tab. 4). There was a significant decrease in fungal attack at an acetyl level of approximately 10%, which means that many of the hydroxyl groups that are required for a fungi to recognize wood as a food source were modified. As the acetyl level reached 15%, the attack by white-rot fungi stopped and very little attack occurred by the brown-rot fungi. At an acetyl level of ca 18%, there was no attack by either the brown- or white-rot fungi.

A possible key to fungal resistance can be seen in table 5. Control and particleboards made from different levels of increasing acetyl content were placed in a fungal cellar in Uppsala, Sweden [Nilsson et al. 1988]. The samples were evaluated at different times, up to 36 months, to determine the level of attack and to measure sample thickness. The first sample check was done at 2 months and already the control had swollen and there was moderate fungal attack. At the same inspection time, the acetylated sample at 7.3 weight percent gain (WPG) was swollen but there was no evidence of fungal attack. By
6 months, the control samples were badly swollen and destroyed as a result of fungal attack.

Table 4. Resistance of acetylated pine to decay fungi in ASTM D-2017-71 soil block test\(^1\) (brown-rot – *G. trabeum*; white-rot – *T. versicolor*)

<table>
<thead>
<tr>
<th>Acetyl weight gain (%)</th>
<th>Weight loss after 12 weeks</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>brown-rot fungus (%)</td>
</tr>
<tr>
<td>0</td>
<td>61.3</td>
</tr>
<tr>
<td>6.0</td>
<td>34.6</td>
</tr>
<tr>
<td>10.4</td>
<td>6.7</td>
</tr>
<tr>
<td>14.8</td>
<td>3.4</td>
</tr>
<tr>
<td>17.8</td>
<td>&lt;2</td>
</tr>
</tbody>
</table>


Table 5. Fungal cellar tests\(^1\) of aspen made from control and acetylated flakes\(^2\)

<table>
<thead>
<tr>
<th>WPG Rating at intervals (months)(^3)</th>
<th>0</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>12</th>
<th>24</th>
<th>36</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.3</td>
<td>S/2</td>
<td>S/3</td>
<td>S/3</td>
<td>S/3</td>
<td>S/4</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>11.5</td>
<td>S/0</td>
<td>S/1</td>
<td>S/1</td>
<td>S/2</td>
<td>S/3</td>
<td>S/3</td>
<td>S/4</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>13.6</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>16.3</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>17.9</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

\(^1\)Non-sterile soil containing brown-, white-, and soft-rot fungi and tunneling bacteria.

\(^2\)Flakeboards bonded with 5 percent phenol-formaldehyde adhesive.

\(^3\)Rating system: 0 – no attack, 1 – slight attack, 2 – moderate attack, 3 – heavy attack, 4 – destroyed, S – swollen [Rowell 2012].

At 3 months, the acetylated sample at 7.3% showed the first signs of fungal attack and was still swollen. This sample continued to be attacked and was destroyed at 12 months. At 4 months, the sample at 11.5% was swollen but no fungal attack was noted. After one more month, this sample showed the first signs of fungal attack. This trend, of a sample first showing swelling before any fungal attack, led to the conclusion that swelling must take place before any fungal attack occurs. This shows the importance of cell wall moisture before fungal attack can take place.

Figure 3 shows the control sample (A) before the 12 week soil bottle test using a brown-rot fungi (see tab. 4). After 12 weeks, the control sample was covered with fungal mycelium (B) and the cell wall was destroyed. After the same length of time in the same experiment, the sample acetylated to 19 WPG showed no weight loss but there was evidence of mycelium growth (fig. 4).
There is a fungal hyphae visible on the radial wall of the acetylated sample and it is growing on the S₃ layer of the cell wall (arrow in fig. 4). This shows that the acetylated wood was not toxic to the fungus, rather that the fungus could not recognize it as a food source.

![Control pine before (top left) and after 12 week soil block test with brown-rot fungi (top right and bottom)](image1)

**Fig 4. Control pine before (top left) and after 12 week soil block test with brown-rot fungi (top right and bottom)**

![Acetylated pine after 12 week soil block test with brown-rot fungi](image2)

**Fig 5. Acetylated pine after 12 week soil block test with brown-rot fungi**

Table 6 shows the results of acetylated wood after 300 days in three different types of soil. The compost soil had mainly brown-rot fungi, the sandy soil had mainly soft-rot fungi, while the forest soil had mainly white-rot and soft-rot fungi.
Table 6. Results of in-ground tests on control and acetylated pine [Rowell 2012]

<table>
<thead>
<tr>
<th>Sample</th>
<th>Weight Loss in:</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>compost</td>
<td>sandy soil</td>
<td>forest soil</td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>74 ±9</td>
<td>50 ±16</td>
<td>27 ±24</td>
<td></td>
</tr>
<tr>
<td>Acetylated</td>
<td>1 ±0</td>
<td>1 ±0</td>
<td>0</td>
<td></td>
</tr>
</tbody>
</table>

Table 7 shows the loss of carbohydrates in the wood fiber. The non-acetylated fiber lost 85.8% carbohydrate with major weight loss in all sugars except the galatans. The acetylated sample at 15% acetyl showed only 13.2% total carbohydrate loss, no loss of cellulose (glucans) but major losses of arabans and rhamnans. In addition, there was no loss of lignin in these experiments.

Table 7. Carbohydrate analysis after brown-rot degradation of wood fiber

<table>
<thead>
<tr>
<th>WPG</th>
<th>Wt loss %</th>
<th>Total carbo lost %</th>
<th>Ar lost %</th>
<th>Gal lost %</th>
<th>Rha lost %</th>
<th>Glu lost %</th>
<th>Xyl lost %</th>
<th>Man lost %</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>51.7</td>
<td>85.8</td>
<td>87.9</td>
<td>71.9</td>
<td>90.0</td>
<td>83.8</td>
<td>90.6</td>
<td>92.5</td>
</tr>
<tr>
<td>15</td>
<td>1.4</td>
<td>13.2</td>
<td>89.0</td>
<td>55.2</td>
<td>70.0</td>
<td>0</td>
<td>38.3</td>
<td>42.0</td>
</tr>
</tbody>
</table>

A – Arabans, Ga – Galatans, Rh – Rhamans, Gl – Glucans, Xy – Xylans, Man – Mannans.

Conclusions

Reductions in the moisture sorption in acetylated wood are due to the substitution of hydroxyl groups with acetyl groups. Increased dimensional stability in acetylated wood is due to the bulking of the cell wall with acetyl groups back to its original green dimension so that the cell wall cannot expand very much more because the elastic limit has not been exceeded. The decay resistance of acetylated wood may be due to the lowering of the cell wall moisture content below that needed to support fungal colonization, therefore the initial enzymatic attack does not take place.

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René HERRERA, Tomasz KRYS'TOFIAK, Jalel LABIDI, Rodrigo LLANO-PONTE

CHARACTERIZATION OF THERMALLY MODIFIED WOOD AT DIFFERENT INDUSTRIAL CONDITIONS

In this study European ash wood (Fraxinus excelsior L.) was modified at 192°C and 202°C in Thermo-Drewno®, Poland; and at 212°C in Termogenik®, Spain. After modification, samples were characterized by wet chemistry according to standard methods (TAPPI) and by instrumental methods (FT-IR); in addition, surface and physical properties were measured (density, acidity, moisture, water uptake, contact angle, colour) in order to quantify changes due to treatment and temperature. The results showed that chemical composition of modified wood presents a gradual variation according to the heating regime, regardless of the industrial process applied; the greatest differences were obtained in treatment at 212°C compared to untreated wood. Furthermore, the density (< 0.68 g·cm⁻³) of modified wood decreased proportionally to treatment temperature. Colour measurements showed proportional changes to darker colours depending on the treatment temperature. On the other hand, some physical properties did not vary significantly between treatments, obtaining similar values of contact angle (97°-99°) and of moisture content (<7%).

Keywords: thermal modification, physicochemical properties, European ash wood

Introduction

Thermally modified wood has been widely used and established by several companies and patents because of the improvements gained, such as durability, hydrophobicity and dimensional stability, while the use of chemical products is minimized [Welzbacher and Rapp 2007]. The modification process has different variants, but all involve controlled pyrolysis performed at temperatures ranging from 170°C to 240°C and within a specific atmosphere (steam, oil, vacuum or inert gas) [Esteves and Pereira 2008a].

The thermally modified wood features are well accepted by researchers and companies due to the improved dimensional stability and restricted accessibility.

René HERRERA (renealexander.herrera@ehu.eus), Jalel LABIDI (jalel.labidi@ehu.eus), Rodrigo LLANO-PONTE (rodrigo.llano-ponte@ehu.eus), University of the Basque Country UPV/EHU, San Sebastian, Spain; Tomasz KRYS'TOFIAK (tomkry@up.poznan.pl), Poznan University of Life Sciences, Poznan, Poland.
to fungal activity and degrading agents [Brischke and Rapp 2006; Junghans et al. 2005]. These modifications prolong the service life of wood and wood-based materials and enlarge the range of outdoor applications [Militz 2002].

The changes occurring in the chemical structure of wood during thermal modification, are mainly due to the autocatalytic reactions of the cell wall constituents [Shen, et al. 2010]. Initially carbonic acids will be formed as a result of cleavage of the acetyl groups of particular hemicelluloses, subsequently the monomeric sugar units will be dehydrated to aldehydes [Hakkou et al. 2005]. The lignin complex reacts in small proportions, the reactivity increases only at high temperatures, then the lignin disintegrates into highly concentrated phenol groups, and several condensation reactions with aldehydes occur [Boonstra and Tjeersdsma 2006].

Together with the chemical reconfiguration, the decrease of cell wall microvoids plays an important role on the interactions between water and substrate by reducing water sorption. The microvoids matrix contains hydroxyl groups that absorb water through hydrogen bonds that expand the cell wall to the point where it becomes saturated with water, and therefore changes the moisture content [Yildiz et al. 2004].

On the other hand, due to the chemical reconfiguration of wood some mechanical properties decrease slightly. Thermally modified wood shows a reduction in impact toughness, modulus of rupture, work to fracture and abrasion resistance, and is not suitable for structural applications [Epmeier et al. 2004, Boonstra et al. 2007].

Regarding the industrial methods of modification, the physicochemical changes are produced by the heating regime, process steps and time of treatment. This work analyses the physicochemical differences and the surface properties of European ash wood industrially treated at three different temperatures.

Materials and methods

Wood and industrial treatment

European ash wood (Fraxinus excelsior L.) samples were thermally modified according to the industrial production standards of Thermo-Drewno® (Poland) and Termogenik® (Spain). The modification process begins with a fast increase of chamber temperature up to 100°C which allows the wood to dry to within 3-4% of moisture content. Subsequently steam is sprinkled in order to avoid damage to the wood and the temperature in the chamber is raised to its maximum level (192°C, 202°C, 212°C respectively); the last stage is the cooling down and stabilizing of the samples at 25°C (about 24 hours). The processes are similar for the two companies and both use steam. The differences are in the specific schedule that they each use to run the modification steps and to achieve the maximum temperature. Untreated samples were used to compare all analytical characterizations.
**Wood macromolecular composition**

The chemical analysis of modified wood and control samples was done by wet chemistry according to the standard methods with samples milled through a 4 mm mesh sieve; ashes (TAPPI T211 om-12), ethanol-toluene soluble extract content (TAPPI T264 cm 07), lignin (TAPPI T222 om-02), holocellulose [Wise et al. 1946], cellulose [Rowell 1984] and hemicelluloses as the difference between holocellulose and cellulose. All analyses were carried out three times.

**Physicochemical characterization of modified wood**

The investigated properties were measured using solid or milled samples depending on the performed test. The moisture content was done in accordance with UNE-EN 13183-1 (oven dry basis), and the basic density (oven dry weight and volume) according to ASTM D2395-14. The water uptake test was performed calculating the weight of water absorbed (%WWA) on samples submitted to a vacuum of 7 mbars for 15 minutes and introduced into a vessel filled with deionized water and maintained fully submerged for 96 hours. The weight of samples was measured at the beginning and after submersion at oven-dry state and at different times (4, 8, 24, 48, 72 and 96 hours) of the experiment. WWA was calculated as shown in Equation 2, where \( w_i \) is the initial weight and \( w_f \) is the weight after each period of impregnation.

\[
\text{WWA} \,(\%) = \left( \frac{w_f - w_i}{w_i} \right) \times 100
\]  

(1)

Moreover, wood pH was measured using 1.25 g of sawdust suspended in 25 mL of distilled water and stirred for 24 hours; after that the pH of the suspension was measured with a CRISON- Basic 20 pH meter. The wood acidity was obtained according to a procedure described by Matsuda [1987], adding 1 mL of 0.1 M HCl to the mixed sample and titrated using 0.01 M NaOH with phenolphthalein as the indicator. The amount of NaOH required to reach the neutralization point was used as a measure for the acidity of dry wood (meq NaOH g\(^{-1}\)); then the following equation was obtained.

\[
A \, (\text{meq/g dry wood}) = (v - v_0) \times 10^{-2} m^{-1}
\]  

(2)

Where \( v \) is the volume (mL) of 0.01 M NaOH titration solution used for a wood sample, \( v_0 \) the volume (mL) of 0.01 M NaOH solution used for neutralizing 1 mL of 0.1 M HCl diluted in 25 mL of distilled water and \( m \) the sample mass (g) used for titration.

In addition, FT-IR spectroscopy was used to analyse the structure of wood components and the chemical changes induced by the treatment. Infrared spectra were collected using the PerkinElmer Spectrum Two FT-IR Spectrometer equipment, by direct transmittance equipped with a Universal Attenuated Total
Reflectance accessory with internal reflection diamond crystal lens. The defined range was from 800 cm\(^{-1}\) to 4000 cm\(^{-1}\) with 32 scans and a resolution of 8 cm\(^{-1}\).

**Wood surface evaluation**

To determine the impact of hydrothermal treatment on the wood surface, samples were cut with the following dimensions: 25 mm \(\times\) 25 mm \(\times\) 60 mm (tangential, radial and fibre directions), and the contact angle (OCA System 20 goniometer, provided by Data Physics Co.) was measured using distilled water, ethylene glycol and methylene iodide as reference liquids. The test was performed by the sessile drop technique with single drops of 10 µL dispensed on surface points and the shape recorded with a digital camera during the first second (25 frames per second); after measurements, the surface free energy of each sample were calculated based on Young’s equation:

\[
\gamma_S = \gamma_{SL} + \gamma_L \cos \theta
\]

Where \(\gamma\) is the surface tension (mJ m\(^{-2}\)) of the solid (\(S\)), the solid-liquid (\(SL\)), and the liquid (\(L\)) interface, respectively.

The optical appearance of modified samples was analysed with a Konica Minolta CM-2600d device and expressed using the CIE-Lab colour space coordinate system \(L^*, a^*, b^*\) (lightness, red-green-axis, and yellow-blue axis). The overall colour change (\(\Delta E^*\)) was also calculated from the \(L^*, a^*, \) and \(b^*\) values for each coating system (Eqn. 3). The surface specular gloss at the incidence angles of 20, 60 and 85 was measured using a gloss meter (Konica-Minolta Multi Gloss 268 plus).

\[
\Delta E^* = \sqrt{\Delta L^*^2 + \Delta a^*^2 + \Delta b^*^2}
\]

**Results and discussion**

The analysis of the structural components of wood (hemicelluloses, cellulose and lignin) and low molecular mass compounds (ethanol-toluene extractives) was done to quantify the influence of treatment on the macromolecular composition of wood. The results obtained (tab. 1) show that macro constituents of European ash wood were gradually altered during the hygrothermal modification according to the treatment intensity.

The proportion of wood components showed remarkable differences compared to untreated wood samples and these differences increased with the treatment temperature. Treatment at 192°C present an increase in extractives of up to 30% and a decrease of about 13% in hemicelluloses, meanwhile the cellulose and lignin show only slight differences. Increasing the temperature of treatment by 10°C (202°C) causes the proportion of extractives to increase by up to
72% and the content of hemicellulose decreases to 39% whilst at the same time the lignin proportion increases by 22%.

At maximum heating regimen (212°C) the differences were intensified between modified and unmodified wood with an increase of over 100% in extractives and 23% in lignin, while the hemicelluloses content decreases by up to 44% and the cellulose content starts to degrade by reducing by 16% compared to the other treatments and control.

Table 1. Changes in the macromolecular composition of wood

<table>
<thead>
<tr>
<th>Analysis [%]</th>
<th>Hemicellulose</th>
<th>α-Cellulose</th>
<th>Lignin</th>
<th>Extracts</th>
<th>Ash</th>
</tr>
</thead>
<tbody>
<tr>
<td>F. excelsior</td>
<td>18.61 ±0.52</td>
<td>45.38 ±0.14</td>
<td>28.88 ±1.29</td>
<td>4.69 ±0.43</td>
<td>1.27 ±0.12</td>
</tr>
<tr>
<td>F. excelsior192</td>
<td>16.29 ±0.22</td>
<td>43.81 ±0.61</td>
<td>29.13 ±1.11</td>
<td>6.11 ±0.93</td>
<td>1.89 ±0.10</td>
</tr>
<tr>
<td>F. excelsior202</td>
<td>11.44 ±0.23</td>
<td>43.93 ±0.66</td>
<td>35.12 ±1.42</td>
<td>8.06 ±0.38</td>
<td>2.16 ±0.21</td>
</tr>
<tr>
<td>F. excelsior212</td>
<td>10.47 ±0.14</td>
<td>38.14 ±0.46</td>
<td>35.54 ±1.99</td>
<td>9.49 ±0.53</td>
<td>2.19 ±0.18</td>
</tr>
</tbody>
</table>

Following the results, the hemicelluloses are degraded to a much greater extent than cellulose; however, the extracts and lignin apparently increase and these differences were specifically highlighted in treatments over 200°C. According to the grade of degradation of the macromolecular components of wood, it is possible to classify the intensity of treatment as mild (192°C), intermediate (202°C) and strong treatment (212°C) depending on the heating regimen used.

The hygrothermal modification of wood degraded the hemicelluloses to a greater proportion compared to other components, even with the mild treatment, being the most thermally labile component of wood. One of the main reasons for this behavior is the presence of acetyl groups in the hemicelluloses which are thermally labile causing acid-catalysed degradation of these components [Herrera et al. 2014]. Moreover, the degradation of hemicelluloses involves dehydration reactions that reduce the hydroxyl groups, with a direct effect on the moisture content of thermally modified wood [Korkut et al. 2012].

A relative stability of the cellulose fraction was observed with slight changes in mild (192°C) and intermediate treatments (202°C) but with noticeable degradation on the strong treatment (212°C); a possible reason for this effect is the high degradation of hemicelluloses at this treatment temperature which leads to increases in the crystalline fraction of wood, diminishing the amorphous cellulose which is unstable [Carvalheiro et al. 2008].

On the other hand, a significant increase of the lignin content and ethanol-toluene extractives was observed. In the case of lignin, the increment of phenolic-OH groups during treatment promoted free ortho-sites from the demethoxylation of guaiacyl and syringyl, allowing cross-linking with fragments of cellulose and hemicelluloses arising from the degradation of polysaccharides. The cross-linking of wood fragments has formed a lignin carbohydrate complex
(LCC) and thus the lignin proportion was established or increased [Choi et al. 2007].

In the case of ethanol–toluene extractives, during the modification process extracts such as fats, waxes and resin acids first migrated to the surface of the wood and above 180°C almost all of the original extractives disappeared from the surface, but new compounds are formed as a result of depolymerization reactions of the cell wall components. These extracts included phenolic-OH groups, monosaccharides and lignin derivatives which can be very soluble in ethanol–toluene mixture [Esteves et al. 2008b].

The physicochemical properties of thermally modified wood are presented in table 2. The results show significant changes in water and the water sorption mechanism with 50% to 60% reduced moisture content (MC) and 40% less water uptake than unmodified wood. This reduction effect is principally due to the removal of accessible water-sorption regions on the wood surface, related to a reduction of OH groups within the wood cell wall as a result of the degradation of macromolecular components [Wikberg and Manau 2004].

<table>
<thead>
<tr>
<th>Analysis</th>
<th>Moisture content [%]</th>
<th>Density [g cm⁻³]</th>
<th>Acidity [meq g&lt;sub&gt;dry&lt;/sub&gt;wood]</th>
<th>pH</th>
<th>WWA [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td><em>F. excelsior</em></td>
<td>11.08 ±0.71</td>
<td>0.69 ±0.04</td>
<td>6.74 10⁻²</td>
<td>4.55 ±0.06</td>
<td>54.65 ±1.95</td>
</tr>
<tr>
<td><em>F. excelsior192</em></td>
<td>5.58 ±0.72</td>
<td>0.68 ±0.04</td>
<td>4.35 10⁻²</td>
<td>4.55 ±0.09</td>
<td>34.59 ±0.42</td>
</tr>
<tr>
<td><em>F.e xcelsior202</em></td>
<td>4.67 ±0.24</td>
<td>0.65 ±0.02</td>
<td>2.79 10⁻²</td>
<td>4.75 ±0.05</td>
<td>33.13 ±0.51</td>
</tr>
<tr>
<td><em>F. excelsior212</em></td>
<td>4.47 ±0.07</td>
<td>0.63 ±0.02</td>
<td>1.59 10⁻²</td>
<td>5.32 ±0.08</td>
<td>33.03 ±0.36</td>
</tr>
</tbody>
</table>

In relation to the density decrease, it is possible to link the elevated degradation on hemicelluloses content into volatile products, which is higher in the intermediate and strong treatment, leading to a progressive diminishing of density in wood. Acidity values of wood decreased with the intensity of treatment, and it was verified by measuring the pH values (pH increased gradually). The most important changes affecting these properties are the decrease of carboxylic acid in the hemicelluloses fraction and the phenolic functions of lignin, showing decreasing acidity values [Willems 2014].

The chemical structure of wood was also characterized by FT-IR to visualize chemical changes caused by hygrothermal modification. The adjusted peaks and their assignments are listed in table 3, and the comparison between FT-IR spectra are presented in figure 3. The FT-IR showed some chemical modifications between treatments and unmodified wood, but their interpretation is very complex as there are several reactions occurring at the same time [Esteves et al. 2013].

There were changes found, however, from the mildest treatment at 192°C with peaks assigned to different stretching vibrations of groups from the main
wood components. Noticeable were the stretching vibrations of different groups O-H, C=O characteristic to carbohydrates (bands at 3340, 1740, 1375 and 1030 cm\(^{-1}\)) and stretching of different groups C-H characteristic to lignin (bands at 2897 and 1595 cm\(^{-1}\)) and the apparent displacement of groups C-O (1235 cm\(^{-1}\)) [Tjeerdsma and Miltiz 2005].

**Table 3. Bands of wood FT-IR spectrum and assignments**

<table>
<thead>
<tr>
<th>Wavenumber [cm(^{-1})]</th>
<th>Assignment</th>
<th>Short description</th>
</tr>
</thead>
<tbody>
<tr>
<td>3340</td>
<td>O-H str. of bonded hydroxyl groups</td>
<td>Primary O-H</td>
</tr>
<tr>
<td>2942</td>
<td>CH stretching in CH(_2)-CH(_3) groups</td>
<td>CH(_2)-CH(_3) Lignin</td>
</tr>
<tr>
<td>2910</td>
<td>CH stretching in CH(_2)-CH(_3) groups</td>
<td>CH(_2)-CH(_3) Lignin</td>
</tr>
<tr>
<td>2897</td>
<td>CH stretching</td>
<td>CH aliphatic Lignin</td>
</tr>
<tr>
<td>2873</td>
<td>CH stretching</td>
<td>CH aliphatic Lignin</td>
</tr>
<tr>
<td>1740</td>
<td>Bond C=O stretching</td>
<td>Acetyl group Xylan</td>
</tr>
<tr>
<td>1734</td>
<td>Bond C=O str. non-conjugated</td>
<td>Carbonyl + esters</td>
</tr>
<tr>
<td>1716</td>
<td>conjugated carboxylic groups</td>
<td>Polysaccharides</td>
</tr>
<tr>
<td>1595</td>
<td>Aromatic skeletal. vibr. plus C=O str.</td>
<td>Lignin</td>
</tr>
<tr>
<td>1424</td>
<td>Aromatic skeletal. vibr. with CH(_2)-CH(_3)</td>
<td>Lignin</td>
</tr>
<tr>
<td>1375</td>
<td>C-H deformation vibration</td>
<td>Polysaccharides</td>
</tr>
<tr>
<td>1235</td>
<td>Syringyl ring plus C-O stretching</td>
<td>Lignin</td>
</tr>
<tr>
<td>1106</td>
<td>Ring asymmetric vibration</td>
<td>Polysaccharides</td>
</tr>
<tr>
<td>1030</td>
<td>C-O-C stretching</td>
<td>Polysaccharides</td>
</tr>
</tbody>
</table>

The FT-IR spectra of the intermediate treatment (202°C) showed a similar contour to that found in the mild treatment, with stretching vibrations of different groups O-H, C=O and conjugated carboxylic groups characteristic from carbohydrates (1740 cm\(^{-1}\) plus the same bands found in the mild treatment). Moreover, the smoothing of the bands at 1595 and 1235 cm\(^{-1}\) corresponds to vibrations in the aromatic ring of lignin and the contributions of syringyl and guaiacyl lignin, suggesting that there was a growth of structural diversity around the aromatic rings [Popescu et al. 2011].

Changes in the spectra of the strong treatment (212°C) are remarkable, and in addition to the differences above, reductions were found in peaks corresponding to hydroxyl groups and the overlapping of the stretch asymmetric and symmetric vibrations of methyl and methylene (2942, 2910, 2873 and 1424 cm\(^{-1}\)). The reduction of the intensity of these peaks indicates that in the modified wood less hydroxyl groups are accessible to be acetylated. Besides, the accessibility of hydroxyl groups for acetylation is highly correlated to the availability of water and leads to a drastic change in the hygroscopicity of wood [Pandey and Pitman 2003].
Fig. 1. FT-IR spectra of modified and control samples; above 4000-800 cm\(^{-1}\) and below the fingerprint region 1800-800 cm\(^{-1}\)

The displacement at 1106 cm\(^{-1}\) and the gradual C-O stretching at 1030 cm\(^{-1}\), indicate an apparent modification in characteristic groups of carbohydrates, which is probably due to the modification of the crystallinity of cellulose which influences the CH and OH stretch frequencies [Akerholm et al. 2004].
The evaluation of the modified wood surface wettability was performed according to the results of the equilibrium contact angle with three liquids: water ($\gamma_{LV} = 72.8 \text{ mN/m}; \ \rho = 998 \text{ kg/m}^3$), ethylene glycol ($\gamma_{LV} = 47.7 \text{ mN/m}; \ \rho = 1113 \text{ kg/m}^3$) and methylene iodide ($\gamma_{LV} = 50.8 \text{ mN/m}; \ \rho = 3325 \text{ kg/m}^3$). In addition, the surface free energy was evaluated from the results of the different liquids tested and all the results are presented in table 4.

In general, the modified woods (from mild to strong treatment) showed higher hydrophobicity than untreated wood. The wettability of modified woods by water was drastically decreased by up to 50% and with ethylene glycol it decreased from 25% to 40%. On the other hand, with methylene iodide it showed an opposite trend increasing the wettability from 2% to 5%.

### Table 4. Measured values of contact angles and surface free energy of wood

<table>
<thead>
<tr>
<th>Samples</th>
<th>Contact angle [°]</th>
<th>Surface free energy [mJ m$^{-2}$]</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Water</td>
<td>Ethylene glycol</td>
<td>methylene iodide</td>
<td>$\gamma_s^P$</td>
<td>$\gamma_s^d$</td>
</tr>
<tr>
<td>$F. excelsior$</td>
<td>66.31 ±3.45</td>
<td>19.54 ±0.64</td>
<td>35.03 ±0.25</td>
<td>6.74</td>
<td>43.94</td>
</tr>
<tr>
<td>$F. excelsior$</td>
<td>98.10 ±1.04</td>
<td>25.26 ±1.12</td>
<td>34.33 ±0.40</td>
<td>0.10</td>
<td>52.19</td>
</tr>
<tr>
<td>$F. excelsior$</td>
<td>99.12 ±1.10</td>
<td>25.29 ±1.22</td>
<td>33.84 ±0.32</td>
<td>0.10</td>
<td>52.61</td>
</tr>
<tr>
<td>$F. excelsior$</td>
<td>99.80 ±1.25</td>
<td>28.72 ±1.20</td>
<td>33.15 ±0.25</td>
<td>0.10</td>
<td>51.58</td>
</tr>
</tbody>
</table>

All the modification conditions (192°C, 202°C and 212°C) considerably improve the performance of wood against wettability increasing contact angles of water. This effect could be explained as a result of molecular reorientation of the surface functional groups and the cell micropores closure [Windstein et al. 2007; Wiedenhoft and Miller 2005; Gérardin et al. 2007]. Moreover, the reductions in free reactive hydroxyl groups present in the hemicelluloses limit the wood wetting phenomena [Kocaefe et al. 2008]. Also, the migration of lignin compounds to the surface, creates a new hydrophobic layout cross-linked on the surface.

The results show decreasing contact angles of methylene iodide in the modified wood along with an increase in the disperse value of the surface energy. These results were somewhat expected, taking in to account that the contact angle of the polar liquid (water) was increased due to the thermal modification of the wood. Another theory relates to the chemical groups presented in the tested liquids that may also exist on wood surfaces, thus, interact with them in varying degrees depending on the surface modification [Cao and Kamdem 2007]. Several studies indicate a wetting behavior of this liquid used in thermally modified wood with values of contact angle near to 0° [Cao and Kamdem 2007; Kutnar et al. 2013; Piao et al. 2010].

The results of wood surface free energy revealed a relatively small polar component (acid-base component) compared to the disperse component, which
dropped drastically in modified wood and is probably caused by the removal of acid components during the acetylation in the modification process [Hochmanska et al. 2014]. The reduction of the acid-base component after thermal modification could be supported by the decrease of acidity and the increase of pH after modification [Gindl and Tschepp 2002].

The optical characterization of thermally modified wood has been defined by the CIE-Lab colour space and glossiness; the measured values are shown in table 5 and graphically in figure 2. A preliminary visual assessment exhibits gradual darkening of the original wood colour to the strong modification. This effect is due to oxidation of the degrading products during treatment [Korkut et al. 2012].

Colour values of untreated ash wood presented a lightness $L^*$ of 82.52, a red/green hue $a^*$ of 3.49, a yellow/blue hue $b^*$ of 20.16, a chroma value $C^*$ of 20.46 and a hue angle of 80.16. After the thermal treatment, the initial colour of ash wood became gradually darker (decreasing the $L^*$ value) with a slight shift from yellow to red (increasing hue $a^*$ and decreasing the hue angle).

The glossiness was measured perpendicular to the grain at 20°, 60° and 85° (tab. 5), and showed a remarkable decreasing trend of gloss at 60° up to 61.2% and at 20°, approximately 82.5% less. In the case of gloss at 85° the results are diverse. According to the results at 60°, the unmodified wood was classified as half-matte (15.50) and in all treatments it was changed to matte (6.87 to 6.04).

In several end-use applications the gloss values at an angle of 60° are very important, commonly used when lower than 90 gloss units. The values obtained across the grain section showed a good linear correlation between gloss at 60° and thermal modification. This tendency is similar to that found in some wood species after thermo-mechanical modification [Bekhta et al. 2014], showing that in general, the glossiness decreased with an increasing heating regime independent of the treated wood specie [Aksoy et al. 2011].

Table 5. Measured values of colour (CIE-Lab) and gloss on modified and control samples

| Samples      | Colour  |  |  |  |  |  |  |  |  |  |  |  |  |
|--------------|---------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|
|              | $L^*$   | $a^*$    | $b^*$    | $\Delta E^{*ab}$ | $C^*$    | $h$      | 20°      | 60°      | 85°      | classification |
| $F.$ excelsior 192 | 55.15±2.31 | 10.75±1.04 | 25.12±1.77 | 30.21 | 27.33±2.36 | 66.83±2.69 | 1.21±0.14 | 6.87±0.22 | 1.89±0.10 | Matte |
| $F.$ excelsior 202 | 42.22±2.65 | 10.11±0.47 | 18.79±0.78 | 42.21 | 21.34±2.78 | 61.67±2.77 | 1.04±0.11 | 6.64±0.33 | 1.81±0.12 | Matte |
| $F.$ excelsior 212 | 37.91±2.01 | 8.94±0.54 | 14.28±0.71 | 46.57 | 16.86±1.77 | 57.89±1.99 | 2.13±0.17 | 6.04±0.31 | 5.88±0.10 | Matte |
Conclusions

The European ash wood thermally modified at different industrial and heating conditions presented significant changes in the macromolecular composition induced by different physicochemical reactions occurring simultaneously. The modified samples were gradually altered during the hygrothermal modification according to the treatment intensity, resulting in a remarkable decrease in hemicelluloses content and an increase in extractives and lignin content. These structural components were altered, especially in treatments over 200°C and in a smaller proportion in the mildest treatment at 192°C; these alterations were evaluated by FT-IR spectra where the stretching of functional groups related to hemicelluloses were noticeable from the spectrum of the treatment at 192°C and the bands of groups associated as lignin components were changing in the spectrum of treatments at 202°C and 212°C.

The changes in physical properties were related to the chemical modifications that occur on wood during treatment. The darker colour after treatment, therefore, is caused by changes in the wood matrix when the treatment temperature is increased beyond 100°C. At this point, the content of hemicelluloses started to decrease, the lignin content increased and simultaneously the less stable extractive compounds were released from the wood. All these effects gradually change the pH and acidity of the wood, and are physically manifested by colour change to darker tones, meanwhile, the release of volatile compounds helps to reduce the free radicals able to attract water to the wood surface varying the acidity, pH and wettability of wood.
All treatments have a better dimensional stability and lower water affinity than unmodified wood, proved by wettability tests. The ash wood thermally modified at different temperatures, showed an interesting increase in quality characteristics, including colour and surface properties, which is an exceptional value in the solid timber market, and can be used as surface layers in outdoor applications as well.

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List of standards

TAPPI T211 om-12 – Ash in wood, pulp, paper and paperboard: combustion at 525°
TAPPI T264 cm-07 – Preparation of wood for chemical analysis
TAPPI T222 om-02 – Acid-insoluble lignin in wood and Pulp

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Henrik Heräjärvi, Juhani Marttila

THE IMPORTANCE OF CLEANTECH BUSINESS FOR THE DEVELOPMENT OF FUTURE WOOD PRODUCTS INDUSTRIES

Cleantech refers to processes which help reduce the environmental load caused by humans. For the present, the wood products sector has been considered a part of the concept of bioeconomy in Finland. This study aimed to analyse the possibilities for closer collaboration between the wood products sector and cleantech. An in-depth interview of 10 experts mostly from outside the wood products sector was carried out. In addition, a web questionnaire sent to 228 experts representing in particular the forest and wood sector received 62 answers. The results revealed many opportunities and benefits that are not being maximized in terms of collaboration between the wood products and cleantech sectors. Timber construction and wood product manufacturing process know-how are among the areas showing the strongest possibilities for closer integration with cleantech. People outside the forest sector seem to be more open to collaboration between cleantech and wood-based industries than those in the forest sector. China, followed by other Asian and European countries, offers virtually unlimited markets for cleantech solutions, and wood may assume a prominent role in those markets.

Keywords: Bioeconomy, cleantech, sustainability, timber construction

Introduction

The global trend for sustainability demands a shift from the exploitation of non-renewable resources to the utilisation of renewable ones. This creates the demand for new products and services which are quite often made of wood [Metsäalan strateginen ohjelma 2012]. At present, the majority of the natural resources used are non-renewable. Due to pollution of the soil, air, and water, as well as the extinction of many species and the depletion of raw materials, the world is facing great sustainability challenges. The development of technology is an attempt to tackle these challenges and as a result, some traditional products and industrial processes may be conceptually re-organised in terms of...
sustainability. For instance, forest-based industries have a wealth of know-how and technologies which could be applied to other sectors in order to improve their environmental profile.

Megatrends are global development paths which influence large parts of the human population. Megatrends linked to the forest sector include population growth, ageing, urbanisation, climate change, loss of biological diversity, pollution, the rising cost of non-renewable resources, and the increased utilisation rate of renewable resources. Furthermore, digitalisation and overall technology development may be considered megatrends influencing the forest sector. All these may be categorised under the umbrella of sustainability. Since the world population is increasing and there is the desire for further economic growth, clean and resource-efficient processes and products need to be developed [Lovio 2013]. The so-called sixth wave of the economy focuses on smart energy production technologies [Wilenius and Kurki 2012] such as environmental technologies, biotechnologies, nanotechnologies, and health service technologies. The change in paradigm towards resource efficiency leads to, among other things, an increase in the price of the main raw materials and stricter environmental legislation [Wilenius and Kurki 2012].

Finnish forest-based industries have faced challenges in terms of profitability and product markets over the past 10 years. Forests are, however, still the most important natural resource in Finland. Therefore, finding new business areas that can utilise the forest resources in a profitable and sustainable manner is a challenge of great national importance.

The environmental performance of wood is, by many measures, superior in comparison to competing construction materials. Even though wood is the only renewable, industrially-utilised construction material, it is surprisingly also the only construction material from which consumers and dealers require transparent certification of sustainability. From an energy efficiency viewpoint, wood product manufacturing processes are very competitive since bark, dust, and chips provide factories with more energy than needed in the production processes. Wood products also store carbon for their entire life cycle. Carbon dioxide emissions generated by the construction of concrete buildings are almost three times higher than those of wooden buildings [Ruuska and Häkkinen 2012]. Globally, construction and building activities consume 50% of natural resources and cause 40% of greenhouse gas emissions, energy consumption, and waste production [Metsäalan stratenginen ohjelma 2012]. Building construction is the most significant single end use of wood products; in the case of Finland, up to 80% of domestic wood product consumption eventually ends up in building construction. The Finnish wood products sector consists to a very large extent of small and medium-sized firms. This sector is also characterised by a low rate of internationalisation and networking, production orientation, and a slow pace of renewal [Metsäalan stratenginen ohjelma 2012].
In addition to its positive environmental performance attributes, wood is technically a unique construction material. It combines many material properties, such as heat and sound insulation, load-bearing capability, and suitability as interior design material. The load-carrying capacity of wood is maintained even in high temperatures. When burning, the strength of wood reduces as a result of charring at a predictable, constant velocity irrespective of temperature. The effects of wooden structures and surfaces on indoor air quality, as well as physical and mental health, are as yet not fully known. However, current knowledge indicates that the effects of wood are positive [see: Simonson et al. 2001, Muilu-Mäkelä et al. 2014].

Climate change and other environmental concerns have changed consumer behaviour and policy making in Europe. The bioeconomy has rapidly taken a primary role within sustainability discourse. The bioeconomy refers to economic growth based on the sustainable use of renewable resources. Many countries, as well as the European Commission, have devised their bioeconomy strategies. The official aim of the Finnish bioeconomy strategy is to increase the output of the bioeconomy sector from €60 to 100 billion by 2025 and create 100,000 new jobs [Suomen biotalousstrategia 2014]. The Finnish bioeconomy strategy largely relies on the forest sector.

As a difference to the bioeconomy which is based on the idea of the economically sustainable utilisation of biomass, the term cleantech refers to technology development. According to Lovio [2013], cleantech includes processes, services and products that offer more environmentally-friendly solutions than competing processes or products. Vanhanen et al. [2012] state that cleantech is neither an industrial nor economic sector, but more likely a set of solutions which cross many conventional sectors. Cleantech has advanced the most in the fields of emission and waste control, measurement, treatment, cleaning, and environmental restoration [Lovio 2013]. Even during the economic recession, the turnover of Finnish cleantech companies steadily increased with an annual rate of 10-15%, clearly more on average than other sectors of Finnish industries. The marketing brand Cleantech Finland, owned by the Confederation of Finnish Industries, was established almost ten years ago. Now Cleantech Finland (www.cleantechfinland.com) is composed of a network of approximately 80 companies as a part of the organisation Export Finland. The Finnish government relies heavily on the future development of the cleantech business; with 40% of public research, development and innovation (RDI) funding allocated to the support and development of cleantech.

One of the objectives of the Finnish Bioeconomy Strategy is to create a strong competence base for the bioeconomy [Suomen biotalousstrategia 2014]. This is partially supported by the current analysis of the synergies between wood products industries and cleantech businesses. So far, the role of wood in cleantech businesses has been insignificant. Cleantech companies strive to minimise the environmental footprints and energy consumption of industrial
processes and consumers. Exactly the same objectives prevail in, for example, the Finnish house-building industries, where wood is the dominant raw material in single-family houses and a material as to which there are great expectations as regards multi-storey houses.

The objective of this article is to analyse the relationships between the bioeconomy and cleantech, particularly the synergy, means, challenges and advantages of closer collaboration between wood products industries and cleantech businesses.

**Materials and methods**

The current knowledge from the literature and through internet searches was gathered, reviewed, and analysed. In addition, two different experimental data sets were collected. The first data set was accumulated using semi-structured in-depth interviews of 10 experts mostly from the cleantech sector (‘interviewed respondents’). The questions concerned Cleantech Finland, the strengths of Finland in cleantech, the relationship between the wood products industries and cleantech, funding possibilities, and export support. The interviews were carried out in the period September-December 2014. The other data set was collected using an internet questionnaire (Webropol) which was sent to 229 respondents from the forest and wood products sectors (‘Webropol respondents’). Altogether 62 responses were obtained (a response rate of 27%) from the industrial and public sector. Besides background information (organisation size and type, and product categories), the questions from the Webropol survey covered the growth expectations of the companies, the concepts of bioeconomy and cleantech, the utility of the cleantech brand for wood products, and the relevant geographic markets for wood-based cleantech products. The respondents were requested to express how far they agreed or disagreed with the given statements using the Likert scale 1-5 (strongly disagree – strongly agree). The questionnaire was accessible from December 2014 to January 2015.

Respondents of the Webropol survey mostly represented the wood products industry, government organisations, and universities. The respondents from the companies (31 in total) were divided between large (7 responses from companies with more than 250 employees), medium-sized (13 responses from companies with 10-250 employees), and small (11 responses from companies with 1-9 employees) companies. Only one response was obtained from the pulp and paper industry, 17 from the wood products industry, and 13 from other companies (consultancy, machinery manufacture, etc.). The respondents from the public sector (31 in total) worked mostly in research and development organisations (22), but also in educational organisations (6) and government administration (3).

The responses both from the in-depth interviews and the Webropol questionnaire were analysed qualitatively. The Webropol questionnaire allowed
quantitative analysis based on the mean values and standard deviation of the variables studied. An independent two-sample t-test was used to determine statistically significant differences between the respondent groups. In the t-test, the assumption of equal variances in both groups existed.

Results and discussion

Global relevance of cleantech

Cleantech is sometimes misunderstood as only a Finnish phenomenon and a marketing brand of the Finnish forest and wood products sectors. In reality, the relevance of cleantech is globally acknowledged as a toolbox to mitigate climate change [e.g., Parad et al. 2014]. Global collaborative institutions, such as the International Cleantech Network, aim at creating new business opportunities, improving competitiveness, and creating new value for companies, institutions, and local cooperatives in terms of cleantech solutions.

Finland and Sweden are relatively similar as regards cleantech businesses. Both countries encompass a wide range of clean technologies and services. The other Nordic countries, on the other hand, are mainly focused on the energy sector in cleantech [see: Strategi för… 2011; Cleantech Strategic… 2013; Om oss… 2015]. Brand management, networking, and business collaboration are carried out through similar organisations ‘Cleantech Finland’ and ‘Cleantech Inn’ in Finland and Sweden, respectively. In Denmark, the ‘Copenhagen Cleantech Cluster’ focuses on supporting the growth and internationalisation of small and medium-sized cleantech companies, as well as the brand management of Danish cleantech, particularly in the energy sector [Energistrategi 2050… 2011; Andersson et al. 2012]. Furthermore, in this study most of the interviewed respondents recognized the strong cleantech sectors in Sweden and Denmark. Cleantech is also of great importance in several other European countries. In the expert interviews 4 respondents out of 10 highlighted Germany as an important cleantech market.

Interestingly, the interviewed respondents representing sectors other than the forest sector had rather a positive attitude towards the idea of the joint marketing brand ‘Nordic Cleantech’, whereas the Webropol respondents from the forestry sector were much more doubtful regarding the benefits of such a brand. Although the Nordic countries are the global frontrunners of cleantech, the resources in individual countries are small. The global aim of cleantech is a cleaner world. Therefore, collaboration in global marketing and building a joint ‘Cleantech Nordic’ brand might provide these countries with competitive advantages, particularly in big markets outside Europe, such as China, India, or Brazil. The potential market for cleantech in such countries is so big that meeting demand without international collaborative business networks is very difficult.
The interviewed respondents indicated a contradiction between the circular economy policy targets of the European commission aimed at the decreased utilisation of all raw materials and the Finnish bioeconomy strategy aimed at increased (though sustainable) use of biomass. It was also noticed by the interviewed respondents that the concept of cleantech varies according to time and location. For instance, the definition of ‘environmentally-friendly energy production’ differs between nations, cultures, societies, and even between researchers. Western populations are turning towards a kind of modern subsistence economy: 3D printing, urban agriculture, local goods, and other trends reduce the dependence of individuals on logistics and industrially manufactured imported goods. The current predominantly large scale industrial production may eventually suffer from this development. New technologies and cultures bring production back to the end users, and the role of individuals in production chains will strengthen as decentralisation becomes more common.

China has really ambitious political objectives in cleantech. In order to clean this highly polluted country, China plans to invest over 500 billion euros in cleantech development by 2020, as its objective is to be the world’s leading country in cleantech [Lin 2014; Parad et al. 2014]. According to one of the interviewed respondents, there are now already more cleantech companies listed in China than anywhere else. The boost to cleantech in China is based on the unbearable pollution problem derived from rapid industrialisation and urbanisation. China is also the only nation already building large-scale eco-cities, however it needs international partners to find solutions to its ecological challenges. Since wood has a good reputation in China, it would be an enormous business opportunity to provide the Chinese with wooden eco-city solutions based on European competence. Such projects would attract clean water and energy solutions, waste management solutions, etc. In the expert interviews, 3 respondents out of 10 admitted that the Chinese market is so large that other Asian countries or Russia might be easier to access and handle. The environmental problems are thus far not as visible in Russia as in China. Furthermore, environmental and industrial legislation does not provide as strong a support for cleantech development in Russia as in China.

Cleantech as a part of the Finnish wood products sector development

According to the interviewed respondents, the relatively small size of Finland was also considered advantageous: a small country can be dynamic and react quickly to fluctuating market needs. This is not, however, necessarily true since cleantech competence is strongly concentrated in Finland. Only a few large companies possess the majority of the know-how. If there is reluctance in process or product development, these companies can paralyse the development of the whole business. In addition, small and medium-sized enterprises in Finland specialising in wood products are traditionally relatively unwilling to grow or expand into international markets. Newer companies and younger
entrepreneurs are more interested in growth and internationalisation. Forest-based industries, in general, are considered to be a conservative sector relying on a production-based approach instead of a solution-based service approach. Forest-based industries, which are dominated by large companies in Finland, process huge material streams in rather conventional ways but typically react slowly to new ideas.

The views of both the interviewed and Webropol respondents strongly indicate that the most promising synergy potential between the cleantech and wood products industries lies in wood-based construction. The same conclusion was drawn by Lovio [2013]. Tightening environmental standards, norms, and laws, supported by economic incentives, can facilitate the development of competitive cleantech products and services both to foreign and domestic markets. Since approximately 40% of public RDI funding in Finland is allocated to cleantech development, access to these public funding instruments may provide immediate benefits for the wood products sector.

According to the interviewed respondents, it would be sensible to determine cleantech as a crucial part of the wood product company strategy in order to be able to profile itself as a cleantech company. However, it is not feasible to forget or reject the existing brands, including the bioeconomy, since they will most likely be useful in future marketing. The usefulness of the brands is also dependent on the market or customer in question.

Of the 31 company responses from the Webropol survey, altogether 75% profiled their company as a bioeconomy company. 53% of the companies even profiled themselves under cleantech, although there were no member companies of Cleantech Finland among the respondents. This indicates a rather liberal attitude towards cleantech within the Finnish wood products sector despite its reputation as a conservative business. Based on the data collected in this study, however, it is evident that the potential benefits of cooperation with Cleantech Finland are not known or understood well enough among the respondents’ companies. The reason for this poor awareness is most likely insufficient communication. Thus, in order to create cooperation and expand both business sectors, more communication is needed between the cleantech and forest industries.

Some of the interviewed respondents strongly supported the idea of closer collaboration between the wood products sector and cleantech businesses. According to their opinions, such collaboration would create successful new clusters which could develop innovative products and services for the markets, for example, solutions to reduce oil dependency. Some of those interviewed also had quite a neutral attitude towards the benefits of closer collaboration between the cleantech and wood products sectors. Both small and large wood products companies often prefer their own individual brands instead of general marketing brands such as cleantech.
Market value of the Cleantech brand

There has been much discussion concerning the positive environmental attributes of wood in Finland and their benefit for the wood products industries. However, it has thus far proven difficult to run a profitable business. Consumers of the future are expected to be more environmentally-conscious and willing to invest more money in environmentally-friendly products compared with consumers of today [e.g., Aquilar and Vlosky 2007]. For the present, ecological aspects affect small purchases more than strategic investments, such as homes. According to the interviewed respondents, it is also true for cleantech markets. In most cases, income level determines willingness to pay for environmentally-friendly products. In low income areas, basic needs have to be met regardless of their friendliness to the environment. Consumers in wealthier nations can make choices on an environmental basis. Eco-friendliness was also seen as a possible threat: consumers may demand it, but it will not necessarily bring any real benefit, i.e., added value for the producer.

The Webropol survey also mapped the respondents’ opinions regarding the benefits of the bioeconomy and cleantech branding on different geographic markets with the following questions (tab. 1):

1. Is the cleantech or bioeconomy brand useful from the viewpoint of competitiveness in your company? (Respondents from companies)

2. Is the cleantech or bioeconomy brand useful from the viewpoint of competitiveness in industry? (Respondents from the public sector)

### Table 1. Mean values and standard deviations of responses to the question: Is the cleantech or bio-economy brand useful from the viewpoint of your company’s competitiveness? (company respondents) / Is the cleantech or bio-economy brand useful from the viewpoint of the competitiveness of industries? (public respondents).

Scale 1-5 (totally useless – very useful). The difference between the mean values of the company and public answers was tested using a t-test, the significance denoted by p-value

<table>
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<tr>
<th></th>
<th>Cleantech brand</th>
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<tr>
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<td>public</td>
<td></td>
<td>p</td>
<td>companies</td>
<td>public</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>(N = 16)</td>
<td>(N = 31)</td>
<td></td>
<td></td>
<td>(N = 22)</td>
<td>(N = 31)</td>
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<tr>
<td>mean</td>
<td>S.d.</td>
<td>mean</td>
<td>S.d.</td>
<td></td>
<td>mean</td>
<td>S.d.</td>
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</tr>
<tr>
<td>Finland</td>
<td>3.80</td>
<td>1.15</td>
<td>3.55</td>
<td>0.93</td>
<td>0.428</td>
<td>4.14</td>
<td>0.77</td>
<td>3.90</td>
</tr>
<tr>
<td>EU</td>
<td>4.25</td>
<td>1.06</td>
<td>4.55</td>
<td>0.57</td>
<td>0.215</td>
<td>4.41</td>
<td>0.67</td>
<td>4.29</td>
</tr>
<tr>
<td>Russia</td>
<td>2.80</td>
<td>1.26</td>
<td>2.97</td>
<td>0.95</td>
<td>0.617</td>
<td>2.95</td>
<td>0.86</td>
<td>2.94</td>
</tr>
<tr>
<td>North America</td>
<td>3.40</td>
<td>1.35</td>
<td>4.19</td>
<td>0.87</td>
<td>0.020</td>
<td>3.48</td>
<td>0.87</td>
<td>3.87</td>
</tr>
<tr>
<td>South America</td>
<td>2.93</td>
<td>1.16</td>
<td>3.19</td>
<td>0.95</td>
<td>0.422</td>
<td>2.95</td>
<td>0.74</td>
<td>3.10</td>
</tr>
<tr>
<td>China</td>
<td>3.00</td>
<td>1.25</td>
<td>3.84</td>
<td>1.13</td>
<td>0.028</td>
<td>3.05</td>
<td>0.89</td>
<td>3.48</td>
</tr>
<tr>
<td>Mean value</td>
<td>3.36</td>
<td></td>
<td>3.72</td>
<td></td>
<td>3.50</td>
<td></td>
<td>3.60</td>
<td></td>
</tr>
</tbody>
</table>

Both the bioeconomy and cleantech branding were estimated to be equally useful by the Webropol respondents irrespective of the given geographic area. In general, there was a minor difference between the company respondents’ and
public respondents’ opinions: respondents from the companies considered bioeconomy branding slightly more beneficial, whereas the attitude in the public sector was somewhat against cleantech branding. Branding, *per se*, was estimated as most beneficial in the Finnish and European markets, and clearly less important in the Russian, South American, and Chinese markets. It appears that the respondents from companies in particular are not fully aware of the scale of cleantech investments and its huge societal relevance in China, for instance.

The Webropol respondents from the public sector recognised the usefulness of both the cleantech and bioeconomy brands in North America more readily than the respondents from the companies. In the case of China, the corresponding difference was discovered only for the cleantech brand.

China, India, Russia, Europe, and North America were seen as the most important markets for combined cleantech and wood product solutions among the interviewed respondents. The predominant view among these respondents was that it is not reasonable to categorize one industrial sector strictly under one brand, but to approach different market segments with divergent marketing strategies. Nevertheless, the differences in attitudes towards eco-friendliness between consumer segments may be bigger than the differences between the countries. The segmentation of consumers according to their potential consumption of cleantech products using anticipatory consumption imaging [see: Christensen et al. 2004] was seen as important. The cleantech brand, *per se*, has created growth in new markets, which indicates the strategic viability of the brand. This chance should be used as efficiently as possible and wood products provide the cleantech market with novel business opportunities.

According to the Webropol respondents, the cleantech brand was considered relatively beneficial or beneficial in all the given wood product categories. The following question was asked (tab. 2):

3. Which product categories would benefit from marketing under the cleantech brand?

Wooden houses and engineered wood products (LVL, glulam, and plywood) were assessed as the most positive categories by the respondent groups in the Webropol survey. The pulp and paper industries already have existing cleantech collaboration, thus their cleantech branding has not as much novelty value as the branding of wood products.

Surprisingly, the benefits obtainable from cleantech marketing were assessed as less significant for wooden doors and windows than for other wood product categories in the Webropol survey. Single assessment values 1 and 2 (poor or relatively poor potential) were given to all the product categories, but most often for sawn timber, doors, windows, and parquets. No statistically significant differences existed between the public and company respondents in any product category.
Table 2. Mean values and standard deviations of responses to the question: *Which product categories would benefit from marketing under the cleantech brand?* Scale 1-5 (no benefit – very beneficial). The difference between the mean values of the company and public answers was tested using a t-test, the significance denoted by p-value.

<table>
<thead>
<tr>
<th></th>
<th>Companies (N=31)</th>
<th>Public (N=31)</th>
<th>p</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>mean</td>
<td>S.d.</td>
<td>mean</td>
</tr>
<tr>
<td>Wooden houses</td>
<td>4.14</td>
<td>1.16</td>
<td>3.94</td>
</tr>
<tr>
<td>Engineered wood products*</td>
<td>3.90</td>
<td>1.08</td>
<td>3.90</td>
</tr>
<tr>
<td>Wood-based panels</td>
<td>3.79</td>
<td>1.29</td>
<td>3.61</td>
</tr>
<tr>
<td>Sawn timber</td>
<td>3.76</td>
<td>1.38</td>
<td>3.61</td>
</tr>
<tr>
<td>Wood pulp</td>
<td>3.72</td>
<td>1.33</td>
<td>3.42</td>
</tr>
<tr>
<td>Paperboards</td>
<td>3.59</td>
<td>1.30</td>
<td>3.58</td>
</tr>
<tr>
<td>Parquet</td>
<td>3.45</td>
<td>1.27</td>
<td>3.52</td>
</tr>
<tr>
<td>Doors</td>
<td>3.48</td>
<td>1.18</td>
<td>3.45</td>
</tr>
<tr>
<td>Windows</td>
<td>3.43</td>
<td>1.23</td>
<td>3.45</td>
</tr>
<tr>
<td>Papers</td>
<td>3.34</td>
<td>1.20</td>
<td>3.39</td>
</tr>
<tr>
<td>Mean value</td>
<td>3.61</td>
<td></td>
<td>3.55</td>
</tr>
</tbody>
</table>

*Engineered wood products refers to glulam beams, laminated veneer lumber (LVL), and cross-laminated timber (CLT).

Regarding the collaboration potential between the cleantech and bioeconomy brands, challenges were also identified by the interviewed respondents. Although in many cases the same enterprises may be easily seen as part of cleantech and the bioeconomy, sometimes these two concepts may be impossible to merge. The rapid conceptual change in terminology, especially in the bio-based sector, was considered a challenge. According to some respondents, it is disturbing that consumers’ minds are confused by the media and policy makers using vaguely defined but fashionable terms, such as bioeconomy, bio-commerce, clean economy, sustainable development, green growth, and circular economy.

**Steps to be taken in wood products industries**

The Webropol respondents predicted which future measures were the most critical for the wood products sector. The following question was asked (tab. 3):

4. What is the meaning of the following measures for the development of your business (respondents from companies) / of your activities (respondents from the public sector)?

All the Webropol respondents emphasised the meaning of competence development and internationalisation. It is notable that finding new business opportunities is more important for the respondents from the public sector than those from companies. Obviously in this study, the respondents from the public sector, often being RDI professionals, focus heavily on recognising new business opportunities in their work. They also considered international networking and
the development of demonstration facilities more important issues than the
respondents from the companies did.

Table 3. Mean values of responses to the question: What is the meaning of the
following measures for the development of your business? Scale 1-5 (very small
meaning – very meaningful). The difference between the mean values of the
company and public answers was tested using a t-test, the significance denoted by
p-value

<table>
<thead>
<tr>
<th>Measure</th>
<th>Companies (N=31)</th>
<th>Public (N=31)</th>
<th>p</th>
</tr>
</thead>
<tbody>
<tr>
<td>Personnel competence development</td>
<td>4.36 0.78</td>
<td>4.32 0.79</td>
<td>0.867</td>
</tr>
<tr>
<td>Production efficiency</td>
<td>4.29 0.76</td>
<td></td>
<td></td>
</tr>
<tr>
<td>International networking</td>
<td>4.11 0.58</td>
<td>4.48 0.77</td>
<td>0.044</td>
</tr>
<tr>
<td>RDI within own organisation</td>
<td>4.07 0.87</td>
<td>4.26 0.93</td>
<td>0.443</td>
</tr>
<tr>
<td>Increasing value-added</td>
<td>4.04 1.93</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Finding new business opportunities for wood</td>
<td>3.96 1.07</td>
<td>4.52 0.85</td>
<td>0.032</td>
</tr>
<tr>
<td>RDI with other firms or RDI organisations</td>
<td>3.93 0.92</td>
<td>4.55 0.72</td>
<td>0.005</td>
</tr>
<tr>
<td>Profile of environmentally-friendly partner</td>
<td>3.86 0.80</td>
<td>4.13 0.88</td>
<td>0.223</td>
</tr>
<tr>
<td>Finding new export markets</td>
<td>3.79 1.03</td>
<td>3.68 1.19</td>
<td>0.712</td>
</tr>
<tr>
<td>Development of demonstration facilities*</td>
<td>3.59 1.12</td>
<td>4.23 1.09</td>
<td>0.033</td>
</tr>
<tr>
<td>Support for export activities</td>
<td>3.39 1.29</td>
<td>3.58 0.99</td>
<td>0.530</td>
</tr>
<tr>
<td>Investment incentives</td>
<td>3.11 1.37</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mean value</td>
<td>3.90</td>
<td>4.18</td>
<td></td>
</tr>
</tbody>
</table>

*Demonstration facilities are reference projects or objects offering the possibility of product
development and cooperation with other companies or RDI institutions.

The modern processes used in forest industries were perceived by the
interviewed respondents as a huge potential for cleantech. Finland is considered
to have a great competence in the value networks of both the chemical and
mechanical forest industries, and the cleantech features can actually be found in
the existing industrial processes. However, the entire forest sector or some
industrial sub-sector of it should not be fully branded as cleantech, but it should
opt for some strategic products, processes, or services. Such areas could be
found, for instance, within timber construction or modern production monitoring
techniques, for example, in the pulp and paper industries or in the sawmilling
industry. Timber construction fulfils many attributes of cleantech with regard to
raw material sustainability, carbon sequestration, recyclability, as well as the
energy efficiency of the production and engineering processes. These attributes
provide the timber construction sector with a vast potential for the export of
products and production processes, advantages which, for the present, have not
been widely exploited.

The interviewed experts agreed that wood and other raw materials should not
be set against each other but new markets should be sought through value
innovations. These innovations refer to novel markets which add value both to
the customer and the manufacturer [Kim and Mauborgne 2005]. In particular, the
wood products sector should pay more attention to customer preferences and needs and, hence, learn how to supplement and develop the conventional production-oriented business strategy.

Public procurements were seen as important drivers for cleantech by the interviewed respondents. Calls for tender should not define the technologies needed but the desired final solutions. Thus, producers of new technology have a chance to participate in bidding and entering markets with their innovative products.

With regards to the development of multi-storey timber construction, creating healthy, sustainable, and comfortable living environments for people should be the key issue instead of the simple production of houses. Cleantech, as well as digitalisation, are enabling techniques necessary for such living environments.

**Conclusions**

This study indicates that:

- People outside the forest sector are more open to collaboration between cleantech and wood-based industries than people from the forest sector.
- Cleantech is a process-based concept, whereas the bioeconomy takes a raw-material-oriented approach. Thus, no major contradiction between the two concepts exists, and collaboration should be the rule rather than the exception.
- It might be beneficial to launch a global joint marketing brand ‘Nordic Cleantech’ among the Nordic countries, since these countries have rather limited resources yet similar strategies and interests in terms of cleantech development.
- China, followed by other Asian and European countries offer virtually unlimited markets for cleantech solutions, and wood may have a prominent role to play in entering those markets.
- The most encouraging subsectors in the Finnish wood products industries to be branded as cleantech in selected markets are: 1) the high-tech timber construction business as a part of the construction value network, and 2) process know-how in wood product manufacturing.
- An interesting topic for future research would be to analyse the means to organise win-win collaboration between the cleantech and the wood-based sectors so that challenges related to traditional competition-based business might be met.
References


Aquilar F.X., Vlosky R.P. [2007]: Consumer willingness to pay price premiums for environmentally certified wood products in the U.S. Forest Policy and Economics 9 [8]: 1100-1112


Acknowledgements

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RESEARCH REPORTS

Magdalena CZAJKA, Ewa FABISIAK

RADIAL VARIATION OF MACROSTRUCTURAL PARAMETERS AND DENSITY OF WOOD IN DOMINANT TREES OF CONIFEROUS SPECIES

This paper presents the results of measurements of wood macrostructure parameters and wood density in dominant trees of Scots pine, Norway spruce, and European larch, originating from an even-aged stand, therefore growing on identical sites and in identical climatic conditions. The influence of the cambial age of annual rings and the cross-section area on the measured features was determined in this study. The measurements of macrostructural parameters were taken using an image analyser. Basic density was determined on samples containing three annual rings. In the analysed species the widths of annual rings demonstrated significant differences in the first 50 years of tree growth. The smallest widths of annual rings throughout the whole period of tree growth were observed in the case of spruce wood. The latewood share, analysed within 25-year increment zones, demonstrated statistically significant differentiation between tested species in all selected zones of the cross-section. For each of the tested species the lowest shares were observed for juvenile wood. The lowest density values were observed, therefore, in this zone. In the mature wood of larch and pine an approximate 20% increase in the density was observed and furthermore, all the way to the circumference, density showed slight fluctuations. In the case of spruce wood, the values of density within the analysed zone increased towards the circumference. Accordingly, larch wood and pine wood were characterised by greater uniformity of properties, compared to spruce wood. This observation has important practical implications, because density is a determinant of many properties that influence the technical quality of wood.

Keywords: macrostructural parameters, wood density, pine, spruce, larch

Magdalena CZAJKA (m_czajka@itd.poznan.pl), Wood Technology Institute Poznań, Poland, Ewa FABISIAK (efabis@up.poznan.pl), Poznań University of Life Sciences, Department of Wood Science, Poznań, Poland
Introduction

The radial variability of wood properties is determined by the dynamics in the formation of annual rings, thus by their width and the share of latewood. These parameters are significant elements of the technical quality assessment of coniferous wood. They depend on many factors, inter alia, genetic predispositions, tree age, the biosocial class of trees in a stand, the type of habitat, climatic conditions, and the geographic region [Zobel and Buijtenen 1989; Antonova and Stasova 1993; Wiemann and Williamson 2002; Mäkinen and Isomäki 2004; Riesco Muñoz et al. 2008; Pärn 2012]. The wood of the same species in one region of the country, obtained from a defined habitat, may have different properties than the wood growing on a different area or habitat [Burczyk and Giertych 1991; Witkowska and Lachowicz 2013; Szaban et al. 2014]. Some reports suggest that the environmental factors have a greater impact on the growth of an annual ring than the genetic factors [Zobel and Jett 1995].

A property directly connected with the macrostructure of wood is its density. Many literature reports promote wood density as the property, determining possible applications of wood. This is connected with a strong correlation between density and mechanical parameters of wood [Bamber and Burley 1983; Dinwoodie 2000; Roszyk et al. 2013] and with the fact, that density is a property which can be relatively easily measured.

Radial variation of many properties within a tree is also significantly influenced by the location of wood tissues at the cross-section (juvenile and mature wood) [Larson 1994; Helińska-Raczkowska and Fabisiak 1991, 1999].

Bearing this in mind, the authors decided to conduct comparative tests of the variability of macrostructural parameters and density of wood of various coniferous species, originating from the same biosocial class and growing in the same habitat and climate conditions. This research will allow determination of interspecies differences in the analysed parameters and together with other properties may provide a more complete description of the technical quality of raw wood material originating from a given geographic region.

Materials and methods

The material used for testing was wood from the Norway spruce (*Picea abies* L.), Scots pine (*Pinus sylvestris* L.) and European larch (*Larix decidua* Mill.). The tests were carried out on wood from the class of dominant trees at an age of 104-106 years, from a stand growing in a habitat of mixed fresh forest. The stand was located in the forest division of Łopuchówko, a commune of Murowana Goślina (52°26′N; 16°43′E). Three trees from each species of the class of dominant trees had been chosen for investigations. Next, approximately 5 cm thick test discs were cut out at the diameter at breast height, and then approximately 4 cm wide slats were cut out from the discs along the north-south
radius. With regards to macrostructural parameters, the width of the annual rings and the share of latewood was determined. The measurements of the width of the zones of earlywood and latewood were taken using an image analyser including a stereoscopic microscope fitted with a CD camera which was connected to a computer. The measurements were conducted using the Micro Scan Plus programme. The share of latewood was calculated as a quotient of the width of the latewood zones and the width of the entire annual ring. Wood density was determined on samples split along the borders of annual rings and including three annual rings each, in the direction from the core to the circumference. The authors determined basic density, i.e. the density, which is a quotient of the mass of oven-dry wood and the volume of maximally swollen wood. The results were analysed using the programme STATISTICA 10.0 PL, descriptive statistics and the single-factor variance analysis ANOVA. All tests were carried out for a significance level of $p < 0.05$.

**Results and discussion**

Table 1 presents basic descriptive statistics of the studied parameter. All the values of the properties determined in this study are averages of the measurements taken along the north and the south radius of the three test trees of each species. The average width of the annual rings was 1.12 mm for spruce wood, and for pine and larch it was very similar and equalled 1.52 mm and 1.53 mm, respectively. Table 1 also presents descriptive statistics of the share of latewood.

**Table 1. Descriptive statistics of annual rings width, percentage of late wood of spruce, larch and pine wood and variance analysis**

<table>
<thead>
<tr>
<th>Species</th>
<th>Mean</th>
<th>Min</th>
<th>Max</th>
<th>Standard deviation</th>
<th>Standard error</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Annual rings width (mm)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Spruce</td>
<td>1.12</td>
<td>0.40</td>
<td>3.30</td>
<td>0.4111</td>
<td>0.0387</td>
</tr>
<tr>
<td>Larch</td>
<td>1.53</td>
<td>0.35</td>
<td>8.65</td>
<td>1.1209</td>
<td>0.1054</td>
</tr>
<tr>
<td>Pine</td>
<td>1.52</td>
<td>0.40</td>
<td>4.90</td>
<td>0.7764</td>
<td>0.0761</td>
</tr>
<tr>
<td></td>
<td>Percentage of late wood (%)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Spruce</td>
<td>38.39</td>
<td>14.04</td>
<td>66.66</td>
<td>11.1155</td>
<td>1.0456</td>
</tr>
<tr>
<td>Larch</td>
<td>33.30</td>
<td>5.83</td>
<td>57.14</td>
<td>10.0873</td>
<td>0.9489</td>
</tr>
<tr>
<td>Pine</td>
<td>36.73</td>
<td>7.05</td>
<td>61.36</td>
<td>11.2128</td>
<td>1.0995</td>
</tr>
</tbody>
</table>

**Results of variance analysis**

<table>
<thead>
<tr>
<th>Source of variation</th>
<th>Value of test function $F_{(103;208)}$ estimated</th>
<th>Empirical level of significance $\alpha$ tabular $\alpha=0.05$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cambial age of annual rings</td>
<td>Annual rings width</td>
<td>3.6316</td>
</tr>
<tr>
<td></td>
<td>Percentage of late wood</td>
<td>1.8291</td>
</tr>
</tbody>
</table>

*Significant differences.
The highest share of late wood was observed for spruce wood (38%), and the lowest for larch (33%). Although the arithmetic averages of the measured features were very similar, the parameters of the rings produced in the same years of tree growth were compared. The analysis of ANOVA variance proved that the interspecies differences in the width of annual rings produced in the same vegetative periods and in the share of latewood were statistically significant (tab. 1).

Analysis of the radial variability of the studied parameters should take into account values and the course of these parameters in the juvenile and mature wood zones [Fabisiak 2005; Karlman et al. 2005; Gryc et al. 2011].

Previous research conducted using the same test material resulted in the determination of the boundary between juvenile and mature wood. Based on the variability of the tracheid length, the width of juvenile wood was determined to have been the first 25 annual rings [Czajka et al. 2015]. A detailed analysis of the properties determined in this study was therefore carried out in 25-year increment zones, counting from the pith to the circumference (further they were marked as zones I, II, III, and IV). Figure 1 presents the widths of annual rings and the shares of latewood in the 25-year increment zones of the cross-section. The differences in the widths of annual rings in the analysed areas of the cross-section of spruce, larch and pine wood were statistically significant in the first two increment zones (tab. 2). In the case of spruce wood, the widths of annual rings within the juvenile wood zone were two times smaller compared to larch and pine wood, and they equalled 1.29 mm, 2.44 mm, and 2.59 mm, respectively. The width of the annual rings for individual species was most even, in the last 50 years of tree growth, i.e. in zones III and IV, where the differences in this feature were statistically insignificant at a level of $p < 0.05$ (the significance level was $p = 0.1592$ and $p = 0.1205$, respectively).

The lack of significant diversity of the radial increment of trees growing in the same vegetative periods is a result of similar dynamics of division of the initial cells of cambium in the tested tree species [Larson 1994].

On the other hand, the share of latewood demonstrated statistically significant differences between the tested species in each of the separated zones of the trunk cross-section (tab. 2). Within the zone of the first 25 annual rings the share was the greatest in spruce wood and equalled 35%, and in pine and larch wood it was 10 percentage points lower (fig. 1). In the next zone of the cross-section the latewood share increased to approximately 35% for larch wood and approximately 40% for pine wood, and in further cross-section zones it demonstrated insignificant fluctuations. Only in the case of spruce wood, was the latewood share very even along the tree radius, with the exception of the last zone of the cross-section, where it increased to approximately 45%, although the widths of annual rings in this zone were very similar to the widths of annual rings in zones II and III. It should be mentioned here, that the variability of the proportion of earlywood and latewood in trees growing in the same habitat
conditions are influenced by the interaction of genetic and environmental factors [Creber and Chaloner 1984].

Fig. 1. The mean of the rings’ width and the percentage of late wood in the test zone of spruce, larch and pine wood; vertical bars denote a confidence interval of 95%

Descriptive statistics of the basic density of the studied wood species are presented in table 3. The radial variability of density was analysed in the 25-year increment zones, as were the previously discussed properties (fig. 2). Spruce wood, compared to pine and larch wood, was characterised by the lowest density throughout the whole period of tree growth. The density of spruce wood, had an average for the whole cross-section, of 423 kg/m³, while in the case of larch it was 478 kg/m³, and for pine it equalled 505 kg/m³ (tab. 3). The obtained density values fell within the scope of previously published literature [Čunderlík et al. 2005; Gryc et al. 2011]. Irrespective of the species, the lowest values of density
Table 2. Analysis of the variance of annual rings width and percentage of latewood for cross-section zone in spruce, larch and pine wood

<table>
<thead>
<tr>
<th>Source of variation</th>
<th>Cross-section zone</th>
<th>Value of test function $F_{(2:72)}$ estimated</th>
<th>Empirical level of significance $\alpha$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ring width in spruce, larch and pine wood</td>
<td>I</td>
<td>7.6024</td>
<td>0.0010</td>
</tr>
<tr>
<td></td>
<td>II</td>
<td>16.5401</td>
<td>0.0000</td>
</tr>
<tr>
<td></td>
<td>III</td>
<td>1.8851</td>
<td>0.1592&lt;sup&gt;ns&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>IV</td>
<td>2.6590</td>
<td>0.1205&lt;sup&gt;ns&lt;/sup&gt;</td>
</tr>
<tr>
<td>Percentage of latewood in spruce, larch and pine wood</td>
<td>I</td>
<td>7.2042</td>
<td>0.0014</td>
</tr>
<tr>
<td></td>
<td>II</td>
<td>6.0922</td>
<td>0.0035</td>
</tr>
<tr>
<td></td>
<td>III</td>
<td>3.2568</td>
<td>0.0442</td>
</tr>
<tr>
<td></td>
<td>IV</td>
<td>6.7379</td>
<td>0.0019</td>
</tr>
</tbody>
</table>

<sup>ns</sup> – insignificant differences.

Table 3. Descriptive statistics of the basic density of spruce, larch and pine wood and variance analysis for the mature wood zone

<table>
<thead>
<tr>
<th>Species</th>
<th>Mean</th>
<th>Min</th>
<th>Max</th>
<th>Standard deviation</th>
<th>Coefficient of variation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spruce</td>
<td>423</td>
<td>366</td>
<td>509</td>
<td>39.3</td>
<td>9.2</td>
</tr>
<tr>
<td>Larch</td>
<td>478</td>
<td>298</td>
<td>550</td>
<td>54.0</td>
<td>11.3</td>
</tr>
<tr>
<td>Pine</td>
<td>505</td>
<td>270</td>
<td>633</td>
<td>79.9</td>
<td>15.8</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Source of variation</th>
<th>Species</th>
<th>Value of test function $F_{(2:16)}$ estimated</th>
<th>Empirical level of significance $\alpha$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Basic density of the cross-section zone in mature wood (zones II, III and IV)</td>
<td>Spruce</td>
<td>6.3361</td>
<td>0.0094</td>
</tr>
<tr>
<td></td>
<td>Larch</td>
<td>0.6704</td>
<td>0.5253&lt;sup&gt;ns&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>Pine</td>
<td>0.5399</td>
<td>0.5930&lt;sup&gt;ns&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

<sup>ns</sup> – insignificant differences.

were characteristic of the close-to-core annual rings. In the subsequent years of tree growth, as the sample grew away from the pith, the density increased and then stabilised at some level, with insignificant fluctuations. In the case of coniferous wood, juvenile wood is characterised by a lower density than mature wood, which is connected with the difference in the proportion of cell wall thickness to cell diameter in both types of wood. Within the juvenile wood zone, the share of latewood is lower than in further zones of the trunk cross-section [Kučera 1994; Sauter et al. 1999].

Analysing this property within one species, it may have been observed, that it increased the most between the zone encompassing the first 25 close-to-core annual rings (juvenile wood) and further zones of the cross-section. This difference was approximately 25% for pine wood, 16% for larch wood, and only
4% for spruce wood. Gryc et al. [2011] in their research on the density of wood from the close-to-core zone and close-to-circumference zone of 80-100 year old trees, observed similar differences, which equalled 18% for pine, 15% for spruce, and 8% for larch. In this study, it was observed that differences in the analysed feature were statistically insignificant between zones II, III and IV (hence in mature wood) in the case of pine wood and larch wood, which suggested high homogeneity of this feature in these zones (tab. 3). On the other hand, in the case of spruce wood, these differences were significant, furthermore, although the share of latewood in the last increment zone was the greatest, compared to the other species, the density values were the lowest among the tested species. This observation proves, that the variability of the latewood share may be a determinant of wood quality, expressed by its density, only within particular species and not between species.

![Graph showing radial variation of basic density in 25-year increment zones of spruce, larch and pine wood](image)

**Fig. 2. Radial variation of basic density in 25-year increment zones of spruce, larch and pine wood**

**Conclusions**

The research conducted indicates that the widths of annual rings in the analysed coniferous species demonstrated significant differences only in the first 50 years of tree growth. Amongst the studied species, spruce wood was characterised by the narrowest annual rings (throughout the whole period of tree growth). In the
case of this species, the width of annual rings in the first selected zone of the
cross-section, i.e. in the juvenile wood, was two times smaller than in the case of
pine wood and larch wood. The share of latwood, analysed within 25-year
increment zones, demonstrated statistically significant differentiation between
the studied species in all selected zones of the cross-section. The lowest values
of this parameter were observed in each of the studied species within the period
of juvenile growth of the trees. Accordingly, these zones of the cross-section
were characterised by the lowest density (390 kg/m³ for spruce, 430 kg/m³ for
larch, and 450 kg/m³ for pine). In the case of larch and pine, an approximate
20% increase in density was observed in the subsequent zone of the cross-
section, i.e. in the mature wood, and in further zones density slightly fluctuated
all the way to the circumference. In the case of spruce wood, a gradual increase
in density was observed in the mature zone (approximately 11%). Although the
share of latwood was the highest in spruce wood, compared to larch and pine,
the density demonstrated the lowest values. This observation proves, that the
variability of the latwood share may be a determinant of wood quality,
expressed by its density, only within particular species and not between species.

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Daniela T. SILVA, Nadia H. BIANCHINI, Marlove F. B. MUNIZ, Berta M. HEINZMANN, Jalel LABIDI

CHEMICAL COMPOSITION AND INHIBITORY EFFECTS OF NECTANDRA GRANDIFLORA LEAVES ESSENTIAL OIL AGAINST WOOD DECAY FUNGI

The environmental toxicity and potential human health problems that can be caused by most common wood preservatives are pushing forward the search of safe preservatives from renewable resources. Consequently, several studies have been realized to assess the potential of plant extracts in terms of fungal resistance for wood products. In this context, the present work aims to evaluate the inhibitory effects of essential oil obtained from the leaves of Nectandra grandiflora against wood decay fungi. Chemical characterization was carried out by gas chromatography and the antifungal activity was performed by the radial growth technique. Firstly, the potato sucrose agar medium was supplemented with essential oil at concentrations ranging from 0 (control) to 5.0 μL·mL⁻¹. Afterwards, mycelial discs of Pycnoporus sanguineus and Gloeophyllum trabeum were transferred to the plates and the results were evaluated by the probit method. Chemical analysis revealed a complex mixture of sesquiterpenoids in the essential oil, which presented dehydrofukinone as a major compound. The essential oil and dehydrofukinone proved to be effective in the mycelial growth control of G. trabeum and P. sanguineus. These preliminary reports demonstrated the suitability of the N. grandiflora essential oil as a component of preservative solutions.

Keywords: antifungal property, wood preservatives, Lauraceae, volatile components

Introduction

Some fungal species can be pathogenic to forest and urban trees and consequently, they deteriorate wood products [Bento et al. 2014]. Wood decay fungi are mainly responsible for the destruction of structural elements of the cell wall that can result in economic and material losses and subsequently, reduces

Daniela T. SILVA (dthomasdasilva@gmail.com), Nadia H. BIANCHINI (nadia bianchini@hotmail.com), Marlove F. B. MUNIZ (marlovenuniz@yahoo.com.br), Berta M. HEINZMANN (berta.heinzmann@gmail.com), Federal University of Santa Maria, Santa Maria, Brazil; Jalel LABIDI (jalel.labidi@ehu.es), University of the Basque Country, San Sebastian, Spain
the wood quality [Stangerlin et al. 2013]. Among the most important saprophytic
wood-rot basidiomycetes, are *Pycnoporus sanguineus* (white-rot fungi) and
*Gloeophyllum trabeum* (brown-rot fungi) which are able to degrade wood
components. The first species attacks the lignocellulosic materials, while the
second destroys the polysaccharide constituents.

Although traditional synthetic fungicides such as arsenate-based wood
preservatives are very effective, its continued employment has led to
environmental pollution, resistance development and human health toxicity
[Yoon et al. 2013]. Plants produce large amounts of secondary metabolites for
protection against adverse environmental conditions and biological pests.
Extractives and isolated components, therefore, from individuals of diverse
botanical families have been studied and have demonstrated promissory
potential to fungal control [Cowan 1999; Schultz and Nicholas 2002; Wang et al.
2005; Sen et al. 2009].

In addition to the low toxicity, the application of substances from vegetal
resources could improve the efficacy of antifungal products through its
synergistic effects including a positive interaction between the components.
Hwang et al. [2007], Schultz and Nicholas [2002] reported the usefulness of the
combined use of tannins, heartwood extractives and synthetic biocides. In this
background, the study aims to investigate the influence of essential oil extracted
from *Nectandra grandiflora* leaves, on the mycelial growth of two species of
wood-rot fungi. Additionally, a comparison of the antifungal property of
essential oil and its major constituent, dehydrofukinone, was performed.

**Materials and methods**

**Essential oil obtainment**

Leaves of the *Nectandra grandiflora* Nees were collected on a native population
located in the Jaguari city, South of Brazil (at 29º 26’ S and 54º 40’ W). The
fresh leaves were fragmented and afterwards the essential oil was extracted by a
hydrodistillation process using a Clevenger-type apparatus for three hours. The
essential oil yield was quantified based on the mass weight (0.7 g per 100 g of
dried leaves). An aliquot of the obtained extractive was chemically analysed by
gas chromatography.

**Chemical characterization and quantification**

The chemical composition of the essential oil was determined by an Agilent
7890A gas chromatograph connected to a mass spectrometer 5075C (GC-MS)
using a non-polar HP5-MS fused silica (5% phenyl, 95% methylsiloxane)
capillary column (30 m × 0.25 mm i.d. × 0.25 mm film thickness), and an
electron ionization mode at 70 eV. The carrier gas was helium at a flow rate of
1.0 mL·min⁻¹, injector and detector temperatures of 150ºC and 280ºC,
respectively, the split inlet injection mode (ratio 1:100), oven temperature at 40°C for four minutes, and up to 320°C at 4°C·min⁻¹ were the employed parameters. Quantitative evaluation was performed in triplicate using a flame ionization detector (GC-FID), according to Silva et al. [2015]. The constituents were then identified by comparison of the retention indices and mass spectra with libraries [NIST-EPA-NIH 2009; Adams 2009].

Following the chemical characterization, three chromatography columns (CC) were performed to fractionate the leaves essential oil and to isolate the main constituent. In the first CC, 11 g of essential oil were added to 630 g of silica gel 60 (7.2 × 30.5 cm) and eluted with hexane-acetone (95:5 v/v). Fractions of 45 mL were gathered in nine main fractions based on the thin layer chromatography (TLC) profile and concentrated under reduced pressure at 40°C. The TLC was carried out on silica gel 60 F₂₅₄ chromathoplates and the spots were detected by vanillin sulfuric acid-UV 365 nm.

The fractions 4 and 5 were grouped (2.8 g) and submitted to another CC (4.1 × 43.2 cm, 260 g silica gel 60, hexane-ethyl ether 95:5 at 1.25 mL/min). From the resulting ten main fractions, 6 and 7 were grouped (1.4 g) and submitted to the third CC (2.4 × 66 cm, 90 g of silica gel sixty impregnated with 10% AgNO₃, hexane-acetone 95:5 at 1.0 mL/min) [Williams and Mander 2001]. Among the four main fractions obtained, the fraction 1 (1 g) was identified as dehydrofukinone (100% purity), according to Schenato et al. [2001], Alkhathlan et al. [2005], Bolzan [2007] and Silva et al. [2015].

**Antifungal activity**

The antifungal assay was evaluated by the radial growth technique [Wang et al. 2005]. All assays were carried out in quadruplicate. Primarily, potato sucrose agar (200 g, 20 and 18 g in 1 L of distilled water) medium was supplemented with essential oil at concentrations of 0 (control), 0.25, 0.50, 1.0, 2.0 and 5.0 μL/mL, which were dissolved in ethanol (1:1), and placed into the Petri dishes. Mycelial discs (1.5 cm diameter) were then transferred aseptically to the center of the plates and incubated at 25 ±4°C in 12 h-photoperiod for seven days.

In another experiment, the inhibition effect of dehydrofukinone was analysed at equivalent concentration to that detected in 5 μL/mL (4.63 μg/mL) of essential oil. Thereby, considering the density of essential oil (0.926 g/mL), the substance purity (100%) and the content of dehydrofukinone in essential oil (26.85%), dehydrofukinone was tested at 1.25 μg/mL. This assay was performed as mentioned above.

The growth of the fungal colony was estimated by an average of two perpendicular measurements and mycelial-growth rate was calculated using the following equation (1).
\[ MGR \ (\text{mm}) = \frac{dt}{n_1} + \frac{dt}{n_2} + \frac{dt}{n_5} + \frac{dt}{n_7} \]  
(1)

where: \( n \) – days of the beginning of the experiment,
\( dt \) – average diameters of fungal colony (mm).

Mycelial-growth inhibition (%) was calculated through the equation (2).

\[ \% \text{ inhibition} = \frac{MGR_c - MGR_t}{MGR_t} \times 100 \]  
(2)

where: \( MGR_c \) – mycelial-growth rate of control,
\( MGR_t \) – mycelial-growth rate of treatment.

The concentration-response data of the first experiment were analysed by probit analysis [Finney 1971] to obtain the 50% lethal concentration (LC_{50}) values and 95% confidence interval (CI). Mycelial-growth rate (MGR) results of dehydrofukinone in comparison with the essential oil were evaluated by t-test. A significant difference was considered at a level of \( P < 0.05 \).

Results and discussion

Chemical characterization

GC-MS and GC-FID analysis led to the identification of twenty-four components, representing 73.16% of the total essential oil obtained from \( N. \) grandiflora leaves. The chemical composition revealed a complex mixture of sesquiterpenoids in the extractive (tab. 1) and the chromatogram of GC-MS peaks is shown in figure 1A. The major volatile components were dehydrofukinone (26.85%, fig. 1B), valencene (6.89%), kaurene (6.03%), aristolochene<4,5-di-epi> (5.41%), selin-11-en-4-α-ol (5.34%) and bicyclogermacrene (5.06%).

In comparison with other studies about the chemical compositions of the essential oils of the \( N. \)ectandra species, differences in the major components could be observed. According to Amaral et al. [2015], \( N. \) megapotamica presented the highest quantities of monoterpene such as α- and β-pinene, similar to those detected for the essential oil of \( N. \) membranacea leaves [Wu et al. 2006]. The sesquiterpenoid atracylone was extracted from the leaves of \( N. \) salicina amounting to 14.6% of essential oil [Cicció et al. 2009]. Conversely, previous reports showed dehydrofukinone as the main component of the leaf extracts from the \( S \)enecio species [Bohllmann et al. 1981; Nachman 1983]. To date, this compound was only described in the Lauraceae target by Garlet et al. [2016].
Table 1. Chemical composition of the essential oil obtained from *N. grandiflora* leaves

<table>
<thead>
<tr>
<th>RT</th>
<th>Component</th>
<th>RP&lt;sup&gt;a&lt;/sup&gt;</th>
<th>RI&lt;sup&gt;b&lt;/sup&gt; calc</th>
<th>RI&lt;sup&gt;c&lt;/sup&gt; lib</th>
<th>Content (%)&lt;sup&gt;d&lt;/sup&gt;</th>
<th>Identification</th>
</tr>
</thead>
<tbody>
<tr>
<td>10.4</td>
<td>α-Pinene</td>
<td>931</td>
<td>930&lt;sup&gt;n&lt;/sup&gt;</td>
<td>0.37</td>
<td>RI, MS</td>
<td></td>
</tr>
<tr>
<td>12.0</td>
<td>β-Pinene</td>
<td>973</td>
<td>975&lt;sup&gt;n&lt;/sup&gt;</td>
<td>0.25</td>
<td>RI, MS</td>
<td></td>
</tr>
<tr>
<td>14.7</td>
<td>Z-β-Ocimene</td>
<td>1039</td>
<td>1038&lt;sup&gt;n&lt;/sup&gt;</td>
<td>0.30</td>
<td>RI, MS</td>
<td></td>
</tr>
<tr>
<td>15.0</td>
<td>E-β-Ocimene</td>
<td>1049</td>
<td>1047&lt;sup&gt;n&lt;/sup&gt;</td>
<td>1.38</td>
<td>RI, MS</td>
<td></td>
</tr>
<tr>
<td>17.1</td>
<td>Linalool</td>
<td>1100</td>
<td>1100&lt;sup&gt;n&lt;/sup&gt;</td>
<td>0.41</td>
<td>RI, MS</td>
<td></td>
</tr>
<tr>
<td>20.3</td>
<td>Z-3-Hexenyl butyrate</td>
<td>1188</td>
<td>1186&lt;sup&gt;n&lt;/sup&gt;</td>
<td>0.95</td>
<td>RI, MS</td>
<td></td>
</tr>
<tr>
<td>27.3</td>
<td>β-Elemene</td>
<td>1393</td>
<td>1391&lt;sup&gt;n&lt;/sup&gt;</td>
<td>3.29</td>
<td>RI, MS</td>
<td></td>
</tr>
<tr>
<td>28.1</td>
<td>β-Caryophyllene</td>
<td>1420</td>
<td>1418&lt;sup&gt;n&lt;/sup&gt;</td>
<td>0.80</td>
<td>RI, MS</td>
<td></td>
</tr>
<tr>
<td>28.8</td>
<td>α-Guaiene</td>
<td>1442</td>
<td>1441&lt;sup&gt;n&lt;/sup&gt;</td>
<td>1.38</td>
<td>RI, MS</td>
<td></td>
</tr>
<tr>
<td>29.2</td>
<td>α-Caryophyllene</td>
<td>1455</td>
<td>1454&lt;sup&gt;n&lt;/sup&gt;</td>
<td>0.49</td>
<td>RI, MS</td>
<td></td>
</tr>
<tr>
<td>29.7</td>
<td>Aristolochene&lt;sup&gt;&lt;4,5-di-epi&gt;&lt;/sup&gt;</td>
<td>1471</td>
<td>1473&lt;sup&gt;a&lt;/sup&gt;</td>
<td>5.41</td>
<td>RI, MS</td>
<td></td>
</tr>
<tr>
<td>30.1</td>
<td>α-Amorphene</td>
<td>1482</td>
<td>1485&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.71</td>
<td>RI, MS</td>
<td></td>
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<tr>
<td>30.3</td>
<td>Valencene</td>
<td>1489</td>
<td>1488&lt;sup&gt;a&lt;/sup&gt;</td>
<td>6.89</td>
<td>RI, MS</td>
<td></td>
</tr>
<tr>
<td>30.4</td>
<td>Z-β-Guaiene</td>
<td>1495</td>
<td>1493&lt;sup&gt;a&lt;/sup&gt;</td>
<td>1.84</td>
<td>RI, MS</td>
<td></td>
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<tr>
<td>30.5</td>
<td>Bicyclogermacrene</td>
<td>1498</td>
<td>1500&lt;sup&gt;a&lt;/sup&gt;</td>
<td>5.06</td>
<td>RI, MS</td>
<td></td>
</tr>
<tr>
<td>30.8</td>
<td>Germacrene A</td>
<td>1506</td>
<td>1509&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.24</td>
<td>RI, MS</td>
<td></td>
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<tr>
<td>31.8</td>
<td>Kessane</td>
<td>1541</td>
<td>1539&lt;sup&gt;a&lt;/sup&gt;</td>
<td>2.68</td>
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<td></td>
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<tr>
<td>32.9</td>
<td>Spathulenol</td>
<td>1579</td>
<td>1578&lt;sup&gt;n&lt;/sup&gt;</td>
<td>1.49</td>
<td>RI, MS</td>
<td></td>
</tr>
<tr>
<td>34.2</td>
<td>Humulane-1,6-dien-3-ol</td>
<td>1624</td>
<td>1619&lt;sup&gt;n&lt;/sup&gt;</td>
<td>0.31</td>
<td>RI, MS</td>
<td></td>
</tr>
<tr>
<td>34.4</td>
<td>Eremoligenol</td>
<td>1632</td>
<td>1631&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.36</td>
<td>RI, MS</td>
<td></td>
</tr>
<tr>
<td>35.1</td>
<td>Selin-11-en-4-α-ol</td>
<td>1657</td>
<td>1660&lt;sup&gt;a&lt;/sup&gt;</td>
<td>5.34</td>
<td>RI, MS</td>
<td></td>
</tr>
<tr>
<td>37.4</td>
<td>Isobicyclogermacrene</td>
<td>1741</td>
<td>1734&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.33</td>
<td>RI, MS</td>
<td></td>
</tr>
<tr>
<td>39.0</td>
<td>Dehydrofukinone</td>
<td>1807</td>
<td></td>
<td>26.85</td>
<td>MS, NMR</td>
<td></td>
</tr>
<tr>
<td>44.8</td>
<td>Kaurene</td>
<td>2039</td>
<td>2043&lt;sup&gt;n&lt;/sup&gt;</td>
<td>6.03</td>
<td>RI, MS</td>
<td></td>
</tr>
</tbody>
</table>

Components identified: 73.16

<sup>a</sup>Retention time; <sup>b</sup>Retention indices relative to n-alkanes (C8-C31) on a HP5-MS capillary column; <sup>c</sup>retention index from libraries: ^Adams [2009]; NNational Institute of Standards and Technology – U.S. Environmental Protection Agency – National Institutes of Health [NIST-EPA-NIH 2009]; <sup>d</sup>Content obtained by GC-FID.
Moreover, *N. grandiflora* is an endemic tree from Brazil and its leaves were employed in local medicine as a diuretic and digestive [Correa 1984]. There are a few studies about the biological activities of this plant [Moreno et al. 1993], however, little is known about its essential oil.

**Antifungal activity**

Regarding the antifungal assay, the essential oil of the *N. grandiflora* proved to be effective in the inhibiting of *P. sanguineus* and *G. trabeum* and the detected activity was in a concentration-dependent manner (tab. 2).
Table 2. Mycelial-growth inhibition (%) of essential oil of from *N. grandiflora* leaves against *P. sanguineus* and *G. trabeum*

<table>
<thead>
<tr>
<th>Fungi species</th>
<th>Concentration (µL/mL)</th>
<th>Inhibition mean ± SEM (%)</th>
<th>LC$_{50}$ ± SEM (µL/mL) 95% confidence limits</th>
<th>Intercept</th>
<th>Slope</th>
<th>$\chi^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td><em>P. sanguineus</em></td>
<td>0.25</td>
<td>26.75 ± 0.99</td>
<td>1.22 ± 0.16 (0.96-1.61)</td>
<td>4.91</td>
<td>1.06</td>
<td>1.50</td>
</tr>
<tr>
<td></td>
<td>0.50</td>
<td>33.34 ± 0.46</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.0</td>
<td>46.57 ± 1.08</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2.0</td>
<td>52.20 ± 0.93</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>5.0</td>
<td>79.03 ± 0.41</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><em>G. trabeum</em></td>
<td>0.25</td>
<td>47.82 ± 0.46</td>
<td>0.39 ± 0.26 (0.015-0.84)</td>
<td>5.42</td>
<td>1.02</td>
<td>2.27</td>
</tr>
<tr>
<td></td>
<td>0.50</td>
<td>55.24 ± 0.75</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.0</td>
<td>61.70 ± 1.40</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2.0</td>
<td>69.53 ± 3.69</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>5.0</td>
<td>94.76 ± 0.63</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

$\chi^2$ values were calculated by probit method [Finney 1971].

The calculated $\chi^2$ values were obtained and compared with tabulated $\chi^2$ to verify the adequacy of results for the probit model. All values showed appropriate fit, with calculated $\chi^2$ lower than tabulated $\chi^2$ (7.81). The CI values obtained after seven days of treatment did not overlap, indicating the significant differences between the effects of essential oil on both fungi species. *G. trabeum*, however, seems to be more vulnerable to essential oil, because it presented the lowest LC$_{50}$ (0.39 ±0.26 µL/mL) and CI values (0.015-0.84 µL/mL). Our results agree with Yen and Chang [2008], who proposed that the antifungal activity is strongly related to the fungal species.

Furthermore, figure 2 shows a comparative analysis of antifungal effects among essential oil and the isolated substance against *P. sanguineus* and *G. trabeum*.

The t-test showed no significant difference between the MGR results of essential oil and dehydrofukinone with respect to *P. sanguineus* (8.03 ±1.05 mm and 9.89 ±0.74 mm, respectively; $P = 0.171$). This similarity, however, was not found in the *G. trabeum* assay, which showed more susceptibility to the essential oil. Such behavior could be explained by the synergistic or additive effects of substances present in *N. grandiflora* essential oil [Schultz and Nicholas 2002; Yen and Chang 2008]. Mycelial-growth inhibition results observed in this work, ranged from 76.06% to 79.45% for dehydrofukinone and it was greater than 80.56% for essential oil. Secondary metabolites such as essential oils are a promising source of active substances that can provide the fungal protection of wood surfaces. Some studies reported the potential use of essential oil from *Cinnamomum osmophloeum* [Wang et al. 2005] and *Eucalyptus camaldulensis* leaves [Salem et al. 2016], as well as pure substances [Marei et al. 2012] with this purpose.
Fig. 2. Comparative analysis of mycelial-growth rate (MGR) concerning essential oil from *N. grandiflora* leaves (4.63 μg/mL) and dehydrofukinone (1.25 μg/mL) against *G. trabeum* and *P. sanguineus*

*Indicates significant differences among essential oil and dehydrofukinone by t-test (*P* < 0.05).

**Conclusions**

The essential oil of *Nectandra grandiflora* leaves was mainly composed of sesquiterpenoids and among them dehydrofukinone was the major one. Preliminary findings display the *in vitro* efficacy of leaves extractives from *N. grandiflora* against two wood-rot fungi. Moreover, the results suggested that the detected effect is partially due to dehydrofukinone. The essential oil and its major component can then be applied as a natural fungicide in wood treatments.

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Patrícia DOS SANTOS, Silvia DA SILVA, Darci GATTO, Jalel LABIDI

FIRE RESISTANCE OF WOOD TREATED BY EMULSION FROM KRAFT LIGNIN

The use of wood for construction is not new, however, in some countries there are many resistances to its use, due mainly to the behaviour of wood during a fire. This study aimed to enhance the fire resistance of wood by treating the wood with Kraft lignin emulsion. An emulsion from Kraft lignin was used for the treatment of wood Pinus sp. by full cell methods. The specific mass, wettability and Brinell hardness were analysed. Fire resistances were determined (ignition, flame and ember time). The obtained results showed that the ignition times of treated wood, were higher than those of untreated wood. The emulsion hinders the ignition of samples in approximately 10 seconds. Conversely, the flame and ember time was significantly longer in wood treated, but there was a lower mass loss, compared to untreated wood. This shows that the treatment improves the behaviour of wood in relation to the ignition of fire and also increases the hardness of the wood. Wettability analysis was possible to observe that treatment reduces the rate of moisture uptake.

Keywords: fire retardancy of wood, Kraft lignin, ignition, flame and ember time

Introduction

The use of wood for construction is nothing new and many countries have a culture of building wooden houses. The wooden buildings provide a warm and comfortable addition. The use of wood in building has several advantages over other materials due to its low cost, excellent mechanical, thermal and acoustic properties, as well as providing a cosy atmosphere. The major factor considered in the utilization of wood in buildings, however, is its behaviour against fire [Tondi et al. 2012]. Because according to Zhang [2011], flammability is the biggest disadvantage of wood.

Fire resistance depends on the thickness of the charred layer of wood and the size of the residual section, which depends on the rate of carbonization of the wood species used [White 1995].

Patricia SOARES BILHALVA DOS SANTOS (patricia.bilhalva@hotmail.com), Silvia FUENTES DA SILVA, (silviashfuente@hotmail.com), Jalel LABIDI (jalel.labidi@ehu.eus), Chemical and Environmental Engineering Department, University of the Basque Country, San Sebastian, Spain, 2018; Darci GATTO, (darcigatto@yahoo.com) Center Engineering, Federal University of Pelotas, Brazil, 96010-290
The pulp industry is the most important source of production of lignin, this waste is primarily used as fuel in furnaces to generate energy and the recovery of reactive inorganic used in the process. Lignin has a bio-protective activity [Dos Santos et al. 2012] and acts as one of the plant protectors against attack by microorganisms and pests [Quesada-Medina et al. 2010]. These properties could provide added value in the use of lignin.

The aim of this work was to study the fire resistance (ignition, flame and ember time) of wood after impregnation with emulsion of Kraft lignin using the full cell method. The specific mass, wettability and Brinell hardness of the resulting materials were analysed.

**Materials and methods**

Tests were performed on *Pinus* sp. sapwood (DBH height, 1.30 m) after sectioning each tree, the central plank was selected and cut according to the recommendation of the [ASTMD 5536:1994]. The samples were stored in a climatic chamber at 20°C and 65% of relative humidity until the equilibrium moisture was reached. The thickness of the log was then reduced from 8.0 to 6.0 cm through a plane and various test samples with different dimensions were cut.

Wood treatment was done with a preservative emulsion of Kraft Lignin (EKL), that was prepared with Kraft lignin (2 g/L) in the presence of NaOH (1%) and 97% of water was used. The treatment was carried out in a laboratory autoclave with 2 L capacity using the Full cell procedure. An initial vacuum of 1.0-1.2 bar for 30 min was applied, after the impregnation with the preservative solution, a pressure of 6.0 to 8.0 bars was applied for 60 min. Weight percent gains (WPG) of the samples due to the treatment with (EKL) were calculated by equation 1.

\[
WPG = \left(1 - \frac{W_m}{W_u}\right) \times 100
\]

where: \(W_m\) = dried weight of sample after treatment, \(W_u\) = dried weight of untreated samples.

The surface hardness (Brinell) was evaluated according to the standard test [EN 1534:2000], using Emco test automatic M4U075 equipment. The test was performed in the tangential surface of ten specimens (80 × 20 × 10 mm³) in three-points for each sample.

The short-term fire tests were done according the procedure described by Tondi et al. [2012]. The radial surfaces of the samples (50 × 25 × 15 mm³) were exposed to the flame of a Bunsen burner for 2 min, with distance at 7 cm between blue flame and wood surface. The ignition, flame and ember time were determined with a stopwatch and realized in quintuplicate for each treatment.

Contact angles (CA) were measured by the sessile droplet method (5 μL distilled water) in all samples at equilibrium moisture content (EMC). The first measurement of CA was made after 5 seconds (time zero). Subsequent CA,
measurements were repeated at intervals of 30 seconds up to 120 seconds, totalling six CA measurements per sample and in five samples for each treatment.

**Results and discussion**

The basic characteristics of aqueous preservative emulsion of Kraft Lignin (EKL), analysed in this work were pH 6.8, density 0.97 g·ml⁻¹ and black colour. Samples presented a weight percentage gain (10.5%) after 24 hours with the utilized treatment.

In table 1 the results of Brinell hardness is shown, where the treated wood showed a higher hardness with (13.44 N·mm⁻²) compared to the untreated wood (5.70 N·mm⁻²). According to Fejfer et al. [2014], the basic density is a standard procedure in the assessment of the degree of the degradation of wood.

<table>
<thead>
<tr>
<th>Samples</th>
<th>WPG (%)</th>
<th>Density 12% (kg·m⁻³)</th>
<th>Brinell hardness (HB) [N/mm²]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Untreated</td>
<td>–</td>
<td>453.8 (7.2)</td>
<td>5.70 (1.38)</td>
</tr>
<tr>
<td>Treated</td>
<td>10.5 (0.15)</td>
<td>476.6 (8.4)</td>
<td>13.44 (1.73)</td>
</tr>
</tbody>
</table>

The values in parentheses are SD. Mean values in the same column followed by the same letter are not statistically different at level of 5% by the Tukey test.

It is known that the hardness varies with the material density, but in this study, it was not possible to observe the increase in hardness due to the variation of the density, because the density increment values were relatively small compared to an increase in hardness, which showed an increase of approximately 135% in *Pinus* wood treatment, compared with untreated wood.

The behaviour against fire is an important factor for the use of wood in the construction of buildings. The short time fire exposure test simulates the ignition process, whilst the weight loss test after longer exposure, provides better information about the strength of a wood species [Tondi et al. 2012].

The ignition time shown in table 2 was significantly different for both treated timber (26.6 s) and untreated (13.2 s). The EKL emulsion retarded the ignition by over 10 seconds compared to untreated wood.

Moreover, the flame was significantly longer in the treated wood (172 s) and with longer glowing time (714 s). Nevertheless, the treated wood presented lower weight loss (26.9%) compared to the untreated wood (33.5%) which had smaller fire time and ember (137.7 and 335.20 s respectively). These results show that treatment improves the performance of the timber in relation to
ignition time and mass loss when the wood is exposed to direct heat for 2 min. According to Li et al. [2014], a fire retardant is seen as any substance that by physical or chemical action inhibits combustion.

**Table 2. Short time fire test and ignition time of the three species studied**

<table>
<thead>
<tr>
<th>Samples</th>
<th>Ignition time (s)</th>
<th>Flame time (s)</th>
<th>Ember time (s)</th>
<th>Weight loss (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Untreated</td>
<td>13.2 (1.6)a</td>
<td>137.7 (18.0)a</td>
<td>335.2 (31.3)a</td>
<td>33.5 (1.4)b</td>
</tr>
<tr>
<td>Treated</td>
<td>26.6 (0.9)b</td>
<td>172.2 (10.2)b</td>
<td>714.2 (24.0)b</td>
<td>26.9 (1.3)a</td>
</tr>
</tbody>
</table>

The values in parentheses are SD. Mean values in the same column followed by the same letter are not statistically different at level of 5% by the Tukey test.

The fire resistance depends on the thickness of the wood carbonized layer and the size of the residual section, which for optimal use of wood species with respect to fire resistance depends on the charring rate [White 1995].

The differences in the wetting characteristics of wood treated and untreated by the sessile droplet method are presented in figure 1.

![Image A](image1.png)

**Fig. 1. The image droplet water in wood surface. A: untreated wood (time = zero), B: treated wood with EKL (time = zero)**

In figure 2, it is possible to observe that the treatment reduces the rate of moisture uptake, but over time the wood absorbs a little of the water droplet reducing contact angle. The treated wood samples became more hydrophobic, that means that the treatment with the emulsion of Kraft lignin by full cell method is also more effective regarding the contact angles (CA). The effect that could have the decrease of the hygroscopicity in the dimensional stability of wood, however, was not studied.
Fig. 2. Values of radial contact angle (°) in wood treated compared with untreated wood

Conclusions

The impregnation with EKL by the full cell method improved the performance of wood in relation to ignition of fire and the hardness of the wood, when compared with untreated wood. Furthermore, it improved the hydrophobic properties of wood species *Pinus* sp.

It is extremely important to perform other types of analyses (such resistance to leaching and evaporation, decay resistance) in future work to verify the possible applications and the limitations of using EKL.

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List of standards


EN 1534:2011 Wood Floring – Determination of resistance to indentation – Test method

Acknowledgements

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Karol Bula, Monika Knitter

PROPERTIES OF HIGH-DENSITY POLYETHYLENE REINFORCED WITH PINE-WOOD FILLERS

Wood-polymer composites based on high-density polyethylene and two types of pine-wood filler were investigated. It is shown that the addition of long wood fibres to common polyethylene leads to a substantial increase in stiffness, i.e. from 504 MPa for PE-HD up to 1526 MPa for PE-HD with 60% wood fibres. Moreover, morphological investigations revealed that superficial wood particles were sufficiently embedded in the base polymer ensuring protection against higher moisture uptake rates.

Keywords: polyethylene, pine wood, mechanical properties

Introduction

Wood-polymer composites (WPCs) are materials which have been on the market for many years, and are used in the construction industry, for example, for moulded components or decking profiles [Błędzki et al. 2006; Zajchowski and Ryszkowska 2009; Oszust et al. 2011; Michalska-Pożoga and Czerwińska 2015]. For furniture application (plates and sheets), thermostetting materials, such as phenol formaldehyde resin, are usually used as a polymeric matrix. Thermoplastic polymers such as polypropylene, polyethylene or polyvinyl chloride are the most common, are characterized by good commercial properties and may be shaped by conventional processing methods such as injection moulding or extrusion [Gozdecki et al. 2011; 2012; 2015; Kociszewski et al. 2012] at moderate temperatures not exceeding 210°C. In comparison with synthetic commodity polymers, wood is a cheaper, stiffer and stronger material, which makes it a good candidate for use as a polymer filler or for reinforcement. The properties of a wood-polymer composite depend on the type of matrix and filler. In the case of a reinforcing agent, not only is the volume fraction important, but also the size, shape and surface area of the particles, as well as the compatibilization method [Bula and Jesionowski 2010; Bula et al. 2015; Knitter and Dobrzyńska-Mizer 2014]. The filler used to prepare WPC can be of any
origin, and have different forms: splinters, chips and flour. It can be obtained from conifers (pine or spruce) and much less frequently from deciduous trees (oak or maple) [Stark and Rowlands 2003; Liber-Kneć et al. 2006; Migneault et al. 2008]. The properties of the composite are formed at the stage of processing where the material in a molten state is characterized by specific rheological properties which affect the accurate shape of the product. The selection of processing tools determines the level of distribution of the wood filler in the polymer matrix as well as its orientation, which in turn impacts the heat dissipation rate of the moulded product.

The main purpose of this study was to compare the reinforcing effect of two kinds of pine-wood particles, differing in particle length, on the mechanical properties of high-density polyethylene.

**Materials and methods/Research methodology**

The high-density polyethylene (PE-HD) used in this study was Hostalen GD 7255 (obtained from Basell Orlen Polyolefin, Poland) with a density of 0.955 g/cm$^3$ and a melt flow rate of 4 g/10 min (190°C/2.16 kg). Ethylene-propylene copolymer grafted maleic anhydride (PE-g-MAH) Fusabond P353 (DuPont) with a density of 0.904 g/cm$^3$, a melt flow rate of 470 g/10 min (190°C/2.16 kg), and a grafting degree of 1.4% wt was used as a coupling agent.

Two kinds of pine-wood particles were used as fillers: Lignocel C120 short wood fillers (SWF), and Lignocel C300 long wood fillers (LWF), (grades with a mean fibre length of 120 and 300 µm, for C120 and C300, respectively). The fillers from softwood, were supplied by J. Rettenmaier & Söhne GmbH, Rosenberg, Germany.

The WPCs were produced in a two-stage process. In the first stage, both wood particles were compounded into pellets at 20%, 40%, and 60% by weight with the PE-HD using a single-screw extruder. The barrel temperatures of the four zones ranged from 180°C to 200°C from the feeding to the die zone. The screw speed was 60 rpm. Following this, the pure PE-HD and composites were injection moulded into dumbbell-shaped specimens using an ENGEL 80/25 HL injection moulding machine, with 20 tons of clamping force. The specimens obtained were 4 mm thick and 10 mm wide. All the dimensions were in agreement with the ISO 527-2 standard. The compositions of the samples, including the percentage contribution of all the materials used, are listed in table 1.

The post-processing shrinkage was measured at room temperature using specimens with an initial width of 10 mm and 150 mm length, after cooling. The change in the linear dimensions of the injection moulded part $S_l = ((l_0 - l)/l_0) \cdot 100\%$ and width $S_w = ((w_0 - w)/w_0) \cdot 100\%$ were calculated; where $l_0$ and $w_0$ are the length
Table 1. Composite formulations

<table>
<thead>
<tr>
<th>Material code</th>
<th>Type of wood particles</th>
<th>Wood content [%wt]</th>
<th>PE-HD content</th>
<th>PE-g-MAH content</th>
</tr>
</thead>
<tbody>
<tr>
<td>PE-HD</td>
<td>--</td>
<td>--</td>
<td>100</td>
<td>--</td>
</tr>
<tr>
<td>PE/20SWF</td>
<td>small wood fillers</td>
<td>20</td>
<td>76</td>
<td>4</td>
</tr>
<tr>
<td>PE/40SWF</td>
<td>small wood fillers</td>
<td>40</td>
<td>57</td>
<td>3</td>
</tr>
<tr>
<td>PE/20LWF</td>
<td>long wood fillers</td>
<td>20</td>
<td>76</td>
<td>4</td>
</tr>
<tr>
<td>PE/40LWF</td>
<td>long wood fillers</td>
<td>40</td>
<td>57</td>
<td>3</td>
</tr>
<tr>
<td>PE/60LWF</td>
<td>long wood fillers</td>
<td>60</td>
<td>38</td>
<td>2</td>
</tr>
</tbody>
</table>

and width of the injection mould cavity, and $l$ and $w$ are the length and width of the specimen after cooling. The melt flow rate (MFR) was determined in a plastometer (model MP-IIRT-M, Russia) according to ISO 1133. The test conditions were set at a load of 2.16 kg and a temperature of 190°C for all the samples. Tensile tests were carried out using an Instron universal testing machine (model 4481, Canton, USA) at room temperature using a crosshead speed of 10 mm/min. The tensile tests were performed as per ISO 527-2. Brinell hardness tests were carried out using an HPK 8206 Brinell tester as per the standard PN-EN ISO 2039-1:2004P. During the tests, a ball indenter with a diameter of 5 mm and a test force of 49 N was used. The investigations of sample surface morphology were carried out and microphotographs were taken using a reflected-light microscope. The morphology was examined using a Nikon Eclipse E300 microscope.

Results and discussion

Processability properties

The melt flow rate and thermal shrinkage are presented in figures 1 and 2. The melt flow rate is inversely related to the sample viscosity and can be used to estimate the interaction between phases in the polymer composites. Figure 1. shows that the unfilled PE-HD had the highest MFR of all the tested materials. The incorporation of lignocellulose fillers led to a gradual decrease in the melt flow rate. This indicates that during injection moulding a very low flow length value could be expected, or higher injection pressure should be applied for thin-walled parts.

In both cases, the dimensional stability, evidenced by longitudinal and transverse shrinkage, decreased substantially with the pine-wood filler content compared to the unfilled PE-HD. The above findings provide important data concerning the dimensional correction of tools for the processing of these composites in comparison to the ones used for pure PE-HD.
Mechanical properties

The selected tensile and hardness properties of the pure matrix and its composites, obtained from the static tensile and Brinell hardness tests, are
presented in figure 3. For both wooden fillers, the Young’s modulus and tensile strength increased according to the composition, while the elongation at break decreased. The noteworthy improvement in stiffness was probably due to the high volume ratio of pine-wood particles in the polyethylene matrix, as well as in the filler anisotropy, especially for the LWF particles [Zhang et al. 2008; Kaseema et al. 2015]. On the other hand, a drastic limitation in the elongation values for the composites, in comparison to the unfilled PE-HD, is attributed to poor adhesion between the matrix and fillers, and the creation of small voids around the rigid filler particles. The large number of inclusions in the PE-HD matrix were potential nucleation sites, which contributed to a decrease in the plastic deformation of the composites.

![Graphs showing the effect of filler on the properties of PE-HD](image)

**Fig. 3.** Effect of filler on the Young’s modulus (a), Brinell hardness (b), tensile strength (c), and elongation at break (d) of PE-HD and its composites

**Surface morphology**

Figures 4a and b present the surface morphology of the sample filled with 40 wt.% of pine wood. The pine-wood particles, covered with the polymer layer, can be clearly seen. Therefore, the lignocellulose filler is probably protected
against moisture and little or no water absorption should take place during exposure to the weather.

![Fig. 4. View of the PE/40LWF composite surface, magnification 40×](image)

**Conclusions**

The application of pine-wood particles as a reinforcement for PE-HD, especially with a filler content of 40 wt.% or more, markedly reduced the flow ability of the PE-HD/pine-wood composites. This means that highly filled PE-HD composites should be used exclusively for thick walled products, such as hollow and siding profiles or garden furniture. Moreover, the composites revealed superior stiffness and markedly improved tensile strength, as well as hardness. Hence, these composites may be used as engineering materials with the potential to replace wood products. This is even more reliable, if one takes into account that the pine-wood particles in the composites were covered by a hydrophobic matrix. Therefore, no additional protection coating is needed for these materials.

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ISO 1133 [2011] Plastics – Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics


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Edyta URBANIAK-KONIK, Danuta KRÓL

WOOD WASTE AS COMPONENTS OF FUELS USED IN CEMENT PLANTS

This paper presents fuel molding technologies (PAS-i and PAS-r) with the participation of wood waste. These fuels are manufactured specifically for cement plants. The final product in the form of fuel derived from waste, meets the requirements of the recipient. In PAS-i fuel (Impregnated Solid Alternative Fuel), wood waste (sawdust) forms a matrix, to which slug and greasy waste materials are applied. Fuel PAS-r (Shredded Solid Alternative Fuel) is formed from combustible industrial and municipal waste which include wood waste groups. Fuel PAS-r is produced in the BMH installation. The presented tables include physical and chemical properties of PAS-r and PAS-i (fuels from waste) with respect to standards imposed by cement plants. Production of fuels from waste provides opportunities for managing types of wood waste that cannot be used otherwise.

Keywords: wood, waste, fuel from waste

Introduction

Increasing demand for consumer goods, results in an increase in the amount of waste. Storage must be the final link in the chain of rational waste management. Waste should be selectively collected and treated as a raw material – economically exploited. Recovery of valuable recyclables in terms of energy is a large part of waste recycling. These materials, as a result of processing, i.e. formation, receive a fuel nature with specific properties. A significant component of fuels formed from waste in terms of quantity is wood waste. Considering that a substantial part of waste originating from wood is contaminated (for example, with impregnating agents, lacquers, paints, oil derivatives. etc.), the appropriate way of dealing with such material is to utilize it as a constituent of waste derived fuels. The production of fuel from waste, therefore, enables managing the kinds of wood waste, which cannot be used otherwise. In Poland, waste derived fuels are used in cement plants as a substitute for conventional fuel. Rotary cement furnaces [Mokrzycki and Eliasz-Bocheńczyk 2004] burn for a considerably long time and with

Edyta URBANIAK-KONIK (edyta.konik@suez.com), SITA Poland, Warsaw, Poland; Danuta KRÓL (danuta.j.krol@polsl.pl), Silesian University of Technology, Gliwice, Poland
temperatures exceeding 1500°C, cause virtually all the components of waste derived fuels to be thermally destroyed. The ash obtained, accounts for a clinker additive. The production of cement clinker requires that the applied fuel is entirely combusted. Reaction of carbon and hydrogen oxidation proceeds correctly if the fuel is properly mixed with air and has a suitably high specific area. The fuel used must, therefore, be adequately crushed and mingled with other fuel types [Willitsch and Sturm 2003]. The annual use of waste derived fuels in the cement industry exceeds 1.2 million Mg and becomes a significant element of fuel economy in Poland. Within the European Union, waste derived fuels are increasingly applied in the energy sector [European Commission – Directorate General Environment 2003]. That is why the European Committee for Standardization (CEN) has introduced a system of classification and quality requirements for waste derived fuels (Solid Recovered Fuels – SRF) [Van Tubergen et al. 2005]. This system categorizes fuels formed from household waste as “solid fuel manufactured from other-than-hazardous waste” applied for energy recovery in combustion and co-combustion installations and fulfilling classifications and specifications given in CEN/TS 15359.

The aim of this work is to present the possibility of managing wood waste in the form of sawdust, chips, railway sleepers and others, also contaminated (often with hazardous substances). The indicated direction of such waste application (for energy recycling) as PAS-r and PAS-i components is to transform waste into a calibrated fuel with standardized properties, fulfilling specified technical rules, which guarantees its negotiability. Waste derived fuel, being a conventional fuel substitute, can be combusted or co-combusted with conventional fuels in various furnaces [Wei et al. 2009; Liu Ya. and Liu Yu. 2005], undergo gasification (forming combustible generator gas) [Arafat and Jijakli 2013, Zhou et.al. 2014] or pyrolysis [Velghea et al. 2011]. Utilizing waste derived fuels for energy recovery can, therefore, increase the share of renewable energy sources in fuel-energy balance.

**Waste derived fuels – PAS-r and PAS-i**

The production of fuels based on waste material, destined to substitute conventional, non-renewable fuels, is targeted at the overall decrease in energy demand throughout the economy by limiting the consumption of energy produced from conventional sources. Technologies for producing waste derived fuels are relatively energy-saving due to the simple technological systems being utilized.

The PAS-r fuel (from Polish „Paliwo Alternatywne Stałe rozdrobnione” – crushed Solid Alternative Fuel), based on household and industrial waste, is composed of various combustible materials, mixed in adequate proportions. Within the PAS-r fuel, waste of wooden origin can be found among others.
These include, for example bulk waste, in the form of furniture, wooden constructions, large used up wooden containers etc.

As for the PAS-i fuel (from Polish „Paliwo Alternatywne Stałe impregnowane” – Impregnated Solid Alternative Fuel), wooden waste, in the form of sawdust, form a matrix, into which various sludge-like and greasy materials are introduced. PAS-r and PAS-i fuels are produced by the SITA STAROL company from Chorzów, which belongs to the SITA SUEZ consortium. A list of types and amounts of wood based waste used in SITA STAROL to produce waste derived fuels in 2014 is shown in table 1. Each waste type has a designated code, according to the Waste Catalogue (Journal of Laws, No. 112, pos. 1206 dated September 27th 2001).

**Table 1. Types and amounts of wood based waste used in SITA STAROL in Chorzów to produce waste derived fuels in 2014**

<table>
<thead>
<tr>
<th>Waste code</th>
<th>Type of waste</th>
<th>Amount [Mg]</th>
</tr>
</thead>
<tbody>
<tr>
<td>03</td>
<td>Waste from wood processing and production of boards, furniture, cellulose mass, paper and cardboard</td>
<td></td>
</tr>
<tr>
<td>03 01 04*</td>
<td>contaminated chips</td>
<td>10.61</td>
</tr>
<tr>
<td>03 01 05</td>
<td>wooden waste</td>
<td>15.53</td>
</tr>
<tr>
<td>03 01 05</td>
<td>chips</td>
<td>0.50</td>
</tr>
<tr>
<td>03 01 99</td>
<td>wooden waste and plywood, contaminated with cardboard</td>
<td>355.30</td>
</tr>
<tr>
<td>03 01 99</td>
<td>sawdust</td>
<td>3967.95</td>
</tr>
<tr>
<td>12</td>
<td>Waste from forming and physical / mechanical treatment of metal and plastic surfaces</td>
<td></td>
</tr>
<tr>
<td>12 01 17</td>
<td>sawdust</td>
<td>1548.46</td>
</tr>
<tr>
<td>15</td>
<td>Container waste; sorbents, wiping textiles, filtration materials and safety clothes not mentioned in other groups</td>
<td></td>
</tr>
<tr>
<td>15 01 03</td>
<td>wooden containers</td>
<td>0.48</td>
</tr>
<tr>
<td>17</td>
<td>Waste from construction, repair and dismantling of buildings and road infrastructure (including soil and ground from contaminated areas)</td>
<td></td>
</tr>
<tr>
<td>17 02 01</td>
<td>demolition wood</td>
<td>1.69</td>
</tr>
<tr>
<td>17 02 04*</td>
<td>wood waste containing or contaminated with hazardous substances – railway sleepers</td>
<td>16.36</td>
</tr>
<tr>
<td>Sum</td>
<td></td>
<td>5916.449</td>
</tr>
</tbody>
</table>

*Indicates hazardous waste.

The primary type of wood waste applied in PAS-i fuel production is sawdust, constituting of a sorbent (40-50% of mass). Among wood waste, being a component of the PAS-r fuel, waste from the code 03 01 99 form the largest group. These are often contaminated with different materials such as cardboard, textiles, and waste from the furniture industry.
Raw material composition of a waste derived fuel must guarantee physical, chemical, fuel and emissive properties imposed and expected by the recipient (in this case, cement plants). The selection of waste types, therefore, and their mutual mass proportions is always preceded by the analysis of their fuel, physical and chemical parameters [Król 2013, Stowarzyszenie Producentów Cementu 2008]. The manufactured fuel undergoes quality control. As previously mentioned, the fuel has to fulfil standards with regards to its fuel, its physical and chemical properties, as well as the amount of heavy metals, imposed by the receiving cement plant [Marszałek Województwa Śląskiego 2010], for example:

- average calorific value of the product: 15 MJ/kg,
- ash content below 30%,
- humidity below 30%,
- bulk density within the range of 0.3 to 0.5 kg/m³,
- maximum sulphur content: 2.5%,
- chlorine and other halogens content: below 0.3%,
- heavy metals content: mercury – below 2 mg/kg of dry mass, chromium – below 400 mg/kg of dry mass, sum of cadmium, mercury and thallium – below 10 mg/kg of dry mass.

In the case of fuel bricking, various kinds of adhesives may need to be applied (which may also be industrial waste), because these types of fuels must possess a specified mechanical strength [Białecka 2006].

**PAS-i fuel formation**

Shredded wood derived waste, is the primary constituent of the PAS-i fuel (tab. 1). These form a matrix. The groundwork of sawdust in relation to sludge-like (for example, from cleaning tanks for oil derived substances and grinding sludges – below 5% in quantity), liquid and greasy waste being applied to it, functions as a sorbent. It is subsequently mixed with (i). shredded waste containers after paints, lacquers, oil emulsions and thinners as well as overdue chemicals; (ii). grinding dusts, polyethylene and polypropylene, as well as other waste containers, for example paper. Figure 1 depicts a block diagram of the technological process of PAS-i fuel production.

The unitary energy demand to produce 1 Mg of Impregnated Solid Alternative Fuel equals 10-16 kWh.

**PAS-r fuel formation**

The PAS-r fuel is manufactured using a Finnish installation provided by BMH Wood Technology (fig. 2), capable of processing 1200 Mg of waste daily.
Considering the composition variety of waste directed to the fuel production, a series of mechanical unit operations is required, such as:

- sorting,
- drying,
- shredding,
- metals separation,
- mineral fraction separation,
- packing.

Fig. 1. Block diagram of the PAS-i fuel production line
Fig. 2. BMH installation scheme

Waste processed into a formed fuel is delivered to the production plant by road transport, unloaded in the production hall, next to a step feeder, or outside the hall, from where they are loaded using a wheel loader on a step feeder (1) or directly to the crusher’s feed funnel. These are subsequently crushed in a Tyrannosaurus coarse crusher (2) into the maximum size of $80 \times 80$ mm. The remaining oversized material is directed to a separate bunker. Crushed waste gets transported by the belt conveyor (3) under a stationary magnetic separator (4), where ferrous metals of less than 80 mm in size are extracted. The following device is a fine fraction separator (5), which removes glass, sand, fine stones and biodegradable waste smaller than 12 mm. The primarily purified material (sized 12 to 80 mm) gets directed onto a vibrating feeder (6), which widens and unifies the waste stream, that first passes under an eddy-current separator (7), where non-ferrous metals are extracted (expected size of this fraction is 12-80 mm), and then falls into an air classifier (8), which separates the stream into light and heavy fractions. Heavy fraction is composed of sand, remainders of metals, glass, ceramics, wood, plastics, biodegradable substances and others. This fraction also has the size of 12-80 mm. The following conveyor directs the light fraction into two parallel Monster fine crushers (9) which shreds the material into the grain size of $30 \times 30$ mm maximum. The final product passes to an ascending conveyor (10) and a stacking conveyor (11), which puts the fuel into several storage bunkers. There is a by-pass within the fine crusher setup, which extracts the overflowed 80 $\times$ 80 mm fraction. The stacking conveyor is equipped with slide gates, which enable filling of a specified storage bunker.

The unitary energy demand to produce 1 Mg of the PAS-r fuel is estimated to be around 23 kWh, what is caused by the necessity of applying more energy – demanding shredding processes and full mechanization and automation of the installation.
Fuel, physical and chemical parameters of PAS-i and PAS-r fuels

The quality of the produced waste derived fuels with respect to fulfilling the plant standard as well as requirements imposed by recipients is assessed by the plant’s Quality Control Laboratory. Basic fuel parameters are measured: humidity, ash content, calorific value, sulphur and chlorine content within the combustible material. Additionally, the concentration of selected heavy metals and their sum is measured.

Example analysis results for two separate batches of PAS-i and PAS-r fuels are given in table 2.

Table 2. Fuel parameters of waste derived fuels and heavy metals content

<table>
<thead>
<tr>
<th>Property</th>
<th>unit</th>
<th>PAS-i</th>
<th>PAS-i</th>
<th>PAS-r</th>
<th>PAS-r</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture content</td>
<td>[%]</td>
<td>16.4</td>
<td>14.6</td>
<td>16.1</td>
<td>20.4</td>
</tr>
<tr>
<td>Ash</td>
<td>[%]</td>
<td>17.0</td>
<td>16.2</td>
<td>6.8</td>
<td>7.9</td>
</tr>
<tr>
<td>LHV [kJ/kg]</td>
<td></td>
<td>23949</td>
<td>24237</td>
<td>17668</td>
<td>16559</td>
</tr>
<tr>
<td>S</td>
<td>[%]</td>
<td>0.48</td>
<td>0.43</td>
<td>0.25</td>
<td>0.31</td>
</tr>
<tr>
<td>Cl</td>
<td>[%]</td>
<td>0.36</td>
<td>0.39</td>
<td>0.66</td>
<td>0.63</td>
</tr>
<tr>
<td>Ni</td>
<td>ppm</td>
<td>304</td>
<td>286</td>
<td>41</td>
<td>44</td>
</tr>
<tr>
<td>Pb</td>
<td>ppm</td>
<td>130</td>
<td>124</td>
<td>103</td>
<td>119</td>
</tr>
<tr>
<td>Cd</td>
<td>ppm</td>
<td>1.5</td>
<td>1.5</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>Cr</td>
<td>ppm</td>
<td>82</td>
<td>85</td>
<td>118</td>
<td>203</td>
</tr>
<tr>
<td>Cu</td>
<td>ppm</td>
<td>425</td>
<td>420</td>
<td>218</td>
<td>121</td>
</tr>
<tr>
<td>Mn</td>
<td>ppm</td>
<td>535</td>
<td>411</td>
<td>160</td>
<td>156</td>
</tr>
<tr>
<td>Co</td>
<td>ppm</td>
<td>11</td>
<td>21</td>
<td>14</td>
<td>27</td>
</tr>
<tr>
<td>Tl</td>
<td>ppm</td>
<td>&gt;5.0</td>
<td>&gt;5.0</td>
<td>&gt;5.0</td>
<td>&gt;5.0</td>
</tr>
<tr>
<td>V</td>
<td>ppm</td>
<td>5.4</td>
<td>5.0</td>
<td>6.0</td>
<td>6.0</td>
</tr>
<tr>
<td>Sb</td>
<td>ppm</td>
<td>68</td>
<td>76</td>
<td>35</td>
<td>41</td>
</tr>
<tr>
<td>As</td>
<td>ppm</td>
<td>0.6</td>
<td>1.4</td>
<td>5.0</td>
<td>1.5</td>
</tr>
<tr>
<td>Hg</td>
<td>ppm</td>
<td>1.4</td>
<td>0.8</td>
<td>0.4</td>
<td>0.4</td>
</tr>
<tr>
<td>Metals in total</td>
<td>ppm</td>
<td>1563.9-1568.9</td>
<td>1431.7-1436.7</td>
<td>704.4-709.4</td>
<td>723.9-728.9</td>
</tr>
</tbody>
</table>

Parameter values from table 2 show, with respect to standards imposed by cement plants (cited in point 2), that the final product – formed waste derived fuel – fulfils the recipient’s requirements.

Conclusions

Wooden waste of various forms, different granularity and homogenization, often contaminated with hazardous substances, may be applied in energy recovery, when used to produce waste derived fuels.

Even though these constitute a multi-material mixture and are considered “difficult” fuels, they still possess strictly calibrated properties. They also count as renewable fuels. When undergoing thermal processing, they can become an
energy source, part of which originates from a biodegradable fraction and, as such, is considered energy from a renewable source. There exists, therefore, a possibility to classify a share of the chemical energy that comes from biodegradable fractions’ within the chemical energy of the total waste mass.

The application of forest biomass in a fuel allows green certificates to be obtained. In Poland, according to the URE terminology (Energy Regulatory Office), the forest biomass covers: clean biomass from forests (usable wood), remainders from forest industry as well as from industries processing its products.

PAS-r and PAS-i fuels and their production technologies presented in this paper prove that in the case of waste with energy potential, the most suitable direction is energy recovery realised through fuel production.

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Dominika Janiszewska, Iwona Frąckowiak, Natalia Bielejewska

APPLICATION OF SELECTED AGENTS FOR WOOD LIQUEFACTION AND SOME PROPERTIES OF PARTICLEBOARDS PRODUCED WITH THE USE OF LIQUEFIED WOOD

This work presents the characteristics of wood liquefied using different types of solvents in terms of its application for binding particleboards. Standard pine particles from barked wood were used for the liquefaction experiments. The liquefaction reaction was carried out in high temperature conditions using a mixture of solvents from the polyhydroxy alcohol group, including glycerine, ethylene glycol, propylene glycol, diethylene glycol, and dipropylene glycol. The microstructure of both the liquefied wood and the liquefaction residues was determined by means of optical microscopy analysis. The basic parameters of the adhesive mixture modified with the liquefied wood, such as viscosity, pH and gel time were determined. Particleboards containing liquefied wood were produced. The following physicochemical and mechanical properties of the particleboards were measured: tensile strength, bending strength, modulus of elasticity, and formaldehyde content. The influence of the liquefying agent on the board properties was investigated. In all the tests, a control particleboard, bonded with a urea-formaldehyde adhesive resin with no inclusion of liquefied wood, was used for the purposes of comparison.

Keywords: liquefied wood, adhesive, particleboard properties, confocal microscopy

Introduction

Recently there has been growing interest in research on liquefied wood and its possible applications. Thus far, liquefied wood has been used, among other things, in the production of polyurethane foams [Ertas et al. 2014, Ćuk et al. 2015a] and coatings [Hrastnik et al. 2014, Cheumani-Yona et al. 2015], for the preparation of activated carbon fibres [Li and Ma 2013, Liu et al. 2015], as
a fuel [Seljak et al. 2012] and as a urea- and melamineureaformaldehyde resin substitute or modifier [Kunaver et al. 2010, Esteves et al. 2015, Janiszewska et al. 2016]. Recently Čuk et al. [2015b] carried out research which involved producing particleboards bonded with melamine-formaldehyde resin modified with liquefied wood. The research indicated that particleboards made with an adhesive mixture containing even 30% liquefied wood displayed the same desired parameters as a board made without the inclusion of liquefied wood. Other similar research has also indicated that liquefied wood affects the emission of formaldehyde from particleboards. Medved et al. [2009] and Kunaver et al. [2010] have proved that a 30% substitution of synthetic resin with liquefied wood can decrease formaldehyde emission by ca 40%. Of the best-known liquefying agents from the polyhydroxyl alcohols, the most used is a mixture of glycerine and diethylene glycol. In this study, different types of liquefying agents were used, including the less toxic propylene glycol, in order to investigate the influence of the tested liquefying agent on the properties of particleboards produced using liquefied wood. A decision was also taken to assess the suitability of laser scanning confocal microscopy, which as yet has not been fully described in the research on liquefied wood.

The aim of the research was to determine selected physicochemical properties, including confocal microscopy analysis of wood liquefied using different types of polyhydroxyl alcohol group solvents and liquefaction residues, as well as to investigate the standard properties of particleboards produced with the addition of liquefied wood.

**Materials and methods**

The raw wood material consisted of standard pine particles from barked wood. The particles were sorted using an Allgaier vibration screening machine with a set of screens with mesh diameters of 8, 2, 1 and 0.5 mm, respectively. Particles ≤1 mm and ≥0.5 mm were selected as the usable fraction for the liquefaction experiments. Before use, the particles were dried at 103°C for 24 h. The polyhydroxyl alcohol mixture of glycerine-G, ethylene glycol-EG, propylene glycol-PG, diethylene glycol-DEG, and dipropylene glycol-DPG were used as a liquefying agent. P-toluenesulfonic acid acted as a catalyst. The glycerol, ethylene glycol, propylene glycol, diethylene glycol, and 1,4-dioxane were obtained from Chempur, while the dipropylene glycol and p-toluenesulfonic acid (monohydrate) were acquired from Alfa Aesar. All the chemicals and solvents were of synthesis grade and were used without further purification. The mixture of glycerine and ethylene glycol G-EG (G-PG, G-DEG etc.) 1:1 by wt and p-toluenesulfonic acid (3% by wt for the liquefying agent) were placed into a 2000 cm³ three-necked glass reactor equipped with a mechanical stirrer. The mixture was heated to 130°C and was stirred constantly. Wood particles were then gradually added. The liquefaction reaction
was carried out for 2 h at 130-140°C. The mixture was diluted with a dioxane/water solution (4:1 v/v) after the reaction was finished. The product was separated from the solid residues by vacuum filtration. The residues were rinsed with the dioxane and oven dried at 103°C for 24 h. The water and dioxane were evaporated under reduced pressure.

The microscopic investigations were conducted in the Department of the Physics of Liquid Crystals at the Institute of Molecular Physics of the Polish Academy of Sciences. A drop of liquefied wood and dry liquefaction residues were put onto the microscopic slides. The microstructural analysis of the samples was performed using an Olympus BX53 polarizing microscope (PM) and an Olympus FluoView FV1200 IX83 laser scanning fluorescence confocal microscope (LSFCM). For all the studied samples, a magnification of 10× was used. As a source of coherent light, a diode laser with two excitation beams of 559 nm and 635 nm was used.

A mixture of an industrial urea-formaldehyde resin (80%) and liquefied wood (20% relative to the dry weight of the resin) was prepared to use as the binder. The industrial urea-formaldehyde (UF) glue resin was characterised by the following parameters: a gel time of 75 seconds, a viscosity of 336 mPa-s, a dry mass content of 69.4%, and a pH of 7.3. Urea-ammonium nitrate solution (46%) was used as a curing agent, constituting 1% of the dry weight of the resin. The viscosity, pH and gel time at 100°C characterising the adhesive mixture were determined according to the following standards: EN 12092:2004, EN 1245:2011 and PN-C-89352-3:1996. Each value is an average from three parallel experiment.

The particle fraction ≤8 mm and ≥1 mm was intended for particleboard production. In order to characterize the raw wood material, a determination of the fraction composition and a measurement analysis of the particles were carried out. After the sorting process, the determination of the fraction composition of the particles was carried out on the particles dried to a moisture content of approx. 8-10%. The test was conducted using a Fritsch screening machine. Approx. 200 g of raw material was taken randomly from a given particle portion. The mean share of the fraction was as follows: 4.00 mm – 4.6%, 2.00 mm – 18.3%, 1.00 mm – 49.4%, 0.50 mm – 25.6%, 0.25 mm – 1.6%, and <0.25 mm – 0.5%. Approx. 200 pieces of raw material were taken randomly from a given particle portion in order to undergo dimensional analysis. The average dimensions of the particles were: length 9.58 mm, width 1.82 mm, thickness 0.83 mm, slenderness coefficient 11.5 and flatness coefficient 2.2.

Single-layer particleboards measuring 510 × 510 × 10 mm with a 20% share of liquefied wood were produced. Pressing was performed at 200°C, at a unit pressure of 2.5 N/mm² reduced to 1 N/mm² after 41 s, to 0.5 N/mm² after 50 s and to 0 N/mm² at the end of the pressing process. The pressing time coefficient was 6.5 s/mm, while the resination rate was 10%. The nominal density of the panels was 650 kg/m³. The standard mechanical and physicochemical properties
of the particleboards were examined according to the standards: EN 323:1999, EN 310:1994, EN 319:1999, and EN 120:1994. Two parallel tests of one type of the board were carried out. A particleboard bonded with urea-formaldehyde adhesive resin without a share of liquefied wood (Control) was used for comparison purposes in all the tests. The boards were conditioned at 20°C and at 65% relative humidity.

**Results and discussion**

The optical microscopy studies conducted on the pine particles liquefied with the G-PG mixture (fig. 1a), as well as for the liquefaction residues (fig. 1b), showed differences in the images of the microstructures of the samples. The microstructure images of the wood liquefied with the aid of propylene glycol and registered by means of a confocal fluorescence microscope (fig. 2a) indicated the liquefaction of almost all the lignin and remaining of the cellulose in the residues. The structure of the liquefied wood was crosslinked and showed strong fluorescence in regions where no objects observed by optical microscope were present. In contrast, the sample of the residue showed poor luminescence in all the investigated regions, indicating a high cellulose content (fig. 2b).

![Fig. 1. Optical microscopy image of G-PG; a – liquefied wood, b – residue (886 × 670 µm), magnification 10×](image)

The physicochemical properties such as the viscosity, pH and gel time of the adhesive mixture based on liquefied wood are given in table 1.

The gel time of the adhesive mixture was in the range of 41-55 s for all the liquefying agents. The inclusion of liquefied wood in the adhesive mixture decreased the gel time by ca 30 s. Additionally, the pH value of the liquefied wood itself was between 2-3, which may increase the adhesive crosslinking process [Medved et al. 2009]. The greatest increase was observed in the viscosity of the prepared adhesive mixture G-DPG in comparison to the standard UF resin.
Fig. 2. LSFCM image of G-PG; a – liquefied wood (1276 × 1276 μm), b – residue (3140 × 3140 μm), magnification 10×

Table 1. Physicochemical properties of adhesive mixture based on liquefied wood

<table>
<thead>
<tr>
<th>Adhesive mixture modified with 20% liquefied wood</th>
<th>pH [-]</th>
<th>Viscosity [mPa·s]</th>
<th>Gel time [s]</th>
</tr>
</thead>
<tbody>
<tr>
<td>G-EG</td>
<td>5.6</td>
<td>299</td>
<td>49</td>
</tr>
<tr>
<td>G-PG</td>
<td>5.4</td>
<td>335</td>
<td>41</td>
</tr>
<tr>
<td>G-DEG</td>
<td>5.9</td>
<td>345</td>
<td>55</td>
</tr>
<tr>
<td>G-DPG</td>
<td>5.7</td>
<td>388</td>
<td>50</td>
</tr>
</tbody>
</table>

Gel time for adhesive mixture without liquefied wood: 75 s

The properties of wood-based panels based on liquefied wood are presented in table 2.

For all tested liquefying agents, it was observed that the tested particleboards displayed desirable properties in comparison to the control particleboard produced without a share of liquefied wood. The density profile of the standard board and the boards prepared with the use of liquefied wood was similar, therefore the influence of that parameter on the mechanical properties of the board was excluded. The research conducted showed that the replacement of urea-formaldehyde resin with 20% of liquefied wood did not cause an increase in the formaldehyde content.
Table 2. Properties of wood-based panels based on liquefied wood

<table>
<thead>
<tr>
<th>Tested property</th>
<th>Value</th>
<th>Measure unit</th>
<th>Board symbol</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Control</td>
</tr>
<tr>
<td>Tensile strength</td>
<td>x</td>
<td>N/mm²</td>
<td>0.86</td>
</tr>
<tr>
<td></td>
<td>s</td>
<td>N/mm²</td>
<td>0.06</td>
</tr>
<tr>
<td></td>
<td>v</td>
<td>%</td>
<td>7.0</td>
</tr>
<tr>
<td></td>
<td>n</td>
<td>pcs</td>
<td>8</td>
</tr>
<tr>
<td>Sample’s density for tensile strength test</td>
<td>x</td>
<td>kg/m³</td>
<td>654</td>
</tr>
<tr>
<td></td>
<td>s</td>
<td>kg/m³</td>
<td>14</td>
</tr>
<tr>
<td></td>
<td>v</td>
<td>%</td>
<td>2.1</td>
</tr>
<tr>
<td></td>
<td>n</td>
<td>pcs</td>
<td>8</td>
</tr>
<tr>
<td>Bending strength</td>
<td>x</td>
<td>N/mm²</td>
<td>15.6</td>
</tr>
<tr>
<td></td>
<td>s</td>
<td>N/mm²</td>
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<tr>
<td></td>
<td>v</td>
<td>%</td>
<td>18.6</td>
</tr>
<tr>
<td></td>
<td>n</td>
<td>pcs</td>
<td>8</td>
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<tr>
<td>Modulus of elasticity</td>
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<td>N/mm²</td>
<td>2663</td>
</tr>
<tr>
<td></td>
<td>s</td>
<td>N/mm²</td>
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</tr>
<tr>
<td></td>
<td>v</td>
<td>%</td>
<td>6.8</td>
</tr>
<tr>
<td></td>
<td>n</td>
<td>pcs</td>
<td>6</td>
</tr>
<tr>
<td>Sample’s density for bending strength and modulus of elasticity tests</td>
<td>x</td>
<td>kg/m³</td>
<td>642</td>
</tr>
<tr>
<td></td>
<td>s</td>
<td>kg/m³</td>
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<td>v</td>
<td>%</td>
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<td>6</td>
</tr>
<tr>
<td>Formaldehyde content</td>
<td></td>
<td>mg/100g dry board</td>
<td>8.1</td>
</tr>
<tr>
<td>Moisture content</td>
<td></td>
<td>%</td>
<td>5.4</td>
</tr>
</tbody>
</table>

x – mean value, s – standard deviation, v – coefficient of variation, n – number of samples taken for the test

Conclusions

The preliminary experimental results showed that the tested particleboards based on liquefied wood displayed mechanical properties which were comparable to the control board without liquefied wood, irrespective of the type of liquefying agent used. A slight decrease in the bending strength was observed when G-DPG
was used as the liquefying agent, but this parameter still fulfilled the demands of the European quality standard. In addition, the usefulness of laser scanning confocal microscopy in the research on liquefied wood was demonstrated. The microstructural image of the wood liquefied with the aid of glycerol and propylene glycol, registered by means of a confocal fluorescence microscope showed strong fluorescence, which indicated that almost all the lignin was liquefied and the cellulose remained in the residues.

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Li D-N., Ma X-J. [2013]: Preparation and characterization of activated carbon fibres from liquefied wood. Cellulose 20 [4]: 1649-1656


List of standards

EN 12092:2004 Adhesives. Determination of viscosity
EN 1245:2011 Adhesives. Determination of pH
PN-C-89352-3:1996 Kleje-Oznaczanie czasu żelowania (Adhesives. Determination of gel time)
EN 310:1994 Wood-based panels. Determination of modulus of elasticity in bending and of bending strength
EN 319:1999 Particleboards and fibreboards. Determination of tensile strength perpendicular to the plane of the board
EN 323:1999 Wood-based panels. Determination of density
EN 120:1994 Wood-based panels. Determination of formaldehyde content

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Anna KACZMAREK, Kazimierz A. ORLOWSKI, Lubomir JAVOREK

A BRIEF REVIEW AND COMPARISON OF SELECTED EXPERIMENTAL METHODS FOR MEASURING NATURAL FREQUENCIES OF CIRCULAR SAW BLADES

Different methods for the empirical determination of the natural frequencies of circular saw blades are presented. Stationary methods, such as the harmonic and impulse tests, are discussed and the results of related comparison are given. The comparison of the methods revealed their degree of practical usefulness and their accuracy in determining natural frequencies. A combination of specific methods is proposed, which should allow optimal results.

Keywords: circular saw blade, measurement methods, natural frequencies

Introduction

Knowledge of the critical rotational speeds of the circular saw blade in use might help the user avoid unstable cutting conditions, which could cause ‘snaking’ of the saw blade in the workpiece, and, as a result, inaccuracy in sawing. The fundamentals of critical rotational speed theory have been described in studies by Stakhiev [1970], Šteucêk [1971], Mote and Nieh [1973] and Strzelecki [1974]. The minimum critical rotational speed is a function of the natural frequencies of the circular saw blade [Strzelecki 1974; Schajer 1986, 1991; Nishio and Marui 1996; Stakhiev 1998]. Nevertheless, the majority of saw blade manufacturers mark their tools with the maximum allowed rotational speed for each saw, and the usual way to determine the maximum rotational speed of a saw is based on the value of the maximum rim speed. According to the literature, sawing speed should not exceed a value of 100 m/s [Li et al. 2000]. This kind of approach may sometimes give misleading information to users, since the actual permissible rotational speeds of some circular saw blades are below those recommended [Stakhiev 2004; Orlowski et al. 2007].

The critical rotational speed theory of circular saw blades has been the subject of many scientific publications. The natural frequencies or critical
rotational speeds of circular saw blades (clamped saws) have mainly been determined experimentally, e.g. Stakhiev [1970, 1998, 2000], Strzelecki [1974], Javorek and Sokołowski [2000], Veselý et al. [2012], and Kaczmarek et al. [2014].

Recently, Mohammadpanah and Hutton [2015a, b] reported on the analytically and empirically determined instability of guided splined circular saw blades, which rotated with speeds higher than their critical rotational speeds due to flutter (the phenomenon of self-excited vibration). Sawing kinematics with guided splined saws is widely used in the North American wood industry [Mohammadpanah and Hutton 2015b].

The optimal rotational speeds of circular saw blades might be defined empirically, even in sawmill conditions on the circular sawing machine equipped with an individual tool, if the methods presented in research by Orlowski and Hyvärinen M. [2007], and Sandak et al. [2007] are applied. It ought to be emphasized that in the above mentioned approach the behaviour of the circular saw blade is examined and the ranges of the lowest values of the blade’s lateral displacements are sought, which correspond to the largest value of dynamic stiffness. Nevertheless, in both cases, there is a need for a stepless driving system for the spindle (arbor) of the circular sawing machine. On the other hand, Finite Element Methods (FEM) have been applied to determine the natural frequencies of circular saw blades, and the results of these analyses have been reported by Gogu [1988], Nicoletti et al. [1996], Cristóvão et al. [2012], Droba et al. [2015], and Svoreň et al. [2015]. Tensioning circular saw blades is a way to increase the critical rotational speed [Schajer and Mote 1983; Schajer 1984, 1992; Schajer and Kishimoto 1996; Chabrier and Martin 1999; Stakhiev 1999, 2000; Cristóvão et al. 2012; Heisel et al. 2015]. Such a saw blade treatment may make it difficult to accurately model the examined circular saw blade. Furthermore, the use of FEM models for complex designs of circular saw blades without their empirical validation could complicate matters.

**Theoretical background**

The range of the permissible rotational speed of a circular saw blade is defined by the critical rotational speed of the tool. It is usually the maximum speed in which the circular saw blade can work with the required stability. The critical rotational speed could be determined with a knowledge of the values of the natural frequencies of the circular saw blade.

For circular saw blades, there exists a theory which states that the resonance phenomenon of circular plates is a result of the superposition of two component waves in which the first is travelling forwards and the second is travelling backwards [Stakhiev 1970; Schajer 1986; Nishio and Marui 1996]. The equations for determining these frequencies have been published in several studies e.g. [Stakhiev 1970; Schajer 1986; Nishio and Marui 1996, Droba et al.
2015]. When the rotational speed of the circular saw (clamped with collars) increases, at a certain rotational speed the frequency of the backward travelling wave becomes zero, which is called the critical (lowest) rotational speed, \( n_{cr} \) [Stakhiev 1970]. At this point the phenomenon of resonance occurs, and even a small lateral force can cause a large lateral deflection of the saw blade [Stakhiev 1970, 2004].

Orlowski et al. [2007] presented a simple measurement method for determining natural frequencies in the impact test (the impulse excitation test). This kind of test is useful for examining circular saw blades with more complex shapes (a large number of slots, unknown tensioning method, etc.). In the impulse test, the measurements of saw blade displacements may be taken with the use of an eddy current displacement sensor [Orlowski et al. 2007], a microphone [Cristóvão et al. 2012], an inductive displacement sensor [Kaczmarek et al. 2014] or a laser [Orlowski et al. 2007]. In the latter, the laser spot position seen by video camera changed according to the saw blade deflection; therefore, it was possible to analyse the amplitude of the circular saw vibrations.

The impulse method seems simple but at the same time very effective. However, if the circular saw blade design is more complex in shape, it may be difficult to gain a proper understanding of the natural frequencies from the Fast Fourier Transform (FFT) of the time course of the circular saw displacement signal [Kaczmarek et al. 2014]. Hence, in some cases the results obtained from the impact (impulse) test might be ambiguous. In such cases, the experiment should be supported by the harmonic test, despite it being extremely time-consuming. The harmonic method is based on the classic Chladni patterns method which allows identification of the modal shapes of the resonances of the plates [Šteuček 1971; Strzelecki 1974; Kaczmarek et al. 2014].

The aim of the paper is to present and compare the empirical results of determining the natural frequencies of a circular saw blade of complex design clamped with collars using both the impulse test and the harmonic test.

Materials and methods

Tool

In both experiments, the harmonic test and the impulse test for determining the natural frequencies of a brand-new circular saw blade (ASPI Tech) were examined. A Multix saw blade was examined with the following measurements: outside diameter \( D = 350 \) mm, hole diameter \( d = 30 \) mm, saw blade thickness \( a = 2.5 \) mm, teeth of cutting number \( z = 18 \) (not tipped with inserts), number of teeth throwing chips \( z’ = 16 \), collar diameter \( A = 90 \) mm, clamping ratio \( A/D = 0.26 \).
Harmonic test

Natural frequencies were empirically determined at the Technical University in Zvolen [Orłowski and Javorek 2009]. The tested saw blade was clamped with collars and sprinkled with semolina, which, for the specific exciting frequencies, created Chladni’s patterns corresponding to the modal shapes of resonances (natural frequencies). Then, the values of the natural frequencies were noted and pictures were taken of each of the patterns obtained.

Impulse test

The impulse test was conducted in a laboratory of the Technical University in Zvolen. The saw blade under examination was mounted using collars measuring \( A = 90 \text{ mm in diameter (clamping coefficient } A/D = 0.26) \). Then the non-rotating saw was excited by hitting it with a small hammer. The transverse displacements were measured using a contactless inductive displacement sensor (Balluff BAW M08EI-UAD15B-BP03) mounted close to the saw surface at the radius close to the gullets. The sampling frequency amounted to 3000 Hz, and the number of samples totalled 16384. The signals obtained were recorded in a form which made it possible to change them by FFT into an amplitude spectrum using software such as Labview (v.8) or AnalizaDAQ. In turn, the values of the natural frequencies of the tested circular saw blade were obtained from its amplitude spectrum [Orłowski et al. 2007].

Results and discussion

The natural frequencies of the examined circular saw blade obtained in both the harmonic \( f_h \) and impulse \( f_i \) tests, together with Chladni’s patterns corresponding to the modal shapes (nodal diameters) \( n = 1-5 \), are presented in table 1.

Determination of the natural frequencies of the circular saw on the basis of the FFT of the time data obtained in the impulse test are presented in figure 1. At first glance, without a knowledge of the values of the natural frequencies quantified in the harmonic test, determination of these frequencies from the FFT transform (fig. 1b) could prove difficult, and in reality ambiguous. Moreover, some frequencies, from the point of view of Chladni’s pattern occurrence, could be disregarded, since their amplitude values in the amplitude spectrum were rather low.

The nodal diameters of the same mode occurred for different values of frequencies. However, Chladni’s patterns (tab. 1) changed their position on the circular saw blades. It should be emphasized that the harmonic method provided proof of the phenomena of quasi-twin resonant frequencies (the same modes but different positions) [Kaczmarek et al. 2014]. This kind of phenomenon has not been observed for circular saw blades with simple designs. Furthermore, the FFT
spectra for the latter were straightforward and determining the natural frequencies was an easy task [Orlowski et al. 2007].

![Graph a) Blade displacement (V) vs Time (s)](image)

![Graph b) FFT (amplitude) (V) vs Frequency (Hz)](image)

**Fig. 1. Determination of the natural frequencies of the circular saw using the impact test: a – time domain data from inductive displacement sensor, b – FFT of the time data, (saw diameter $D = 350$ mm, hole diameter $d = 30$ mm, saw blade thickness $s = 2.5$ mm, clamping diameter $A = 90$ mm, $A/D = 0.26$)**

In the last column of table 1, the data showing the difference between the frequencies from both tests is given, calculated as follows:

$$\Delta f = |f_h - f_i|$$  \hspace{1cm} (1)

In general, the modulus of $\Delta f$ gradually increased with an increase in the mode number from 1.81 Hz to 12.04 Hz, with the exception of $n = 2'$, for which a minimum value was obtained.

In figure 2, the gaps between the quasi twin frequencies of the compared modal shapes (Chladni’s patterns) obtained in the harmonic test are presented. The value of the gap $g_n$ in Hz for the $n$ modal shape is given by:

$$g_{n-j} = f_h(n = j) - f_h(n' = j)$$  \hspace{1cm} (2)

where: $j = 2, 3, \ldots, 5$. It must be emphasized that for $n = 5$ the gap between the frequencies for the modal shapes $n'$ and $n''$ was also determined. An increase can be observed in the gap values for the modal shapes from $n = 2$ to $n = 5$, where
the maximum occurred. For the larger values of the frequencies of the modal shape \( n = 5' \) and \( n = 5'' \), an inverse phenomenon emerged (a decrease in gaps).

Table 1. Chladni’s patterns corresponding to the modal shapes, natural frequencies of the examined circular saw blade \( D = 350 \, \text{mm} \), \( d = 30 \, \text{mm} \), \( a = 2.5 \, \text{mm} \) (clamped with collars \( A = 90 \, \text{mm} \)) from the harmonic \( f_h \) and the impulse \( f_i \) tests

| Modal shape number \( n \) [-] | Natural frequency \( f_h \) [Hz] | Natural frequency \( f_i \) [Hz] | Modulus \( |\Delta f| \) [Hz] |
|-------------------------------|-------------------------------|-------------------------------|-----------------------------|
| 1                             | 123.26                        | 125.074                       | 1.81                        |
| 2                             | 165.82                        | 167.558                       | 1.74                        |
| 2’                            | 194.66                        | 194.477                       | 0.18                        |
| 3                             | 293.30                        | 289.610                       | 3.69                        |
| 4                             | 332.50                        | 329.440                       | 3.06                        |
| 4’                            | 444.00                        | 437.483                       | 6.52                        |
| 5                             | 548.40                        | 540.581                       | 7.82                        |
| 5’                            | 623.30                        | 616.395                       | 6.90                        |
| 5”                            | 799.40                        | 789.996                       | 9.40                        |
|                               | 846.90                        | 834.863                       | 12.04                       |

Fig. 2. The gaps between quasi twin (natural) frequencies of compared modal shapes (Chladni’s patterns) obtained in the harmonic test (saw diameter \( D = 350 \, \text{mm} \), hole diameter \( d = 30 \, \text{mm} \), saw blade thickness \( s = 2.5 \, \text{mm} \), clamping diameter \( A = 90 \, \text{mm} \), \( A/D = 0.26 \))

Conclusions

Although the harmonic test is time-consuming, it makes it possible to unambiguously determine the natural frequencies of a circular saw blade of complex design.
The analyses of the results obtained in the harmonic test revealed the existence of similar modal shapes (Chladni’s patterns) for different frequencies. Nevertheless, the registered shapes had dissimilar positions on the saw blade. For this reason they have been called quasi twin natural frequencies.

Gaps between quasi twin frequencies depend on the modal shape. An increase was observed in the gap values for the modal shapes from \( n = 2 \) to \( n = 5 \), where the maximum occurred. For the larger values of the frequencies of the modal shape \( n = 5' \) and \( n = 5'' \), an inverse phenomenon emerged.

The experiments carried out to determine the natural frequencies of a circular saw blade of complex design revealed the limited usefulness of the impulse test, since, in the FFT spectrum of the lateral saw blade displacements, natural frequencies appeared which were difficult to unequivocally identify. This was caused by the phenomenon of quasi twin resonant frequencies.

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Sławomir POSKROBKO, Danuta KRÓL, 
Aleksandra BORSUKIEWICZ GOZDUR

GASIFICATION OF WASTE WOOD BIOMASS

This paper presents the results of the gasification of sawmill waste – pine sawdust, deciduous sawdust, bark pine, wet sawdust and wood pellets. The moisture content of the waste in the test varied from 9 to 48%. The higher the moisture of biofuels, the lower the quality of the energy. This was confirmed by the results of the fuel property analysis, which are presented in the paper. The wood biomass gasification process was carried out in a compact bed gasifier with a power of 5 kW. The gasifying factor was the air fed to the bed through a grid sieve from the bottom of the gasifier. The research showed that the shares of flammable components in the resulting gas generator varied, resulting in differing calorific contents. Studies have shown that the gasification of wood biomass (even with a large moisture load, allows it to be transformed into a low-calorie fuel gas. The gasification of wood waste with a moisture content level of 9-11% only resulted in a larger share of combustible components in the gas generator. In the case of the pellets, the syngas contained 30% carbon monoxide, 12% methane and 8% hydrogen.

Keywords: wood waste, gasification, syngas

Introduction

Of the many types of biofuel, plant-based biomass is the most important, particularly forest biomass. However, this use of forest biomass should only apply to waste biomass. When such waste has a significant moisture load, it is classified as “difficult”.

Methods for the use of biomass for energy include, among others, direct combustion in boilers and co-combustion with coal. Some of the technologies used to convert biomass-based energy are gasification processes. They are the most technologically advanced. The agent used for biomass gasification is usually air. It creates a low or medium calorific generator gas, which can be used as a substrate for chemical syntheses, a fuel in gas-steam systems, motor fuel,
and as a reburning gas to reduce NOx in power boilers. During the gasification of biomass using air, a gas which comprises large amounts of nitrogen is obtained, which reduces the heat of the gas combustion to a value of 4-7 MJ/Nm³. Gasification using pure oxygen produces a gas with a calorific value of 10-12 MJ/Nm³. The use of steam as a gasifying agent increases the amount of hydrogen in the gas [Piskowska-Wasiak 2011]. The process achieves good results with improvements in the construction of the gasification chambers (furnaces) e.g.: gasification in chambers with a fluidized bed [Li et al. 2010], double-layer gasification [Wang et al. 2007], and gasification in a cyclone chamber [Guo et al. 2009]. The gasification of Brazilian Pinus elliottii in a downdraft gasifier with air as the gasification agent was studied by Mendiburu et al. [2014]. The input parameters considered were: (a) equivalence ratio (0.28-0.35); (b) moisture content (5-20%); (c) gasification time (30-120 min) and carbon conversion efficiency (80-100%). Gasification can become more efficient with the use of catalysts to increase the reactivity of the components involved in the process [Brown et al. 2008; Zhu et al. 2008]. The increased efficiency of the biomass gasification process can also be achieved by using various organic additives, among others, resin biomass pine [McKendry 2002]. The fuel additives used do not act as catalysts but as additional fuel substrate involved in the thermal change processes which occur in the reaction chamber of the thermal gasifier. Abroad biomass gasification technologies have been developing at a rapid pace due to constantly diminishing fossil fuel resources along with a constant increase in the demand for electricity. In contrast to the Polish market, gas generators are commercially produced overseas and occupy a stable position as heating elements [Kirkels and Verbong 2010]. In research by Gunarathne et al. [2014], bio-coal pellets were gasified in an updraft high-temperature agent gasification unit with air preheated to 900°C in order to study the performance of the air gasification of hydrothermal carbonized biomass. Through the process of biomass carbonization, the share of coal rose from 46 to 66% and the calorific value increased from 19 to 29 MJ/kg. The calorific value of the syngas reached 7.9 MJ/Nm³.

The aim of this study was to gasify various types of waste wood biomass in a laboratory gasifier with a capacity of 5 kW and with a compact bed, where the gasification agent was air. The process was conducted under atmospheric pressure. The gasification test results indicate the differences in the composition and calorific value of the gas produced from different types of wood biomass, with varying degrees of density and moisture load.

**Materials and methods**

The research material consisted of a forest waste wood biomass in the form of bark, sawdust and wood pellets: (i) pine sawdust, wood sawdust from deciduous trees (except oak), and pellets from coniferous tree sawdust coming
from forest within the Podlasie Province; (ii) mixed wet sawdust, bark pine, wood pellets made from mixed sawdust – coming from the TRAK Timber Production Plant in Garbatka Długa, in Mazowieckie province.

### Table 1. Fuel properties of wood waste

<table>
<thead>
<tr>
<th>Properties</th>
<th>Pine sawdust</th>
<th>Deciduous tree sawdust</th>
<th>Mixed wet sawdust</th>
<th>Coniferous tree pellets</th>
<th>Mixed wood pellets</th>
<th>Coniferous bark</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture content [%]</td>
<td>11.7</td>
<td>9.3</td>
<td>42.1</td>
<td>9.1</td>
<td>9.0</td>
<td>47.4</td>
</tr>
<tr>
<td>Flammable fraction* [%]</td>
<td>98.5</td>
<td>97.0</td>
<td>98.4</td>
<td>99.5</td>
<td>99.4</td>
<td>98.9</td>
</tr>
<tr>
<td>Ash* [%]</td>
<td>1.5</td>
<td>3.0</td>
<td>1.6</td>
<td>0.5</td>
<td>0.6</td>
<td>1.1</td>
</tr>
<tr>
<td>LHV* [kJ/kg]</td>
<td>19050</td>
<td>19101</td>
<td>17958</td>
<td>18151</td>
<td>18145</td>
<td>18680</td>
</tr>
<tr>
<td>LHV [kJ/kg]</td>
<td>17433</td>
<td>17542</td>
<td>8794</td>
<td>16170</td>
<td>16165</td>
<td>8031</td>
</tr>
<tr>
<td>Carbon** C [%]</td>
<td>48.54</td>
<td>51.70</td>
<td>45.07</td>
<td>49.1</td>
<td>48.93</td>
<td>47.86</td>
</tr>
<tr>
<td>Hydrogen** H[%]</td>
<td>6.10</td>
<td>6.10</td>
<td>6.26</td>
<td>6.32</td>
<td>6.48</td>
<td>6.16</td>
</tr>
<tr>
<td>Nitrogen** N[%]</td>
<td>0.32</td>
<td>0.21</td>
<td>1.44</td>
<td>0.20</td>
<td>0.93</td>
<td>1.27</td>
</tr>
<tr>
<td>Sulphur** S [%]</td>
<td>0.05</td>
<td>0.10</td>
<td>0.01</td>
<td>0.04</td>
<td>0.02</td>
<td>0.03</td>
</tr>
<tr>
<td>Chlorine** Cl[%]</td>
<td>0.11</td>
<td>0.10</td>
<td>0.16</td>
<td>0.07</td>
<td>0.01</td>
<td>0.12</td>
</tr>
<tr>
<td>Oxygen** O [%]</td>
<td>44.88</td>
<td>41.79</td>
<td>47.06</td>
<td>44.27</td>
<td>43.54</td>
<td>44.56</td>
</tr>
</tbody>
</table>

*Expressed on a dry free basis.

**Expressed on a dry ash-free basis.

Samples of the research material were collected according to the procedures used for sampling solids. After homogenization (in order to obtain representative samples), a fuel analysis was carried out. For this purpose, the following was determined: humidity, flammable and non-combustible parts, heat of combustion – in accordance with the PN-ISO standards [PN-Z-15008-02:1993; PN-ISO 1171:2002; PN-ISO 1928:2002] for the elemental composition of flammable substances - carbon, hydrogen, nitrogen, and sulphur by means of a Perkin Elmer 2400 series II CHNS elemental analyzer, and chlorine according to PN-ISO 587/2000. Mixed wet sawdust and coniferous tree bark are loaded with a substantial amount of moisture (42 and 47%, respectively). The combustion of these biofuels causes technical difficulties, and the fuel is known as “difficult”. In the gasification process, fuel moisture can be a factor advantageously influencing the process, however more than 40% of water in the fuel significantly decreases its calorific value. The ash content and elemental composition of the combustible substance of analyzed forest residue (sawdust, wood pellets, and bark) did not differ from the typical values for wood biomass. Fuels from waste pulp with the characteristics of the fuel shown in table 1 were converted into heat in the gasification process, in order to obtain combustible generator gas.
Fig 1. Laboratory test stand – gasifier

Gasification was carried out in a laboratory reactor with a fixed bed (fig. 1). The maximum heat output of the gasifier was 5 kW. The gasifying agent was air (0.56-0.93 g/s), which was fed to the bed through the sieve grate from below the gasifier. Fuel was supplied to a feeder and then transported to a reactor using a screw conveyor. The air was heated by a system of electric heaters. Self-ignition of the fuel layer was initiated by heating the reactor chamber with air to a temperature of 320°C. Gasification was carried out at 375-435°C – the temperature of the fuel layer. In contrast, above the bed, in the gas phase, the temperature was 870-940°C. At a temperature of 375-435°C, the autothermal process occurred in the fuel layer. The intensive evaporation of high-calorific condensing substances, including tar, took place. It is suggested that these high-calorific compounds were burned over the layer of the fuel, in the atmosphere (in the lower layer) of unreacted oxygen, which resulted in a temperature increase to the value T= ~940°C. The lack of high-calorific CnHm hydrocarbons in the generated gas indicated this. Once the temperature in the reaction chamber was stabilized, the gas was collected from the upper part of the chamber using an aspirator. Having passed through the scrubber system, the gas was transported to the analyzer. Measurement of the concentrations of gaseous products was carried out using a GAS 3000 synthesis gas analyzer.

The ash was removed during the process by blowing it out of the gasification chamber with a stream of air from the exhaust fan. The ash was deposited in the bottom part of the secondary combustion chamber. The resulting gas was burned in a secondary combustion chamber and before using the exhaust fan, the exhaust gases were cooled to a temperature of ca 180°C. For cooling, ambient air was sucked through the flue exhaust duct. The gas generator and channel air blower were equipped with an electrical heating system, which was used to
provide warmth to the reaction zone. The stabilization process was performed by regulating the flow of fuel and the gasification air flow.

**Results and discussion**

Gasification is a complex chemical process and the main reactions leading to the formation of gaseous flammable products are [Littlewood 1997]:

**Oxidation reactions:**

\[
\begin{align*}
C + \frac{1}{2} O_2 &= CO \quad \text{(exothermic reaction)} -110.6 \text{ kJ/mol} \\
C_nH_m + n/2 O_2 &= nCO + m/2 H_2 \quad \text{(exothermic reaction)}
\end{align*}
\]

**Boudouard reaction:**

\[
C + CO_2 = 2CO \quad \text{(endothermic reaction)} 172.6 \text{ kJ/mol}
\]

**Steam gasification:**

\[
\begin{align*}
C + H_2O_{(steam)} &= CO + H_2 \quad \text{(endothermic reaction)} 131.4 \text{ kJ/mol} \\
C_nH_m + nH_2O &= nCO + (n + m/2) H_2 \quad \text{(endothermic reaction)}
\end{align*}
\]

**Methanation reactions:**

\[
\begin{align*}
C + 2H_2 &= CH_4 \quad \text{(exothermic reaction)} -74.9 \text{ kJ/mol} \\
2C + 2H_2O &= CH_4 + CO_2 \quad \text{(exothermic reaction)} -15.3 \text{ kJ/mol} \\
CO + 3H_2 &= CH_4 + H_2O \quad \text{(exothermic reaction)} -201.9 \text{kJ/mol} \\
2CO + 2H_2 &= CH_4 + CO_2 \quad \text{(exothermic reaction)} -247 \text{kJ/mol}
\end{align*}
\]

The results of the gasification of the wood biomass in variable inlet air, refer to the composition of the produced generator gas. The change in the air flow in the conducted experimental studies was obtained by adjusting the air blow. Figs. 2a-2f show the volume fractions in % of the flammable components of the syngas: carbon monoxide CO, hydrogen H2, and methane CH4, as a function of the the oxygen content ratio \(O_2[%_{vol}]\) in the oxidizer (blast) and carbon C share (mass fraction) in the fuel. The quality (calorific value) of the generated gas is decided by the share of flammable components.

The calorific value of the generated gas was calculated using the following formula (its source is found in the instruction manual for the analyzer):

\[
\text{LHV}\text{syngas} = 126 \text{ [%CO]} + 108 \text{ [%H}_2\text{]} + 359 \text{ [%CH}_4\text{]} + 665 \text{ [%C}_n\text{H}_m\text{]} \text{ [kJ/Nm}^3\text{]}
\]

The greatest number of combustible components were found in the generated gases resulting from the gasification of the pellets - mixed and pine (figs. 2a and 2b): carbon monoxide CO at up to ca 28-30%, 11-14% methane CH4, and 5-6% hydrogen H2. This gas composition was reflected in the calorific
value (fig. 3) – 7600 to 8700 kJ/Nm³. The gasification of the sawdust regardless of its type (figs. 2c-2e) resulted in a gas production with a share of flammable components lower by more than half, and half the calorific value. A significant amount of methane in the gas (up to 14% when gasified pellets) was also observed. It should be noted that in the gasification of wood biomass in the compact fixed-bed gasifier, the share of methane in the synthesis gas is approx. 2-3% and the heating value of synthesis gas typically reaches a level of

![Graphs](https://via.placeholder.com/150)

**Fig. 2.** [% vol] CH₄, H₂, CO = f(O₂/C_fuel): a – mixed pellets, b – pine pellets, c – deciduous tree sawdust, d – pine sawdust, e – wet sawdust, f – coniferous bark
Fig. 3. The calorific value of gas generated from the gasification of wood

2000 kJ/Nm³ to 4000 kJ/Nm³ [Saravanakumar et al. 2007]. The bark of conifers (fig. 2f) proved to be the worst fuel for gasification. Several percentages of methane and carbon monoxide, and a fraction of hydrogen in the gas, resulted in a low calorific value of ca 1600 kJ/Nm³. Here, the process occurred at the highest values of O₂/Cₐₚ (>).1. Gasification of the mixed pellets and pine sawdust was carried out under conditions with the least oxygenation – O₂/Cₐₚ 0.001-0.008. Research on waste wood material gasification enabled the production of low-calorific gases (fig. 3).

Conclusions

1. Wood waste is a solid fuel which, during the gasification process, can be converted into a gaseous fuel.
2. The type of biomass determines the conduct of the process and affects the performance of the gasification products.
3. The concentrated wood waste (pellets) were characterized by the highest calorific value. In the gasification process in a compact fixed-bed gasifier, the heating value of the synthesis gas was 7600 and ~ 9000 kJ/Nm³, while the CH₄ content ranged from 11 to 14%. The gas obtained using this technology - due to its calorific value - may be used to drive gas turbines.
4. The gasification of poorly carbonized fuels (and wood waste is an example), characterized by a high moisture level (wet sawdust and coniferous bark), does not require the introduction of steam to the generator, however excess water often interferes with the process and needs to be evaporated off.
5. An increase in the methane and hydrogen content in the syngas is obtained using fluidized gasification technologies and dual-layer gasification technology, which are complicated from a construction point of view.
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Saravanakumar A., Haridasan T.M., Reed T.B., Bai K. [2007]: Experimental investigation of long stick wood gasification in a bottom lit updraft fixed bed gasifier, Fuel Processing Technology 88: 617-622


List of standards

PN-ISO 587/2000 Oznaczanie zawartości chloru z zastosowaniem mieszaniny Eschki (Determination of chlorine using Eschka mixture)

PN-ISO 1171:2002 Oznaczanie popiola (Determination of ash)

PN-ISO 1928:2002 Oznaczanie ciepła spalania metodą spalania w bombie kalorymetrycznej i obliczanie wartości opalowej (Determination of gross calorific value by the bomb calorimetric method, and calculation of calorific value)

PN-Z-5008-02:1993 Oznaczanie wilgotności całkowitej (Determination of moisture content)

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Tomasz ROGOZIŃSKI

WOOD DUST COLLECTION EFFICIENCY IN A PULSE-JET FABRIC FILTER

The performance of pulse-jet bag filters is characterised by two contradictory parameters: separation efficiency and pressure drop. Both parameters depend on the filtration time when the dust layer is created. The time between cleaning pulses – the filtration cycle – is also of great importance for the separation efficiency of the filter. This paper describes the results of experimental studies on the wood dust separation efficiency of a pilot scale pulse-jet filter during the initial phase of the process when the number of particles penetrating the filter visibly decreased.

Keywords: wood dust, filtration efficiency, pulse-jet filter

Introduction

The rational use of industrial pulse-jet filters is related to the need to balance two contradictory requirements. The aim is to achieve the highest possible separation (dust collection on the filter surface) efficiency while avoiding an excessive increase in pressure drop. During the filtration process carried out at constant parameters – filtration velocity and dust loading – separation efficiency and pressure drop depend on the properties of the dust layer formed on the surface of the filter material. Industrial filtration devices very often operate with a higher separation efficiency at the cost of increased airflow resistance [Mukhopadhyay 2009].

Total separation efficiency can be taken to evaluate the performance of the filter. However, if the particle size distribution of the dust flowing into the filter is known, more information can be obtained by calculating the fractional efficiency.

According to the theory described by Leith and Ellenbecker [1980], the mass of dust penetrating through the filter depends on the areal density of the dust collected on the filter surface, the filtration face velocity and the time between cleaning pulses (filter cycle duration). The characteristics of the collected dust and used filter medium also have an indirect impact. However, there are also

Tomasz ROGOZIŃSKI (trogoz@up.poznan.pl), Department of Furniture Design, Faculty of Wood Technology, Poznań University of Life Sciences, Poznań, Poland
a number of interactions between the accumulating dust and the surface of the filter material, which manifest in the variability of all kinds of factors taken into account in theoretical studies on filter performance. In practice, the properties of the dust and filter medium should not be regarded separately because during filter operation the dust creates a layer permanently associated with the medium structure. Therefore, all design activities concerned with the selection of the filter construction, the filter medium and the parameters of the filtration process must take into account the results of experimental studies on the relationship between the dust layer and the filter surface [Mračková et al. 2015].

Dolny [2005] observed the decrease in wood dust particle concentration during the filtration process. The number of particles decreased rapidly in the first phase of the process. The dust concentration in the air flowing out of the filter then stabilized in the subsequent cycles. However, thus far it is not known how this affects the fractional separation efficiency of the filter in terms of wood dust collection.

The aim of the study was to determine the separation efficiency of a non-woven polyester filter fabric in terms of wood dust collection during the initial phase of the filtration process, when the number of particles penetrating the filter rapidly decreases, and within a single selected filtration cycle of this phase, when the properties of the accumulated dust layer greatly change due to an increase in thickness.

Materials and methods

The experiments were carried out using a pilot-scale test bag filter. The operation of this testing device and the impressive results obtained with it were described in previous papers [Dolny 1998; Dolny and Rogoziński 2014]. The parameters of the experimental filtering process are given in table 1. The properties of the filter material, type KYS – PROGRES series, and the characteristics of the inlet dust were also described by Dolny and Rogoziński [2014].

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filtration velocity</td>
<td>0.077 m/s</td>
</tr>
<tr>
<td>Dust concentration at inlet</td>
<td>10 g/m³</td>
</tr>
<tr>
<td>Air pulse pressure</td>
<td>5 MPa</td>
</tr>
<tr>
<td>Duration of filtering cycle</td>
<td>5 min</td>
</tr>
</tbody>
</table>

The separation efficiency in the 5th and 45th filtration cycle was calculated based on the average mass concentrations of dust at both the inlet and outlet. However, the separation efficiency in the 35th cycle was calculated based on the same constant inlet mass concentration and the outlet concentrations obtained in
the first and fifth measurements during this cycle. The general formula is expressed as follows:

\[ \eta = \frac{C_1 - C_0}{C_1} \]

where: \( \eta \) – general separation efficiency,
\( C_1 \) – mass concentration of dust at the inlet of the filter,
\( C_0 \) – mass concentration of dust in the air at the outlet.

For the wood dust (polydisperse dust) used in the test, the fractional separation efficiency \( \eta_i \) of the filter was determined:

\[ \eta_i = f(d_i) \]

where: \( d_i \) – particle size of fraction \( i \).

Then the general separation efficiency is a sum of the efficiencies in particular size ranges:

\[ \eta = \sum q_i \eta_i \]

where: \( q_i \) – mass fraction of dust.

For this purpose the number concentration of the dust particles in the outlet was determined using a HR5250A laser particle counter. The measuring range of the counter included 8 channels with the upper dimensional limits 0.5, 1, 2, 3, 5, 10, 15, and 25 \( \mu m \). The air flow rate of the counter was 0.0283169 \( m^3/min \) (1 ft\(^3\)/min). A sample of cleaned air was taken from the outlet tube using the isokinetic probe. The air sample was then diluted with an isodilutor (dilution factor 1:10). The sampling time was set at 30 s, and the delay time between successive samples was also 30 s. The integral software of the counter calculated the results expressed as counts of particles in \( m^3 \) based on these assumptions. At these settings, five measurements of particle content were carried out in each filtration cycle and then the average values were calculated.

The mass concentration at the outlet of the filter was calculated using the data obtained by the particle counter as follows:

\[ C_0 = \sum_{i=1}^{8} n_{0i} \frac{\pi d_i^3}{6} \cdot \rho \]

where: \( n_{0i} \) – the number concentration of dust measured on channel \( i \) of the counter,
\( d_i \) – average size of channel \( i \),
\( \rho \) – density of wood substance 1500 kg m\(^{-3}\).

The mass concentration of dust particles at the filter inlet in the assumed channels was calculated on the basis of the empirical function of the particle size distribution of dust and the general dust concentration at inlet \( C_1 = 10 \, g/m^3 \). The particle-size distribution of the tested dust was determined using an Analysette 22 MicroTec Plus laser particle sizer. Then, based on this function obtained
during the particle size analysis by the sizer, the \( q \) fractions were calculated. Therefore, the mass concentration of particular dust fractions is as follows:

\[
C_i = C_i q_i
\]

Similar procedures for calculating the fractional efficiency of filter materials and separators were also described in detail by Maus and Umhauer [1996]; Warych [1998]; Simon et al. [2014].

**Results and discussion**

The particle size distribution of the beech wood dust used in the test was previously presented in the paper of Dolny and Rogoziński [2014]. The fractions of the inlet dust calculated in reference to assumed dimensional channels according to the empirical function of particle size distribution are shown in table 2.

**Table 2. Fractions of the inlet dust**

<table>
<thead>
<tr>
<th>Upper limit [( \mu m )]</th>
<th>0.5</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>5</th>
<th>10</th>
<th>15</th>
<th>25</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass fraction of the inlet dust ( q ) [%]</td>
<td>0.60469</td>
<td>0.0954</td>
<td>0.06238</td>
<td>0.02597</td>
<td>0.04274</td>
<td>0.58091</td>
<td>1.5075</td>
<td>5.27633</td>
</tr>
</tbody>
</table>

Figure 1. shows the average values of total particle number concentration in the air at the outlet recorded during all the filtration cycles of the experimental process. A gradual decrease in the concentration of particles during the conditioning of the filter material – the formation of a permanent dust layer on its surface – can be noticed. After the 50\(^{th}\) cycle, only a small decrease in the particle number in the cleaned air can be observed. This is an effect of the stabilization of the separation efficiency of the filter material in this phase of the process. Figure 2. presents the results of measurements of the particle concentration in the air at the outlet within the 35\(^{th}\) filtration cycle. These values were taken to calculate the separation efficiency in this cycle. An increase in the mass areal density of dust on the filter surface during the cycle caused a decrease in particle content in the air at the outlet.
Fig. 1. Total particle number concentration in air at outlet

Fig. 2. Particle concentration in air at outlet during the filtration cycle
Fig. 3. Separation efficiency in the filtration process

Fig. 4. Separation efficiency in the filtration cycle

The fractional filtration efficiency related to the average concentration of dust particles in the 5\textsuperscript{th} and 45\textsuperscript{th} filtration cycles is shown in figure 3. The results
shown in figure 4 refer to the fractional efficiency within the 35th cycle immediately after the cleaning pulse and just before the end of the cycle when the accumulated dust layer on the filter surface was the thickest. The curves of the fractional separation efficiency have a typical V shape. The lowest point of a curve represents the size of the most penetrating particles. The combination, interaction and simultaneous operation of the main capture mechanisms (diffusion and interception) are the weakest at this point. The total efficiency of the capture mechanisms depends on the particle, gas and fiber properties and is characteristic of particular filtration conditions. In addition, there were some problems with particle measurement, because the counters represented the results in the form of distribution according to equivalent diameter. However, wood dust particles are irregularly shaped and their length is often greater than other dimensions.

The increase in the separation efficiency shown in figure 3 refers mainly to particles measuring 2-3 μm. The number of these particles was mostly reduced under the influence of the conditioning of the filter material during the first phase of the process.

Changes in the dust layer characteristics are the reason for the increase in separation efficiency within the cycle shown in figure 4. This increase related to cycle time covered a wider dimensional range of dust particles. However, the results relating to the average dust concentration suggest that the described increase occurred in every filtration cycle. Nevertheless, this should be confirmed by more detailed research. These changes are related to particle puffs particularly described by Simon et al. [2014]. Sharp increases in downstream dust concentrations during and immediately after each cleaning pulse were observed in this work, but its authors did not calculate the separation efficiency during the cycles. Results obtained in different baghouse dust collectors at different operating parameters, filter and dust properties, cannot be directly compared but the changes observed in the fractional separation efficiency are a confirmation of the phenomenon of momentary increased particle penetration caused by the sudden impact of a pulse.

Conclusions

The results described in this paper illustrate the effect of the time of dust layer accumulation during the filtration process on the separation efficiency of the tested filter medium. In the initial phase, between the 5th and 45th cycles, this effect is most noticeable for particles measuring 2-3 μm, which are the most penetrating particles. Changes in the separation efficiency within a single filtration cycle occurred with a wider range of particle size. However, this was not transferred to the separation efficiency in the 5th and 45th cycles, which was related to the average particle concentration.
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Danuta Król, Aleksandra Borsukiewicz Gozdur, Sławomir Poskrobko

THE STUDY OF WASTE WOOD BIOMASS
AS A BIOFUEL IN THE CONTEXT OF BOILER
OPERATIONAL PROBLEMS – SLAGGING
AND HIGH-TEMPERATURE CORROSION

This paper presents the melting characteristics of ash from different residual forest biomass - wood pellets, oak chips, coniferous bark, deciduous tree bark, wet sawdust and non-sorted blueberries. The melting temperatures of ash in reducing and oxidizing atmospheres were similar and did not differ by more than 10°C to 30°C. These values were within a range of 1230°C to 1330°C, and for the blueberry it was more than 1500°C. Only ash pellets melted in a reducing atmosphere at a temperature of 1280°C, and in an oxidizing temperature at 1430°C. To compare, determination tests were also performed for ash from "agro" biomass waste. Ash melting temperatures for biomass composites of agricultural origin (made in an oxidizing atmosphere), turned out to be lower than the temperatures of ash from wood biomass. In the results of the presented elemental analysis, the share of chlorine and sulphur and the impact S/Cl on high temperature corrosion processes were indicated. On the basis of the results for all the tested types of biomass, the calculated ratio S/Cl was below the value of 2.0. This means that the tested biofuels do not meet the safe use of biofuels detailed in the energy sector criterium (from the point of view of boiler operation).

Keywords: wood waste, combustion, slagging, high-temperature corrosion

Introduction

The process of high-temperature thermal transition of fuel generates a solid mineral residue – ash. The amount of mineral substance in biomass is usually smaller than in coals. Although the amount of ash after the combustion of biofuel is smaller, its composition differs from the composition of the coal ash. This is the reason why boilers have many operational difficulties: on heat

Danuta Król (danuta.j.krol@polsl.pl), Silesian University of Technology, Gliwice, Poland; Aleksandra Borsukiewicz Gozdur (aborsukiewicz@zut.edu.pl), West Pomeranian University of Technology, Szczecin, Poland; Sławomir Poskrobko (drposkrobko@wp.pl), Białystok University of Technology, Białystok, Poland
transfer surfaces deposits are formed, followed by their agglomeration and the phenomenon of high-temperature corrosion occurs.

The paper [Vassilev et al. 2013a, b, c] presented a series of excellent reviews on the composition and application of biomass ashes and their behaviour during co-combustion. Analysis of different kinds of ash, from wood biomass (eucalyptus, rockrose, pine, olive tree pruning, cork, and poplar), agro-industrial biomasses (almond shells, olive stones and another two by-products of the olive oil extraction industry called ‘alperujo’) and herbaceous biomasses (rice straw, wheat straw, sunflower, thistle and brassica) were conducted by [Fernández Llorente and Carrasco García 2005, 2006]. To assess the degree of chloride corrosion risk based on the composition of the biomass, the fuel corrosion chloride rate (ICV) is used. It is set by determining the chlorine and sulphur content in biomass and potassium in the ash [Born 2005].

The small particle size of the fly ash produced from the combustion of biomass, means the biomass co-combustion with coal typically does not substantially increase the risk of erosion. If the biomass is contaminated with grains of sand, erosion may increase. With some simplification, it can be concluded, that the risk of slagging when burning a fuel depends on the characteristics of the melting ash, defined by three temperature values: softening – TD, melting – HT and flowing – FT. If, in case of biomass co-combustion with coal, the softening temperature of ash lowers (in comparison to the softening temperature of coal ash), this results in an increase in the speed of deposits build-up on boiler heating surfaces. In turn, this process causes a rise in the temperature of exhaust gas, and that reduces the gross efficiency of the boiler (compared to the efficiency obtained during the combustion of the coal). The study of ash changes, the building up of deposits on the heating surfaces during the combustion of wood and straw biomass and co-firing with coal was conducted by [Hansen et al. 1999; Robinson et al. 2002; Nordgren et al. 2013].

A relationship exists between the melting point of the ash and the content of chlorine, sulphur, silicon and alkali metals. Sticky fuel-derived ash may form in cases where fuel ash chemistry is governed by a high content of potassium and organically bound silicon, combined with a high content of chlorine and a low content of other ash-forming elements [Bartels et al. 2008; Brus et al. 2005]. Zheng et al. [2007] combusted bituminous coal, lignite coal and straw. They analysed fly ash, bottom ash and deposits, determining the concentration of Cl, S and K.

The aim of the study was to determine (in reducing and oxidizing atmospheres) the characteristic melting temperatures of ash from forest residues. To compare, determination tests were also performed for ash from “agro” biomass waste [Król 2013]. Biomass as a fuel in the power industry, creates many operational difficulties. In order to avoid adverse agglomeration of the bed during the combustion process, (the formation of residues on the heating surfaces) the HT temperature of the ash should be higher than the temperature in
the furnace during combustion of the fuel. The aim of the study of chlorine and sulphur loads was to assess the slagging factors and corrosion of chloride for plant, wood and agriculture biomass.

**Materials and methods**

The subject of the study was forest biomass and agricultural waste. Forest biomass, which consisted of: wood pellets, wet sawdust, oak chips, bark coniferous, deciduous tree bark and unsorted blueberry came from TRAK a Timber Production Plant in Garbatka Długa, in the Mazowieckie province. The waste material is agricultural straw and oat bran (derived from individual agricultural crops, from the area of the Podlasie province) and potato pulp (waste from the manufacture of starch and potato starch, came from the Food Industry Enterprises PEPEES SA in Łomża). With agricultural waste two fuels were produced:

1. twenty percent from potato pulp plus eighty percent from oat bran;
2. twenty percent from potato pulp plus eighty percent from oat straw.

Characteristic ash melting temperatures in reducing and oxidizing atmospheres were determined using microscopic-photographic methods, according to CEN / TS 15370-1: 2007. The concentration of chlorine in the biomass was determined in accordance with ISO 587/2000. Sulphur was measured using CHNS elemental analyzer model 2400 series II Perkin Elmer.

**Results and discussion**

The results of determining the melting temperature of ash from forest biomass are shown in table 1.

SST shrinking temperature, determined in a reducing atmosphere (tab. 2), ranged from 740°C (wood pellets), 790°C (oak chips), 840°C (coniferous shoulder) and 1250°C, 1260°C (wet sawdust, deciduous tree bark) to > 1500°C (unsorted blueberry). In contrast, the SST determined in an oxidizing atmosphere was the lowest for ash from pellets – 720°C, the highest (> 1500°C) for ash from the unsorted blueberry sample, and for other materials it the value was between 1200 and 1270°C.

The first signs of shaped ash softening were visible at the deformation temperature called softening temperature TD. In a reducing atmosphere (as in the case of the shrinking temperature) the lowest TD was recorded for ash originating from the wood pellets (820°C) and the highest for unsorted blueberry ash (> 1500°C). The TD for the remaining biomass was contained within a range of 1240°C to 1290°C. The oxidizing atmosphere changed the TD value a little, and so: for wood pellets it was 1420°C, deciduous tree bark 1310°C, wet sawdust 1290°C, coniferous bark 1280°C and oak chips 1230°C. For unsorted blueberry, the softening temperature (as all characteristic ash temperatures
determined) regardless of the atmosphere (oxidizing or reducing), was higher than 1500°C.

**Table 1. The characteristic melting temperatures of ash from forest residues**

<table>
<thead>
<tr>
<th>Type of forest biomass</th>
<th>Wood pellets</th>
<th>Wet sawdust</th>
<th>Oak chips</th>
<th>Coniferous bark</th>
<th>Deciduous tree bark</th>
<th>Unsoured blueberry</th>
</tr>
</thead>
<tbody>
<tr>
<td>SST°C, shrinking temperature atmosphere</td>
<td>reducing 740 720</td>
<td>reducing 1250 1270</td>
<td>reducing 790 1200</td>
<td>reducing 840 1260</td>
<td>reducing 1260 1260</td>
<td>reducing &gt;1500 &gt;1500</td>
</tr>
<tr>
<td>DTC, deformation temperature atmosphere</td>
<td>reducing 820 1420</td>
<td>reducing 1290 1290</td>
<td>reducing 1240 1230</td>
<td>reducing 1270 1280</td>
<td>reducing 1290 1310</td>
<td>reducing &gt;1500 &gt;1500</td>
</tr>
<tr>
<td>HT°C, hemisphere temperature atmosphere</td>
<td>reducing 1280 1430</td>
<td>reducing 1290 1300</td>
<td>reducing 1240 1230</td>
<td>reducing 1280 1290</td>
<td>reducing 1300 1330</td>
<td>reducing &gt;1500 &gt;1500</td>
</tr>
<tr>
<td>FT°C, flowing temperature atmosphere</td>
<td>reducing 1300 1440</td>
<td>reducing 1300 1310</td>
<td>reducing 1250 1240</td>
<td>reducing 1290 1300</td>
<td>reducing 1320 1340</td>
<td>reducing &gt;1500 &gt;1500</td>
</tr>
</tbody>
</table>

In the boiler, unfavourable phenomena such as slagging or fouling of the heating surfaces (which is associated with the ash change temperature) are the result of insufficient values for their melting points HT (temperature of the hemisphere). Having the HT temperature higher than the temperature in the furnace during combustion of the fuel is preferred. The melting temperatures of ash in reducing and oxidizing atmospheres were similar and did not differ by more than 10°C to 30°C. These were the values in the range, from 1230°C to 1330°C, for unsoured blueberry, it was more than 1500°C. Only ash pellets melted in a reducing atmosphere at a temperature of 1280°C, and in an oxidizing temperature at 1430°C. The ability of sticking (adhesion) did have ashes already in the temperature range between the softening point and melting point. FT temperature of ash flow is the one at which the ash melts completely.

Ash melting characteristics determined by their melting temperatures based on laboratory tests alone, do not give all the information regarding the nature of the threat of slagging on heat exchange surfaces. Ash melting temperatures indicated by the laboratory tests do not include boiler design and operating conditions, which has a major impact on fouling and slagging. In the laboratoty, melting temperatures are determined in a short time, heating up the ash with a several degree increase rate of the temperature per min., while deposits in the boiler accumulate over long periods and are subject to alternate heating and cooling processes in the environment of exhaust gases.
Melting temperatures of ash from biomass of agricultural origin composites (performed in an oxidizing atmosphere – table 2), proved to be lower than the temperatures of ash from wood biomass.

**Table 2. Characteristic ash melting temperatures (in an oxidizing atmosphere) of agricultural biomass rolniczej [Król 2013]**

<table>
<thead>
<tr>
<th>Type of “agro” biomass</th>
<th>Shrinking temperature, SST (°C)</th>
<th>Deformation temperature, DT (°C)</th>
<th>Hemisphere temperature, HT (°C)</th>
<th>Flowing temperature, FT (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>80% oat bran + 20% potato pulp</td>
<td>700</td>
<td>990</td>
<td>1110</td>
<td>1180</td>
</tr>
<tr>
<td>80% oat straw + 20% potato pulp</td>
<td>580</td>
<td>990</td>
<td>1080</td>
<td>1110</td>
</tr>
</tbody>
</table>

FT temperatures, in which the ash melted, varied from 1110°C to 1180°C. These values are about 100°C to more than 500°C lower, than the value corresponding to the temperature of ash forest waste. Ash from “agro” biomass softened at DT = 990°C. With respect to ash from the forest biomass, they were lower in range from 240°C to more than 500°C. They melted at a temperature of HT 1000°C. In case of loading the biomass with chlorine, alkali and heavy metals (when forming eutectics), melting of the ash is already observed at around 250°C [Ferrer et al. 2005].

The melting and slagging of biomass is dependent on the concentrations of F, Cl, S, Al and Si in ash [Arvelakis and Frandsen 2007]. Chlorine in plant biomass is present predominantly in the form of chlorides and, in small quantities organic bindings. The direct burning of plant biomass may cause (because of the participation of chlorine) the occurrence of high temperature corrosion, slagging and the pollution of the heating surfaces of boilers. It is generally accepted, that if the share of chlorine does not exceed 0.3 percent, then the combustible has a low (Cl < 0.2%) or medium tendency to slagging. In contrast, when it is larger than 0.3 percent, this tendency is significant, or very large if the proportion of Cl > 0.5 percent.

On the basis of determining chlorine and sulfur in biofuels (tab. 3), the classification according to their propensity to form deposits, and the risk of high temperature chloride corrosion was shown. The molar ratio of S/Cl can be used as an indicator for the high-temperature corrosion risk [Sommersacher et al. 2012].

Forest biomass and agricultural biomass composites are characterized by a low tendency to slagging (Cl < 0.2%), in contradiction to oak chips. In oak chips, the chlorine content is within a range of 0.3 < 0.26 > 0.2, and therefore, the tendency of slagging is at an average level. As mentioned above, chlorine is present in the biomass in the form of chlorides, the presence of which (especially potassium chloride) is important due to the high temperature corrosion. These
Table 3. The size of slagging and chloride corrosion factors for plant biomass

<table>
<thead>
<tr>
<th>Type of biomass</th>
<th>Chlorine Cl</th>
<th>Slagging tendency</th>
<th>Sulfur S</th>
<th>S/Cl</th>
<th>Chloride corrosion preponderance S/Cl &lt; 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>wood pellets</td>
<td>0.10</td>
<td>&lt;0.2% small</td>
<td>0.02</td>
<td>0.20</td>
<td>very high</td>
</tr>
<tr>
<td>wet sawdust</td>
<td>0.16</td>
<td>&lt;0.2% small</td>
<td>0.01</td>
<td>0.06</td>
<td>very high</td>
</tr>
<tr>
<td>oak chips</td>
<td>0.26</td>
<td>&lt;0.3% average</td>
<td>0.02</td>
<td>0.08</td>
<td>very high</td>
</tr>
<tr>
<td>coniferous bark</td>
<td>0.12</td>
<td>&lt;0.2% small</td>
<td>0.03</td>
<td>0.25</td>
<td>very high</td>
</tr>
<tr>
<td>deciduous tree bark</td>
<td>0.09</td>
<td>&lt;0.2% small</td>
<td>0.05</td>
<td>0.56</td>
<td>very high</td>
</tr>
<tr>
<td>unsorted blueberry</td>
<td>0.07</td>
<td>&lt;0.2% small</td>
<td>0.03</td>
<td>0.43</td>
<td>very high</td>
</tr>
<tr>
<td>oat bran + potato pulp</td>
<td>0.08</td>
<td>&lt;0.2% small</td>
<td>0.13</td>
<td>1.63</td>
<td>yes</td>
</tr>
<tr>
<td>oat straw + potato pulp</td>
<td>0.17</td>
<td>&lt;0.2% small</td>
<td>0.01</td>
<td>0.06</td>
<td>very high</td>
</tr>
</tbody>
</table>

chlorides, residing in sediments forming on the boiler heating surfaces, can cause intense chloride corrosion. The presence of sulfur in biofuel reduces the risk of chloride corrosion by replacing the chloride ion in potassium chlorides or sodium in sulfate ion, determining higher stability of the sulfates of chlorides. Salmenia [2000] reports that only when the state in which the ratio of the weight fractions of the S/Cl in the fuel falls below 2.0, is it dangerous. In [Aho and Ferrer 2005], this value is given as 2.2. From the results of the research presented in table 4 for all the examined types of biomass, the S/Cl is below the value of 2.0. This means that the tested biofuels do not meet the safe use of biofuels detailed in the energy sector criterium (from the point of view of the boiler operation).

Conclusions

In the processes of energy use of biomass waste, we can observe unfavourable events in the form of slagging or build-up of deposits on the heat transfer surfaces. This is related to the ash temperature changes, which are usually lower than the corresponding temperatures of the ashes from bituminous coals. This is particularly true for insufficient HT melting temperatures (hemisphere temperatures) which for carbon, is somewhere around 1500°C.

For the ash from wood waste, HT temperatures defined under reducing and oxidizing conditions, ranged from 1240°C to 1440°C. Only another forest biomass (unsorted blueberry), was characterized by a HT temperature value as in case of coal, i.e. more than 1500°C. In technical terms these values are acceptable.

Agriculture biomass, due to a very different quantitative composition of ash (in terms of low melting components) compared with the ash from coal, generates ash during combustion with lower values of temperature change. This was confirmed by studies of fuel formed from “agro” waste. Melting of ash was observed at 1000°C. These temperatures were lower than for wood ash, by several hundred degrees Celsius. Typical agricultural biomass, however, for
example in the form of wheat straw, is characterized by significantly lower ash melting temperatures, around 772°C. In practice, if agro biomass is combusted, the combustion temperature is allowed to be at 700°C to 750°C. Under these thermal conditions, ash rich in alkali chlorides does not readily lend itself to the softening process, thus preventing corrosion and slagging of boilers. All tested biomass materials were characterized by a low tendency to slagging (Cl < 0.2%).

The sulphur and chlorine introduced into the furnace boiler with fuel, especially such as straw or wood, play a key role both in corrosion processes, which, in the high temperature atmosphere of the steam boiler constitute a threat to its structural steel materials, screens, superheater or evaporator and in staining (fly ash) and the slag on heat transfer surfaces. The criterion of safety for boiler corrosion processes is the ratio of sulfur to chlorine S/Cl 0> 2. The biofuels tested do not meet the safe use in energy industry criterium (from the point of view of the boiler operation). The S/Cl is less than two.

Plant biomass fuel is such a difficult fuel, because its combustion or co-combustion with coal requires the prevention of these undesirable processes, which significantly hinders ensuring trouble-free operation of boilers.

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