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RESEARCH PAPERS

Jadwiga ZABIELSKA-MATEJUK, Anna STANGIERSKA,
Elżbieta GRABIŃSKA-SOTA, Katarzyna CZACZYK,
Agnieszka DROŻDZYŃSKA

BIODEGRADATION OF NEW IONIC LIQUID-BASED
WOOD PRESERVATIVES IN SOIL AND WATER
ENVIRONMENTS

The ability of structurally different ionic liquids with cocotrimethylammonium,
didecyldimethyloammonium and bis-dimethyloctylammonium cations to degrade in water and soil environments were investigated. The test in water was conducted according to OECD guidelines 301 A and 302 B. Secondary effluent from a domestic sewage treatment plant in Zabrze, Poland was used as the inoculum. The ready biodegradability of the test compounds was monitored via DOC (dissolved organic carbon) for 28 days. Biodegradation by moulds in soil were carried out on Scots pine Pinus sylvestris L. using the soil-block method. The two-phase titration and HPLC chromatography methods were used to detect and determine the metabolites of the ionic liquids. All the tested compounds showed ready biodegradability in the water environment, as well as decomposition by the test fungi species in the treated wood exposed in the soil. The degree of decomposition of the ionic liquids by moulds during the 12-week test was dependent on their structure, reaching a maximum of 55% for the cocotrimethylammonium nitrate and 52% for the bis-dimethyloctylammonium nitrate.

**Keywords:** ionic liquids, biodegradation, moulds, water, soil, HPLC
Introduction

The use of impregnated wood in conditions where biotic and abiotic factors are involved is linked to the risk of migration and leaching of the components of chemical preparations into the environment, leading to the accumulation of toxic substances in the natural environment. The spectrum of biocidal action, the effectiveness of the protection of the lignocellulosic material, as well as possibilities of binding active ingredients with wood chemical substances, are among the main considerations when developing new wood protection preparations. However, overlooking the biodegradation problem of active substances during exploitation and, subsequently, in the course of utilisation, may have a detrimental impact on the environment. That is why chemical compounds, widely considered environmentally-friendly, have attracted the interest of scientists involved in research on wood protection against microbiological degradation. Ionic liquids (ILs), also referred to as green solvents and obtained from quaternary ammonium salts, are employed with increasing frequency in industry due to their softening, anti-electrostatic as well as bacterio- and fungicidal properties. The multifunctionality (the multi-task nature) of these organic compounds is the result of their ionic chemical structure and, practically speaking, their unrestricted synthetic possibilities. Growing interest in the investigation of ionic liquids has resulted in the development of manufacturing technologies for these compounds, characterised by novel functional properties, including technologies for application in wood protection [Takahashi et al. 1993; Thang and Ruddick 2000; Kartal et al. 2005; Pernak et al. 2006]. The toxicity of new ionic liquids has been thoroughly described in the literature [Pretti et al. 2005; Pernak et al. 2006; Garcia-Lorenzo et al. 2008; Latała et al. 2009, 2010; Grabińska-Sota 2010; Petrović et al. 2011; Zhang et al. 2011]. Ecotoxicity studies conducted by Grabińska-Sota [2011], as well as investigations on mutagenicity, revealed that ammonium ionic liquids were not genotoxic and mutagenic, which is an important consideration from the point of view of ecological safety as well as protection of human health. The predicted possibility of the bio-elimination of ionic liquids in an aqueous environment has only been confirmed in a small number of investigations [Harjani et al. 2009; Ford et al. 2010]. Earlier studies on the biodegradation of quaternary ammonium chlorides by bacteria demonstrated some influence of the cation structure, especially of the quantity of long-chain alkyl substituents, on this process [Van Ginkel and Kolvenbach 1991; Van Ginkel et al. 1992; Doyle and Ruddick 1993; Nishiyama et al. 1995; Nashihara et al. 2000; Patrauchan and Oriel 2003]. Likewise, in investigations conducted by Bürgel et al. [1996], Zheng and Ruddick [1995] as well as Zabielska-Matejuk and Czaczyk [2006], possibilities for the degradation of quaternary ammonium chlorides, acetates and propionates by some species of mould fungi were reported.
Biological investigations of the structures of newly developed ionic liquids with a cation of natural origin obtained from plant fats, especially from coconut oil, and containing a mixture of alkyl substituents of C$_8$, C$_{10}$, C$_{12}$ and C$_{14}$ length and a nitrate or nitrite anion, revealed the high effectiveness of their action against fungi of brown and white degradation, as well as against fungi causing blue stained wood [Zabielska-Matejuk and Pernak 2009]. The fungicidal values of these ionic liquids with respect to Coniophora puteana ranged from 2.7 to 4.3 kg/m$^3$. In the case of bis-quaternary ammonium-based ionic liquids, N,N-dimethyloctylamine derivatives with a nitrate anion, the effectiveness of the protective action was higher and ranged from 1.8 to 2.9 kg/m$^3$.

This study presents research results concerning the sensitivity of new ionic liquid structures in aqueous solutions to biodegradation on the basis of OECD recommendations in accordance with the 92/32EEC directive [1992], as well as the migration and degradation of these compounds in treated wood by mould fungi in the soil environment. Quantities of degradation products were determined using high performance liquid chromatography (HPLC). Investigations were carried out on the above-mentioned ionic liquids with a cation obtained from: coconut oil, with a didecylidimethyl ammonium cation, as well as with a “gemini” ammonium cation containing two alkyl substituents with a chain length of C$_8$. Sodium benzoate was used as a reference substance in the biodegradation experiments performed in an aqueous environment.

Materials and methods

Chemicals

Five ammonium ionic liquids with nitrite or nitrate anions and different cation structures, tj didecylidimethylammonium, cocotrimethylammonium and N,N’-[1,10-(2,9-dioxadecane)] bis(dimethyloctylammonium) cations were selected for the biodegradation study. The structure of the examined compounds are presented in scheme 1. All the chemicals were prepared at the Faculty of Chemical Technology of Poznań University of Technology, while all the reagents for synthesis were purchased from commercial sources, Sigma-Aldrich and Akzo-Nobel. The chemical purity of the ILs was determined by a direct two-phase titration procedure [PN-EN ISO 2871-2:2000].

The prepared ILs were characterized by their $^1$H NMR and $^{13}$C NMR spectra. $^1$H NMR spectra were recorded on a Varian Model XL 300 spectrometer at 300 MHz with tetramethylsilane as the standard. $^{13}$C NMR spectra were recorded on the same instrument at 75 MHz. NMR spectra and elemental analyses were performed at A. Mickiewicz University, Poznań.
Table 1. The studied ionic liquids

<table>
<thead>
<tr>
<th>ILs</th>
<th>Purity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>[DDA][NO₃]</td>
<td>Didecyldimethylammonium nitrate</td>
</tr>
<tr>
<td>[DDA][NO₂]</td>
<td>Didecyldimethylammonium nitrite</td>
</tr>
<tr>
<td>[ArqC₃₅][NO₃]</td>
<td>Cocotrimethylammonium nitrate</td>
</tr>
<tr>
<td>[ArqC₃₅][NO₂]</td>
<td>Cocotrimethylammonium nitrite</td>
</tr>
<tr>
<td>[bis(AmC₄)][NO₃]</td>
<td>N,N’-[1,10-(2,9-dioxadecane)]bis[dimethyloctylammonium nitrate]</td>
</tr>
</tbody>
</table>

*Applied in ACQ preservative system (the amount of bis-IL in the preparation – 10% m/m)

Didecyldimethylammonium nitrate [DDA][NO₃]: $^1$H NMR (DMSO-$d_6$) δ ppm = 3.24 (m, 4H), 2.99 (s, 6H), 1.62 (m, 4H), 1.26(m, 28H), 0.86 (t, J = 7 Hz, 6H); $^{13}$C NMR δ ppm = 62.7, 49.9, 31.2, 28.8, 28.7, 28.6, 25.7, 22.0, 21.6, 13.9. Elemental analysis: Found: C 68.12 H 12.71 N 7.03. Calc. for C$_{22}$ H$_{48}$ N$_2$O$_3$ (388.6): C67.99, H 12.45, N 7.21%

N,N’-[1,10-(2,9-dioxadecane)]bis[dimethyloctylammonium nitrate]: $^1$H NMR (CDCl$_3$) δ ppm =5.14 (s, 2H), 3.94 (m, 2H), 3.55 (m, 4H), 3.29 (s, 12H), 1.84( m, 2H), 1.39 (m, 28H), 0.9 (t, J = 7 Hz, 6H); $^{13}$C NMR (CDCl$_3$) δ ppm = 90.2, 73.2, 60.9, 47.1, 31.4, 29.0, 28.8, 28.7, 26.2, 24.7, 22.3, 22.1, 13.8.

The ionic liquids under investigation were thermally stable. The decomposition temperatures of the [NO₃]$^-$-based compounds ranged between 215 and 230°C, and for [DDA][NO₂] they were between 160 and 190°C. Thermal analysis was performed by simultaneous thermal gravimetric and differential thermal analysis (TG/DTA) measurement using a Setsys 12 Setaram thermobalance, under air atmosphere.
Microorganisms

Fungal strains

Gliocladium roseum (Bainier) species No, 62726, obtained from Deutsche Sammlung von Mikroorganismen and Zellkulturen GmbH in Braunschweig, were used in the biodegradation studies of the ionic liquids by mould fungi in a soil environment, as well as mixtures of the following mould: Penicillium brevicompactum (Dierckx), Penicillium funiculosum (Thom), Phialophora fastigiata (Lagerb. & Melin) Verticillium lecani (Zimm.) Viégas, and G. roseum. The species were obtained from the collection at the Institute of Wood Technology, Poznań, Poland.

Methods

Biodegradation in a water environment

The tests for the susceptibility of the ionic liquids to biodegradation were performed in two stages (ready biodegradability and inherent biodegradability). The ready biodegradability test was conducted according to OECD guideline 301 A. The ready biodegradability of the test compounds was monitored via DOC (dissolved organic carbon) for 28 days. Secondary effluent from a domestic sewage treatment plant in Zabrze, Poland was used as the inoculum.

A measured volume of inoculated mineral medium, containing a concentration of the test substance 40 mg DOC/L as the nominal sole source of organic carbon, was aerated in diffuse light at 22±2°C. The mineral medium was composed of 8.5 mg L⁻¹ KH₂PO₄, 21.75 mg L⁻¹ K₂HPO₄, 33.4 mg L⁻¹ Na₂HPO₄·2H₂O, 0.5 mg L⁻¹ NH₄Cl, 27.5 mg L⁻¹ CaCl₂, 22.5 mg L⁻¹ MgSO₄·7H₂O, 0.25 mg L⁻¹ FeCl₃·6H₂O (pH 7.4). The degree of biodegradation was calculated by expressing the concentration of DOC removed (corrected by the concentration in the blank inoculum control) as a percentage of the concentration initially present. As a reference compound, sodium benzoate was used.

The inherent biodegradability tested whether the test compound complied with the condition of “ready biodegradability”, i.e. that it was not removed in 70% (DOC) in a 28 day time period. At this level the test was conducted according to OECD guideline 302 B (Zahn-Wellens method) for assessing inherent biodegradability. The principle of the test was to aerate the mixture containing the tested compound (50 mgDOC/L), culture medium and relatively high amounts of activated sludge in the water medium for 28 days at a temperature of 20-25°C.

The test for the blank probe containing activated sludge and culture medium without the tested compound, while testing of sodium benzoate as the reference substance was conducted simultaneously. The biodegradation process was monitored viaDOC measurement in the filtered probes collected at suitable time intervals. The ratio of the vanishing to primary DOC value, corrected by the value of the blank probe for the collected samples, was stated as the biodegradation percentage.
Soil-block test – biodegradation in a soil environment
A sample of Scots pine sapwood *Pinus sylvestris* L., measuring 22 × 17 × 12 mm (L × T × R) and saturated under vacuum conditions with studied ionic liquids with concentrations of 0.63 and 2.5%, was used for the degradation study of ionic liquids by moulds in treated wood. Twenty-four samples were saturated for each concentration of ionic liquid. After three weeks of conditioning in a closed container, as well as seasoning and sterilization by overheated steam, the wood samples were exposed to mould fungi for 12 weeks in a soil-block test. Garden substrate containing biohumus with a pH of 5.5 and relative humidity of 62% was applied. The procedure of the soil-block test were described in the earlier work of Zabielska-Matejuk and Czaczyk [2006] The quantity of ILs in the treated wood was determined, while at the same time a direct two-phase titration procedure [PN-EN ISO2871-2:2000] was carried out using sodium lauryl sulphate as the titrant, and a HPLC procedure [AWPA Standard A 16-93: 1993] was conducted. The ionic liquids were extracted from refined wood (particle size φ < 0.5 mm) using an ultrasound bath for three hours at 35 kHz frequency.

HPLC analysis
Determination of the ionic liquids was carried out on an Agilent Technologies 1200 series system consisting of an autosampler (model G1329B), a pump (model G1312B) and a diode array detector (model G1315C) set at 262 nm. The analysis were performed on a Waters SCX Ion-Exchange Column (100 × 8 mm). Acetonitrile and water (70:30 for [DDA][NO₃], [DDA][NO₂] and 75:25 for [ArqC₃₅][NO₃]) acidified with 1.2% acetic acid and containing 0.8 g/l benzyltrimethylammonium chloride were used as the eluent [Bürgel et al. 1996]. The flow rate was 1.5 mL/min, and the injection volume – 60 μL. Standards were used to identify peaks in the chromatograms, and a peak area was used to determine the sample concentrations. This was carried out by computer integration (ChemStation for LC 3D Systems, Agilent) operated in external standard mode. The method was based upon the indirect UV analysis of the mobile phase after passing through the column (added benzyltrimethylammonium chloride was used to give a positive background). The chromatographic peaks appeared negative and their integration was made after computer transformation into positive.

Results and discussion

Biodegradation in a water environment
The results of the first phase of ready biodegradation testing showed that there was 69.54% removal of the didecyl dimethylammonium nitrite ([DDA][NO₃] 99%) within 28 days. The reduction percentage of the compound marked as DOC was close to the required value of the temporary level for significant ready
biodegradability of approx. 70%. This level was achieved within a 10 day period from the first day of the test, hence the compound was classified as readily biodegradable. However, two other ionic liquids did not comply to this condition, since 44.16% of [ArqC\textsubscript{35}][NO\textsubscript{3}] 97% was removed on the 28\textsuperscript{th} day of the ready biodegradability test, and only 8.62% of [ArqC\textsubscript{35}][NO\textsubscript{3}] 75% (fig.1). In this situation, according to OECD guideline 302 B (Zahl-Wellens method), the two liquids underwent further testing to confirm these results. The results of the second phase of the biodegradation tests are presented in figures 2 and 3. The reference substance was completely biodegraded in 14 days from the beginning of the test, however the biological decomposition of ArqC\textsubscript{35}[NO\textsubscript{2}] 75% in the test for inherent biodegradation amounted to 93% in 28 days. [ArqC\textsubscript{35}][NO\textsubscript{3}] 97% underwent biodegradation in a similar manner. In the test for actual biodegradability, 96% was removed within 28 days.

Based on the tests performed, it was demonstrated that 99% of the didecyldimethylammonium nitrite ([DDA][NO\textsubscript{2}]) ionic liquid was readily biodegradable, however the [ArqC\textsubscript{35}][NO\textsubscript{2}] 75% and [ArqC\textsubscript{35}][NO\textsubscript{3}] 97% ionic liquids were found to be prone to inherent biodegradation.

![Graph showing degradation over time](image)

Fig. 1. The course of decomposition of ionic liquids and the reference substance in biodegradability study according to OECD 301 A

**Biodegradation in a soil environment**

The mean degree of [DDA][NO\textsubscript{2}] biodegradation in the treated wood (17.93 kg/m\textsuperscript{3}) by *Gliocladium roseum* amounted to only 4.27% and by a mixture of mould fungi to 7.24% (tables 2 and 3). In the case of the lower level of wood treatment (4.58 kg/m\textsuperscript{3}), the degree of biodegradation of this ionic liquid was
higher and amounted to approximately 20.5%. The didecylidimethyl nitrite migration from the treated wood to the sterile soil was less than 1% due to the insolubility of this compound in water. The ionic liquid with a nitrate anion and a [DDA] cation exhibited a slightly greater biodegradability (from 10.1% to 11.4%) in the case of higher wood retention. On the other hand, a change in the cation structure of the ionic liquid to cocotrimethylammonium made it possible to increase the sensitivity of the ionic liquid [Arq C33][NO3] to the degradation
caused, in particular, by a mixture of the test mould fungi (tables 2 and 3). The mean degree of degradation ranged from 52.17% to 55.33%. This indicates that the cation with the alkyl substituent from coconut oil, which was a mixture of hydrocarbons of C₈ to C₁₄ carbon lengths, underwent biodegradation more easily in comparison with the didecyltrimethylammonium cation in the [DDA][NO₃] and [DDA][NO₂] ionic liquids. Likewise, in the case of the bis-ammonium nitrate with two alkyl C₈ substituents, the recorded biodegradability reached, depending on the wood retention, levels ranging from 33.15% to 51.98%. The migration of the water soluble ionic liquids [bis(AmC₃)][NO₃] and [ArqC₃₅][NO₃] from the treated wood into the soil fluctuated from 9.73% to 17.62%.

Table 2. Changes in retention of ionic liquids in treated Scots pine wood (Pinus sylvestris L.) as a result of migration into the soil and degradation by mould fungi (soil-block test)

<table>
<thead>
<tr>
<th>Ionic liquid</th>
<th>Retention in the wood</th>
<th>Average content of ionic liquid in treated wood</th>
<th>Average content of ionic liquid in treated wood after 12-week soil test</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>kg/m³</td>
<td></td>
<td>Sterile soil</td>
</tr>
<tr>
<td>[ArqC₃₅][NO₃]</td>
<td>4.43</td>
<td>1.0442</td>
<td>0.9426</td>
</tr>
<tr>
<td></td>
<td>17.68</td>
<td>3.9942</td>
<td>3.3212</td>
</tr>
<tr>
<td>[DDA][NO₃]</td>
<td>4.49</td>
<td>1.0959</td>
<td>0.9930</td>
</tr>
<tr>
<td></td>
<td>17.52</td>
<td>4.1350</td>
<td>4.0058</td>
</tr>
<tr>
<td>[DDA][NO₂]</td>
<td>4.58</td>
<td>1.0981</td>
<td>1.0792</td>
</tr>
<tr>
<td></td>
<td>17.93</td>
<td>4.0134</td>
<td>3.9883</td>
</tr>
<tr>
<td>[bis(AmC₃₅)][NO₃]</td>
<td>4.52*</td>
<td>0.1326</td>
<td>0.1115</td>
</tr>
<tr>
<td></td>
<td>18.37*</td>
<td>0.4155</td>
<td>0.3423</td>
</tr>
</tbody>
</table>

*The retention of ACQ preparation (the content of the ionic liquid – 10% m/m)

Table 3. Average degree of biodegradation by mould fungi of ionic liquids in treated Scots pine wood (Pinus sylvestris L.) after 12-week soil test (soil-block test)

<table>
<thead>
<tr>
<th>Ionic liquid</th>
<th>Concentration of ionic liquids in impregnation solution</th>
<th>Retention in the wood</th>
<th>Average content of ionic liquid in treated wood</th>
<th>Average degree of biodegradation by mould fungi of ionic liquid in treated wood after 12-week soil test</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>%</td>
<td>kg/m³</td>
<td></td>
<td>Sterile soil</td>
</tr>
<tr>
<td>[ArqC₃₅][NO₃]</td>
<td>0.63</td>
<td>4.43</td>
<td>1.0442</td>
<td>36.81</td>
</tr>
<tr>
<td></td>
<td>2.50</td>
<td>17.68</td>
<td>3.9942</td>
<td>36.06</td>
</tr>
<tr>
<td>[DDA][NO₃]</td>
<td>0.63</td>
<td>4.49</td>
<td>1.0959</td>
<td>17.37</td>
</tr>
<tr>
<td></td>
<td>2.50</td>
<td>17.52</td>
<td>4.1350</td>
<td>10.10</td>
</tr>
<tr>
<td>[DDA][NO₂]</td>
<td>0.63</td>
<td>4.58</td>
<td>1.0981</td>
<td>20.21</td>
</tr>
<tr>
<td></td>
<td>2.50</td>
<td>17.93</td>
<td>4.0134</td>
<td>4.27</td>
</tr>
<tr>
<td>[bis(AmC₃₅)][NO₃]</td>
<td>0.63</td>
<td>4.52</td>
<td>0.1326</td>
<td>33.61</td>
</tr>
<tr>
<td></td>
<td>2.50</td>
<td>18.37</td>
<td>0.4155</td>
<td>33.15</td>
</tr>
</tbody>
</table>
Fig. 3. HPLC chromatograms of extracts from wood samples treated with cocotrimethylammonium nitrate: a – standard, b – after test in sterile soil, c – after incubation with Gliocladium roseum strain no 62 726
The concentration values of the ionic liquids in the extract obtained from the treated wood presented in table 4 and determined with the assistance of high performance liquid chromatography (HPLC), confirmed a decline in the quantities of the examined compounds after the 12 week incubation period in the soil infected by the tested microorganisms. Moreover, the research results obtained also indicated a slight migration of ionic liquids into the soil. The HPLC chromatogram obtained of the nitrate with a cocotrimethylammonium (standard) cation showed a characteristic peak for the chain with a mixture of hydrocarbons C_{10}, C_{12} and C_{14} and with a retention time of 16.797 min (fig. 3). The analysis performed of the extracts obtained from the wood after incubation with the *Gliocladium roseum* fungus and with the mixture of mould fungi showed peaks of similar retention times on the chromatograms (15.63 min. and 15.51 min, respectively.) as in the case of the extract from the treated wood (the initial sample had a retention time of 16.18 min.) but this peak did not reflect the chain with a mixture of C_{10}, C_{12} and C_{14} hydrocarbons but only the C_{12} hydrocarbon. This may indicate the uneven degradation of this alkyl substituent by mould fungi.

Table 4. Changes in concentration of ionic liquids in extract from treated Scots pine wood after 12-week soil test, determined using HPLC method

<table>
<thead>
<tr>
<th>Ionic liquid</th>
<th>Retention in the wood</th>
<th>Average concentration of ionic liquid in extract from treated wood</th>
<th>Average concentration of ionic liquid in extract from treated wood after 12-week soil test</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>kg/m³</td>
<td>g/L</td>
<td>Sterile soil</td>
</tr>
<tr>
<td>[ArqC₃₀][NO₃]</td>
<td>4.43</td>
<td>0.564</td>
<td>0.5050</td>
</tr>
<tr>
<td>[DDA][NO₃]</td>
<td>4.49</td>
<td>0.4495</td>
<td>0.3044</td>
</tr>
<tr>
<td>[DDA][NO₃]</td>
<td>4.58</td>
<td>0.458</td>
<td>0.3964</td>
</tr>
<tr>
<td>[bis(AMC₃)][NO₃]</td>
<td>4.52</td>
<td>0.1685</td>
<td>--</td>
</tr>
</tbody>
</table>

In all the cases examined, no degradation metabolites with distinctly longer retention times (shorter alkyl chains in relation to the standard) were observed on the chromatographs, indicating the absence of defragmentation by mould fungi of the alkyl substituents from the examined compounds.

**Conclusions**

The examined ionic liquids characterised by different structures of anions and cations exhibited varying sensitivity to biodegradation in aqueous and soil environments, depending on the microorganisms activating these compounds. In conditions of limited opportunity for the biodegradation to occur (screening test), didecyltrimethyl ammonium nitrite exhibited 69.54% degradation, whereas in soil conditions, its biological degradation in treated pine wood (at a retention
of 4.58 kg/m$^3$) as a result of the action of mould fungi amounted only to approximately 20%. Nitrate and nitrite with a cocotrimethyl ammonium cation proved to be sensitive to true biodegradation, amounting to 96% and 93%, respectively. Biodegradation of the cocotrimethyl ammonium nitrate in the wood caused by mould fungi ranged from 36.1% to 55.33% and was greater in comparison with the ionic liquids containing didecylidimethyl ammonium cations. The chromatogram runs of the extracts from wood after exposure to mould fungi were similar to the chromatograms of the examined compounds, with the exception of the ionic liquid containing a cocotrimethyl ammonium cation, developed from coconut oil, the alkyl substituents of which were a mixture of C$_{10}$, C$_{12}$ and C$_{14}$ hydrocarbons. This may indicate a disappearance of the compounds with C$_{10}$ and C$_{14}$ substituents.

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List of standards


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OECD guideline for the testing of chemicals. Inherent Biodegradability: Zahn-Wellens/EMPA. Test Guideline 302B

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AUTOMATION OF ANNUAL TREE INCREMENT MEASUREMENT USING VISION SYSTEM

The objective of the study was the development of an automatic measurement method for tree ring increments using a vision system combined with an XYZ scanner. The elaborated method allows for the measurement of increments on cross-section a three discs or on increment bores. The solution utilizes the XYZ scanner equipped with a vision system, enabling increment measurement during sample scanning. The vision system collects and analyses images of a wood sample. The splicing of partial images enables the construction of a full image of rings, visible on the entire sample surface. Image construction of the studied sample based on a set of magnified partial pictures enables the attainment of very high resolutions, which in top end optical systems, greatly surpasses the resolutions obtained using traditional scanners. The system works automatically and the algorithm of the increment measurement is based on the image analysis of the sample area and is realized in the acquisition time of partial pictures by the vision system. Configuration of the measurement system enables selection of the field of view and measuring resolution. The maximum obtained in the presented system is 0.001 mm.

Keywords: dendrochronology, radial increment, tree-ring measurement, vision system, image analysis

Introduction

One of the most serious challenges of the 21st century is climate change and global warming caused by increasing concentrations of carbon dioxide in the atmosphere. Therefore, increasing interest in and the importance of studies concerning biomass and carbon sink by forest ecosystems has been observed [Ochał et al. 2013; Botkin et al. 2014; Zasada et al. 2014]. One of the most important tree characteristics measured during such studies is tree height, the diameter or tree volume increment, which could be obtained by the analysis of tree rings width at various tree heights [Vaganov et al. 2006; Socha and Orzel 2011]. Tree ring measurement becomes increasingly important in studies concerning the effect of forest management and environmental factors on forest
ecosystems [Socha 2012, Seidl et. al. 2013]. The difference in diameter increments resulting from the intensity of silvicultural treatments may result in different values of merchantable timber of the forest stands [Bembenek et al. 2014]. Today, however, specialists from other fields also recognize and are investigating the problems of dendrochronology. Tree-ring research has acquired a permanent role in the various sciences of archeology, history, geology, ecology, and climatology [Schweingruber 1993].

The development of computers, X-ray and optic systems have accelerated the evolution of wood surface and tree ring width measurement methodology [Vila-Lameiro and Diaz-Maroto 2007]. Thetford et al. [1991] designed a system that digitizes sample images on an X-ray negative. The developed system required a microtome to take samples from a specimen core. Small ring widths (0.06 mm) were then measurable with a video camera and microscope. The use of X-ray, and HF-densitometry, however, which are primarily dedicated to measurements within annual tree ring density suffer from relatively high equipment costs, time-consuming preparation and measuring procedures [Schweingruber 1990; Schinker et al. 2003]. Digital image methods, reducing subjectivity in the assessment of ring width are examples of solutions used most frequently nowadays [Guay et al. 1992; Simpson and Denne 1997; Bucur 2003; Vila-Lameiro 2003]. These solutions are based on computer software for the analysis of digital images of wood samples to obtain information concerning the number and width of tree rings. This information is necessary to determine the age and diameter, volume or biomass increment of individual trees or stands [Courbet 1999; Vila-Lameiro and Diaz-Maroto 2007].

Measurements taken with the use of digital images require the scanning resolution to be set correctly as well as correct sample preparation. Samples should be ground and cleaned prior to measurement. To increase the measurement resolution and contrast between the rings, increased scanning resolution, (for example up to 2,400 dpi) is used, but it results in an increase of the file sizes, in which the digital picture of samples is saved. An increase in the contrast between the rings is also obtained by technological processing with the use of sample impregnation, fast evaporation or chemical etching [Rose 1957; Campbell 1981] or “shadow technique” [DeRose and Gardner 2010]. Pictures with a high resolution can be also obtained using photographic cameras, but then calibration of each camera setting and each increase of picture magnification with the use of a lens is necessary. Furthermore, correct preparation of samples, including sanding and increasing contrast is also necessary. An alternative group of methods, which gives the possibility of tree ring measurement are vision systems, which allow fast acquisition and an analysis of images, using various methods intended to recognize and locate different image characteristics. Most vision systems can be described as 2D systems, due to information available from the image and the method of performing measurements on the image. The most commonly used vision method is the analysis of greyscale or colour [Kim
and Koivo 1994; Hu et al. 2004; Sandak and Tanaka 2005, Fournier et al. 2010, Jakubowski 2014]. Also used are solutions involving the analysis of tomographic or thermographic images [Meinlschmidt 2005; Wei et al. 2009]. Three dimensional (3-D) vision systems may have an especially promising future [Olszyna et al. 2013, 2014; Romaszko et al 2015; Sioma 2015]. One of
the advantages of the 3-D system is that it allows many more measurements and tests to be performed, particularly in the scope of spatial measurements, supplementing and expanding the applied inspection methods.

The objective of the presented study was the elaboration of a new, fully automatic vision system, enabling identification of boundaries and measurement of the width of annual rings on wood samples obtained in the form of bores made with increment bore or disks cut out from the trunk of a cut down tree. Realization of the established aim, required the elaboration of acquisition method and analysis of the images of samples with the vision system combined with the sample positioning system. For the building of a three dimensional representation of ring arrangement, monochrome image was used. The elaborated method includes 3 sets of algorithms, which are used to: 1 – image preparation for the measurements, 2 – determination of ring boundaries and 3 – measurement realization. The study discusses the obtained results and presents the elaborated method compared to measurement methods, which are currently most commonly used for the measurement of the annual tree rings in practice.

Material and methods

The elaborated measurement system consists of the mechanical system, ensuring the realization of sample movement and the vision system with software realizing the image analysis (fig. 1). Three numerically controlled axes were installed in the mechanical system. The measurement table onto which the tested sample is mounted, enables its movement in the XY axis of the stand. The axes of the measurement table are driven by stepping motors, and controlled by the PLC driver managing the operation of the stand. The drive axis Z is responsible for setting the camera height to the surface of the measurement table and the tested sample. The regulation of the camera placement to the measurement table allows control of the FOV (Field of View) on the tested sample. The vision system IVP with 1/3” CCD sensor with a resolution of 640 × 480 was used in the measurement system. The measurement resolution depends on the distance of the vision system from the sample, establishing the field of view defined on the sample. A measurement lens, allowing sharpness to be obtained within a large range of DOF (Depth of Field) was used in the solution. The elaborated measurement system was tested on samples of Scots pine.

The configuration of the vision system was performed to the requirements established at the stage of project preparation, of the automatized measurement system. The following parameters of the vision system were assumed:
Fig. 1. View of the test stand

- the optical system and the sensor of the vision system enable measurement with adjustable resolution of at least 0.05 mm or higher
- the measurement is realized automatically in time under 10 s.
- the measurement results are saved in the database and at the same time, a graphic preview of the results is available to the operator of the test stand.

Measurement of increments on bores or increment disks

In the first stage of the measurement, the grinding sample is mounted on the measurement table and the basing is performed, which enables the adjustment of the pith in the optical axis of the vision system. Then, the field of view of the camera is selected by regulation of the height of the camera positioning to the sample surface. For the selected field of view, the vision system image sharpness check and regulation is performed. Measurements for samples in the form of flat disks, which are flat cross-sections, are realized from the midpoint of a disk, i.e. from the pith. For samples in the form of a "cutout" obtained using test bores in the trunk, the measurements are performed in the mode assumed by the operator, most often from the point of contact between the bark and trunk. The system was tested on Scots pine wood samples.

The analysis of the ring width allows for the use of measurements of a given sample at different field of view sizes. Regulation of the field of view enables the adjustment of the required measuring resolution and its adjustment to the size of rings visible in a sample. Figure 2a presents an image with the largest size and the lowest measurement resolution at the same time. Figures 2b and 2c present smaller FOV and measurement resolutions obtained on the selected surface area. For each camera setting in the Z axis, calibration of the vision
system should be performed, which enables conversion of the results of measurements carried out in pixels to the results provided in the engineering units. If the measurements are realized at the same resolution on subsequent samples, the preparation procedure of the measurement stand is realized only for the first sample.

![Image 1](image1.png)

Fig. 2. Vision system resolution presented in relation to FOV size: a – FOV = 30 mm with resolution 0.05 mm/pixel, b – FOV = 10 mm with resolution 0.016 mm/pixel, c – FOV = 6 mm with resolution 0.012 mm/pixel

The image analysis in the vision system begins by performing the preliminary transformations, aiming at the preparation of the wood surface image for measurements. For the image recorded by the vision system, visible in figure 3a, point and morphological transformations were conducted, as the result of which a contrasting image of the pith visible in figure 3b was obtained. Thus, the prepared image is used in the determination of the pith midpoint, which was assumed as the measurement basis for the determination of increments visible in the sample.

![Image 2](image2.png)

Fig. 3. Preliminary image transformations: a – image recorded by vision system, b – image after preprocessing
For the identification of the pith midpoint, the location of the centre of gravity of the area of pith image was used. The pith area was defined using intensity thresholds enabling the determination of pixels belonging the pith, visible in figure 3a. The determination of the centre of gravity is based on the analysis of the location of all pixels creating the area describing the pith (fig. 4). The centre of gravity is determined on the basis of \( x \) and \( y \) coordinates of the location of each pixel creating the area assigned to the object. In the calculations all points of the area or points creating the contour can be included.

**Fig. 4. Methods of centre of gravity determination for area: a – analysis of all area points, b – analysis of contour points**

Using coordinates of the points creating the area, the centre of gravity location \((C_X\text{ and } C_Y)\) are determined from equations 1 and 2:

\[
C_X = \frac{1}{P} \sum_{1}^{P} P_x \\
C_Y = \frac{1}{P} \sum_{1}^{P} P_y
\]

where:  
\( P \) – number of points creating the area,  
\( P_x \) – coordinate \( X \) of points creating the area in the image,  
\( P_y \) – coordinate \( Y \) of points creating the area in the image.

The next stage of the image processing is the determination of the edges describing the ring location on the wood image. Figure 5a presents an image of the real wood surface and 5b an image after point and morphological transformations. After that, transformations aiming at averaging and scaling of the intensity range visible in figure 5c were performed. Preprocessing and determining the edge involves removing wood texture, morphological transformations, image smoothing, intensity scaling and edge detection with the use of derivative intensity. The boundary determination visible in figure 5d was then carried out. Figure 5d presents clear ring boundaries, undisturbed by the presence of information on the texture of the wood surface. Thus, the prepared image was used in the algorithm of increment measurement.
Results and discussion

The increment measurement is carried out based on the analysis of the distance between boundaries describing following increments visible in the sample image. For the implementation of the measurements, a coordinate system was defined, which was located in the middle of the pith, determined at the preparation stage of the image for measurements. The identification of the subsequent boundaries is then implemented along the X axis of the coordinate system with the use of image 5d. Using the information on the location of the subsequent boundaries, the distance of each ring from the pith is determined. The first measurement was performed at a resolution of 0.05 mm/pixel, resulting from the assumed FOV. The resolution was obtained at a large FOV, allowing for observation of half of the trunk. Each of the tests was performed 1000 times, for each of the random sample sets using several dozen samples of Scots pine. The paper presents measurements performed for three FOV sizes at three measurement resolutions.

The presented measurement system allows for the automation of the annual ring width measurement. The main advantage of the solution is the possibility of
current adjustments of the measurement resolution to the measured wood samples, which contributes to an increase in the measurement precision. Following the widely known regularities of the course of growth [Assmann 1970] increments layered by trees diminish with age, and thus, the width of rings found on the outer circumference of a trunk is smaller. Furthermore, in the entire life of a tree, a considerable difference in the width of late wood can be observed. The possibility of using variable resolution levels out the problem, which allows for its adjustment for the properties of the studied samples. The example below presents measurements performed at a resolution of 0.027 mm (fig. 7). In order to present the results, only part of the sample image is visible, for which a local coordinate system was assumed, allowing the presentation of the results visible in figure 6.

Fig. 7. Increment measurements carried out with 0.027 mm resolution

The developed measurement system also allows the automatic identification of shape and size of a pith. The pith area is used for the determination of the midpoint of a wood disk subjected to measurement. A decrease in the FOV and an increase in measurement resolution to 0.012 mm enables a detailed assessment of the pith shape and determination of the parameters describing the pith. Figure eight presents the pith image with a determined circumference describing the pith shape. On the basis of the pith area analysis, the circumference of the pith was determined, expressed in pixels and mean, minimum and maximum radius (fig. 8b). The surface area of the pith image was additionally calculated.

Vision systems were also used in tree ring measurements. Guay et al. [1992] used a line-scan camera to build an image directly from the sanded core or disk specimen. The entire sample image was stored before interactive analysis. At that time, however, the practical limit of the resolution was six rings per millimetre (0.17 mm ring width) for the contrast range of conifer rings. Thanks to the development of the vision systems, which resulted in improved resolutions, it is considered to be very precise at present. Vision systems which are supported by operator's choice concerning resolution and light seem to be the
best solution, and could fulfil the need for an accurate measure of growth increment on many samples of increment cores or stem discs. We developed a nearly automatic method, which could be sufficiently reliable, especially in the case of large samples.

![Image of tree core with measurements](image)

**Fig. 8. Measurements of pith shape are carried out with a resolution of 0.012 mm**

**Conclusions**

The realization of measurements on the automatized test stand using the vision system, allows the measurement of increments for one sample to be carried out in approximately ten seconds. Analysis of the image obtained during construction of the surface image is realized in under one second. Implementation of the measurement of samples with large widths of sample rays up to 300 is possible mm. The sample is moved under the vision system, which allows for construction of the image with a high resolution, calculated along the shift axis. It stems from the possibility of automatic splicing of the partial images, carried out for the established field of view. During the sample shift, measurement is carried out with the use of the vision system and information collected in the control system from encoders controlling the current sample location.

Selection of the surface area observed by the vision system and the possibility to control the measurement resolution enables adjustment of the image parameters, i.e. resolution and field of view for the sample size. At the same time, it is possible to splice images, enabling a decrease in the field of view and an increase in the measurement resolution, which allows for the observation of wood surface characteristics which are not visible in the image produced with a lower resolution. The resulting image, created from spliced images with high resolution is characterized by a wide field of view and at the same time high resolution. Measurements can be disrupted by the presence of knots, cracks and other damage. The sample containing the disturbances should be measured several times and algorithm removing damage edges must be used.

The observation and implementation of measurements of pith, and increments with resolution enabling evaluation of pith shape as well as the shape of increment boundary shape are also possible. For such measurements, a small
field of view is selected, enabling a resolution of 0.012 mm or higher to be obtained, depending on the used lens and sensor of the vision system. The presented solution of this measurement system enables the image construction of a wood sample with selected measurement resolutions. The resolution is selected by the adjustment of the field of view of the vision system. The advantage of the system consists of the ability to perform measurements for the same sample with different resolutions, enabling the evaluation of other surface properties on each of the obtained images. It is possible to measure samples in the form of increment disks, as well as samples obtained using bores. The total measurement time is approximately ten seconds for a sample of any type. The device enables simultaneous measurement of several samples obtained in the bore technology, which increases the efficiency of the measurement work. This version of the measurement system was elaborated on Scots pine species. The system, however, can be easily adapted to other coniferous and ring porous species.

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VISUAL GRADING OF SMALL-DIAMETER POPLARS FOR PEELING USE

Small-diameter poplars (Populus euramericana, or Populus deltoids, or Populus nigra) are an important raw material resource for the wood and paper industry. Both manufacturers and governments are increasingly interested in obtaining this raw material from poplar plantations. A small-diameter poplar is between fifteen and thirty five centimetres in diameter at breast height. In this study, small-diameter poplars were graded visually for use in peeling. Traditional rules currently in use were applied when grading small-diameter poplars, and standard layer-sorting rules were used on the outputs from the peeling process. The field study has shown that standard sorting by layer doesn't correspond well when small-diameter poplars are sorted. For instance, the best small-diameter poplar is rarely classified as class A or B by standard layer sorting. In this research, small-diameter poplars are classified accurately using specific, alternative rules, and the grading scale ranges are presented. Some defects, such as knot and splits, have a strong influence on the quality of the final product, but often, existing standard-sorting methods do not reflect the degree to which the defects have affected the final product. A new method for classifying small-diameter poplars is presented, which groups the wood by its value in the final product.

**Keywords:** visual grading; small-diameter poplar; defect; output

**Introduction**

Visual grading is essential for selecting the type of application

Wood scaling regulations are used to classify raw materials by grouping them by their quality. The quality of a product, which includes both visible and invisible characteristics, determines the use to which the wood can be applied. Each sorted group is appropriate for a particular application. Visible characteristics are important in classifying logs and timbers because their application is affected by defects. Unfortunately, invisible characteristics (mechanical and chemical properties) also have a key role in material use, and visible defects are affected

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by invisible characteristics. Knots and the slope of the grain, for example, influence the mechanical properties of wood and decrease its bending strength. The output and efficiency of sawmills and plywood mills are improved when the wood contains fewer visible defects. Even though visible characteristics are assessed during the machine stress rating of lumber, visual grading is also essential for selecting the type of application for the lumber. Forest product suppliers, attend to the visible characteristics of the lumber, and their requirements are determined by process technology. Product processes are designed based on the wood material available. Some of those processes match factories that are designed to use small-diameter poplar (SDP; *Populus nigra* or *Populus alba*). Some of the poplar bulks, however, have visible defects, and therefore, those bulks are not accepted by product processes for those factories using SDP. Frost-check defects, for instance, inhibit the application of SDPs in those factories. In this study, the scaling regulations for sorting log groups were investigated, and standard regulations were used to classify SDP. Research was performed to grade and sort the forest products for the wood-based panel and cellulose industries. In addition, structural products made with small-diameter log material also included oriented strand boards and parallel strand lumber; all of which are now commonplace in the timber marketplace. Poplar plantation trees are an important resource for a variety of lumber production processes. Trees from those plantations are considered small diameter if they are less than thirty centimetres in diameter at breast height [Paun and Jackson 2000; Howard 2001; Willits and Roos 2004].

**Visual Inspections and Classifying SDP**

Visual inspection is a simple, convenient, and non-destructive evaluation method. Southern pine (*Pinus taeda* L.) lumber has a higher visual grading yield than Japanese cedar (*Cryptomeria japonica* (L. f.) D. Don), Taiwania (*Taiwania cryptomerioides* Hayata), and Douglas-fir (*Pseudotsuga menziesii* (Mirb.) Franco) do. Most of the defects in these four softwood lumbers are knots, whereas the other defects, such as stains, decay, wanes, crooks, warps, pitting, and twists, are few. Knot types include both loose knots (about 25%) and tight knots (about 75%). Hence, knots are the most important factor in judging lumber grades by visual inspection (the Chinese National Standard) because lumber cut from small-diameter Japanese cedar and Taiwania plantation trees include many knots (in number and in size) in Taiwan. The mechanical properties of wood are significantly influenced by the number and size of its defects.

Southern pine lumber has a higher machine stress rating (MSR) than Japanese cedar, Taiwania, and Douglas-fir. Higher visual grades mean higher dynamic modulus of elasticity (DMOE) and modulus of elasticity (MOE) values for the lumber. Most of the criteria used in the visual grading of the lumber do not correlate closely with specific properties, and visual grading is not as precise as machine grading [Wang et al. 2008].
The logs used in the study [Fernandez-Golfin et al. 2007] have a standard strength group rating of C14, and visual grading was performed on a combination of strength groups including, C14, C16, C18, and C20. Strength grade standards DIN 4074-1, VPS-SRT-2, and PR EN 14544 were calculated for the logs and were compared with the standard BS EN 14081-2. Most of the logs fit a standard and visual grading of VPS-SRT-2. The efficiency and mechanical properties of small-diameter timber (SDT) were classified as first grade, second grade, and out-grade [Fernandez-Golfin et al. 2007].

Incorrect classification by visual grading standards

The PR EN 14544 visual grading standard exaggerated the number of rejects, greatly underestimating the real strength of the material. The DIN 4074-1 grading standard identified the first-quality timber acceptably and correctly graded reject timber. It performed poorly, however, with the second-quality grade, strongly underestimating its quality and incorrectly classifying many second-class pieces as rejects. The VPS-SRT-2 standard correctly identified the first quality material and adequately graded reject material. Second quality material, however, was undervalued or overvalued, almost to the same extent. The VPS-SRT-2 standard not only identified a greater percentage of timber as first quality than the DIN 4074-1 standard, it also identified a smaller percentage of reject timber. The most evident errors with both standards, therefore, involve the identification of second-quality material, although these errors are of less importance with DIN 4074-1 standards because it undervalues, rather than overvalues, the material.

Visual grading is tedious because of the number of variables to be measured, and it is not very efficient, given the high number of rejects obtained. The most suitable course of action might be to focus on designing mixed-grading systems (as used for sawn timber), integrating measurements made using non-destructive systems (ultrasounds, penetrometers, etc.) and rapid visual evaluations of different features (knottiness, curvature, and the presence of juvenile wood, etc.) [Fernandez-Golfin et al. 2007].

There is a need to design a mixed-grading method based on the use of non-destructive tests and the visual evaluation of specific wood features. Erikson et al. [2000] studied the mechanical properties of lodgepole pine (Pinus contorta Douglas ex Loudon), grand fir (Abies grandis (Douglas ex D. Don) Lindl.), and Ponderosa pine (Pinus ponderosa Lawson & C. Lawson) and their corresponding economic value as dimensional lumber produced from typical overstocked forest stands in northern Idaho. The lumber was visually graded and tested for MOE and modulus of rupture, and each piece was sorted into two categories: visual structural light framing and MSR. This study indicated that two of the three species tested had good visual and mechanical characteristics.
Limitations of log-grading standards for final lumber quality

Results show that the strength of small-diameter round timber is high, corresponding to the highest-quality sawn timber. Small-diameter timber can be used in load-bearing structures, and it is, in fact, a strong material. Simple, visual rules are adequate for strength grading. The results provide a basis for the start of an international standard for the structural use of small-diameter, round timber [Ranta-Maunus 1999].

Small-diameter timber is often of lower quality and lesser value than larger saw timber. Small-diameter hardwood timber, therefore, has traditionally been used for pulpwood, but it can also be used for lumber and residue production [Perkins et al. 2008].

Research [Craft and Emmanuel 1981; Craft 1982] has indicated that short-length logs with fewer sweeps have a greater yield than longer-length logs with more sweeps. Clearly, to maximize the profitability of sawing small logs, one must maximize the product yield from SDT.

Grading rules exist for logs with less than an eight inch diameter at the small end. For logs with a diameter at the small end of more than eight inches, the log-grade rules of the U.S. Department of Agriculture, Forest Service (USFS), are used [Rast et al. 1973]. Mills have to develop their own grading criteria for six inch and seven inch logs. Logs with large portions of unsound wood or obvious sweeps have a lower yield.

When dealing with large, crooked, or irregular logs—especially hardwood—a simpler machine might be a better choice, although both productivity and lumber recovery will be less [Michael et al. 2009].

In fact, the recovery rate obtained with the Economizer Small Log Mill is quite high for a small, mobile plant and exceeds that obtained from stationary, Chip-N-Saw mills under similar small-log conditions [Fahey and Hunt 1972].

Grading for sawmills is usually carried out in log-grading facilities. The main criteria for grading logs are the dimensions of the log (normally, the length of the log and the diameter of the smaller end are measured); also graded are the quality parameters of the log (wood species, knots, curvature of the log, etc.). Petutschnigg and Katz [2005] developed a model to analyse the effects of the quality characteristics of logs on the quality of the lumber. Based on 112 logs, it was not possible to make definitive statements about the connection between log quality and lumber quality in general. This is because it is not the characteristics of individual logs that are important, but rather, the combinations of different log-quality characteristics which affects the quality of the lumber produced. In that study, the main log-quality factor influencing lumber quality was the combined occurrence of curvature and discoulouration. By grading the logs according to the log-grading rules deduced from their study, a more-efficient
cutting yield was achieved. Sawmills can use the results of that study to define new criteria for log grading as well as to make differentiated log purchases.

Hecker et al. [2000] investigated the connection between log grading according to various national and European log-grading standards and the quality of the lumber produced. They found no adequate forecast method, however, for predicting lumber quality on the basis of the log grade.

In Italy, poplar plantations represent an important source for wood products, particularly high-quality veneer logs. Italian plantations offer favourable conditions for the introduction of forest mechanization, especially for ease of access and industrial management. Mechanized log-making in Italian poplar plantations has not caused any significant reduction in the value of the veneer recovered, compared with traditional motor-manual log-making. Length-measurement errors are smaller with mechanized processing, whereas the frequency and severity of log surface damage for both treatments was the same [Spinelli et al. 2011].

Increasingly, SDTs are used as posts along highways. That trend will continue to increase in the future because of the high cost for large-diameter timber stands. Most motorway round-beam diameter is between 10 to 12.5 cm for smaller posts and 15 to 17.5 cm for larger posts [Paun and Jackson 2000]. The application SDTs by sawmills were analysed by Becker [1998], and the volume of product boards from SDTs was less than the volume of boards from large-diameter timber in an eight hour period.

Visual grading can be made according to certain criteria in the standard NF52001. The specific criteria in that standard include the width of annual rings, knot dimensions, checks, resin pocket, bark pocket, grain angle, and stain and sap stain defects. The standard includes three-class grades: ST1, ST2, ST3, which is in accordance with the defects allowed in C18, C24, and C30 in the EN 388 standard [CEN 2003]. Grain slope, wane, and knots are the most critical defects observed in visual grading [Bodig and Jayne 1989].

The MOE of structural-lumber-grade logs was reviewed by Edund et al. [2006]. They found a good relationship between the MOE of logs and the construction grade of lumber, and low MOE logs produced construction lumber of a low quality.

**Uses for SDPs**

SDTs have a variety of uses. Experimental observations are necessary to make optimal use of their products, so that these materials can be appropriately classified and selected for each use. Nowadays, SDPs are classified based on the specific requirements of each factory. Groups of SDPs have to be used for certain applications because their output is different. If these groups and their outputs were identified, SDPs could be more optimally used by the wood industry. Visual grading rules for SDTs aren’t available for peeling use. The visual grading process of poplar bulks is based on the use of those bulks at local
plants. Visual grading rules for SDPs should be prepared. The number of ply grades (grading groups) should correspond to the number of bulk grades in the proposed rules because production efficiency of the ply is increased with proper grading groups for SDP. Ply groups of A and B, for example, should result in the first grades of poplar bulks. Another objective of this study was to determine those defects that have an important effect on the grading of poplar bulks. The frequency of the defect and its size were measured, and the production efficiency related to various groups of SDP will be determined. The goals presented in this article include sorting of SDPs for peeling and calculating the outputs as a percentage of each group.

**Materials and methods**

This research was carried out at the Amol peeling mill (Iran). Visual grading was performed according to scientific references [ISIRI-1275, APA-PSI-95], along with the criteria of plant suppliers. The standard number 1275 from Iran and the Product Standards of the American Plywood Association were used to grade logs and to sort ply. First, the SDP was graded, and then, each log had its wood layers extracted and sorted, and finally, production efficiency was calculated for the different ply classes. The average dimension and frequency of the defects was measured for SDP, and the average defects were calculated for different groups of logs. This allowed a proposed range of defects to be determined for each of the different grades. The limits of the defects allowed were specified according to the average measured defects of the SDP, the rules of the national standard [Standard number 1275 in Iran and the Product Standard of American Plywood Association], and a field survey (criteria of plant suppliers). Because the visually graded groups of SDPs were determined by the methods outlined, the grading criteria were usable for SDP. The stages of this method are shown in figure 1. In addition, the shopping criteria from peeling mill suppliers and the grouping criteria in scientific sources were identified for SDTs. Because the SDP sorting was theoretical, the visual scales of poplar bulks survived for peeling use. The defect types for poplar bulks were then measured, and the production efficiency of the ply was calculated for the sorted groups. At that point, the defects that had an important influence on the sorting were identified. By determining a dimension range for the defects, the poplar bulk grading becomes simple and practical in a field survey. Sorting performance can be measured based on the new rules, with clear layers from poplar bulks having the highest quality.

Log volumes were calculated using the Smalian formula, as follows:

\[
V = \frac{f(ds^2 + dl^2)}{2} L
\]  
(1)
where $V$ is the log volume (in cubic meters), $f$ is the metric ratio (0.7854), $ds$ is the diameter of the small end, $dl$ is the diameter of the large end, and $L$ is the log length.

\[
V = L \times b \times t
\]  

(2)

where $V$ is ply volume (in cubic meters), $L$ is the ply length, $b$ is the ply width, and $t$ is the ply thickness.

Outputs from the SDP were converted to layers with the following formula:

\[
\text{Ply extraction efficiency} = \frac{\text{Ply volume total}}{\log \text{volume total}} \times 100
\]  

(3)

**Results and discussion**

The total volume and the number of SDPs were determined for each grading group, and then, the total volume of the ply production and the efficiency percentage were calculated. Sorting performance related to previous rules, showed that grading groups of production layers did not correspond to the visual quality of poplar bulks. The production layer efficiency of the poplar bulks equalled 64.14, 61.32, and 64.83 percent from the first to the third grade, respectively. First-grade poplar bulks produced the greatest number of C-grade ply, and the production efficiency of the ply related to first-grade poplar bulks were as follows: $A = 17.34$, $B = 12$, $C = 30$, and $D = 4.8$ percent. Second-grade
poplar bulks produced the greatest number of A-grade ply, and production efficiency of the ply related to second-grade poplar bulks were as follows: A = 26.02, B = 14.86, C = 8.92, and D = 11.52 percent. Third-grade poplar bulks produced the greatest number of C-grade ply, and production efficiency of ply related to third-grade poplar bulks were as follows: A = 18.72, B = 17.80, C = 28.31, and D = 0 percent. Production efficiency of poplar bulks, therefore, isn’t proper, based on the current grading rules, and the rule should be revised based on new rules with clear layers produced by quality bulks.

The number and the dimensions of defects were measured as sorted bolts. The defects in SDP included knots (unsound or sound, loose or tight, dead), cavities or holes, stains or discoloration specks, surface decay, splits or checks (star, compound, heart check or ring shake, frost cracks), abnormal section conditions or ovateness, irregularly shaped logs, tapers in long logs, and sweepers. Splits or checks, in particular frost cracks, were the most frequent defects in poplar bulks, followed by stains or discoloration specks. Peeling mills do not purchase poplar bulks with frost cracks, sweepers, or that are irregularly shaped.

The number and dimensions of defects were also measured at the production layers. Defects in the production layers were the same as those in the poplar bulks. Stained streaks, extraction, and surface damage (loose side, rupture, swirls, raised grains, and gaps) were the most frequently seen defects in the production layers.

The proposed grading rules for SDP are presented in table 1. The range of defect measurements and the number of defects allowed or unacceptable in each group were determined according to the mean average of the defect measurements, the national standard rules for grading (Iran national grading 1275 for logs and the Product Standards from the American Plywood Association), and by the criteria of the peeling mills.

The clear cutting length is completely free of defects. The number of permitted clear cuttings is small for grade one and the percentage of the log length required for clear cuttings is large for grade one. On the other hand, the number of clear cutting is unlimited for grade three, and the log length required in grade three clear cuttings is smaller than that in grade one.

The dimensions of the product layers were equal to 244 by 122 by 0.22 ±0.01 cm (length by width by thickness). The PS1 rules can be simplified to four categories as follows: (1) knots; (2) splits or open defects and gaps; (3) surface situations, such as loose side, raised grain, grain rupture, rough cuts, uneven surface, swirls, and the slope of the grain; and (4) patches and defect total.

In conclusion, the production efficiency percentage based on the new sorting rules and proposed ranges offer the best performance in field surveys, for example, ply groups A and B were produced by the first grade of poplar bulks (tab. 2).
### Table 1: Proposal grading range of small-diameter poplar

<table>
<thead>
<tr>
<th>Defect</th>
<th>Grade</th>
</tr>
</thead>
<tbody>
<tr>
<td>Length to centimetre</td>
<td>First</td>
</tr>
<tr>
<td></td>
<td>160</td>
</tr>
<tr>
<td>Knot diameter (unsound, dead, sound and tight) at each meter length several knot or one knot</td>
<td>3 centimetre for sound knot, unacceptable for dead knot</td>
</tr>
<tr>
<td>Surface decay</td>
<td>None</td>
</tr>
<tr>
<td>Checks, ring shake and splits</td>
<td>None, except of slight checks or slight shakes</td>
</tr>
<tr>
<td>Frost cracks</td>
<td>None</td>
</tr>
<tr>
<td>Abnormal conditions of sections (distance from the largest to smallest average diameter at one end, to centimetre)</td>
<td>Bolts diameter is larger than 26 centimetre, only, 1 centimetre allowed and 0.5 centimetre allowed for smaller bolts diameters</td>
</tr>
<tr>
<td>Taper of the long log (the difference between the two end diameters), reduce diameter for each meter to centimetre</td>
<td>1 centimetre</td>
</tr>
<tr>
<td>Sweeper (the distance between the tape and the geometric centre, for each meter to centimetre)</td>
<td>1 centimetre</td>
</tr>
<tr>
<td>Off centre hearts (offset or removed pith) (distance from pith to geometrical centre to centimetre)</td>
<td>1.5 centimetre</td>
</tr>
<tr>
<td>All of defects (above mention, and unmentioned)</td>
<td>The maximum number of clear cutting permitted</td>
</tr>
<tr>
<td>The minimum length of clear cutting permitted (minimum proportion of bolts length required in clear cuttings)</td>
<td>The proportion of 5 to 6 or (5/6) (for bolt 1.60 metric is equal 1.33 meter)</td>
</tr>
<tr>
<td>Number of bolts grade (table 4)</td>
<td>Number of bolts total number</td>
</tr>
<tr>
<td>--------------------------------</td>
<td>-------------------------------</td>
</tr>
<tr>
<td>First</td>
<td>9</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>31</td>
</tr>
<tr>
<td>Second</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>20</td>
</tr>
<tr>
<td>Third</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>14</td>
</tr>
</tbody>
</table>

Applications for poplar wood spans a wide range of uses. The special demands of consumers or plant suppliers are sorted based on the characteristics of SDP. Groups of SDP selected by consumers by the quality or production outputs are increased by this method. Production efficiency decreases with some SDP defects. SDP sorting is important for peeling mills because they need logs from the highest-grade group. Effective defects for peeling are empirically understood by processing operators. SDP that has frost splits are not purchased by plant suppliers. These effects and SDP grading groups were determined in this study. Grading rules, such as USFS, APA-PS 1, and 1275 in Iran, aren’t related to SDP. Bolt numbers of the first and third groups produce more of grade two when based on the Standard 1275 grading rules. SDPs have more layer production in group C than in group A when based on APA-PS 1. Dead knot and frost cracks (or splits) have a great influence on the SDP grading and their layer outputs. These defects were taken into account by dealing with the rules of plant suppliers. Unsound or dead knots and splits were defects that were prevented for first-grade logs, and frost cracks were also unacceptable in all sorting groups. The limiting defects have been presented in table 1. The number, location, and diameter of knots from SDP are very important during peeling, whereas
discolouration streaks are more often seen with SDPs, and this defect has a small role in SDP grading.

When SDP has a diameter larger than thirty centimetres, and it has been first graded, the peeling output is more efficient than with other SDPs (tab. 1). Peeling machines cannot use thickset SDP (diameters over thirty centimetres) converted to layers because these machines are designed for light-weight use. Heavyset SDPs cannot be rotated by the peeling machine. Heavy trunks and defects, such as frost cracks, sweepers, and abnormal conditions of any bolts, stop the continuous peeling process. When SDP is sorted by an accounting criteria [BayatKashkoli et al. 2008], the output and speed of layer production was increased by thickset (from twenty five to thirty centimetres) SDP. The diameter of SDP is usually more than thirty centimetres. Peeling machines, therefore, have to use a special technology for SDP. When SDPs are smaller or larger than the specified diameter range, the trunks have not been used for peeling because of the output efficiency and machine technology. Fast-growing wood species or plantation trees, such as poplars, have different defects than those of forest species.

**Visual grading of defects with the new rules:**

Insect cavities are observed after the initial stage of decay because decaying wood is a favourite food of insects such as beetles, and rotted wood hosts insect worms. Insect cavities are like the decay that is addressed in Table 1 and are measured as part of all of the defects or surface decay. Additionally, trunks of poplar can suffer decay because of their wood texture and density.

The ring shake defect is seen in third-grade bolts, and reduces peeling output. The depth of the ring shake is slight in first-grade bolts (tab. 1). A layer rupture in peeling is caused by splits and ring shakes. Splits, gaps, and checks in SDP are unacceptable for A grade ply. Frost cracks on the trunk surface are important in SDP sorting. Changes in temperature, the low thermal conductivity of wood, texture loss, and thin bark of bolts are causes of frost cracks. SDP is capable of developing this type of defect. Frost cracks have a strong influence on the use of poplar. Some of the wood industry, such as peeling mills and match-production factories, do not use poplar with this type of defect. This defect, therefore, isn’t allowed in any of the sorting groups in the new proposed rules (tab. 1).

Sound knots in SDP are rated more highly than dead knots are because plantation trees are pruned, and tissue repair in poplar plantations is more rapid than it is in natural forest trees because poplar is fast growing. The spiral grain of a knot is caused by layers that have separated because the knot shrinks at a different rate than the surrounding texture. The SDP knot texture applies weak pressure against the peeling knife. Spiral grain of the branch texture creates strength against gravity, but the defect is the most important for SDP and
production layers. Quality classifications of SDP and its production layers are reduced by this defect or by patches in the new rules (tab. 1).

Discolouration specks and stain streaks have been seen with SDP. This defect is natural and inborn. Peeling outputs are not decreased by this defect, but it does influence finishing, painting, and gluing. Discolouration specks and stain streaks have a different acidity (PH) than sound-textured wood. Pitch pockets and discolouration defects have caused weak finishes and poor adhesion. This allowable limit of the defect is slight in the grade A classification.

Extraction or damage to the surface, loose side, patches, open defects, and gaps should be limited in all layer classifications, but these defects have only been slightly limited with previous sorting standards. Slope of the grain can strongly influence plywood quality. Production layers easily separate in the grain direction, but are resistant to vertical force. Usually, layers are joined together by sew and punch. Grain direction of adjacent layers should be parallel. In figure 2, the punches and sewing thread are perpendicular to the grain direction, and junction has been sewn up with a tight coupling. Otherwise, the connection will be torn. Production layers with a slope in the grain will rupture during the drying and transport process. The defect can be limited during group sorting.

![Diagram](image)

**Fig. 2. The parallelism of grain direction at adjacent layers (tight junction) (A), and easily separation of connection at the perpendicular of grain direction (B)**

Low width layers result from the taper of long logs. This defect is the same as the slope of the grain because of the above processing problems (fig. 2). If the difference between the diameters of the two ends of the trunk is large, primary rotation of the peeling will be a product of the layers with the lowest width, and additional layers will have to be sewn into the final product layer.
Irregularly shaped logs are an abnormal condition of the various log sections, and the distance from the largest to the smallest average diameter is more than a log with a circular section. The distance between the tape and the geometric centre is also larger in sweeper logs. Outputs are decreased by SDPs with large diameters and those that are irregularly shaped or sweepers caused by bolt shaking and the centrifugal force of peeling. These defects stop the continuous peeling, and production efficiency, process speed, and production quality are decreased (fig. 3).

**Fig. 3. Production layers of bolts with oval section (A) and sweeper (B)**

Large-diameter bolts, irregularly shaped sections, and sweepers that impact the peeling machine blade can cause trunks to be dropped from the peeling machine. Sweeper bolts are associated with reaction wood (tension wood) and off-centre hearts. Additionally, layer surfaces that result from defective bolts have surface damage, loose sides, grain ruptures, rough cuts, and uneven swirls and slopes in the grain. These defects influence finishing and painting. Logs with large portions of unsound wood or obvious sweeps have a lower yield [Rast et al. 1973; Michael et al. 2009]. This is an important defect in the difference between graded logs. The main criteria for grading logs are the dimensions of the log and the quality parameters of the log (the knots, the curvature of the log, discolouration: [Petutschnigg and Katz 2005]). Grain slope, wane, and knots are the most critical defects during visual grading [Bodig and Jayne 1989].
Smooth and glossy surfaces are necessary for plywood, but product cost, raw materials for finishing, and finishing time are increased by defects. The primary stage of polishing is caused by raised grains, and the finishing stages should be increased until the surface is perfected. These defects, therefore, are limited during sorting classifications (tab. 1), and the proposed range of sorting groups is related to the defects effects on the final products.

Ply production and the efficiency percentage of peeling were calculated based on the data in table 1. These results provide a new sorting scheme, as shown in table 1.

Output and sorting results from previous rules classify more first-grade products as grade C, but the proposal in table 2 classifies more products as grade A. In addition, B and C layers classify trunk products as second and third grade, respectively (tab. 2). This proposed sorting standard, therefore, is more precise than previous grading rules (tab. 2). The requirements specified in the standards ISIRI-1275 and APA-PS1-95 are available as published data. Factory operators complete the sorting of log defects with excessive detail. These scaling regulations cannot be used to classify small diameter poplars. The classifying rules should determine the use to which the logs can be applied. This proposed sorting standard is based on the application type, and after this, the peeling output is more efficient than with previous sorting standards.

Plantation trees have many knots (number and size; [Wang et al. 2008]), but poplar trees also have many splits, surface decay, and discoloration. Visual grading is required to select trees for their use in non-destructive systems. The most suitable course of action might be to focus on designing mixed-grading systems [Fernandez-Golfin et al. 2007]. Species with good visual grading have proper mechanical characteristics [Erikson et al. 2000; Wang et al. 2008]. The strength of small-diameter, round timber corresponds highly with the highest-quality sawn timber [Ranta-Maunus 1999]. In conclusion, sorted layers correspond to new scales of grading, and superior layers result from the highest grade of poplar bulks (tab. 2).

Conclusions

Applications for SDP change, depending on the defects of the various sorted groups. Visible defects are affected by invisible characteristics. Standard regulations are used to classify SDP and its plywood, and poplar trunks are sorted for peeling and output percentage is calculated for each group. Grading rules, such as USFS, APA-PS 1 and 1275 in Iran, aren’t proper for experimental applications. Splits or checks, in particular frost cracks, occur with greater frequency than other defects do in poplar bulks, followed by stain or discolouration specks. Stained streaks and extraction or damage to the surface (loose side, rupture, swirls, raised grain, and gap) is more frequent than other types of defects in production layers. In the proposed grading rules, SDP with
frost splits, sweepers, and irregularly shaped bolts will not be purchased by plant suppliers. Frost cracks are not allowed in any of the sorting groups. Dead knots and frost cracks (or splits) have great influence on SDP grading and their layer outputs. Sound knots in SDP are more frequent than dead knots. Quality classification of SDP and production layers have been reduced by this defect or by patches. Ring shake defects reduce peeling output, and the depth of a ring shake should be slight in first-grade bolts. Defects such as splits, gaps, and checks in SDP are unacceptable for grade A layers. Heavy trunks and defects such as frost cracks, sweepers, and abnormal conditions of the bolts stop continuous peeling and reduce production efficiency, process speed, and production quality. Trunks of poplar can decay because of wood texture and density. Discolouration specks and stain streaks have been seen in SDP. Pitch pocket and discolouration defects have caused weak finishing and poor adhesion. These defects should be slight in grade A layers. Extraction or damage of the surface, loose side, patches, open defects, and gaps are limited in all layer classifications, but these defects have only been slightly limited under previous sorting rules. The grain slope strongly influences plywood quality. Production layers with grain slope defects will rupture during the drying and transport process. Grain slope defects should be limited to specific sorting groups. These defects (slope of grain and loose side) influence finishing and painting. These defects, therefore, are limited in the new sorting classification, and proposed ranges of sorting groups are related to the effect of the defect on the final product. The proposed sorting method is more precise than previous grading systems because the factors that affect the final product are considered when grading SDP.

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List of standards


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DIN 4074-1: Strength grading of wood - Part 1: Coniferous sawn timber
ISIRI Number 1275: 1994 Institute of Standards and Industrial Research of Iran – acceptable defects for logs (and gradation)
PR EN 14544: Timber structures - Strength graded structural timber with round cross-section – Requirements
VPS-SRT-2: Structural round timber – grading – requirements for visual strength – grading standards
Janusz ZAWADZKI, Jakub GAWRON, Andrzej ANTCZAK, Teresa KŁOSIŃSKA, Andrzej RADOMSKI

THE INFLUENCE OF HEAT TREATMENT ON THE PHYSICO-CHEMICAL PROPERTIES OF PINEWOOD (PINUS SYLVESTRIS L.)

Pinewood (Pinus sylvestris L.) was thermally modified at temperatures of 160°C and 200°C for 2, 6 and 10 hour periods, respectively. The severe conditions of the heat treatment resulted in decreased mechanical properties (compressive strength and modulus of elasticity) and a decrease in the degree of cellulose polymerization. A comparison of selected mechanical properties and the degree of cellulose polymerization indicated that there was a relationship between them and the temperature and duration of the heat treatment.

Keywords: pine, heat treatment, cellulose, degree of polymerization, mechanical properties

Introduction

The properties of thermally-modified wood have been examined since 1930s. One of first research studies was conducted by Stem and Hansen [1937] and concerned the dimensional stability of wood. Numerous technologies to obtain thermally-modified wood have been created, of which the best-known are: Dutch PlatoWood®, German Thermoholtz®, Finnish Thermowood® and French RectificatedWood®. These processes differ as regards the conditions in which the treatment is conducted. Some of the physical properties of thermally-modified wood change irreversibly. In addition to changes in the durability of the wood, the heat treatment of wood results in a change of wood colour [Schneider and Rusche 1973; Viitaniemi and Jämsä 1996; Kubojima et al. 2000]. The high temperature also causes a number of changes in the structural composition of the wood and, in particular, in the polysaccharide contents
[Zawadzki et al. 2007; Gawron et al. 2011, 2014]. Furthermore, a number of chemical processes take place [Rowell et al. 2002; Yildiz and Gumuskaya 2007]. Only higher temperatures lead to a significant decrease in the degree of cellulose polymerization, which is connected with its degradation. The processes and reactions result in an alteration of the structural and non-structural substances content in the wood [Yildiz et al. 2006]. More visible changes can be observed where such treatment is conducted with the use of superheated steam or oxygen (e.g. in atmospheric conditions) as compared to the modification of dried wood in the presence of an inert gas. Mitchell [1988] found that the heat treatment of wood in the presence of atmospheric oxygen additionally influences the process of carbohydrate depolymerisation. The objective of this work was to discover whether the treatment of pine wood under oxygen conditions allows a correlation between the degree of cellulose polymerization obtained from the wood, particularly at a temperature of 200°C and heat treatment times of 6 and 10 hours, and the mechanical properties.

Materials and methods

A pinewood (Pinus sylvestris L.) board, without defects, was used for the analysis. Samples were collected from the sapwood zone and measured 20×20×30 mm (the longest edge parallel to grain). There were 38 samples in each group.

The first group of samples was stored at room temperature and no further action was taken with them. The second group was dried at 105°C for 12 hours. The following three groups were dried first and then thermally modified at 160°C for periods of 2, 6 and 10 hours, respectively. Similarly, the last three groups underwent heat treatment at 200°C for periods of 2, 6 and 10 hours, respectively.

The treatment process

1. Raising the temperature to 105 ±1°C; maintaining the temperature for a period of 12 hours.
2. Raising the temperature to the final level of 160 ±1°C or 200 ±1°C; maintaining the temperature for the given period of 2, 6, or 10 hours.
3. Taking the samples out and achieving constant weight in a desiccator.

Preparation of samples for chemical analysis

The pinewood control samples and samples after heat treatment were ground, obtaining a sawdust fraction of 0.5-1.0 mm used for testing. The sawdust underwent extraction using a chloroform and ethanol mixture (93:7) w [Antczak et al. 2006]. From the samples which had undergone heat treatment, the cellulose was separated with the use of the Kürschner-Hoffer method.
Following this, 3 cm$^3$ of water was added to a test tube containing 15 mg (0.015 g) of cellulose, and the suspension of cellulose in water was left for 12 hours. After this time, the water was separated from the swollen cellulose using a syringe and 2.5 cm$^3$ of methanol was added. After 45 minutes, the methanol was filtered using a syringe and replaced with a new portion of the solvent. The cellulose was then washed twice with 2.5 cm$^3$ of DMAc at identical time intervals. Following the addition of 2.5 cm$^3$ of DMAc for the third time, the suspension of cellulose in DMAc was left for 12 hours. After the removal of the DMAc, 1.25 cm$^3$ of 8% (w/v) LiCl/DMAc was added to dissolve the cellulose. After complete dissolution of the sample, it was diluted to a concentration of 1% (w/v) LiCl/DMAc [Dupont and Mortha 2004]. Subsequently, all the samples were analyzed with a SHIMADZU LC-20 liquid chromatograph. During the test, a Nucleogel M-10 (Macherey-Nagel) column was used. For all the analyses, identical conditions were applied: eluent – 0.5% solution of LiCl in DMAc, filtered through the 0.2 μm PTFE membrane; eluent flow rate was 1.5 cm$^3$/min and temperature 80°C. For the purpose of calibration, polystyrene standards with a molar mass from 580 Da to 6850 kDa were used. A Mark-Houwink universal calibration was used to establish the molar mass distribution, with the application of the following properties:

\[
k_{ps} = 17.35 \times 10^{-3} \text{ cm}^3/\text{g}, \quad \alpha_{ps} = 0.642 \quad \text{for polystyrene [Timpa 1991]}
k_{cel} = 2.78 \times 10^{-3} \text{ cm}^3/\text{g}, \quad \alpha_{cel} = 0.957 \quad \text{for cellulose [Bikova and Treimanis 2002].}
\]

**Preparation of samples for mechanical properties test**

The mechanical properties of the samples collected from the sapwood zone which underwent heat treatment were measured, as well as those of the control samples which had been stored in ambient conditions. The compressive strength and elastic modulus of the wood compressed in the direction along the fibres were measured.

The consolidated results of the physical and mechanical properties – the density, compressive strength and elastic modulus of the pinewood samples, according to treatment temperature and time, were presented in an earlier publication [Zawadzki et al. 2013].

**Results and discussion**

The weight average molar mass was established with regard to the cellulose contained in the pinewood which underwent heat treatment with the application of different durations and temperatures. It is possible to observe, on the basis of the data provided in figure 1, that the temperature and time of the wood treatment influenced the degree of cellulose degradation. Changes in the weight average molar mass of the cellulose extracted from the pinewood occurred in both cases – at a temperature of 160°C and 200°C.
The duration of the heat treatment also influenced the degree of cellulose polymerization. The extension of the heat treatment time from 2 h to 6 h, and then to 10 hours, at the given temperatures consequently resulted in a decrease in the weight average molar mass of the cellulose. In relation to 380 kDa molar mass of cellulose in reference samples, the influence of a treatment temperature of 160°C and 10 h time resulted in its decrease to 180 kDa, i.e. a decrease of 53%. Treatment temperature of 200°C and 10 h duration caused a decrease in the weight average molar mass of the cellulose molecules to 120 kDa, i.e. a decrease of approx. 69%. The results obtained correspond well with the results of Kacikova et al. [2013].

Similar results were obtained by Fengel [1967], who tested Norway spruce wood which was subjected to thermal modification for 24 hours. The degree of cellulose polymerization remained almost unchanged up to 120°C, however, at a temperature of 200°C, it rapidly decreased to 40% of initial value.

A kinetic model of pseudo-zero-order reaction [Zou et al. 1994; Emsley et al. 1997] was used in order to examine the kinetics of the degradation process. Taking into account the dependence between the percentage yield and the degree of cellulose polymerization, an equation was obtained, which was used to interpret the experimental data. On the basis of the parameters of molar mass distribution of the cellulose from degraded samples (previously extracted from the wood which underwent heat treatment), the weight average polymerization degree ($P_w$) were determined and the kinetic model described above was applied. The dependence of the reciprocal polymerization degree of cellulose on the time of wood treatment is illustrated in figure 2.

Fig. 1. Changes in the weight average molar mass of cellulose molecules contained in pinewood during heat treatment
The influence of heat treatment on the physico-chemical properties of pinewood (*Pinus sylvestris* L.) 53

![Graph showing the kinetic dependence of degradation of cellulose contained in thermally modified pinewood](image)

**Fig. 2. The kinetic dependence of degradation of cellulose contained in thermally modified pinewood**

The observed dependence seems to be linear, therefore the degradation mechanism may be interpreted as random.

The cellulose depolymerisation reaction constant was established on the basis of the observed dependence. For wood treatment at 160°C, the following value was obtained: \( k = (1.85 \pm 0.16) \times 10^{-8} \text{ s}^{-1} \). At 200°C, the progress of cellulose degradation was faster: \( (3.25 \pm 0.27) \times 10^{-8} \text{ s}^{-1} \), i.e. by 76%.

The Arrhenius equation in the logarithmic form gives linear dependence of the reaction rate constant on temperature. Knowing the process rate values of the experimentally determined degree of cellulose depolymerisation at the treatment temperatures 160°C and 200°C, it is possible to determine the activation energy of the cellulose degradation process. Under the conditions of the applied heat treatment process, it was \( (23.9 \pm 2.3) \text{ kJ} \cdot \text{mol}^{-1} \). In most chemical reactions, the activation energy levels are between 50-300 kJ·mol⁻¹. In the case of cellulose, the activation energy of the thermal decomposition reaction changes significantly according to the experiment conditions. Hill et al. [1995] provides the values of 79-92 kJ·mol⁻¹ for thermal decomposition, and higher values of 113-120 kJ·mol⁻¹ for hydrolytic degradation. At the higher range of temperatures, between 300°C and 360°C, the activation energy for thermal degradation is higher and reaches up to 112 kJ·mol⁻¹ [Zickler et al. 2007]. According to Strlič et al. [2001], values for the initial phase of decomposition at 180°C, obtained on the basis of chemiluminescence measurements, are approx. 49 kJ·mol⁻¹ under anaerobic conditions and 78 kJ·mol⁻¹ under aerobic conditions. On the other hand, Ding and Wang [2008] concluded that the values obtained for sulphate paper degradation at temperatures under 100°C depend on the environment. The activation energy was only 11.5 kJ·mol⁻¹ in the presence of air, 34 kJ·mol⁻¹ in oil,
and it reached 120 kJ·mol$^{-1}$ after oxygen and humidity elimination. In addition, it should be stressed that in the case examined of the heat treatment of wood, the cellulose degradation process did not function as a closed system. Wood is a complex system, which comprises substances of various natures, such as resin acids or phenolic compounds (including lignin). These compounds may catalyze or inhibit cellulose reactions, thus significantly altering the activation energy.

It was decided that the interdependence between the physical and mechanical properties of the modified wood and the measured degree of cellulose polymerization extracted from the thermally modified pine wood would be analyzed.

The results of tests to determine the density, compressive strength and elastic modulus of the pinewood (*Pinus sylvestris* L.) samples are presented in an earlier publication [Zawadzki et al. 2013]. A significant decrease in the compressive strength and the modulus of elasticity was clearly visible at 200°C and with a duration time of 10 hours; a large decrease in wood density was also visible under these conditions. The results obtained also correspond well with the results of Kacikova et al. [2013] and Yildiz and Gumuskaya [2007]. The resulting dependence between the level of cellulose polymerization in the pinewood after heat treatment and its compressive strength and elastic modulus is shown in figures 3 and 4.

According to the data provided in figures 3 and 4, the degree of cellulose polymerization changed as a result of the heat treatment applied. The temperature of 160°C and heat treatment durations of 2, 6 and 10 hours did not significantly influence the compressive strength and modulus of elasticity, though the process of cellulose degradation took place and the degree of polymerization fell from 2000 to approximately 1000. The increase in treatment temperature to 200°C resulted in a significant decrease in the degree of polymerization. This change was particularly apparent at a temperature of 200°C and 10 hours of heating time. In such conditions, a significant decrease in the durability and elastic properties was observed, which was a result of the decrease in the degree of cellulose polymerization to 650.

According to the data provided, there was a direct dependence between the degree of polymerization of the cellulose extracted from the thermally modified pine wood and the examined mechanical properties of the wood. This dependence resulted, among other things, from the changes in the composition and amount of cellulose, which was subjected to degradation under the heat treatment conditions.
Fig. 3. Dependence between the degree of cellulose polymerization in pinewood after heat treatment and its compressive strength

Fig. 4. Dependence between the degree of cellulose polymerization in pinewood after heat treatment and its elastic modulus

Conclusion

To sum up, it can be stated that temperatures of 160°C and 200°C and the duration of the process, resulted in changes in the wood and the mechanical properties of the cellulose. A change in the molar mass and decrease in the degree of cellulose polymerization could be observed, as well as the lower compressive strength and elastic modulus of the pinewood. There was a direct and strong correlation between these data.
Increasing the treatment duration from 2 to 10 hours significantly influenced the degree of cellulose polymerization. The temperature of the heat treatment influenced the degree of cellulose polymerization more than the duration of the process. The decrease in the degree of cellulose polymerization did not cause a significant decrease in the compression strength and modulus of elasticity. For this reason, the heat treatment at a temperature of 160°C with duration up to 6 hours did not cause a decrease in the polymerization degree below 1000. Therefore, such a wood may still be used as construction wood but with increased resistance to biological factors.

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APPLICATION OF ADHESIVE JOINTS IN REINFORCEMENT AND RECONSTRUCTION OF WEAKENED WOODEN ELEMENTS LOADED AXIALLY

The paper concerns the formulation and analysis of an adhesive joint model, aimed at reinforcing or reconstructing weakened wooden elements. The joint is modeled as a plane stress problem of the theory of elasticity. It is assumed that wood is an orthotropic material. The reinforcement of an element is achieved by means of attaching a covering plate, while reconstruction is carried out by introducing an insert into the weakened (deteriorated) zone of an element. The influence of varying thickness of plates and inserts on the stress states in the adherends and adhesive is analyzed. The analyses are related to axially loaded elements.

Keywords: wood, orthotropy, adhesive joint, element reinforcement, reconstruction of weakened element, stress concentrations

Introduction

An adhesive joint is made of two adherends in a state of plane stress connected at common surfaces by an adhesive. It is assumed that the adherends and the adhesive have constant or moderately changing thickness.

The adhesive joint is modeled as a two-dimensional plane element parallel to the 0XY plane in a Cartesian set of co-ordinates 0XYZ. Projections of the adherends and adhesive onto the 0XY plane form identical figures of an arbitrary shape.

It is assumed that the bending effects in adherends are small and negligible. Thus, it is further assumed that stresses are constant across adherend thickness and form plane stress states parallel to the 0XY plane. The layout of an adhesive joint is presented in figure 1.

The thickness of the adherends is represented by functions \( g_1 = g_1(x, y) \) and \( g_2 = g_2(x, y) \). The mid-plane of the adhesive is given by the function \( s = s(x, y) \). Adhesive thickness \( t = t(x, y) \) is always larger than zero.

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Fig. 1. Layout of adhesive joint. 1 – adherend 1, 2 – adherend 2, 3 – adhesive

The adherends are made from orthotropic materials and the principal axes of orthotropy coincide with the X and Y axes. An orthotropic material in a plane stress state is described using the moduli of longitudinal deformation $E_{kx}$, $E_{ky}$, the shear modulus $G_{xy}$ and Poisson’s ratios $v_{kx}$, $v_{ky}$. The adhesive is modeled as a linearly elastic isotropic medium described using the following material constants: Young’s modulus $E_s$, shear modulus $G_s$ and Poisson’s ratio $v_s$, where $E_s = 2(1 + v_s)G_s$. The adhesive is subjected to shear stresses $\tau_x = \tau_x(x, y)$, $\tau_y = \tau_y(x, y)$ tangential to the adhesive mid-plane and stress $\sigma_N = \sigma_N(x, y)$ normal with respect to it. It is assumed that the stresses in the adhesive are constant across its thickness.

If an adherend thickness at its edge is more than zero, then we describe the edge as *unsharp*. Stresses acting at the unsharp edges of an adherend $k$ are denoted as $p_{kx}$ and $p_{ky}$ ($k = 1, 2$). It is assumed that stresses $p_{kx}$ and $p_{ky}$ are parallel to the X and Y axes, respectively, and that they are constant across the thickness. They are understood as external loading to the adherend edges. The thickness of an adherend along the entire edge or its fragment can be zero. In this case, the edge is called *sharp*.

Fig. 2. Cross-section at two types of sharp edges $K$: a – obtuse sharp edge, b – tangential sharp edge

If a sharp edge $K$ is defined by the external surfaces of two adherends forming an angle $\alpha > 0$, then the edge is called an *obtuse sharp edge* (fig. 2a). If a sharp edge has the external surfaces of two adherends, which are mutually tangential ($\alpha = 0$), then the edge is called a *tangential sharp edge* (fig. 2b). No boundary loading is defined at sharp edges.

Displacements of adherends 1 and 2 are given by the functions $u_1 = u_1(x, y)$ and $u_2 = u_2(x, y)$ in the direction of the X axis and by the functions $v_1 = v_1(x, y)$ and $v_2 = v_2(x, y)$ in the direction of the Y axis.
and \( v_2 = v_2(x, y) \) in the direction of the Y axis. The displacements \( u_1, u_2, v_1, v_2 \) are considered as unknowns. Equations of the theory of elasticity in displacements and boundary conditions for a plane stress state were formulated in research by Rapp [2010, 2015]. Having found the functions of the displacements \( u_1, u_2, v_1, v_2 \), the stress and strain states for the adhesive and adherends may be expressed.

The subject of the paper

An adhesive joint is considered with an adherend 2 loaded axially by a force \( N \), with an unloaded adherend 1 attached to it. If adherend 2 of a constant thickness has adherend 1 attached, then the total thickness of both adherends 1 and 2 is more than that of adherend 2 alone.

![Diagram of adhesive joint configurations](image)

**Fig. 3. Variants of reinforcement of adherend 2 using covering plates with: a – constant thickness, b – obtuse sharp edges, c) tangential sharp edges**

Such an adhesive joint can be treated as a reinforcement of adherend 2 using a *covering plate* (adherend 1). The considered variants of such reinforcement using covering plates with various edge shapes are presented in figure 3.

If the material in adherend 2 is locally damaged or there are voids, then these zones can be replaced with a new adherend 1 in such a way that the total thickness of the adhesive joint is equal to the original thickness of element 2. Such an adhesive joint can be considered a reconstruction of the cross-section of element 2 by means of an insert (element 1). Some variants of reconstruction with inserts with variously shaped edges are presented in figure 4.
Fig. 4. Variants of reconstruction of adherend 2 using inserts with: a – constant thickness, b – obtuse sharp edges, c) with tangential sharp edges

Zones for the anchoring of covering plates or inserts should be short. Stresses in adherends between these zones should be constant and equal and stresses in the adhesive equal to zero. The adhesive in the anchoring zones should be free of stress concentrations.

Meeting these conditions greatly depends on the edge type and the varying thickness of inserts and covering plates in the anchoring zones.

In this paper, the influence of the shapes of the covering plates and inserts on the stress state in the adhesive and adherends is analyzed. In addition, formulae for the anchoring zone length for inserts and covering plates are derived.

**Influence of covering plate and insert shape on stress state in joint**

Adherend 2 carries all the loading at the edges determined by \( x = \pm l_x \). In the range \( -l_x < x < l_x \) both adherends 1 and 2 carry the load. Stresses in the adhesive at the edges \( x = \pm l_x \) are relatively high. This section of the adhesive surface is considered the anchoring zone of the covering plate or insert. It is assumed that the adhesive joint in the anchoring zone carries a suitably large part of the load.

A stress state in the adhesive in the anchoring zone depends on adherend thickness at the edges \( x = \pm l_x \). For further analysis it is assumed that covering plates and inserts may have constant thickness \( g_1 = \text{const} \), as in figures 3a and 4a, varying thickness \( g_1(x, y) \) with obtuse sharp edges given by formulae (1) – as in figures 3b and 4b, or with tangential sharp edges given by formulae (2) – as in figures 3c and 4c.
An adhesive joint made of wooden adherends with two planes measuring $2l_1 \times 2l_2 = 10.0 \text{ cm} \times 8.0 \text{ cm}$, $g_1 = 0.2 \text{ cm}$ and $g_2 = 1 \text{ cm}$ is analyzed. In a timber trunk one can distinguish an element approximately characterized by plane orthotropy – for instance, a plank cut from a trunk in a radial plane (fig. 5b).

In such a plank, in a plane stress state, the principal directions of orthotropy coincide with the direction parallel to the wood grain $X = L$ and the radial direction perpendicular to the wood grain $Y = R$. It is assumed that in both the adherends the wood grain direction is parallel to the $X$ axis. The material constants for spruce wood were taken from Neuhaus [1994]:

- elasticity modulus in the direction parallel to the wood grain $E_x = 1.2 \cdot 10^6 \text{ N/cm}^2$,
- elasticity modulus in the direction perpendicular to the wood grain $E_y = 0.8 \cdot 10^5 \text{ N/cm}^2$,
- shear modulus $G_{xy} = 0.6 \cdot 10^5 \text{ N/cm}^2$.
Fig. 5. Wood anisotropy: a – anatomical directions, b – a plank in a radial plane

- Poisson’s ratios $v_{xy} = 0.03$ and $v_{zx} = 0.45$ (notation of $v_{xy}$, $v_{zx}$ by Rapp [2015]).

The following data were assumed for the adhesive: thickness $t = 0.04$ cm, $G = 0.45 \times 10^5$ N/cm², $E = 1.215 \times 10^5$ N/cm². Then $v = 0.35$.

It is assumed that the adhesive joints are loaded axially by forces $N = 8 \text{ N}$ in a form of a normal stress $1 \text{ N/cm}^2$ uniformly distributed along the edges $x = \pm l$, of adherend 2.

The loading $N$ yields the stresses $\tau_x$, $\tau_y$, and $\sigma_N$ in the adhesive and plane stress states $\sigma_{kx}$, $\sigma_{ky}$, and $\tau_{kxy}$ in the adherends ($k = 1, 2$). Force $N$ is carried by the adhesive as a stress $n$ as a resultant of the shear and normal stresses $\tau$ and $\sigma_N$, respectively, which are parallel to the X axis, and by the adherends as the normal stresses $\sigma_{1x}$, and $\sigma_{2x}$.

A two-dimensional boundary value problem for each adhesive joint presented in figures 3 and 4 was solved using the finite difference method. The presented results are restricted to the stresses $n$, $\sigma_{1x}$, and $\sigma_{2x}$ related to axial force $N$. They are given in figures 6-10 (no results for the joint in fig. 4a were given as they only differ from those in fig. 6 in magnitudes, see fig. 12c).

The extreme adhesive stress values $n$ are found at the covering plate or insert edges in the case of constant thickness (fig. 6a). At the obtuse sharp edges stress $n$ decreases by ca 50-60% (figs. 7a and 9a), while the extreme values are still located at the edges. However, stress $n$ at the adhesive surface is more flattened. Stress $n$ at the tangential sharp edges is equal to zero (figs. 8a, 10a) [Rapp 2015].

Covering plates and inserts with tangential sharp edges take the stresses from adherend 2 in a moderate way and the extreme stress $n$ is found in the anchoring zone. Thus, the risk of adhesive debonding at the edge is reduced. Extreme values of stress $n$ are lower than for obtuse sharp edges.

In the case of adherends of constant thickness the stress distributions $\sigma_{1x}$ and $\sigma_{2x}$ take the known shape. Stress $\sigma_{1x}$ increases from zero at the edge $x = \pm l$, and quickly reaches an approximately constant level between the anchoring zones (fig. 6b). In loaded adherend 2 stress $\sigma_{2x}$ at the edges $x = \pm l$, assumes the boundary values: $1 \text{ N/cm}^2$ (in the case of the covering plate) or $1.25 \text{ N/cm}^2$ (in
the case of the insert), then decreases and quickly levels up to a constant value as in adherend 1 (fig. 6c).

a – stress $n_x$  

b – stress $\sigma_{1x}$  

c – stress $\sigma_{2x}$

Fig. 6. Stresses due to axial force $N = 8$ N in a joint with a covering plate of constant thickness as in figure 3a. $n_x(\pm L_x, 0) = \pm 0.38215$ N/cm$^2$, $\sigma_{1x}(0, 0) = 0.83332$ N/cm$^2$, $\sigma_{2x}(\pm L_x, 0) = 1$ N/cm$^2$, $\sigma_{2x}(0, 0) = 0.83334$ N/cm$^2$

a – stress $n_x$  

b – stress $\sigma_{1x}$  

c – stress $\sigma_{2x}$

Fig. 7. Stresses due to axial force $N = 8$ N in a joint with a covering plate with obtuse sharp edges as in figure 3b. $n_x(\pm L_x, 0) = \pm 0.18173$ N/cm$^2$, $\sigma_{1x}(\pm L_x, 0) = 0.90865$ N/cm$^2$, $\sigma_{1x}(0, 0) = 0.83244$ N/cm$^2$, $\min \sigma_{1x}(x, 0) = 0.74923$ N/m$^2$, $\sigma_{2x}(\pm L_x, 0) = 1$ N/cm$^2$, $\sigma_{2x}(0, 0) = 0.83248$ N/cm$^2$

a – stress $n_x$  

b – stress $\sigma_{1x}$  

c – stress $\sigma_{2x}$

Fig. 8. Stresses due to axial force $N = 8$ N in a joint with a covering plate with tangential sharp edges as in figure 3c. $\max |n_x(x, 0)| = 0.14749$ N/cm$^2$, $\sigma_{1x}(\pm L_x, 0) = 1.5943$ N/cm$^2$, $\sigma_{1x}(0, 0) = 0.83339$ N/cm$^2$, $\min \sigma_{1x}(x, 0) = 0.71944$ N/m$^2$, $\sigma_{2x}(\pm L_x, 0) = 1$ N/cm$^2$, $\sigma_{2x}(0, 0) = 0.83345$ N/cm$^2$
Fig. 9. Stresses due to axial force $N = 8$ N in a joint with an insert with obtuse sharp edges insert as in figure 4b. $n_1(±L_e, 0) = ±0.18958$ N/cm$^2$, $\sigma_{1x}(±L_e, 0) = 0.96667$ N/cm$^2$, $\sigma_{1x}(0, 0) = 0.99971$ N/cm$^2$, min $\sigma_{1x}(x, 0) = 0.88205$ N/m$^2$, $\sigma_{2x}(±L_e, 0) = 1$ N/cm$^2$, max $\sigma_{2x}(x, 0) = 1.0246$ N/cm$^2$

Fig. 10. Stresses due to axial force $N = 8$ N in a joint with an insert with tangential sharp edges as in figure 4c. max$|n_3(x, 0)| = 0.17134$ N/cm$^2$, $\sigma_{3x}(±L_e, 0) = 1.5942$ N/cm$^2$, $\sigma_{3x}(0, 0) = 1.0001$ N/cm$^2$, min $\sigma_{3x}(x, 0) = 0.85296$ N/m$^2$, $\sigma_{2x}(±L_e, 0) = 1$ N/cm$^2$, max $\sigma_{2x}(x, 0) = 1.0330$ N/cm$^2$

Except for small fluctuations at the anchoring zones, stress level $\sigma_{1x}$ is flat and only very slightly exceeds the stress in the adhesive joint in both cases of obtuse sharp edges – with a covering plate or with an insert. Such a model is a most convenient way to reinforce or reconstruct a cross-section of adherend 2 (figs. 7b, 9b).

In the case of tangential sharp edges for covering plates or inserts, a convenient distribution of stress $n_3$ is accompanied by a large local increase in stress $\sigma_{3x}$ at the edges of adherend 1. In the case of the covering plate, it is ca 100% (fig. 8b), and for the insert – ca 75% (fig. 10b) of the mean stress value. This is due to the fact that adherend 1 is less thick at the sharp edges.
Anchoring length for covering plate and insert

For a joint between adherends of constant thickness (figs. 3a and 4a), the anchoring length of a covering plate or an insert can be assessed analytically using a one-dimensional model, where adherend 2 is under axial tension due to edge stresses $p_{2x}^o = \sigma$ and $p_{2x}^l = -\sigma$ ($\sigma > 0$), and adherend 1 is not loaded. The function of the shear stress in adhesive $\tau_2$ is given by a known relation

$$\tau_2(x) = -\frac{G_s\sigma}{tk_0E_{2x}\cosh k_0 l_x} \sinh k_0 x,$$

where

$$k_0^2 = \frac{G_s}{t} \left( \frac{1}{g_1 E_{1x}} + \frac{1}{g_2 E_{2x}} \right).$$

The distribution of function (3) is given in figure 11. There are regions limited by curve $\tau_2$ and the X axis along sections $l_{anch} = l_x - l_k$. An area of each region is a measure of the force carried by the adhesive joint between covering plate 1 and adherend 2 on the left and right ends of the joint. The sections $l_{anch}$ determine the anchoring zones on the adhesive surface and the length $l_{anch}$ is the anchoring length for a covering plate attached to the loaded element.

The length of an anchoring zone can be determined in various ways, for instance as a ratio $\tau_s(l_x) : \tau_s(l_k)$ between shear stresses at the ends of the section $l_{anch}$ or as a ratio between the area bounded by curve $\tau_2$ and the X axis along $l_{anch}$ and the entire area along the section [0, $l_k$].

According to the criterion defined by the ratio $\tau_s(l_x) : \tau_s(l_k)$ one gets

$$\tau_2(l_x) = -\frac{G_s}{tk_0E_{2x}} \sinh k_0 l_x \cdot \sigma, \quad \tau_2(l_k) = -\frac{G_s}{tk_0E_{2x}} \cosh k_0 l_x \cdot \sigma$$

and

$$\frac{\tau_2(l_x)}{\tau_2(l_k)} = \frac{\sinh k_0 l_x}{\sinh k_0 l_k} = \frac{e^{k_0 l_x} - e^{-k_0 l_x}}{e^{k_0 l_k} - e^{-k_0 l_k}}.$$

Usually $e^{-k_0 l_x}$ and $e^{-k_0 l_k}$ are small when compared to $e^{k_0 l_x}$ and $e^{k_0 l_k}$. If one neglects $e^{-k_0 l_x}$ and $e^{-k_0 l_k}$ in the expression (4) the following relation yields

$$\frac{\tau_2(l_x)}{\tau_2(l_k)} = \frac{e^{k_0 l_x}}{e^{k_0 l_k}} = e^{k_0(l_x - l_k)}.$$
Fig. 11. Stress distribution $\tau_x$ in adhesive for a joint with a covering plate loaded axially as in figure 3a

Introducing the anchoring length $l_{anch} = l_x - l_k$, one gets from (5)

$$l_{anch} = \frac{1}{k_0} \ln \frac{\tau_x(l_x)}{\tau_x(l_k)}.$$  \hspace{1cm} (6)

If $N_1$ denotes an axial force in a covering plate at the point $x = 0$, then in the one-dimensional model

$$N_1 = \int_{0}^{l_x} |\tau_x(x)| \, dx.$$  \hspace{1cm} (7)

According to the second criterion the anchoring force, denoted by $N_{anch}$, is defined as part of the force $N_1$

$$N_{anch} = p \cdot N_1,$$  \hspace{1cm} (8)

where $0 < p < 1$. It means that the anchoring zone carries $p \cdot 100\%$ of force $N_1$. The anchoring force $N_{anch}$ as a result of stress $\tau_x$ in the adhesive in the anchoring zone $l_{anch}$ can be given by the following formulae

$$N_{anch} = \int_{l_k}^{l_x} |\tau_x(x)| \, dx.$$  \hspace{1cm} (9)

Substitution of function $\tau_x$ from (3) to the relations (7) and (9) yields

$$N_1 = \int_{0}^{l_x} |\tau_x(x)| \, dx = \frac{G_s \sigma}{tk_0^2 E_{2x} \cosh k_0 l_x} (\cosh k_0 l_x - 1) =$$

$$= \frac{G_s \sigma}{tk_0^2 E_{2x} \cosh k_0 l_x} \left( \frac{e^{k_0 l_x} + e^{-k_0 l_x}}{2} - 1 \right),$$  \hspace{1cm} (10)
\[
N_{anch} = \int_{l_k}^{l_x} \tau_x(x) dx = \frac{G_s \sigma}{tk_0^2 E_{2x} \cosh k_0 l_x} (\cosh k_0 l_x - \cosh k_0 l_k) = \\
= \frac{G_s \sigma}{tk_0^2 E_{2x} \cosh k_0 l_x} \left( \frac{e^{k_0 l_x} + e^{-k_0 l_x}}{2} - \frac{e^{-k_0 l_k} + e^{k_0 l_k}}{2} \right). \tag{11}
\]

Neglecting the terms \(e^{-k_0 l_x}\) and 1 in (10), and \(e^{-k_0 l_k}\) in (11) one gets approximate formulae

\[
N_1 = \frac{G_s \sigma}{tk_0^2 E_{2x} \cosh k_0 l_x} \cdot \frac{e^{k_0 l_x}}{2} \quad \text{and} \quad N_{anch} = \frac{G_s \sigma}{tk_0^2 E_{2x} \cosh k_0 l_x} \left( \frac{e^{k_0 l_x}}{2} - \frac{e^{-k_0 l_k}}{2} \right)
\]

Substitution of these relations to condition (8) leads to

\[e^{k_0 l_k} = (1 - p)e^{k_0 l_x} \quad \text{and} \quad k_0 l_k = \ln(1 - p) + k_0 l_x.\]

From the equation \(l_{anch} = l_x - l_k\) one gets the following relation

\[l_{anch} = \frac{1}{k_0} \ln \frac{1}{1 - p}. \tag{12}\]

In this way two formulae, (6) and (12), defining the anchoring length for a covering plate in an axially loaded adhesive joint, depending on the definition of the anchoring zone, have been formulated. If the following condition is met

\[
\frac{\tau_x(l_x)}{\tau_x(l_k)} = \frac{1}{1 - p}. \tag{13}
\]
then they yield the same value of anchoring length.

For instance, if \(\tau_x(l_x) : \tau_x(l_k) = 100\), then relation (13) leads to \(p = 0.99\). This means that the anchoring zone carries 99% of the entire load acting on the adhesive joint. In this case, the anchoring length is

\[l_{anch} = \frac{\ln 100}{k_0} \approx 4.6 \cdot \frac{t g_1 g_2 E_{1x} E_{2x}}{k_0} \sqrt{G_s (g_1 E_{1x} + g_2 E_{2x})}. \tag{14}\]

If the width \(2l_x\) of the adhesive joint is large enough to have a plane stress strip in its central zone, then in the one-dimensional model with an orthotropic material one has to substitute

\[E'_{1x} = E_{1x} / (1 - \nu_{1xy} \nu_{yy}) \quad \text{and} \quad E'_{2x} = E_{2x} / (1 - \nu_{2xy} \nu_{yy}) \quad \text{for} \ E_{1x} \ \text{and} \ E_{2x}.
\]

Formulae (6), (12) and (14) give good approximations of the anchoring length of a covering plate \(l_{anch}\) for the one-dimensional model loaded axially,
a) One-dimensional plane stress model of a joint with a covering plate of constant thickness as in fig. 3a

\[
n_s(\pm l, 0) = \pm 0.39260 \text{ N/cm}^2 \\
l_{anch} = 1.95495 \text{ cm} \approx 1.955 \text{ cm}
\]

**Two-dimensional models**

b) A joint with a covering plate of constant thickness as in figure 3a

\[
n_s(\pm l, 0) = \pm 0.38215 \text{ N/cm}^2
\]

c) A joint with a covering plate with obtuse sharp edges as in figure 3b

\[
n_s(\pm l, 0) = \pm 0.18173 \text{ N/cm}^2
\]

d) A joint with a covering plate with tangential sharp edges as in figure 3c

\[
\max |n_s(x, 0)| = \pm 0.14749 \text{ N/cm}^2
\]

e) A joint with an insert of constant thickness as in figure 4a

\[
n_s(\pm l, 0) = \pm 0.46752 \text{ N/cm}^2
\]

f) A joint with an insert with obtuse sharp edges as in figure 4b

\[
n_s(\pm l, 0) = \pm 0.18958 \text{ N/cm}^2
\]

g) A joint with an insert with tangential sharp edges as in fig. 4c

\[
\max |n_s(x, 0)| = \pm 0.17134 \text{ N/cm}^2
\]

Fig. 12. Stress profiles \( n_s \) in adhesive of joints with covering plates and inserts loaded axially by force \( N = 8 \text{ N} \). Graphical comparison of anchoring lengths for covering plates and inserts.
if the adhesive joint has medium or small deformability. Then the values \( k_0 l_s \) and \( k_0 l_a \) are sufficiently high and the anchoring length is relatively short, which is important from a practical point of view. The accuracy of these remarks can be checked in the adhesive joint presented in figure 3a. For the plane stress state, one gets \( k_0 = 2.35564 \) l/cm. Then

\[
e^{k_0 l_s} = e^{11.7782} \approx 130380 \quad \text{and} \quad e^{-k_0 l_s} = e^{-11.7782} \approx 0.00000767.
\]

For \( p = 0.99 \), one gets \( l_{anch} = 1.95495 \) cm from (11). The anchoring length calculated for the adhesive joint with an insert, presented in fig. 4a, is \( l_{anch} = 1.91545 \) cm.

The anchoring length \( l_{anch} \) from the one-dimensional model represents a good approximation of the anchoring lengths for covering plates and inserts in two-dimensional models of adhesive joints loaded axially. For verification purposes, the distributions of stress \( n_x \) in the adhesive for the one-dimensional model and two-dimensional adhesive joints shown in figures 3 and 4 are presented in figure 12 on the same scale.

In figures 12a-e, the adhesive is parallel to the OXY plane, therefore \( n_x = \tau_x \). In the cases shown in figures 12f and 12g, the adhesive surfaces in the anchoring zones are curved. Thus, the distributions of stress \( n_x \) parallel to the X axis (stress \( n_x \) is a result of stresses \( \tau_x \) and \( \sigma_N \) ) are shown to enable a comparison of the results.

It can be seen in figures 6-10 that stresses \( \tau_x \) and \( \sigma_N \) (as well as \( n_x \) ) in the adhesive are almost constant along the Y axis. Thus, the graphs of the function of \( n_x \) along the X axis, presented in fig. 12, are representative. The anchoring length \( l_{anch} = 1.955 \) cm is measured to scale in figure 12a as calculated from a plane stress strip in the one-dimensional model, and it is depicted by dashed lines in the remaining figures 12b-g.

The length \( l_{anch} \) calculated from the one-dimensional model is a good approximation of the anchoring length in two-dimensional models.

**Reinforcement and reconstruction zones for an element**

For the assumed value \( p = 0.99 \) in the anchoring zone, the joint carries 99\% of force \( N_1 \), i.e. the total force carried by the joint along \( 0 \leq x \leq l_s \). It can be concluded from the equality \( N_1(x) + N_2(x) = N \) that in section \( 0 \leq x \leq l_s - l_{anch} \) the following inequalities hold:

\[
N_1 \geq N_1(x) \geq 0.99 \ N_1 \quad \text{and} \quad N - N_1 \leq N_2(x) \leq N - 0.99 \ N_1.
\]

Similarly, in section \( -l_s + l_{anch} \leq x \leq 0 \) the inequalities

\[
0.99 \ N_1 \leq N_1(x) \leq N_1 \quad \text{oraz} \quad N - 0.99 \ N_1 \geq N_2(x) \geq N - N_1
\]
are correct. Thus, in region \(-l_x + l_{anch} \leq x \leq l_x - l_{anch}\) stresses \(\sigma_{1x}, \sigma_{2x}\) in adherends 1 and 2 are approximately constant. In this zone of the joint, stress \(n_x = \tau_x\) is negligible or equal to zero (fig. 12), so displacements and strains in both adherends are approximately equal: \(u_1 \approx u_2\) and \(\varepsilon_{1x} \approx \varepsilon_{2x}\). Hence, for normal stress in the adherends the relation \(\sigma_{1x} : \sigma_{2x} \approx E_{1x} : E_{2x}\) is true.

If the adherends are made of identical materials, then in zone \(-l_x + l_{anch} \leq x \leq l_x - l_{anch}\), the normal stresses \(\sigma_{1x}\) and \(\sigma_{2x}\) in adherends 1 and 2 are approximately identical and constant, as illustrated in figs. 6-10.

The internal zone of adhesive joint \(-l_x + l_{anch} \leq x \leq l_x - l_{anch}\) can be considered a reinforcing zone for element 2 in the case of the covering plate or a reconstruction zone for the cross-section of element 2 in the case of the insert.

Conclusions

In the case of axial loading, extreme values of stress \(n_x\) in the adhesive occur at the edges of the covering plates and inserts. In the case of obtuse sharp edges, stress \(n_x\) is reduced by ca 50-75%. For tangential sharp edges, adhesive stress \(n_x\) is zero. A covering plate or an insert with tangential sharp edges takes stresses from adherend 2 in a moderate way and extreme stress \(n_x\) in the adhesive is located in the anchoring zone. Thus, the risk of debonding at the edges is reduced. The maximum values of stress \(n_x\) are then lower than the stress in the case of the obtuse sharp edges.

In the cases of adherends with constant thickness, stress \(\sigma_{1x}\) in the covering plates or inserts increases from zero at the edges \(x = \pm l_x\) and quickly stabilizes at an approximately constant level between the anchoring zones. Stress \(\sigma_{2x}\) at the edges \(x = \pm l_x\) of the loaded adherend 2 assumes boundary values and then decreases to a constant level as in adherend 1.

The level of stress \(\sigma_{1x}\) in the cases of covering plates and inserts having obtuse sharp edges is flattened except for insignificant fluctuations and it only very slightly exceeds the values of the stresses acting on the adhesive joint. Such a case is most efficient for reinforcing and reconstructing element 2.

In the cases of covering plates or inserts with tangential sharp edges, stress distribution \(n_x\) in the adhesive is advantageous. However, a local increase in stress at the edges of the covering plates and inserts is not. In the case of the covering plate, it amounts to ca 100%, and in the case of the insert – to ca 60% of the mean stress. The reason for this increase is the fact that adherend 1 is less thick at the sharp edges.

The anchoring length for the covering plates and inserts calculated from the one-dimensional model is a good approximation of the anchoring length in two-dimensional models.

An overview of problems related to reinforcing and reconstructing weakened elements in various technical fields can be found in Ahn and Springer [2000];
Bahei-El-Din and Dvorak [2001]; Kaye and Heller [2002]; Boss et al. [2003]; Kumar et al. [2006]; and Wang and Gunnion [2008].

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STRENGTH ENHANCEMENT OF TIMBER BEAMS USING STEEL PLATES – REVIEW AND EXPERIMENTAL TESTS

This paper reviews selected historical and modern methods of strengthening timber structures using steel plates. The results of experimental testing are also presented, emphasizing that the differences in the results obtained were a result of the application of different strengthening methods, test specimens, etc. The need to develop design guidelines related to reinforcement methods is also emphasized. Appropriate reinforcement depends on the application, arrangement and ratio of the reinforcement. The increase in the load-bearing capacity of the beams reinforced by bonded or glued-in plates in comparison to the unreinforced elements was found to be similar to the models reinforced with fibre reinforced polymer (FRP). The increase in stiffness was, however, higher than in the case of the FRP reinforcement. Due to the influence of temperature and humidity on the strength and deformation of the bonding if glued-in or bonded plates are used, additional strengthening of the connection using a mechanical connector is recommended. These connectors also protect against rapid destruction in the case of fire.

Keywords: timber structures, strengthening, repair, steel plates, epoxy

Introduction

Wood is the oldest building material used by man. In early times, timber structures were deeply rooted in human culture to be partially replaced later by stone, then by steel and concrete. Yet wood continues to be widely used as a building material. Numerous examples demonstrate that, in many cases, timber structures are better or safer to use – for example, when fire resistance is important. It is also significant that wood is a natural and renewable material and that the production of timber constructions consumes less energy than in the case of other building materials.

The mechanical properties of different wood species have been tested to indicate which constitute the best construction material [Krzosek et al. 2008].
Natural wood strength depends not only on anisotropic structure but also on the application, duration and conditions of loading, as well as on the wood type.

In some situations, a strengthening of the wood structure is essential. The need for strengthening is related to the natural properties and structure of the wood and the need to reinforce weaker areas with another material [Nowak et al. 2010].

The repair of timber structures usually involves the use of mechanical connectors, such as nails, screws, etc. The development of engineering materials has significantly influenced building and conservation technologies. Heavy-duty epoxy resins produced at the end of the 1960s were introduced in structural reinforcement interventions.

The reinforcement of timber structures using steel plates (both mechanically connected and bonded) is carried out in the following situations:

1. for the restoration of historical buildings,
2. to improve existing structures, where a planned extension may result in increased loading in relation to the original structure,
3. for the restoration of elements damaged during structural failure,
4. for seismic strengthening in both newly designed and existing timber structures (for improvement or restoration),
5. for newly designed structures, where the strengthening of weak areas is needed (for example, shear reinforcement).

Section 2 of the paper presents selected reinforcement methods involving the installation of steel plates with mechanical connectors, whereas Section 3 deals with strengthening with the use of glued-in or bonded plates.

Since the 1970s, research in Poland has been strongly associated with the restoration of historical buildings. Starting in the 1980s, tests were carried out on glued-in and bonded plates [Jasieńko 2003].

**Reinforcement with steel plates using mechanical connector techniques**

One of the oldest and simplest ways to reinforce damaged timber beams is to attach steel-plates (or steel profiles) to the sides. Hoath [2006] identified the Middle Ages as a time of historical precedent with respect to the strengthening of timber using steel elements. Plates were bolted or nailed onto the members. This method is commonly used to repair areas of support, for example, when the beam end is rotten. One example showing repair using a mortise and tenon connection between a primary beam and purlins that had been destroyed by insects, is the case of a roof structure built in 15th century [Hoath 2006]. Steel plates are connected to the top of the beam and placed on the sides of the purlins and additionally fastened by a mortise and tenon joint.

The 1960s marked the onset of investigations and tests concerning the use of steel and aluminum plates to reinforce timber elements. The goal of the tests
and associated research was to increase the strength and stiffness of wooden elements serving as reinforcement. The application of steel plates or steel profiles was one of the solutions tested, inter alia, by Stern and Kumar [1973], Coleman and Hurst [1974], and Hoyle [1975].

Hoyle [1975] tested elements discovered and patented by S.W. Lindal (steelam). The steelam beam was composed of lumber with toothed steel plates between the lumber pieces (fig. 1). One characteristic of steelam is that there are no visible fasteners on the sides or edges. Pressing is necessary to install the steel plates. The goal of the test was to confirm the method selected for a calculation of the stresses, fastener loads and deflection. Beams used in the test were made of seasoned western hemlock lumber, with maximum moisture content ranging from 17.5% (manufacture conditions) to 8.5% (test conditions). Drying was provided in an uncontrolled way, typical for indoor environments and using a force-dried method. The beams were 4.88 m (16ft) and 6.10 m (20 ft) in length with each one composed of two members and two A36 steel plates measuring 0.106 inches × 3.0 inches. The steel plates were situated in the top and bottom areas of the beams, along the entire length of the beam (except for the end-areas). The beams were tested cyclically (30 times), then some of them were tested to destruction. Examination after disassembling showed the partial buckling of the steel plates (as a result of spreading lamination) but failure was in the main due to lateral instability. No disconnection of the steelam beams was observed. The application of steel plates resulted in an increase in stiffness of 36% according to calculations, and of 34% according to the test. It can be concluded that the proposed method of calculation was correct and was verified by the test.

Steel plates were used by Coleman and Hurst [1974] as a local reinforcement in the form of:

- steel plates installed between timber members (fig. 2) which were used to resist shear stresses,
- sandwich beams with steel profiles on the top and the bottom of the beam (fig. 3) which were used to resist bending stresses.

The beams were produced from Southern Pine and connected together using nails or – glue-nail connections. The conclusions of the test provided by Coleman, when compared to unreinforced beams (for the moment and shear), were:

- that the ultimate strength of the beams connected using nails was 8-22% higher,
- that the ultimate strength of the glued and nailed beams was 22-37% higher.

In addition, a decrease in deflection (nailed beams – 30-35%, glued and nailed beams – 40%) was obtained through strengthening. Studies have shown the possibility of applying partial reinforcement to reduce both costs and labor input in order to achieve the desired result.
Another well known method, used since the end of the 19th century, involves flitch beams with a sandwich configuration: timber-steel-timber (fig. 4a). In this type of beam, the timber takes the load in proportion to the EI values. The sandwich is connected together using mechanical fasteners. The earliest beams were connected using bolts and the height of the steel plate was lower than the height of the timber members. Nailed connections were subsequently examined [Alam and Ansell 2012].

Tests on flitch beams have continued. Hairstans et al. [2004] presented the results of tests conducted on sandwiches made of timber or Kerto S Laminated Veneer Lumber (LVL) and 3 mm steel plates. Elements were connected using shot fired dowels with a diameter of 3.6 mm and a length of 60 mm. Unlike the traditional flitch beam (fig. 4a), dowels penetrated the second timber member to a depth of ca 12 mm (fig. 4b). Compared to typical flitch beams, where pre-drilling is necessary, the method described by Hairstans et al. [2004] is less time-consuming and therefore cheaper.

The effects of varying nailing density upon the flexural properties of flitch beams were presented by Alam and Ansell [2012]. The results of both the tests and calculations showed a relationship between the density of the nails and flexural stiffness. An increased density of nailing
increases the flexural stiffness but, on the other hand, also causes a reduction in the flexural strength.

Wood is anisotropic which results in different mechanical properties depending on the direction of the grain. For example, the compressive strength perpendicular to the grain is a few times lower than the corresponding strength values parallel to the grain. Tests investigating the differences between wood enhanced by nail plates and unreinforced elements were carried out by Kevarnamäki [1992]. The tests were conducted using three different sorts of nail plates, installed near the end support and on the intermediate support of multi-span beams. 133 enhanced and 42 reference (unreinforced) specimens were tested. It was concluded that the plates placed in the direction of the force offered the best reinforcement capacity. The load carrying capacity of the support area of members 45 mm in width, strengthened in this way, increased by 96%. In the case of plates installed horizontally in the area of support, an increase of 60% in the load capacity was observed. Kevarnamäki also showed that an increase in the load capacity of reinforced elements depends on the width of the members and the type of reinforced material. Reference tests were performed on elements measuring 45 mm in width, elements 70 mm in width, on glulam elements 45 mm in width and on elements made of LVL with a width of 39 mm.

**Strengthening with glued-in or bonded steel plates**

Epoxy adhesives injected into timber constructions are not only used for structural reinforcement, injections, consolidation or to restore geometric cross-sections [Avent 1986; Cruz and Custódio 2010; Custódio et al. 2011; Bertolini et al. 2013], but also to bond the connections between the reinforcing elements and the elements being reinforced [Arriaga et al. 2013; Cavalli et al. 2014; Yang et al. 2015].

One of the methods used to repair or reinforce timber structures involves the application of steel bars and plates [Leijten 1987; di Alamio et al. 2010; Jasieńko and Nowak 2014], steel cords [Borri and Corradi 2011], FRP bars and tapes [Nowak et al. 2013] or composite materials based on natural fibres [Ratery and Kelly 2015]. The reinforcement can be applied to outer surfaces or inserted inside elements: along the entire length, only in the weakened parts or at the ends [Jasieńko 2001, 2003]. Inserting the reinforcement inside the elements enables this method to be used in the restoration of old historical structures – for example, to repair and reinforce decorative ceilings. Placing the reinforcement inside the beam sections reduces the possibility of delamination of the bonding between the timber and the reinforcement [Metelli et al. 2016], as well as increasing fire resistance compared to an element without external reinforcement.
One of the oldest ways of reinforcing timber structures (using adhesives) involves bonding steel plates, aluminum sheets, as well as rods, placed inside the beam sections. This method was used by Silker [1962] in the early 1960s. Static bending tests were carried out on 15 beams of 2133.6 mm (84 in) in length and cross-sections, which is presented in figure 5. The beams were made of light-weight softwood lamelas: True fir, Eastern Spruce and Lodgepole Pine, glued together using resorcinol adhesive. The aluminum sheets used as reinforcement were 1.6 mm (1/16 in) thick, 127 mm (5 in) wide and 2133.6 mm (84 in) long, and were bonded into the wood with epoxy resins. The beams were tested horizontally (fig. 5 – beams A, B, C, D) and vertically (fig. 5 – beams E, F, G) laminated. Analysis of the composites showed an increase in stiffness in comparison to the beams without any reinforcement, as well as an increased loading capacity at the proportional limit. A reduction in the stiffness and strength variation among beams of the same size was also observed. The strength of the beam reinforced for tension and compression was determined by the shear strength of its section. Horizontally laminated beams with reinforcement were found to be in line with the theoretical stiffness. Vertically laminated beams did not demonstrate such a good correlation with the theoretical assumptions. The difficulty also involves inter alia finding ways of connecting two materials which have very different structures and mechanical properties.

![Fig. 5. Sections of beams reinforced with aluminum [Silker 1962], (mm)](image)

The idea of reinforcing glued laminated timber beams appeared in 1960s and 1970s. Research was carried out by Borgin et al. [1968], who placed steel plates inside the beam section using epoxy adhesives. The reinforcement was placed only in the compressed part or symmetrically, on the top and the bottom (fig. 6). Beams reinforced only in the compressed zone were abruptly destroyed in the tension zone. Borgin concluded that it is necessary to use laminations with the highest mechanical properties in the bottom, outer zone of the laminated beam, as reinforcement. The research also showed a significant increase in shear strength in one-sided reinforced beams compared to beams with symmetrical
reinforcement. This reinforcing method, however, increases the strength and stiffness of the elements. Borgin recommended the symmetrical reinforcement of beams using steel with a low module of elasticity and a large elastic range.

Steel plates can be also used to pre-stress the wood sections. As the pre-stressing of beams using steel does not improve the stiffness, Peterson [1965] devised a method of strengthening glued laminated beams with a pre-stressed strip of high-strength steel applied to the bottom face of the beam (fig. 7). This way of installing the reinforcement was supposed to increase the stiffness of the beam. According to Ganowicz and Latusek [1978], the lack of visible influence of the strips’ pre-stressing on beam stiffness was due to the fact that the stressing force was simply transferred to the end face of the beam, instead of to the whole length (as is the case with the tape). Tests were carried out on 6 reference (without reinforcement) and 6 prestressed beams. Figure 7 presents the section and the test stand scheme of the beams analyzed. The pre-stressed beams compared to the reference beams showed an increase in stiffness of approximately 26% and an increase in strength of ca 76%. It was also noted that the coefficient of variation decreased in the pre-stressed beams.

The need to strengthen glue joints with mechanical connections to guarantee the transmission of the load from the steel plates to the timber beam in case of loss of adhesion (fire or delamination) was pointed out by Metelli et al. [2013]. The paper presents an example of the application of bonded steel plates to reinforce ceiling beams (made of larch) in a floor dating back to the 15th century in Palazzo Calini (Brescia, Italy).

Rheological deformation is an important issue in the process of calculating the deflection of beams reinforced with glued-in steel plates. Neglecting rheological influence can lead to an underestimation of the final deflection, and rheological deformations can have values similar to elastic deformations [Misztal and Socha 1996].

Fig. 6. Sections of beams strengthened with steel tapes [Borgin et al. 1968], (mm)

Fig. 7. Section and test stand scheme of the analyzed beam [Peterson 1965], (mm)
The influence of temperature, humidity and dynamic loading (10,000,000 load cycles) on the connection of perforated steel plates bonded to timber elements was tested by Bathon et al. [2014]. The conclusion of the test was that this innovative coupling system can also be used in timber structures under fatigue loading conditions.

It is worth noting that the methods mentioned above are also used to reinforce concrete structures. The issue of bonding external steel plates with epoxy compositions to repair and strengthen concrete beams was researched for example by Swamy et al. [1987].

**Research concerning bending of timber beams strengthened with glued-in or bonded plates – materials and method**

Tests were carried out on timber beams measuring $100 \times 200 \times 4000$ mm made of pine wood (*Pinus sylvestris* L.). The beams for research were made of new wood (NW) and old wood (OW). The old wood was estimated to be 80-100 years old.

The models were strengthened using steel plates with a thickness of 2, 3, and 4 mm. Before their application the plates were sandblasted and carefully degreased with acetone. The epoxy composition used in the test contained the following:

- Epidian 5 resin – 100 parts by weight,
- quartz flour – 230 parts by weight,
- dibutyl phthalate – 10 parts by weight,
- Z-1 amine hardener – 11 parts by weight,
- Aerosil (optional) – 2 parts by weight.

Aerosil is a substance which stabilises the adhesive composition (thixotropic) and is only used to apply reinforcement to the outer surfaces of the beams.

The modulus of elasticity values of the used materials were determined in a bending test for wood, in a tension test for steel and a compression test for epoxy adhesive:

- new wood (NW) – 10 500 MPa (Std. dev. 1510),
- old wood (OW) – 9 500 MPa (Std. dev. 990),
- steel plates – 210 000 MPa (Std. dev. 6700),
- epoxy adhesive – 9800 MPa (Std. dev. 470).

The 3900 mm span beams were tested in a four point bending test (fig. 8). Fork supports (laterally positioned) were used to prevent bending stability loss (lateral buckling). For the strength test, a 600 kN capacity hydraulic jack was used to apply loading pressure. The rate of the monotonic pressure applied was 3 kN/min.
The research was carried out on 23 models made of new wood (NW) and 23 models made of old wood (OW). Each group of models comprised 7 different types. The beams were as follows:
- series A – reference (unreinforced) beams,
- series B – beams with epoxy adhesive introduced into the cross section,
- series C – beams with 2 steel plates inserted into the cross section;
- series D – beams with 2 steel plates with a height less than the full beam height, bonded to both side faces,
- series E – beams with 2 steel plates covering the full beam height, bonded to both side faces,
- series F – beams with 1 steel plate of differing thicknesses ($t_{plate} = 2; 3; 4 \text{ mm}$), bonded to the upper face,
- series G – beams with 1 steel plate bonded to the bottom face.

Figure 9 shows the cross sections of the tested specimens. Models B, C and F are useful in restoration work when it is possible to apply the reinforcement from the top. This method allowed the reinforcement of the construction without destroying the historical value and decorations (painted decorations, wood carvings, etc.) on the bottom and side faces.
The proposed method of gluing-in plates from the top into the section (series C) reduces the possibility of delamination of the bond between the steel plates and the timber, and increases the element’s fire resistance.

Results and discussion

Tables 1 and 2 present the average values of the ultimate force (the $F_u$ mean) for each individual series, as well as the proportional increase in strength ($\Delta F$) in comparison to the reference beams from set A, based on equation (1). The beams from series F were named F-2, F-3 and F-4 according to the thickness of the plate used ($t_{plate} = 2; 3; 4$ mm). Tables 1 and 2 also show the mean increase in stiffness ($\Delta E I$) of the reinforced beams in comparison with the unreinforced reference beams for force ranging from 6 kN to 18 kN.

$$\Delta F_u = \frac{F_{u,mean,F} - F_{u,mean,A}}{F_{u,mean,A}} \times 100\%$$  \hspace{1cm} (1)

<table>
<thead>
<tr>
<th>Beam series</th>
<th>Number of specimens</th>
<th>Ultimate force $F_u$(kN)</th>
<th>Increase in load-bearing capacity $\Delta F_u$ (%)</th>
<th>Increase in stiffness $\Delta E I$ (%)</th>
</tr>
</thead>
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<tr>
<td></td>
<td></td>
<td>min value</td>
<td>max value</td>
<td>mean value</td>
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<tr>
<td>A</td>
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</tr>
<tr>
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<td>3</td>
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<td>50.7</td>
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<tr>
<td>C</td>
<td>3</td>
<td>57.6</td>
<td>60.4</td>
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<tr>
<td>D</td>
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<td>54.4</td>
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</tr>
<tr>
<td>E</td>
<td>3</td>
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</tr>
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<td>G</td>
<td>2</td>
<td>35.7</td>
<td>38.9</td>
<td>37.3</td>
</tr>
</tbody>
</table>

Figures 10 and 11 show the load-deflection plot for the beams from all the series. For a comparison, the area designated by the load-deflections plot of the reference (unreinforced) beams (series A) made of new wood (NW) and old wood (OW) are shown. The vertical lines on the graphs show the limit deflection for ceilings with the limit value of $L/250$. Additionally, the graphs show deflection in the value of $L/167$, which increased 50% for the old renovated buildings, in accordance with the Polish annex to Eurocode 5 [PN-EN 1995-1-1: 2010].
Table 2. Ultimate force, increase in load-bearing capacity and increase in stiffness of tested beams made of old wood (OW)

<table>
<thead>
<tr>
<th>Beam series</th>
<th>Number of specimens</th>
<th>Ultimate force $F_u$ (kN)</th>
<th>Increase in load-bearing capacity $\Delta F_u$ (%)</th>
<th>Increase in stiffness $\Delta EI$ (%)</th>
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</thead>
<tbody>
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<td></td>
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<tr>
<td>B</td>
<td>3</td>
<td>34.5</td>
<td>36.9</td>
<td>35.4</td>
</tr>
<tr>
<td>C</td>
<td>3</td>
<td>51.0</td>
<td>53.1</td>
<td>52.1</td>
</tr>
<tr>
<td>D</td>
<td>3</td>
<td>48.5</td>
<td>50.7</td>
<td>49.7</td>
</tr>
<tr>
<td>E</td>
<td>3</td>
<td>48.7</td>
<td>50.6</td>
<td>49.7</td>
</tr>
<tr>
<td>F-2</td>
<td>2</td>
<td>34.2</td>
<td>36.1</td>
<td>35.2</td>
</tr>
<tr>
<td>F-3</td>
<td>2</td>
<td>35.0</td>
<td>36.4</td>
<td>35.7</td>
</tr>
<tr>
<td>F-4</td>
<td>2</td>
<td>40.4</td>
<td>42.1</td>
<td>41.3</td>
</tr>
<tr>
<td>G</td>
<td>2</td>
<td>36.1</td>
<td>37.3</td>
<td>36.7</td>
</tr>
</tbody>
</table>

Fig. 10. Load–deflection plot for specimens from new wood (NW)

The tests results were more qualitative than quantitative in nature because of the small number of samples in each series (especially series F and G - 2 beams each).

The increase in the strength of the beams tested (up to 100% – tabs. 1 and 2) when compared to the reference beams – both made of new wood (NW) and old wood (OW) – was comparable to the models reinforced with FRP [Borri et al. 2005; Nowak et al. 2013]. The increase in stiffness was higher than in the beams reinforced with FRP. In the tests described here, the increase in strength in the beams reinforced with FRP was 10-77% (tabs. 1 and 2). However, there are other aspects other than the increase in stiffness and strength that determine the advantage of using FRP over steel, such as the weight and corrosion resistance, which is important for the quality of steel-wood bonding [Ishiyama et al. 2014]. Placing steel plates inside the wood section also provides corrosion resistance.
The technical simplicity and the test results suggest that the method of strengthening beams used with series C is likely to become common in the restoration of historical buildings. Failure of series C beams proceeded as follows: the first break occurred in the tension zone, followed by successive fractures in the compressed zone. No separation of the steel plates occurred. Due to the steel reinforcement, no rapid destruction was observed.

Conclusions

The following general conclusions regarding timber member reinforcement using steel plates were formulated based on analysis of the available literature and on the basis of research and experimental testing:

1. Appropriate strengthening depends on the reinforcement ratio and the arrangement of the reinforcement (steel, FRP, etc.) [Raftery and Whelan 2014]. As Kliger et al. [2007] stated, reinforcement can be used to control the strength, stiffness and failure modes.

2. Glue joints assure the continuity of the cross section and a uniform interaction between the original elements and the reinforcing ones, which is practically impossible to achieve using mechanical connectors only.

3. The application of steel plates connected mechanically is less effective than those glued-in. Lower efficiency is caused by, inter alia, the weakening of a section by drilling holes for bolts. It should be mentioned here that using glued-in plates or bolts - the glue penetration in the eventual cracks can help maintain the structure of the beam but can also change the local stiffness of the member. Coleman and Hurst [1974] indicated that the stiffness of the nailed steel-reinforced beams was ca 50% higher than when using glued and nailed steel-reinforced beams,
which caused a doubling of the stiffness. Nonetheless, mechanically connected plates can be easier to install in building site conditions and regardless of weather conditions.

4. The increase in the load-bearing capacity of the beams reinforced with bonded or glued-in plates in comparison to the unreinforced elements was comparable to the models reinforced with FRP. The stiffness increase was, however, higher than in the case of FRP reinforcement [Alam et al. 2009].

5. FRP reinforcement is more expensive than steel and the load-bearing capacity utilization of this reinforcement does not usually exceed 20% [Jankowski et al. 2010], whereas with steel plates it is up to 80% [Jasieńko 2003]. It is also worth clarifying that steel plates need to undergo the difficult process of sandblasting in order to achieve the necessary adhesion to wood.

6. Due to the influence of temperature and humidity on the strength and deformation of the bonding if glued-in or bonded plates are used, additional strengthening of the connection by mechanical connector is recommended. These connectors also protect against rapid destruction in the case of fire.

7. It is extremely important to take into account the properties of the wood and materials used for reinforcement, inter alia, differences in the linear thermal expansion in wood and steel. These differences may cause checks in the timber members. External steel plates are not recommended for use in the area of skylights or other places, where plates can be exposed to strong sunlight.

8. Research on the behaviour of timber elements must take into account not only technical issues, but also the historical and artistic value of the object. Decisions to replace old structures with new ones are made too often and too easily. Such interventions are against the principles of historical building conservation. Changes in the loading of the structure, including the foundations, must be taken into consideration in all such cases prior to deciding to replace old elements. The impact resulting from the replacement of historical elements with new ones can cause cracks in the walls, an uneven settlement of the foundations, etc.

9. In conclusion, it is worth noting that work on reinforcement calculations is on-going, with the potential goal of adding essential design recommendations to the revised version of Standard Eurocode 5, as clear rules on reinforcement design are crucial.
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List of standards

Małgorzata KAJDA-SZCZEŚNIAK, Tomasz Józef JAWORSKI

ANALYSIS OF THE PROCESS OF COMBUSTION OF POST-CONSUMER WOOD USING QUANTITATIVE ASSESSMENT INDICATORS

The paper presents an analysis of the fuel and emission properties of selected types of post-consumer wood. The tested materials include flooring, wood door frames, and used furniture, as well as wood as a material for comparison. In addition, through the results of tests carried out in the laboratory, the paper presents possibilities related to the optimization of waste combustion plants equipped with combustion chambers with mobile grates. For this purpose, indicators of quantitative assessment were determined, i.e. the reaction rate, the fire point, mass loss during combustion, and the heat load of the grate.

Keywords: post-consumer wood, fuel properties, combustion, emission of gas combustion products, quantitative assessment indicators of waste combustion

Introduction

Together with a growing consumption of furniture and home decoration products in Poland, a growth in the amount of wood and wooden board waste has been observed. Problems with the proper management of post-consumer wood waste result among other things from its large dispersion and its variety in respect of amount and form [Cichy and Wróblewska 2003; Ratajczak and Szostak 2003; Danecki 2007]. Post-consumer wood waste includes packaging wood, pallets, wood processing waste, furniture, floors and door frames, and wooden construction elements. Due to the large variety of post-consumer wood waste, knowledge of chemical contamination is necessary. Substances most frequently used to treat wood include urea-formaldehyde, melamine-formaldehyde and phenol-formaldehyde resins, other glues, paraffin waxes, varnishes, veneers, foils and wood preservatives [Roffael et al. 2005; Nicewicz 2006; Danecki 2007; Wasilewski and Hrycko 2010]. Knowledge of which chemicals have been applied would make it possible to sort waste into groups and to suggest which is the best waste management method – recycling, thermal utilization or deposit [Danecki 2007; Cichy and Pawłowski 2009, 2010; Cichy

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This is an important issue, because in Poland an increase of 4% in the demand for wood and wood-like products has been observed. At the same time, a decrease in wood resources has been accompanied by an increase in wood and wood-like waste [3spare: reading on 29.05.2015]. It is estimated that the supply of post-consumer wood which comes only from the replacement of furniture and interior furnishings amounts to approximately 1.44-1.68 Mio m³ per year. On the basis of European indicators of the post-consumer wood market, it may be assumed that with the acquisition in 2005 of 29.7 Mio m³ of wood from forests, the amount of post-consumer wood for reuse in the period of one year would reach 2.8 Mio m³ and 5.7 Mio m³ for energy use [Danecki 2007]. The appropriate management of such wood waste could significantly contribute to a limitation of forest logging, a decrease in the amount of waste directed to landfills, and the recovery of energy and raw materials [3spare: reading on 29.05.2015].

One method for recovering energy from biomass and wood waste is combustion or co-combustion with traditional fuel. The combustion process seems to be simple. However, in reality, it is a complex process due to a number of physical and chemical phenomena taking place during combustion. The combustion of waste in industrial plants has been carried out for nearly 100 years. The characteristics of this process are totally different from those in the combustion of conventional fuels, such as coal or lignite. These differences are caused by the heterogenic content of waste, the large graining variety (different fractions) and regional and seasonal variations in the physiochemical properties of the waste. The combustion of waste is characterized by an unspecified and non-homogeneous process, as well as by the generation of detrimental substances. The possible, detrimental consequences of this process consist of slag formation, as well as the corrosion and erosion of the combustion device. Hence, it is necessary to assure constant optimization of the waste combustion plant operation [Marutzky and Schriever 1986; Nussbaumer 2003; Jaworski 2005, 2007; Cichy 2012; Nadziakiewicz et. al. 2012; Czop 2014; Kajda-Szcześniak and Nowak 2014].

Due to the above, this article deals with the issues related to the optimization of the combustion process, the goal of which is to decrease the risk of detrimental impact on the natural environment. Tests were carried out in the laboratory and they were pertained to a real combustion plant with application of quantitative assessment indicators.

**Indicators of quantitative evaluation of waste combustion**

The replacement of classic fuels with fuels based on wood waste allows, on the one hand, a reduction in CO₂ emissions (especially in respect of biodegradable mass, for example biomass) and, on the other, helps to avoid the deposition of natural fuels, following the rules of sustainable development. For the thermal
disposal of fuels produced from wood waste in incineration or heat plants operating with coal and biomass, etc, it is necessary to define and determine certain characteristic indicators in order to evaluate the combustion process. These indicators should serve as a basis for a comparison between waste fuels and with classic fuels. In addition, perhaps more importantly, they could serve as some sort of “criteria numbers” allowing the transfer of values from the laboratory device, which works on an irregular basis, to the actual industrial device, which works constantly with determined parameters and is equipped with grated firing. It would provide many opportunities for the optimization of exploitation parameters of incineration plants, especially plants equipped with a mobile grate. The following indicators of the quantitative evaluation of the combustion of fuels have been proposed: reaction rate, ignition rate, combustion mass loss and heat load on the grate [Jaworski 2012].

Temperature sensors were placed at the height of the layer which was the subject of the process of combustion on the grate, in order to determine the maximum increase in temperature:

$$\frac{\Delta \theta}{\Delta t} = \left(\frac{\Delta \theta}{\Delta t}\right)_{\text{max}}$$

in each thermo-element of the fuel layer indicating the location of the area of reaction or flame. Such a reaction in the fuel deposit is described as the reaction rate and marked as:

$$u_{FR} = \frac{dx_{FR}}{dt} \left[ \frac{\text{m}}{\text{s}} \right]$$

where: $x_{FR}$ – location of the front of reaction, [m].

The velocity of ignition (SZ) indicates the mass stream of fuel per unit time, which undergoes ignition per unit area.

$$SZ = u_{FR} \cdot \rho_n \left[ \frac{\text{kg}}{\text{m}^2 \cdot \text{s}} \right]$$

$\rho_n$ – bulk density, [kg/m$^3$].

This indicator describes the combustion parameters in grated industrial devices. For fuels with greater ignition velocity, the mass flux per area unit should be increased in order to maintain the combustion process. For fuels with lower ignition velocity, the mass flux should be decreased or the air should be heated for faster drying [Jaworski 2012].

Mass loss rate (SUM) is determined by the mass loss in time and per unit area of the grate

$$SUM = \frac{\Delta m_{\text{fuel}}}{A_R} \left[ \frac{\text{kg}}{\text{m}^2 \cdot \text{s}} \right]$$

$A_R$ – area of the grate, [m$^2$].
With this indicator, the length of the combustion zone in industrial devices can be calculated, if the stream of fuel and the area of the grate are known. This indicator describes the relationship between the ignition velocity and the related real fuel mass loss. For fuels with a much faster ignition than the value of the mass loss rate, there is the risk that the unburned fuel would be moved to the end of the grate.

The heat load of the grate (OCR) indicates how much of the energy from the fuel will be released during its oxidation in a given period of time and on a given area of the grate (it may determine the way of cooling) [Jaworski 2012].

$$OCR = \text{SUM} \cdot \frac{W_d}{m^2} \quad (5)$$

$W_d$ – lower calorific value of the fuel, [kJ/kg].

Observation of the indicator of the heat load of the grate prompts technical service staff to react in case of grate overload.

**Investigated materials and test methods**

**Scope of the research**

The paper presents an analysis of the physicochemical properties of selected post-consumer wood waste. The goal of the analysis was to determine specific fuel properties, such as moisture, ash content, content of volatile components, flash point, calorific value and elemental composition (C, H, O, N, S, Cl).

In addition, possibilities for the optimization of the exploitation parameters of industrial devices equipped with a mobile grate are presented. This is estimated through the results of tests carried out in the laboratory. For this purpose, previously defined indicators of the quantitative evaluation of waste combustion were determined, i.e. the reaction rate, ignition rate, loss of combusted mass and heat load on the grate.

**Material for tests**

The tests covered the following types of post-consumer wood:

- O-I – post-consumer furniture, box furniture based on wood derived slabs with a matt front. The exploitation time of the furniture was approximately 40 years.
- O-II – mixed floor panels of wear ratings AC3, AC4 and AC5 in the proportions 1:1:1,
- O-III – mixed door frames, in the proportions 1:1 (a door wing made of MDF, covered with veneer on the outer side, mixed with a door wing made of MDF frame, filled with honeycomb shaped cardboard, covered with varnish on the outer side),
- O-IV – wood from coniferous trees.
The wooden materials used for the tests were crushed using a Trymer T45,5SW mill equipped with a mesh measuring Ø14 mm in diameter.

**Physicochemical properties**

The moisture content was determined according to the standard PN-Z-15008-02:1993 by drying method at a temperature of 105°C. The ash content was determined according to standard PN-Z-15008-03:1993, and the content of volatile matter was determined in compliance with standard PN-G-04516:1998. The ignition temperature was determined in accordance with standard PN-EN ISO 2592:2008 using a Cleveland open-cup tester. The heat of combustion was determined following the procedure described in PN-ISO 1928:2002.

The carbon and hydrogen content was determined according to standard PN-Z-15008-05:1993. It consisted of the total combustion of a sample in a stream of oxygen, and determination of the mass, water and carbon dioxide content in the flue gas.

The nitrogen content was determined by Kjeldahl method, according to standard PN-G-04523:1992.

The sulphur content was determined using the Eschka method, on the basis of the standard PN-ISO 334:1997. The chloride content was determined by the Mohr method using an Eschka mixture in compliance with standard PN-ISO 587:2000.

**Experimental stand**

A schematic diagram of the testing stand used is presented in figure 1.

The stand consisted of an FCF 30 RP chamber furnace with 5 kW power, according to the DTR (operation and maintenance manual). The furnace was equipped with a specially designed grate with equipment for measuring the parameters inside the layer of combusted waste. Furthermore, the post was equipped with an exhaust analyzer allowing the measurement of the composition of the exhaust gas. It also had a weighbridge which registered the mass loss of waste during the combustion process.

**Determination of the content of exhaust gases**

For the measurement of exhaust fumes, the Madur GA-40Tplus portable analyzer was used. The analyzer enabled an analysis of the concentration of six gases (O₂, CO₂, CO, NO₃, SO₂). Additional measurements during combustion process

Temperature measurements inside the layer of the combusted post-consumer wood waste were taken with the use of three thermocouples, placed in the body of the working grate at layer height. The first thermocouple was placed at a height of 50 mm from the bottom of the grate, the second at 150 mm, and the third at 250 mm. The distance between the thermocouples was 100 mm. The
temperatures were recorded using an electrical measuring device with a testing interval of 1 minute.

![Diagram of the testing stand for assessment of the fuel combustion process](image)

**Fig. 1. Schematic of the testing stand for assessment of the fuel combustion process [Jaworski 2012]**

**Testing process**

Tests of the combustion process within the layer were carried out in devices in a laboratory scale. The tests were performed according to the following scheme:

a. preliminary tests, which consisted of:
   - preparation of fuel for combustion, determination of the mass of combusted waste,
   - determination of the bulk density of the layer of combusted waste,
   - determination of the area of the grate,
   - determination of the parameters of the combustion process, i.e. temperature, process time, amount of air supplied to the combustion chamber,
   - necessary preliminary calculations of the combustion process in order to determine the stream of air for the assumed process time and the coefficient of the surplus air,

b. actual tests covered:
   - attainment of the process temperature within a range of 850-900°C, accounting for 20 minutes of furnace relief at 350°C,
- activation of the primary air fan; setting primary air stream with a rotameter within a range of 18-19 m³/h,
- removal of the dummy grate and placing of the working grate with the waste in the furnace,
- measurement of temperatures inside the layer of combusted waste,
- measurement of the concentration of exhaust fumes,
- measurement of the mass loss of the combusted waste,
- constant measurement of the time which enabled measurement of the parameters at the same time,
- the test was terminated by disconnecting the furnace from the power supply, disconnecting the measuring devices, disconnecting the air fan, removal of the working grate from the furnace, and placing the dummy grate in the furnace.

Results and discussion

Physicochemical properties of tested waste

The results of the physicochemical analysis of the selected post-consumer wood waste are shown in table 1.

Table 1. Physicochemical properties of tested waste

<table>
<thead>
<tr>
<th>Property</th>
<th>Unit</th>
<th>O-I</th>
<th>O-II</th>
<th>O-III</th>
<th>O-IV [Kaltschmitt and Hartmann 2001]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture content</td>
<td>%</td>
<td>7.03</td>
<td>5.08</td>
<td>6.36</td>
<td>–</td>
</tr>
<tr>
<td>Ash, A²</td>
<td>%</td>
<td>0.88</td>
<td>1.02</td>
<td>0.73</td>
<td>0.60</td>
</tr>
<tr>
<td>Volatile components, V⁴</td>
<td>%</td>
<td>77.60</td>
<td>79.03</td>
<td>79.29</td>
<td>82.90</td>
</tr>
<tr>
<td>Flash point</td>
<td>°C</td>
<td>187.00</td>
<td>201.50</td>
<td>219.00</td>
<td>–</td>
</tr>
<tr>
<td>Heat of combustion, W⁄₂ [MJ/kg]</td>
<td>22.07</td>
<td>20.16</td>
<td>16.29</td>
<td>20.20</td>
<td>–</td>
</tr>
<tr>
<td>Calorific value, W⁄₃ [MJ/kg]</td>
<td>20.95</td>
<td>19.22</td>
<td>15.40</td>
<td>18.80</td>
<td>–</td>
</tr>
<tr>
<td>Carbon, C⁴</td>
<td>%</td>
<td>51.51</td>
<td>51.24</td>
<td>51.22</td>
<td>49.80</td>
</tr>
<tr>
<td>Hydrogen, H²</td>
<td>%</td>
<td>5.00</td>
<td>4.40</td>
<td>4.22</td>
<td>6.30</td>
</tr>
<tr>
<td>Oxygen, O⁴</td>
<td>%</td>
<td>35.29</td>
<td>29.55</td>
<td>33.75</td>
<td>43.20</td>
</tr>
<tr>
<td>Nitrogen, N⁴</td>
<td>%</td>
<td>6.88</td>
<td>13.23</td>
<td>9.59</td>
<td>0.13</td>
</tr>
<tr>
<td>Sulphur, S⁴</td>
<td>%</td>
<td>0.26</td>
<td>0.19</td>
<td>0.28</td>
<td>0.02</td>
</tr>
<tr>
<td>Chloride, Cl⁴</td>
<td>%</td>
<td>0.18</td>
<td>0.36</td>
<td>0.22</td>
<td>0.01</td>
</tr>
</tbody>
</table>

– No data
– The oxygen content was determined by calculation

The results show that the tested post-consumer wood waste O-I – O-III were similar regarding their physicochemical properties. The moisture content of all the tested waste was low and fell within the range, from 5.08% for waste O-II to 7.03% for waste O-I. All the tested waste had very low ash content, not exceeding 1.02%. Analysis of the fly ash content showed slight differences between the waste O-I, O-II and O-III. The content of volatile components in the tested waste ranged from 77.60% to 79.29%.
One of the most important parameters was the calorific value calculated on the basis of the determined combustion heat, hydrogen content and moisture content. It was noted that waste O-I had the highest calorific value equalling 20.95 MJ/kg. For the other waste, the calorific value was lower and amounted to 19.22 MJ/kg for waste O-II and 15.40 MJ/kg for waste O-III. The marked flash point fell within a range of 187°C for waste O-I to 219°C for waste O-III.

The analysis concerning a determination of the content of chemical elements proved that the basic elements which formed part of the selected waste from the wood-based panels were C, H, O and N. The carbon content of the tested waste (O-I - O-III) was equal 51%. The content of hydrogen in the tested waste was below 5%. On the basis of the executed tests, a high content of nitrogen was identified in all the analyzed waste. This value was the lowest for waste O-I at 6.88% and the highest for waste O-II at 13.23%. The nitrogen content may have resulted from the existence of urea- and melamine-based resins, lacquers and coatings, as Wandrasz and Wandrasz [2006] found that the content of nitrogen in urea-formaldehyde resins amounts to 37%, and in melamine-formaldehyde resins it reaches 48%. According to Cichy [2012], the content of nitrogen in post-consumed timber from Polish manufactured products falls within a range of 1.5% to 7%, while for urea-resin bonded plywood this value is approximately 8%.

The sulphur content of the tested waste was at a low level, below 0.28%. The recorded chloride content ranged from 0.18% to 0.36%. The waste assortments from the wood-based panels had similar properties to wood O-IV. An exception from this finding was the nitrogen content. The wood also had a lower content of sulphur and chloride.

**Emission properties of tested waste**

The results of the measurements are presented in figures 2-5. The emissions from the combustion of wood-derived waste were compared to the results of the combustion of pure wood.

During the combustion process, an increased emission of nitrogen oxides for waste O-I, O-II and O-III was recorded. This was due to the application of adhesive resins at the production stage of the wood-derived products, which generated a high nitrogen content in the elemental composition. The results confirm that waste of this type should be burned in devices with a properly selected exhaust purification system, which will bring advantages in terms of the environment and energy. In figure 2 it can be seen very clearly, and also in figures 3 and 4, a sufficiently elongated afterburning stage (extended process time) which is shown in the slow decrease in CO₂ concentrations until the values approach zero. The reason was the extended presence of solid combustible particles in the combusted layer of waste, which came from more complex resins and resins which were more difficult to decompose – a component of the tested waste.
Fig. 2. Change in composition of exhaust gases during the combustion process of waste O-I

Fig. 3. Change in composition of exhaust gases during the combustion process of waste type O-II
Fig. 4. Change in composition of exhaust gases during the combustion of waste type O-III

Fig. 5. Change in composition of exhaust gases during the combustion of waste type O-IV

**Distribution of temperatures in solid and gas phases in the combusted waste layer**

The tests of the distribution of temperatures in solid and gas phases in the combusted waste layer on the grate are shown in figures 6-9. The measurements were taken at three given heights – 50 mm from the bottom of the grate, 150 mm and 250 mm.
Fig. 6. Distribution of temperatures in solid and gas phases in the combusted layer of waste type O-I during the combustion process at a temperature of 850°C.

Fig. 7. Distribution of the temperature in solid and gas phases in the combusted layer of waste O-II during the combustion process at a temperature of 850°C.
Fig. 8. Distribution of the temperature in solid and gas phases in the combusted layer of waste O-III during the combustion process at a temperature of 850°C

Fig. 9. Distribution of the temperature in solid and gas phases in the combusted layer of waste O-IV during the combustion process at a temperature of 850°C
Initially the temperatures increased over a certain period of time and then decreased, all reaching similar values in the final phase of the combustion process. This phenomenon was caused by the combustion of the waste in the layer. On the basis of the measurements, it was possible to calculate the following indicators: the reaction rate and the ignition rate. In each case, the highest temperature was noted on a thermocouple placed at the highest position in the layer. However, the largest increase in temperature in the initial phase (up to approximately 600°C) was recorded by the middle thermocouple. The most probable explanation for this finding is that this location had the best combustion conditions, i.e. a suitable temperature and an availability of oxygen, whose deficiency in the top layer of the waste suppressed the total and complete combustion process.

Figure 10 shows how the mass of tested waste was reduced during the combustion process.

![Figure 10](image-url)  
**Fig. 10. Mass loss of the combusted layer of waste O-I – O-IV during combustion, at a temperature of 850°C**

For waste O-I – O-III, the biggest mass losses were noted in the first 16 minutes of the combustion process, but for waste O-IV they were recorded after no more than 5 minutes. At the end of the process, the waste mass stabilized at a steady level, which was evidence of waste burnout. The residue was mineral fractions. Waste O-IV (pure wood without chemical contamination) exhibited, in accordance with the fastest mass loss, the fastest burnout.

Measurement of the mass loss of the waste during combustion together with measurement of the concentration of exhaust fume components provides
important information about termination of combustion. Hence, it helps to
determine the best process time.

The tests conducted acted as a basis for a calculation of quantitative
evaluation indicators – see table 2.

Table 2. Results of the tests of quantitative evaluation indicators for the combustion
of waste

<table>
<thead>
<tr>
<th>Type of waste</th>
<th>Process temperature $T$ [°C]</th>
<th>Combustion time $t$ [s]</th>
<th>Coefficient of surplus air $λ$</th>
<th>Bulk density $ρ$ [kg/m$^3$]</th>
<th>Reaction rate $u_{r}$ [m/s]</th>
<th>Ignition rate $S$ [kg/m$^2$]</th>
<th>Mass loss rate $SUM$ [kg/m$^2$]</th>
<th>Heat load on grate $OCR$ [kW/m$^2$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>O-I</td>
<td>850</td>
<td>2400</td>
<td>–</td>
<td>1.60</td>
<td>340</td>
<td>0.0004</td>
<td>1.143</td>
<td>0.031</td>
</tr>
<tr>
<td>O-II</td>
<td>850</td>
<td>2100</td>
<td>2.07</td>
<td>250</td>
<td>0.0002</td>
<td>0.143</td>
<td>0.050</td>
<td>0.033</td>
</tr>
<tr>
<td>O-III</td>
<td>850</td>
<td>2100</td>
<td>2.49</td>
<td>220</td>
<td>0.0005</td>
<td>0.104</td>
<td>0.027</td>
<td>0.031</td>
</tr>
<tr>
<td>O-IV</td>
<td>850</td>
<td>960</td>
<td>2.92</td>
<td>76</td>
<td>0.0017</td>
<td>0.127</td>
<td>0.016</td>
<td>0.031</td>
</tr>
</tbody>
</table>

All the tests were conducted with an approximate surplus air coefficient
which accounts for the process time in conditions of total and complete
combustion. It made it possible to objectively compare all the analyzed
post-consumer wood waste and formulate the basic parameters of the process of
their combustion in a chamber equipped with a mobile grate. On the basis of the
determined indicators of the quantitative evaluation of the waste combustion
(tab. 2), the following can be stated:

- waste O-IV had the fastest reaction front rate, which is not a surprise
  when considering its clean and porous form and lowest bulk density. The
  lowest value of this parameter was recorded for waste O-II containing the
  biggest quantity of resins,
- the low value of the reaction front rate in the case of waste O-II also
  showed the low rate of ignition,
- in all the cases of tested waste, the ignition rate was higher than the mass
  loss, which may have left residue of the unburned fuel at the end of the
  grate,
- the very high heat load of the grate in the case of waste O-I and O-II
  resulted from the very high calorific value of this waste. It may have
  required the use of additional grate cooling.

Conclusions

Through the ignition rate we may determine parameters in industrial devices
with grates, especially under conditions when different fuels are combusted –
waste at the same time as in the case of incinerators, or when waste is
a participant of the co-combustion process.

In the case of fuels with a high ignition rate, the mass flux per unit area of
the grate should be increased in order to maintain the reaction flame. In the case
of fuels with a low ignition rate, the mass flux should be lowered or the air should be heated in order to dry them faster. Through the rate of mass loss the length of the combustion zone in industrial devices can be calculated, provided that the fuel mass flux and the area of the grate are known. This indicator, when compared to the fire point, gives information about the relationship between the mass ignition and real, released fuel mass loss. In the case of fuel which has a much higher fire point than the rate of mass loss, there is a risk that at the end of the grate, parts of the fuel will remain unburned. The heat load on the grate is an important issue from the point of view of exploitation of grated devices for the combustion of waste. Observation of this indicator prompts technical service staff to react to any overloading. At the incinerator design stage it provides information as to whether the grate cooling system, among other measures, needs to be accounted for.

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List of standards

PN-EN ISO 2592:2008 Oznaczanie temperatury zaplonu w tyglu otwartym metodą Clevelanda (Determination of fire point in open crucible with Cleveland method)

PN-G-04523:1992 Oznaczanie zawartości azotu metodą Kjeldahla (Determination of nitrogen with the Kjeldahl method)

PN-G-04516:1998 Oznaczanie zawartości części lotnych metodą wagową (Determination of the content of volatile matter)

PN-ISO 334:1997 Oznaczanie siarki całkowitej metodą Eschki (Determination of sulphur with the Eschka method)

PN-ISO 587:2000 Oznaczanie zawartości chloru z zastosowaniem mieszaniny Eschki (Determination of chloride using Eschka mixture)

PN-ISO 1928:2002 Oznaczanie ciepła spalania metodą spalania w bombie kalorymetrycznej i obliczanie wartości opalowej (Determining combustion heat with the method of combustion in calorimetric bomb and calculating calorific value).

PN-Z-15008-02:1993 Oznaczanie wilgotności całkowitej (Determination of moisture content)
PN-Z-15008-03:1993 Oznaczanie zawartości części palnych i niepalnych (Determination of combustible and incombustible elements)

PN-Z-15008-05:1993 Oznaczanie zawartości węgla i wodoru (Determination of carbon and hydrogen)
Sebahattin Tiryaki, Abdulkadir Malkoçoğlu, Şükrü Özşahin

ARTIFICIAL NEURAL NETWORK MODELING TO PREDICT OPTIMUM POWER CONSUMPTION IN WOOD MACHINING

This paper investigates and models the effects of wood species, feed rate, number of cutters and cutting depth on power consumption during the wood planing process. For this purpose, the samples were planed at a feed rate of 7 and 14 m/min, a cutting depth of 0.5, 1.5, 2.5 and 3.5 mm, and using 1, 2 and 4 cutters, with measurements taken during this process. According to the results, power consumption increased with increasing feed rate, cutting depth and number of cutters. In artificial neural network model, the mean absolute percentage error values between the actual and predicted values were 0.32% for the training data set and 1.15% for the testing data set. In addition, the values of $R^2$ were found to be 0.99 and 0.97 in the training and testing data sets, respectively. It is evident from the results that the designed model may be used to optimize the effects of process parameters on power consumption during the planing process of different wood species. Thus, the findings of the current study can be effectively applied in the wood machining industry in order to reduce the time for further experimental investigations, to lower energy consumption and avoid high machining costs.

Keywords: neural network modeling, optimization, planing, power consumption, wood machining

Introduction

The manufacture of wood products mostly requires a series of transformation processes. Each of these processes enables the reduction of the wood size by machining [Aguilera and Martin 2001]. Planing is possibly one of the main processes in wood machining [Gurleyen and Budakci 2015]. The machines and cutters used during the planing process should be properly designed and operated in order to achieve a wood machining process with high productivity and lower costs [Korkut et al. 1999]. To do this, it is essential to have a basic knowledge of factors related to the machining process, such as wood density,
chip formation, cutting tool geometry, feed rate, and cutting depth [Ilhan et al. 1990; Gurleyen 2010]. An optimum level of these parameters is also necessary for a reduction in production costs without reducing the quality of the product [Gunay 2003]. However, non-uniform characteristics of wood play a significant role in its effective machining during the planing process. In particular, the behaviour of different wood species in contact with the cutter tools significantly affects power consumption during the machining process [Gurleyen and Subasi 2009]. Therefore, an evaluation of the parameters relating to machining and wood properties is very important in order to achieve an economical machining process.

There have been several attempts to examine the influences of various parameters on power consumption during the machining of different wood species. Steawert [1974] compared the factors affecting machine force during hardwood planing. The results showed that increasing the feed rate, cutting depth and wood density led to higher power consumption within the planing process. Mendoza [1988] determined that an increase in cutting depth led to an increase in power consumption. Aguilera and Martin [2001] investigated power consumption during the planing of spruce and beech wood. They reported that power consumption increased with increasing cutting depth. In a study carried out to determine the cutting forces in wood species from tropical regions (Pseudolachnostylis prunifolia and Swartzia madagascariensis), Cristovao [2012] observed that chip thickness had a significant effect on the main cutting force.

The experimental studies conducted have revealed that a large number of process parameters significantly influence power consumption during wood machining. However, investigating the effect of each parameter on power consumption through experiments is both costly and wearisome. It is clear that such a laborious procedure would consume a lot of time. Therefore, it is absolutely crucial to predict the output values according to different levels of variables using a sufficient number of experimental results. Moreover, determining the optimal values of process parameters by modeling can provide information to help improve the economics of the machining process. Otherwise, numerous experiments have to be carried out to detect the desired optimum value of each parameter considered within the machining process. As already mentioned, this results in a loss of time and energy, as well as high costs, which is not desirable industrially. In the last few decades, an artificial neural network (ANN) modeling approach has drawn the attention of many researchers because of its capability in simulating the relationship between the variables of a process or system [Joo et al. 2014]. This approach has been successfully employed in the field of wood science, for example in research on the optimization of process parameters in the production of wood-based composites [Cook et al. 2000; Ozsahin 2013], calculating thermal conductivity [Avramidis and Iliadis 2005], moisture analysis [Zhang et al. 2006; Avramidis and Wu 2007], the drying
process [Wu and Avramidis 2006; Ceylan 2008], classifying wood and veneer defects [Kurdthongmee 2008; Yuce et al. 2014], wood recognition [Khalid et al. 2008], the prediction of mechanical properties [Yang et al. 2015; Tiryaki et al. 2015], predicting wood surface roughness in the machining process [Tiryaki et al. 2014; Sofuoglu 2015], and predicting various properties in the bleaching process [Okan et al. 2015].

Although many applications of the ANN approach in wood science are available, information on modeling the power consumption in wood planing is very limited. In a study conducted to detect the strain of wood against machining tools during the planing process, Gurleyen [2010] observed a very high correlation ($R^2=0.92$) between the experiment results and regression model results. The goal of the current study was to develop a neural network model capable of simulating the influences of process parameters on power consumption during the planing process by using experimental results.

Materials and methods

Sample preparation

In this study, the aim was to study a low-density wood species as well as a higher-density species. The species selected were spruce (*Picea orientalis* (L.) Link.) and beech (*Fagus orientalis* Lipsky.) for low and high density, respectively. The samples for the experiments were harvested from the Black Sea Region of Turkey. Special care was taken to select the samples without any defects. The sample sizes were trimmed to a length of 910 mm, width of 102 mm and 20 mm thickness. Thirty samples were taken for each species. Thus, a total of sixty samples were used for the power consumption experiments. The samples were conditioned at a temperature of 20 ±2°C and a relative humidity of 65 ±5% to reach a moisture content of approx. 12%. The average density of the wood species was also determined: 0.704 g/cm³ for the beech wood and 0.417 g/cm³ for the spruce wood.

Power consumption tests

The wood machining process was conducted using a planer machine. The samples were planed at feed rates of 7 and 14 m/min, cutting depths of 0.5, 1.5, 2.5 and 3.5 mm and using 1, 2 and 4 cutters. The current intensity drawn by the machine motor during the planing process was measured using an ammeter. As the current which reached the engine was the same in 3 phases, an analogue ammeter was connected to one phase and an experimental setup was prepared. When the planer machine was operated for the first time, an ammeter capable of measuring high voltages was used due to the high current amount drawn from the engine. Moreover, it is important to mention that the power consumption was not linear during the planing process. At the beginning of planing, the power
consumption was generally higher. However, it was seen that the power consumption reached a steady value after some time. To ensure the reliability of the measurement values, when the engine acceleration and the values shown by the ammeter and voltmeter were stable, the current and voltage values were recorded. Furthermore, to ensure the values recorded were reliable, five samples were used for each experimental variation. Special attention was paid to prevent the vibration of the ammeter and voltmeter used during measurement. Following this, the electrical power consumed was calculated using equation (1).

\[ P = \sqrt{3} \times U \times I \times \cos \varphi \times 10^{-3} \]  

(1)

where: \( P \) is electrical power consumed (kW), \( U \) is voltage drawn by the device, \( I \) is electrical current drawn by the device, \( \varphi \) is power coefficient (0.85).

**Data analysis**

The analysis of variance (ANOVA) was performed to evaluate the influence of the considered parameters on the power consumption during the planing of the wood. A total of 240 measurements obtained from 60 samples of beech and spruce were used for the analysis. As a relationship at the \( P \leq 0.05 \) level was available between the factors, Duncan’s multiple mean comparison test was applied and homogenous groups were detected. The analysis was performed using SPSS 11.5 (Statistical Package for the Social Sciences).

**Artificial neural network analysis**

*Artificial Neural Network (ANN)*

ANN is an intelligent modeling method that consists of many nonlinear and densely interconnected processing elements called neurons [Ozsahin 2013]. The popularity of ANN has grown recently because of its ability to deal with nonlinear relationships between variables of any process [Khayet and Cojocaru 2013]. It can be said that ANN is a more effective tool when compared with conventional statistical techniques [Ceylan 2008]. Among different architectures of ANNs, the multilayer perceptron (MLP) is the most widely used network architecture to make predictions. The MLP architecture consists of an input layer, an output layer and one or more hidden layer(s) depending on the degree of complexity of the problem under consideration [Scott and Ray 1993; Panda and Tripathy 2014; Tiryaki and Hamzacebi 2014]. A typical example of the MLP is shown in figure 1. In addition, equation (2) calculates the output of the MLP in figure 1.

\[ Y = g \left( \theta + \sum_{j=1}^{m} v_j \left( \sum_{i=1}^{n} f \left( w_{ij} X_i + \beta_j \right) \right) \right) \]  

(2)
Fig. 1. Typical multi-layered ANN architecture

In equation (2), $Y$ is the predicted value of the dependent variable; $X_i$ is the input value of $i^{th}$ independent variable; $w_{ij}$ is the weight factor between the $i^{th}$ input neuron and $j^{th}$ hidden neuron; $\beta_j$ is the bias value of the $j^{th}$ hidden neuron; $v_j$ is the weight factor between the $j^{th}$ hidden neuron and output neuron; $\theta$ is the bias of the output neuron; $g(.)$ and $f(.)$ are the activation functions.

The first layer of the ANN is the input layer, which gathers the incoming information for the ANN. This layer then transmits the information to the intermediate (hidden) layer. The hidden layer processes this information and then sends the processed information to the output layer. The output layer takes the information and finally produces output data [Canakci et al. 2012]. This flow of information from one neuron to the other is provided by the connection weights between the neurons. On the other hand, all the layers in the ANN architecture include different numbers of neurons. The number of neurons in the input and output layers is equal to the number of the input and output variables, respectively [Tiryaki and Hamzacebi 2014]. Unlike the input and output layers, detecting the number of hidden layer neurons is an important task. They can perform nonlinear mapping between input and output and allow neural networks to capture unknown information regarding the modeled property. However, too many hidden neurons can lead to an overfitting of the model. Such a situation leads to a gain in memorization capability rather than the generalization capability of the network. Obviously, this is not a desirable situation. On the other hand, too few hidden neurons are not enough for the network to uncover complex relationships between the input and output. Hence, the most popular
way to find the optimum number of hidden neurons is to adopt a trial and error procedure [Zhang et al. 1998].

As mentioned above, each neuron in the ANN layers is connected to the neurons of the next layer with a weight factor. These weights have no meaning initially. However, they gain meaningful information after undergoing a training process [Qazi et al. 2015]. The training can be defined as the calibration of the connection weights of the network employing a training algorithm to reach the desired solution [Tiryaki and Hamzacebi 2014]. The back-propagation algorithm is highly popular in training neural networks [Zhang et al. 1998]. By employing this algorithm to produce a desired output, the weights are calibrated until the degree of error between the model output and the actual output is as close as possible to the targeted error [Srisaeng et al. 2015].

**Data collection and preparation**

The initial step in designing a neural network is to collect data of parameters that may affect the modelled property [Kalogirou 2001]. Therefore, the database of the present study derived from the experiments investigating the effects of machining parameters on power consumption. The data obtained as a result of the experiments were randomly divided into two data sets (training and testing data sets). Among this data, 32 pieces of data were considered to train the network, while the remaining 16 were used to test the model. The data sets are shown in table 1. Table 2 shows the predicted values of the experimental samples of power consumption and their percentage errors. In tables 1 and 2, the data in bold represent the testing data while the other data represent the training data.

**Performance evaluation**

The mean square error (MSE) was employed as the performance function. Training of the network terminated as the MSE reached an acceptable value. The value of the MSE is calculated with equation (3).

$$\text{MSE} = \frac{1}{N} \sum_{i=1}^{N} (t_i - \hat{t}_i)^2$$  \hspace{1cm} (3)

where: \(t_i\) is the measured values, \(\hat{t}_i\) is the predicted values, and \(N\) is the total number of training patterns.

A normalization procedure for the data is often applied before training of the ANN starts [Zhang et al. 1998]. The existing data were therefore normalized to equalize the importance of the parameters prior to the training process within a range of -1 to 1 due to the use of hyperbolic tangent sigmoid function in the model.

The predictive ability of the designed network was assessed by the mean absolute percentage error (MAPE), the root mean square error (RMSE) and
coefficient of determination \( (R^2) \), defined by equations (4), (5) and (6). The network configuration giving the best output was selected to predict the power consumption.

\[
\text{MAPE} = \frac{1}{N} \left[ \frac{\sum_{i=1}^{N} \left| \frac{t_i - td_i}{t_i} \right|}{\sum_{i=1}^{N} \left| \frac{t_i - td_i}{t_i} \right|} \right] \times 100
\]  

\[
\text{RMSE} = \sqrt{\frac{1}{N} \sum_{i=1}^{N} (t_i - td_i)^2}
\]  

\[
R^2 = 1 - \frac{\sum_{i=1}^{N} (t_i - td_i)^2}{\sum_{i=1}^{N} (t_i - \overline{t})^2}
\]

where: \( t_i \) is the measured values, \( td_i \) is the predicted values, is the average of the predicted values, and \( N \) is the total number of training patterns.

**Neural network architecture**

Designing a neural network is a crucial stage that can considerably affect the output of the model [Ritchie et al. 2003]. In the present study, a trial and error procedure was adopted to decide the network parameters, such as the number of hidden layers and their neurons, activation functions, the learning rule, the weights and the biases, etc. In this regard, different network architectures were tried until the level of the error between the experimental output and the model response became acceptable. Figure 2 depicts the optimal architecture of the network developed to predict the power consumption. It is possible to see from figure 2 that the optimal architecture of the network involved one input layer, two hidden layers and one output layer.

As can be obviously seen from figure 2, the optimum model had 4, 3, 3 and 1 neurons for the input layer, a first hidden layer, a second hidden layer and an output layer, respectively. In the ANN model, the wood species, cutting depth, number of cutters and feed rate consisted of model input, while power consumption (kW) was the output of the model. As stated previously, the optimum neuron configuration of the ANN was obtained by trying many different networks in terms of hidden layers and their neurons. Moreover, it is worth mentioning that the number of connections in the model was lower than the amount of data available for training. Therefore, it is possible to describe the model mathematically.
Fig. 2. The architecture of the ANN prediction model

**Network training**

A neural network is trained by adjusting the values of the connection weights between the neurons [Tiryaki and Hamzacebi 2014]. As a result of this process, the network learns complex relationships in the data structure. In the current study, a multilayer ANN was used to learn the relationship between the input and output data of the wood machining process. The Levenberg-Marquardt algorithm (trainlm) was considered in training the network. During the training process, the weights of the connections among the neurons were iteratively adjusted, and thus the error between the model output and the experimental output was minimized. The learning rule was a gradient descent with a momentum back propagation algorithm (traindm). Figure 3 shows graphically the variation of the error during the training of the neural network model proposed to predict the power consumption.

As seen in figure 3, the training of the network was stopped after 22 epochs because the targeted error was reached.

**Results and discussion**

**Effects of process parameters on power consumption**

Table 1 shows the average values of power consumption acquired as a result of the experimental study and the results of the variance analysis.

The cutters and machines used in wood machining are exposed to different forces by different wood species during the planing process. In this study, it was determined that a lower wood density and feed rate, as well as a lower number
of cutters and cutting depth applied in the planing process, generally reduced the current consumption and resistance of the wood to the machine and cutters.

Examining table 1 in terms of the wood species, it can be seen that the power consumption of the high density beech samples was generally higher than that of the low density spruce. Several researchers have reported similar results [Stewart 1974; Bozkurt 1985; Aguilera and Martin 2001]. The reason for the higher power consumption in the beech wood may have been due to higher resistance in planing a wood with increased density. For the effects of the process parameters on the power consumption, it was determined from the present study that power consumption was generally lower when the feed speed, cutting depth and number of cutters decreased. Bozkurt [1985] stated that, in general, power consumption increases as a result of increasing feed rate, cutting depth and number of cutters in the planing process. Mendoza [1988] and Aguilera and Martin [2001] found that power consumption was greater when cutting depth increases. Similarly, Stewart [1974] determined that increased feed rate and cutting depth led to higher power consumption during the planing process. According to Gurleyen [2010], this situation as regards the feed rate might be due to an increase in the amount of work corresponding to each cutter per unit of time in the planing process. The formation of thicker chips due to increasing cutting depth may be a reason for an increase in power consumption originating from cutting depth. In such cases, the planer machine encounters more resistance in machining. In a related study, Cristovao [2012] reported that the increased chip thickness during the planing process had a great effect on the power consumed.
Table 1. The average power consumption values (kW) obtained by experiments and the results of variance analysis

<table>
<thead>
<tr>
<th>WS*</th>
<th>FR*</th>
<th>NC*</th>
<th>N</th>
<th>Cutting depth (mm)</th>
<th></th>
<th></th>
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<th></th>
<th></th>
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<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Avg.</td>
<td>HG</td>
<td>SD</td>
<td>Avg.</td>
<td>HG</td>
<td>SD</td>
<td>Avg.</td>
</tr>
<tr>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td>7</td>
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<td>3.086</td>
<td>A</td>
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</tr>
<tr>
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<td>FGHII</td>
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<td>3.567</td>
<td>HIJ</td>
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<tr>
<td>7</td>
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<td>20</td>
<td>3.146</td>
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<td></td>
<td>3.487</td>
<td>FGHII</td>
<td>0.081</td>
<td>3.986</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>20</td>
<td>3.455</td>
<td>EFGH</td>
<td>0.149</td>
<td></td>
<td>3.576</td>
<td>HIJ</td>
<td>0.091</td>
<td>4.085</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>20</td>
<td>3.431</td>
<td>EFGH</td>
<td>0.033</td>
<td></td>
<td>3.555</td>
<td>GHJ</td>
<td>0.044</td>
<td>3.998</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>20</td>
<td>3.515</td>
<td>FGHII</td>
<td>0.042</td>
<td></td>
<td>3.606</td>
<td>IJKL</td>
<td>0.078</td>
<td>4.105</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>20</td>
<td>3.585</td>
<td>HIJK</td>
<td>0.087</td>
<td></td>
<td>3.655</td>
<td>JKL</td>
<td>0.054</td>
<td>4.071</td>
</tr>
</tbody>
</table>

*WS, FR and NC denote wood species, feed rate and number of cutters, respectively; N – number of measurements.
Data in **bold** were used for ANN testing, whereas the other data were used for ANN training.
Avg. – average, SD – standard deviation, HG – homogeneity groups.
The same letters in columns indicate that there is no statistical difference between the samples, according to Duncan's multiple range test at P < 0.05.
Optimizing power consumption by ANN

Table 2 presents the ANN output for the training and test data sets, percentage error ratios and the values of the performance indicators such as the RMSE and MAPE.

A regression analysis between the experimental output and neural network output is often useful to evaluate the validity and accuracy of the networks. For the present study, the graph of the relationship between the experimental and predicted output is shown in figure 4. The $R$ (correlation coefficient) values were 0.99885 for training and 0.98671 for testing. Thus, the $R^2$ (determination coefficient) values obtained were 0.99 and 0.97 for the training and testing data, respectively. It is well known that if $R^2$ values approach 1, the accuracy of the prediction increases. This means that there is a perfect fit between the experimental (measured) output and network output. These results indicated that the designed network was capable of explaining at least 0.97% of the measured data. A high correlation between the model prediction and measured results confirmed the use of the ANN in predicting power consumption.

![Graph showing relationship between measured and predicted values](image)

**Fig. 4. Relationship between the measured and predicted values of power consumption**

Figure 5 indicates the comparative plots of the measured and predicted power consumption values.
<table>
<thead>
<tr>
<th>Wood species</th>
<th>Feed rate (m/min)</th>
<th>Number of cutters</th>
<th>Cutting depth (mm)</th>
<th>0.5</th>
<th>1.5</th>
<th>2.5</th>
<th>3.5</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>p(^a)</td>
<td>e(^a)</td>
<td>p</td>
<td>e</td>
</tr>
<tr>
<td>Spruce</td>
<td>7</td>
<td>1</td>
<td>3.080 0.19</td>
<td>3.243</td>
<td>0.26</td>
<td>3.567</td>
<td>-0.18</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2</td>
<td>3.096 -0.47</td>
<td>3.244</td>
<td>0.28</td>
<td>3.692</td>
<td>0.78</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4</td>
<td>3.215 2.63</td>
<td>3.365</td>
<td>-0.35</td>
<td>3.802</td>
<td>-0.08</td>
</tr>
<tr>
<td></td>
<td>14</td>
<td>1</td>
<td>3.384 0.96</td>
<td>3.552</td>
<td>-0.08</td>
<td>3.892</td>
<td>-0.85</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2</td>
<td>3.466 0.58</td>
<td>3.526</td>
<td>-0.79</td>
<td>3.911</td>
<td>0.56</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4</td>
<td>3.528 -0.32</td>
<td>3.565</td>
<td>0.05</td>
<td>3.957</td>
<td>-0.28</td>
</tr>
<tr>
<td>Beech</td>
<td>7</td>
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<td>3.476</td>
<td>-0.16</td>
<td>3.843</td>
<td>1.08</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2</td>
<td>3.219 -1.23</td>
<td>3.492</td>
<td>-0.14</td>
<td>3.964</td>
<td>0.56</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4</td>
<td>3.464 -0.26</td>
<td>3.639</td>
<td>-1.76</td>
<td>4.068</td>
<td>0.42</td>
</tr>
<tr>
<td></td>
<td>14</td>
<td>1</td>
<td>3.461 -0.86</td>
<td>3.639</td>
<td>-2.35</td>
<td>4.007</td>
<td>-0.22</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2</td>
<td>3.494 0.59</td>
<td>3.597</td>
<td>0.24</td>
<td>4.027</td>
<td>1.91</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4</td>
<td>3.565 0.56</td>
<td>3.656</td>
<td>-0.03</td>
<td>4.064</td>
<td>0.16</td>
</tr>
<tr>
<td>MAPE training</td>
<td></td>
<td></td>
<td></td>
<td>0.322</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>RMSE training</td>
<td></td>
<td></td>
<td></td>
<td>0.015</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*\(^a\)p and e denote predicted values and errors in %, respectively*
Fig. 5. Comparison of the measured and ANN predicted values: a – training, b – testing

From the plots presented in figure 5, it can be seen that the predicted output were very close to the measured output. The high similarity between the outputs supports the usability of the developed model in predicting power consumption.

The maximum absolute percentage errors obtained as a result of prediction by ANN were 0.86% and 2.63% for the training and the testing stages of the proposed model, respectively. The MAPE values were 0.32% for the training data and 1.15% for the testing data. Furthermore, the RMSE values were 0.015 for the training data and 0.052 for the testing data. Prediction becomes more accurate when the MAPE and RMSE are low because it implies there is a small difference between the network response and the experimental data. In this respect, for the present study, low values of MAPE and RMSE demonstrated that
the network was sufficiently accurate and reliable to model power consumption within planing process.

Neural network models gain the ability to yield the desired intermediate values for optimization studies when they are properly trained [Ozsahin 2013]. This is one of the most distinct characteristics of ANNs. In the optimization of power consumption for the current study, the wood species (spruce) and number of cutters (two) were fixed, and the feed rate and cutting depth were changed. The intermediate power consumption values not provided from the tests were determined by means of the proposed model for different feed rates and cutting depths, and are shown graphically in figure 6.

![Figure 6](image.png)

**Fig. 6. The effect of cutting depth and feed rate on power consumption**

The effect of the other parameters on the power consumption can be predicted by analysing the responses of the model. This model enabled us to better understand how different machining conditions affect power consumption.

**Conclusion**

This study investigated and modelled the influence of some machining parameters on power consumption during the planing of wood. According to the results of the study, power consumption increased with an increase in feed rate, cutting depth, and number of cutters. Likewise, high density beech showed greater power consumption values compared to low density spruce. Therefore, it is possible to say that wood materials should not be processed at high feed rates
and cutting depth in order to avoid high electricity expenditure caused by excessive power consumption. In the ANN analysis, the MAPE and $R^2$ values were found to be 1.15% and 0.97 for the testing phase, respectively. The high values of the MAPE and $R^2$ were extremely effective in predicting power consumption. Based on these results, it can be said that the designed model may be employed to efficiently describe the relationship between machining parameters and power consumption. Further, it can be concluded that the approach presented is an applicable tool to predict with a high degree of accuracy power consumption in the wood planing process.

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EFFECTS OF INORGANIC NANOPARTICLES ON MECHANICAL AND MORPHOLOGICAL PROPERTIES OF WOOD FLOUR-POLYPROPYLENE NANO COMPOSITES

This research was conducted to examine the effect of SiO₂ and TiO₂ nanoparticles on the mechanical and morphological properties of wood flour-polypropylene nanocomposites. For this purpose, 60 (wt.%) wood flour was mixed with polypropylene. 4% maleic anhydride polypropylene was also used in all the compounds as a coupling agent. In addition, SiO₂ and TiO₂ nanoparticles were used as mineral fillers at 0, 1, 3 and 5%. The mixing process was performed inside an extruder and the test specimens were prepared by injection molding method. Bending and tensile tests were then performed on the specimens according to the ASTM standard. Scanning electron microscopy (SEM) was also used to show the distribution of nanoparticles over the composite substrate. The results showed that the composites containing nano-SiO₂ had more favorable mechanical properties compared to those containing nano-TiO₂. On the other hand, increasing the nanoparticles from 0 to 3% led to an increase in mechanical strength, however, the addition of more nano-fillers resulted in a significant decrease in mechanical strength. The results of SEM also showed a proper dispersion of nanoparticles at 1 and 3% levels, but using 5% nanoparticles caused the particles to aggregate on the composite substrate.

Keywords: wood plastic composite, inorganic nanoparticles, reinforcing additives, particle dispersion

Introduction

Wood plastic composites, a new class of composites classified as green composites, have been drawing the attention of researchers and industries in
recent years. In the technology by which this class of composites is prepared, there is no material that can improve all properties of the composites. For this reason, nano-scale particles (1-50 nm) are commonly used. Polymer nanocomposites are a class of materials in which inorganic nanoparticles with planar or spherical structures are dispersed in the polymer matrix. This class of composites (nanocomposites) has more acceptable properties compared to pure composites and polymers. Although small amounts of nanoparticles are used to produce polymer nanocomposites, this small amount significantly increases their thermal and mechanical properties [Soon et al. 2012; Njuguna et al. 2008].

Nowadays, polymers reinforced with inorganic nanoparticles are very important: adding these inorganic nanoparticles to the compounds of composites brings many advantages, such as a light weight, enhanced mechanical properties, improved physical properties and easy formability. However, disadvantages include the aggregation of particles in the polymer matrix and a decrease in the impact strength of the composites [Yang et al. 2006; Krueenate et al. 2004].

In recent years, some research has been carried out on the effect of mineral nanoparticles on polymer composites. Deka and Maji [2012] studied the effect of silica and clay nanoparticles on the properties of wood-plastic composites. Their results showed that an increase in nanoparticles of 3% resulted in an increase in flexural and tensile strength, as well as flexural and tensile moduli. However, an increase of the nanoparticles to 5% led to a decrease in all the above-mentioned properties. SEM results also showed that adding a large quantity of nanoparticles causes them to accumulate in the polymer matrix and the mechanical strength decreases. Kord and Taghizadeh Haratbar [2014] studied the effect of nanoparticles on wood-plastic composites. Their results showed that by increasing nanoclay to 3%, flexural strength and modulus increased, but with an addition of 6% nanoclay, the mechanical properties decreased. Ismaeilimoghadam et al. [2015] studied the effect of silica nanoparticles on wood-plastic composites. Their results showed that by increasing nanosilica to 3%, the mechanical properties increased, including the flexural and tensile strength and moduli. However, with the addition of 5% nanoclay, the mechanical strength decreased. SEM results also showed that an increase in silica SiO$_2$ nanoparticles to 5% resulted in aggregation in the polymer matrix. Altan and Yildirim [2010] examined the mechanical and morphological properties of the composite of high density polypropylene and polyethylene strengthened by titanium nanoparticles. Their results showed that increasing the nanoparticles to 5% caused an increase in the flexural strength and modulus of elasticity. The morphological results showed that adding large amounts of nanotitanium causes them to accumulate in the polymer matrix.

Many studies have been conducted concerning the use of SiO$_2$, ZnO and CaCO$_3$ inorganic nanoparticles. Among them, SiO$_2$ nanoparticles are widely used in polymer industries due to their very small size and large surface area. Researchers believe that SiO$_2$ particles can improve the strength, hardness,
modulus, crystallinity, viscosity, creep resistance and inter-structural adhesion of polyethylene, polypropylene and thermoplastic elastomers [Zhang et al. 2003a, b; Rong et al. 2004; Guyard et al. 2006; Khatibzadeh et al. 2010; Parvinzadeh et al. 2010]. In some studies, researchers concluded that SiO₂ nanoparticles do not act as the transition core material and barely affect the crystallinity [Bikiaris et al. 2006; Tian et al. 2006; Chae and Kim 2007; Zhang et al. 2008]. But in some other research, a significant improvement of mechanical properties of SiO₂ nanoparticles were noted, in addition to their transition core functionality [Liu et al. 2012]. On the other hand, TiO₂ nanoparticles, used as polymer reinforcing, are inorganic materials with high mechanical strength [Garakani et al. 2007; Ishak Mohd et al. 2008]. In this study, inorganic transition core nanoparticles were compared, and the independent and interaction effects of the factors on the mechanical properties of the composites were studied. Since no research has as yet been devoted to a comparison of the mechanisms of transition core nanomaterials and their effects on nanocomposite properties, it is necessary to study the subject matter in question.

**Materials and methods**

In this study, grade Pi 800 polypropylene with a melt flow index (MFI) of 8 g/10 min, produced by Arak Petrochemical Company, was used as a polymer matrix, and a 60-mesh wood flour mixed with hardwoods from the northern Iran was used as the reinforcer. Polypropylene grafted maleic anhydride (PP-g-MA) with a melt flow index of 10g/10 min, produced by Krangin Co., was also used, as well as 0.2% maleic anhydride, which was used as a coupling agent. SiO₂ and TiO₂ nanoparticles, produced by US Research Nanomaterials Inc, were used as fillers and their characteristics are presented in table 1 below.

<table>
<thead>
<tr>
<th>Table 1. Characteristics of SiO₂ and TiO₂ nanoparticles</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Type of nanoparticle</strong></td>
</tr>
<tr>
<td>-------------------------</td>
</tr>
<tr>
<td>TiO₂</td>
</tr>
<tr>
<td>SiO₂</td>
</tr>
</tbody>
</table>

The mixing process was performed using an extruder with two counter current screws at Iran Polymer and Petrochemical Institute according to table 2. The hot formable material obtained from the mixing process was collected after leaving the extruder, and a Wieser WG-LS 200/200 semi-industrial crushing machine (Wieser, Germany) was used on the cooled material to prepare granules. In order to remove the moisture, the granules were transferred to a dryer at 85°C
for 24 hours, and the specimens for the mechanical tests were prepared by injection molding. For this purpose, a semi-industrial injection machine produced by Tehran Imen Machine Company and available at Iran Polymer and Petrochemical Institute was used. 7 treatments were selected, 5 replications of each treatment were considered and, overall, 35 samples were made \((7 \times 5 = 35)\). Finally, before performing any test, the specimens were left at room temperature \((23^\circ C)\) with a relative humidity of 50% for 40 hours according to the ASTM standard and D618-99 code.

### Table 2. Scheme of the studied mixing treatments

<table>
<thead>
<tr>
<th>Treatment codes</th>
<th>Wood flour (%)</th>
<th>Polypropylene (%)</th>
<th>Coupling agent ((\text{PhC})^*)</th>
<th>Nano-TiO(_2) ((\text{PhC}))</th>
<th>Nano-SiO(_2) ((\text{PhC}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>CS</td>
<td>60</td>
<td>40</td>
<td>4</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>S-1</td>
<td>60</td>
<td>40</td>
<td>4</td>
<td>–</td>
<td>1</td>
</tr>
<tr>
<td>S-3</td>
<td>60</td>
<td>40</td>
<td>4</td>
<td>–</td>
<td>3</td>
</tr>
<tr>
<td>S-5</td>
<td>60</td>
<td>40</td>
<td>4</td>
<td>–</td>
<td>5</td>
</tr>
<tr>
<td>T-1</td>
<td>60</td>
<td>40</td>
<td>4</td>
<td>1</td>
<td>–</td>
</tr>
<tr>
<td>T-3</td>
<td>60</td>
<td>40</td>
<td>4</td>
<td>3</td>
<td>–</td>
</tr>
<tr>
<td>T-5</td>
<td>60</td>
<td>40</td>
<td>4</td>
<td>5</td>
<td>–</td>
</tr>
</tbody>
</table>

*Per hundred compounds

A bending test was performed on the specimens with a 2 mm/min loading speed, according to the ASTM standard and D790 code, while a tensile test was performed on the specimens with a 5 mm/min loading speed, according to the ASTM standard and D 638 code. For this purpose, a HOUNS H 25 KS machine with N 25000 cell capacity was used in the Wood Mechanics Laboratory of the Department of Natural Resources, Zabol University (Iran). After testing each treatment, the computer connected to the machine provided information including the maximum resistance, changes in the specimen's length, proportional limit strength and modulus of elasticity.

Microscopic images were provided to examine the distribution and transmittance of SiO\(_2\) and TiO\(_2\) nanoparticles in the polymer matrix by scanning electron microscopy (SEM, device EM 3200). In this way, cross sections of the fracture point of the bending test specimens were provided. The samples were covered by a thin layer of gold to prevent static charges.

The results were analyzed using SPSS Software and ANOVA. In the case of a significant difference between the levels, Duncan's multiple range test was used at a 95% confidence level to compare the means. Excel software was also used to draw the diagrams.

### Results and discussion

The results of the variance analysis showed that there were significant differences between the treatments in terms of bending and tensile strengths at
a 95% confidence level. Figure 1 shows the mutual effect of SiO$_2$ and TiO$_2$ nanoparticles on the bending strength of the prepared nanocomposites. As seen in this figure, the highest bending strength was 63.55 MPa for 3 PhC nano-SiO$_2$, while the lowest was 49.62 MPa for 5 PhC nano-TiO$_2$. It is worth noting that the composites containing SiO$_2$ nanoparticles had a higher bending strength than those containing TiO$_2$ nanoparticles, and increasing these nanoparticles up to 3 PhC resulted in an increase in the bending strength, but beyond this level, it decreased significantly. Figure 2 shows the mutual effect of SiO$_2$ and TiO$_2$ nanoparticles on the tensile strength of the prepared composites. According to the figure, the highest tensile strength of the prepared nanocomposites was for 3 PhC SiO$_2$ and TiO$_2$ nanoparticles and the lowest tensile strength was for those without nanoparticles (the control).

![Fig. 1. Mutual effect of SiO$_2$ and TiO$_2$ nanoparticles on bending strength](image_url)

There are, therefore, two mechanisms to increase the bending and tensile strength of nanoparticles up to 3 PhC: the first mechanism is related to the fact that SiO$_2$ and TiO$_2$ nanoparticles increase the tension between the fibres and matrix by tolerating stress [Deka and Maji 2012; Ramos et al. 2005] and the second mechanism also suggests that SiO$_2$ and TiO$_2$ nanoparticles act as a transition core in crystal growth; hence, the introduction of these nanoparticles increases the number of crystals [Tian et al. 2006; Chae and Kim 2007]. On the other hand, these nanoparticles tend to accumulate and absorb each other due to their high surface energy. Through their chemical groups, these nanoparticles can accumulate on the surface of a polymer by creating hydrogen bonds [Albala et al. 2004; Oburoğlu et al. 2012]. Therefore, the decrease in bending strength by using 5 PhC nanoparticles is due to the accumulation of nanoparticles and their non-uniform distribution, as well as the resulting agglomeration of particles in the composite substrate [Yang and Gu 2007]. Another issue concerns the decrease in polymer moisturizing ability due to the use of a large quantity of
nanoparticles leading to the improper bonding between the lignocellulosic fibres and the matrix., These factors reduce the bending and tensile strength of the nanocomposites made using high doses of SiO$_2$ and TiO$_2$ nanoparticles [Deka and Maji 2012]. When the composite materials are finer and their specific surface increases, their static resistance increases significantly. Thus, it seems that the higher bending strength of SiO$_2$ nanocomposites compared to that of TiO$_2$ nanocomposites is due to the smaller size of the SiO$_2$ nanoparticles and their higher specific surface.

![Graph showing the mutual effect of SiO$_2$ and TiO$_2$ nanoparticles on tensile strength.](image)

**Fig. 2. Mutual effect of SiO$_2$ and TiO$_2$ nanoparticles on tensile strength**

The results of the variance analysis show that, at a 95% confidence level of there were significant differences between the treatments in terms of bending and tensile moduli. Figure 3 shows the mutual effect of SiO$_2$ and TiO$_2$ nanoparticles on the flexural modulus. According to the figure, the highest flexural modulus (6980.7 MPa) was with the use of 3 PhC Nano-SiO$_2$, and the lowest (3855.9 MPa) was for the control specimens. Figure 4 shows the mutual effect of SiO$_2$ and TiO$_2$ nanoparticles on the tensile modulus. It can be seen that the highest tensile modulus (5787.7 MPa) was with the use of 3 PhC nano-SiO$_2$ and the lowest (3116.6 MPa) was for the control specimens. Note that the nano-SiO$_2$ composites had higher flexural and tensile moduli compared to the nano-TiO$_2$ composites. On the other hand, an increase in the nanoparticles from 0 to 3 PhC resulted in an increase in the moduli, while an increase to 5 PhC led to a decrease in the moduli.

The increase in moduli in polymer nanocomposites largely depends on the dispersion of nanoparticles in the matrix substrate. Hence, the increase in the flexural and tensile moduli when using 3 PhC SiO$_2$ and TiO$_2$ nanoparticles may be related to the proper distribution of the particles in the polymer substrate, the decreased mobility of the polymer chains and the subsequent decrease in the
relative elongation of the specimens through loading. On the other hand, the decreased flexural modulus due to the use of larger quantities of these nanoparticles is related to their agglomeration and aggregation in the composite substrate [Chen et al. 2003; Deka and Maji 2012].

![Graph showing the mutual effect of SiO₂ and TiO₂ nanoparticles on flexural modulus](image1)

**Fig. 3.** Mutual effect of SiO₂ and TiO₂ nanoparticles on flexural modulus

![Graph showing the mutual effect of SiO₂ and TiO₂ nanoparticles on tensile modulus](image2)

**Fig. 4.** Mutual effect of SiO₂ and TiO₂ nanoparticles on tensile modulus

Figure 5 shows the distribution of SiO₂ and TiO₂ nanoparticles in a polymer matrix. It can be seen that when using 1 PhC nanoparticles, their distribution was formed well in the polymer matrix. The particle sizes measured in the images related to 1 PhC are less than 20 nm, which is fairly consistent with the manufacturer's claim concerning particle diameter (11-14 and 10-25 nm). This means that the 1 PhC nanoparticles were separately distributed in the polymer matrix without interacting with each other. When using 3 PhC nanoparticles, the distance between the particles decreased compared to the 1 PhC nanoparticles,
Fig. 5. Distribution of SiO$_2$ and TiO$_2$ nanoparticles in a polymer matrix: a – 1 PhC NanoTiO$_2$, b – 1 PhC Nano SiO$_2$, c – 3 PhC Nano TiO$_2$, d – 3 PhC Nano SiO$_2$, e – 5 PhC Nano TiO$_2$, f – 5 PhC Nano SiO$_2$
but their distribution improved in the matrix. However, when using 5 PhC nanoparticles, the particles aggregated (agglomeration). The particle size measured in these images is much higher than the manufacturer's claim. This means that SiO₂ and TiO₂ nanoparticles tend to attract each other through hydrogen bonds due to their high surface energy and attracting forces. Thus, with large quantities of these nanoparticles, agglomeration occurs and the crystal size increases, which reduces the mechanical strength of the nanocomposites [Deka and Maji 2012].

Conclusions

In this research, the effects of inorganic nanoparticles on the properties of wood flour–polypropylene nanocomposites were examined. The results showed that:

- By increasing the nanoparticles from 0 to 3 PhC, the mechanical strength increased, including the flexural and tensile strength and moduli. However, an increase in nanoparticles to 5 PhC caused a decrease in mechanical strength.
- The effect of the SiO₂ nanoparticles on the improvement in the mechanical properties was more marked than that of the TiO₂ nanoparticles.
- By increasing the nanoparticles to 3 PhC, their dispersion and distribution improved in the polymer matrix, but the use of 5 PhC nanoparticles resulted in an increase in crystal size and this caused difficulties in their distribution.

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List of standards


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THERMAL AGEING OF CELLULOSE WITH NATURAL AND SYNTHETIC ANTIOXIDANTS UNDER VARIOUS CONDITIONS

Studies were undertaken of the influence of both the natural stabilizers present in wood (lignin and extractives) and synthetic antioxidants on the thermal ageing of cellulose. Among the synthetic antioxidants, butylated hydroxytoluene, propyl gallate and 6-ethoxy-1,2-dihydro-2,4-trimethylquinoline (ethoxyquin) were examined. In order to study the thermal ageing of cellulose with antioxidants, accelerated ageing tests were carried out under various conditions. The ageing tests were performed at a temperature of 95°C in an air and nitrogen atmosphere in anhydrous conditions and at 65% relative air humidity. To study the degradation of the cellulose, size exclusion chromatography was used. The results revealed that ethoxyquin was the best synthetic stabilizer. In addition, butylated hydroxytoluene had stabilizing properties and slowed down the depolymerisation of the cellulose. In turn, the behaviour of the propyl gallate under elevated temperature conditions was the most interesting. This antioxidant, relating to the cellulose degradation process, showed inhibitory as well as catalytic properties under specific conditions. In the ageing conditions applied, the smallest decrease was observed in the average molar mass of the cellulose in the wood without extractives. The results indicate that in this case, lignin played a very important role as a hidden antioxidant. In the presence of the lignin, oxidative the cellulose depolymerisation process proceeded more slowly than with the participation of synthetic antioxidants. The extractives, under elevated temperature conditions, did not show stabilizing properties, and furthermore, they accelerated the degradation of the cellulose.

Keywords: thermal ageing, cellulose degradation, wood, antioxidants, SEC

Introduction

Wood has played an important role in society for a long time. Due to its complex structure, it has unique mechanical, physical and chemical properties. It is
characterized by its considerable strength, its low price and its interesting decorative and utilitarian value. In addition, it is a readily available and renewable resource. However, in spite of so many advantages, it is also much more exposed to degradation than synthetically obtained materials. Factors that cause wood degradation include elevated temperature, humidity and an oxidizing environment. Cellulose is the main component of wood. Wood strength mainly depends on the physicochemical properties of cellulose. In the literature, some information may be found on the possibility of using natural and synthetic antioxidants for polymer stabilization [Kovárová et al. 1995; Schultz and Nicholas 2000; Pouteau et al. 2003; Gregorová et al. 2006; Košíková and Lábaj 2009; Košíková and Sláviková 2010; Ambrogi et al. 2014]. One of the methods to inhibit or at least reduce the oxidative depolymerisation of cellulose is the application of antioxidants [Schmidt et al. 1995; Strlič et al. 2001, 2004; Vízárová et al. 2014]. The most important antioxidants present in wood include extractives (tannins, flavonoids, stilbenes, lignans) and lignin [Kähkönen et al. 1999; Pan et al. 2006; Pietarinen et al. 2006; Redzynia et al. 2009; Faustino et al. 2010]. Whereas the most widely used synthetic antioxidants in different industries include butylated hydroxytoluene (BHT), butylated hydroxyanisole (BHA), tertiary-butylhydroquinone (TBHQ), propyl gallate (PG) and 6-ethoxy-1,2-dihydro-2,2,4-trimethyquinoline (ethoxyquin, EQ). 2,4,5-trihydroxybutyrophenone (THBP), nordihydroguaiaretic acid (NDGA) and 2,6-diterbutyl-4-hydroxymethylphenol (IONOX 100) are less commonly used because of their high cost.

There is little research on the use of the above-listed synthetic antioxidants to study cellulose stability. However, the idea of their application seems to be interesting both from a scientific and a practical view point. In addition, the behaviour of stabilizing compounds at elevated temperatures and their effect on cellulose depolymerisation are still poorly understood. Therefore, an attempt to determine the influence of the natural stabilizers present in wood (lignin and extractives) as well as synthetic antioxidants on the degradation of cellulose was undertaken.

**Materials and methods**

Ageing tests were carried out on the following materials:
- the non-extracted sawdust of pinewood (*Pinus sylvestris* L.) from the sapwood zone;
- the extracted sawdust of pinewood (*Pinus sylvestris* L.) from the sapwood zone (without extractives) treated with a mixture of chloroform (Chempur, Poland) and 96% ethanol (Chempur, Poland) (93:7) \(w\) in a Soxhlet extractor according to the authors’ own method [Antczak et al. 2006];
- cellulose – isolated from the above-mentioned extracted pinewood (*Pinus sylvestris* L.) using the Kürschner-Hoffer method [Krutul 2002].
The method consists of the mild nitration (65% nitric acid; Chempur, Poland) of lignin to obtain an alcohol (96% ethanol) soluble nitro derivative. Hemicelluloses and cellulose with a lower degree of polymerisation undergo hydrolysis at reaction conditions;
- the above-mentioned pinewood cellulose with antioxidants – PG, BHT and EQ (Supelco, USA), which were coated on the cellulose fibre using a stirring method [Antczak et al. 2007]. The method consists of immersing a sample of the cellulose in 0.2% antioxidant solution in methanol (Chempur, Poland) and then the total evaporation of the solvent under vacuum while stirring using a vacuum evaporator (Rotavapor R-215, Büchi company).

**Accelerated ageing tests in normal atmosphere**

At the beginning, the non-extracted and extracted pinewood from the sapwood zone, as well as the cellulose and cellulose with antioxidants (PG, BHT and EQ) were placed in a hermetically-sealed desiccator with P₂O₅ (Sigma-Aldrich, Germany) (in anhydrous conditions). At the same time, for the purposes of comparison, the above-mentioned research material was placed in another hermetically-sealed desiccator with a saturated water solution of NaNO₃ (Chempur, Poland) (in conditions of 65% relative air humidity) [Greenspan 1977]. In these vessels the samples were submitted to accelerated ageing tests. The ageing tests were carried out in the thermal chamber (KC 100/200, Elkton company) at 95°C. The samples were collected every 14 days over a 70 days period and were prepared for SEC (Size Exclusion Chromatography) analysis in order to study the cellulose degradation.

**Accelerated ageing tests in a nitrogen atmosphere**

In these studies, only the cellulose samples and the samples of cellulose with antioxidants (PG, BHT and EQ) were used. They were placed in a hermetically-sealed vacuum desiccator with P₂O₅. A vacuum pump (V-700, Büchi company) was used to remove the air to 0.66 kPa, and the desiccator was then filled with nitrogen. The procedure was repeated three times. After this, the samples in the desiccator were subjected to accelerated ageing tests. The ageing tests were also carried out in a thermal chamber (KC 100/200, Elkton company) at 95°C. The samples were also collected every 14 days over a 70 days period and were prepared for SEC analysis in order to examine the cellulose degradation.

**Preparation of cellulose samples for SEC analysis**

The cellulose samples were prepared using the authors’ own procedure, presented in earlier publications [Antczak 2010b; Radomski et al. 2011]. A detailed description of the procedure is as follows: in order to study the cellulose degradation in the aged non-extracted and extracted wood, the
cellulose was isolated using the Kürschner-Hoffer method. All the cellulose samples (50 mg of each) were treated with 50 cm³ 0.01 M NaBH₄ (Sigma-Aldrich, Germany) at room temperature (25°C). After ca 24 h, the cellulose was filtered through a glass filter (G3), washed with 5% acetic acid (20 cm³) (Chempur, Poland) and then washed with distilled water until it reached a neutral pH. Following this, the cellulose samples were treated with 1% NaOH (20 cm³) (Chempur, Poland) in a nitrogen atmosphere for 1 hour using a magnetic stirrer at room temperature (25°C). The cellulose was then filtered and washed as before. For the next step, the air-dry cellulose samples prepared according to the aforementioned method were subjected to an activation and dissolution procedure. The procedure was carried out in a Baker SPE-12G vacuum system at room temperature (25°C) and was as follows:

- the cellulose samples (15 mg) were placed in test-tubes (6 cm³), poured over with distilled water (3 cm³) and allowed to swell overnight;
- the following day, the samples were transferred to polypropylene tubes with a narrow outlet (8 mm and 0.5 mm – inlet and outlet internal diameters, respectively) and subsequently washed with methanol (Chempur, Poland), filtered and poured over with the next portion of methanol and left for 1 hour; this procedure was repeated twice;
- following this, the samples were washed with DMAc (N,N-dimethylacetamide) (Sigma-Aldrich, Germany), filtered and poured over with the next portion of DMAc and left for 1 hour; this procedure was repeated once and the cellulose with the DMAc was left until the following day;
- the following day, the samples were filtered and poured over with 8% LiCl in DMAc (4 cm³) (Sigma-Aldrich, Germany);
- the cellulose dissolution in an 8% LiCl/DMAc solvent system was realised using an RM-2M mixer (Elmi company);
- after 1-2 days of continuous mixing, part of the sample (0.2 cm³) was diluted to 0.5% LiCl (Sigma-Aldrich, Germany) concentration with pure DMAc (3 cm³);
- finally, the prepared samples were submitted for SEC analysis.

Conditions of SEC analysis
The conditions of the SEC analysis of the cellulose samples were adopted from earlier studies [Antczak 2010b; Radomski et al. 2011]. The analysis was carried out using a HPLC (High Performance Liquid Chromatography) system (LC-20AD, Shimadzu company), which was equipped with a differential refractive detector (RID-10A, Shimadzu), pump (LC-20AD, Shimadzu), degasser DGU-20A (Shimadzu), oven (CTO-20A, Shimadzu) and controller (CBM-20A, Shimadzu). The SEC analysis conditions were as follows:

- 0.5% LiCl/DMAc as the eluent,
- column – cross-linked polystyrene-divinylbenzene gel (PSS GRAM 10000, 10 µ, 8 × 300 mm) connected with a guard column (PSS GRAM 10 µ),
- oven temperature: 80°C,
- flow rate: 2 cm³/min (the high flow rate was compatible with the column specification),
- injection volume: 0.2 cm³.

The chromatographic data were processed using PSS WinGPC scientific 2.74 software. Twelve narrow molar mass polystyrene standards (Polymer Laboratories) were used to calibrate the column. The polystyrene standards were prepared as mixed standards in four separate solutions in a 0.5% LiCl/DMAc solvent system. The first standard solution contained polystyrene of the following molar masses: 6 850 000, 565 000 and 11 300 Da, the second: 3 950 000, 170 600 and 2 960 Da, the third: 3 150 000, 66 000 and 1 700 Da, and the fourth: 1 290 000, 28 500 and 580 Da. These polystyrene standards were used to calculate the molar mass of the cellulose according to Mark-Houwink universal calibration:

$$[\eta] = K \times M^\alpha$$  \hspace{1cm} (1)

where K and α are parameters, which depend on the polymer type, solvent and temperature. For the chromatographic conditions in this study, these parameters were as follows: for the polystyrene K = 17.35 × 10⁻³ cm³/g and α = 0.642 [Timpa 1991] and for the cellulose K = 2.78 × 10⁻³ cm³/g and α = 0.957 [Bikova and Treimanis 2002].

**Results and discussion**

The thermal degradation of the cellulose was examined by SEC after the ageing tests. The relationships between the weight average molar mass (Mₘ) of the cellulose and the ageing time at 95°C in normal atmosphere (in anhydrous conditions) are presented in figures 1 and 2.

The results of the studies presented in figure 1 indicate that the cellulose with the addition of ethoxyquin was the most resistant to thermal degradation at 95°C in the air (in anhydrous conditions). Furthermore, BHT revealed stabilizing properties and effectively slowed down the rate of cellulose degradation. In turn, the addition of propyl gallate in these conditions had a catalytic influence on the cellulose thermal degradation process. There is some information in the literature that propyl gallate, apart from having antioxidant and stabilizing properties, has pro-oxidizing properties under certain conditions contributing to the acceleration of the degradation of substances such as DNA and carbohydrates [Smith et al. 1992; Aruoma et al. 1993]. These findings were also confirmed by previous studies performed at 130°C. According to these studies,
under aerobic conditions, propyl gallate proved to be the catalyst for the thermal degradation of cellulose [Antczak et al. 2008; Antczak 2010b].

Fig. 1. The relationship between the weight average molar mass (M_w) and ageing time for cellulose and cellulose with antioxidants (EQ, PG and BHT) aged at 95°C in normal atmosphere and in anhydrous conditions.

Fig. 2. The relationship between the weight average molar mass (M_w) and ageing time for cellulose, cellulose in wood without extractives and cellulose in wood with extractives aged at 95°C in normal atmosphere and in anhydrous conditions.
The results of the chromatographic analysis for the aged cellulose and cellulose in the aged extracted and non-extracted wood are presented in figure 2. These results indicate that at 95°C in normal atmosphere (in anhydrous conditions) the smallest changes in the weight average molar mass of the cellulose occurred in the extracted wood. The presence of the extractives significantly accelerated the thermal degradation of the cellulose. However, the most significant drop in the weight average molar mass of the cellulose, from the beginning of the ageing process, took place in the case of the pure cellulose.

On the basis of the results of the thermal ageing of the wood at 95°C (fig. 2), it can be concluded that the most likely cause of the lower degree of cellulose degradation in the wood was the lignin. Barclay et al. [1997], on the basis of their studies, showed that lignin acts as a hidden antioxidant. Phenolic groups of lignin can protect the cellulose, and as a consequence the wood, from the harmful action of radicals formed during ageing. Additionally, a change in the structure due to the cellulose isolation process may have been the reason for the greater degradation of the pure cellulose. It is possible that the chemicals (especially the concentrated nitric acid) modified the crystallinity leading to a material with less thermal resistance. This was confirmed by the results previously obtained using the FT-IR technique [Antczak 2010a]. Based on the results [Antczak 2010a], it appears that pure cellulose subjected to thermal ageing at 130°C in the air had a higher crystallinity index than cellulose in aged extracted and non-extracted wood. This proves that the cellulose separated using the Kürschner-Hoffer method was, to a great extent, composed of amorphous regions, which at elevated temperatures were more easily degraded.

Figure 3 shows the results of the weight average molar mass of the cellulose and the cellulose with antioxidants (EQ, PG and BHT), which were aged at 95°C under aerobic conditions in 65% relative air humidity. Based on these results, it can be concluded that the thermal ageing of cellulose under aerobic conditions in 65% relative air humidity caused a sharp reduction in the weight average molar mass of the cellulose. It is certain that the cause of such a large decrease in the molar mass was elevated humidity. In these conditions, the initiation of the hydrolysis reaction occurred, resulting in the rapid disintegration of the cellulose chains.

The addition of antioxidants to the cellulose matrix slowed down the degradation to a certain extent, but its complete inhibition was not possible. The curves describing a decrease in the weight average molar mass of the cellulose with a stabilizer (EQ, PG and BHT) followed a similar course regardless of the antioxidant used (fig. 3).

Figure 4 shows the molar mass distributions of the cellulose and the cellulose with the selected antioxidant – propyl gallate subjected to ageing at 95°C in the air (1008 h) in various relative air humidity conditions. The results confirm that air humidity influenced the degradation process of the cellulose. Based on the results (fig. 4), it can be observed that an increase in relative
humidity caused a shift in the cellulose distribution curve towards a lower molar mass. Firstly, the cellulose chains of the highest molar mass were degraded (the largest loss of the cellulose fraction on the right side of the distribution curve). The addition of the synthetic antioxidant (PG) to the cellulose matrix protected the cellulose to some degree (in a particular part of the fraction of the highest molar mass), but also in this case there was considerable degradation.

![Graph](image-url)

**Fig. 3.** The relationship between the weight average molar mass (\(M_w\)) and ageing time for cellulose and cellulose with antioxidants (EQ, PG and BHT) aged at 95°C in normal atmosphere and in 65% relative air humidity

![Graph](image-url)

**Fig. 4.** The molar mass distributions for cellulose and cellulose with antioxidant (PG) aged at 95°C (1008 h) in normal atmosphere and in various (anhydrous and 65%) relative air humidity conditions
Regarding wood ageing at 95°C in the air with 65% relative humidity, it can be seen that the slowest thermal degradation of cellulose occurred in the wood without extractives (fig. 5). This probably means (as earlier) that the lignin had a stabilizing effect on the cellulose degradation process. Furthermore, it appears that in these conditions, the lignin, which may have acted as a natural antioxidant, protected the cellulose more effectively than the synthetic antioxidants (a smaller decrease in the weight average molar mass of the cellulose in the aged wood without extractives than in the presence of the synthetic antioxidants) (figs. 3 and 5). Additionally, changes in the structure due to the cellulose isolation process may have been the cause of the greater degradation of the pure cellulose (as earlier). In turn, the presence of the extractives (as in anhydrous conditions) significantly accelerated a decrease in the weight average molar mass of the cellulose. The high temperature was probably the cause of the extractives’ much less efficient stabilizing activity. The decomposition of the unstable low-molecular extractives may have occurred under the influence of the high temperature. As a result of this decomposition, very reactive substances of a radical character may have formed, which accelerated the depolymerisation of the cellulose. Similar results were obtained by other researchers, who observed that extractives reduce the thermal stability of wood and wood composites [Shebani et al. 2008; Poletto et al. 2012; Sheshmani et al. 2012].

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**Fig. 5.** The relationship between the weight average molar mass ($M_w$) and ageing time for cellulose, cellulose in wood without extractives and cellulose in wood with extractives aged at 95°C in normal atmosphere with 65% relative air humidity
Figure 6 presents the molar mass distribution curves of various celluloses aged at 95°C (1008 h) in normal atmosphere with 65% relative air humidity conditions. The distribution of the curves confirms that the cellulose in the aged wood without extractives was the least degraded (the curve is the most shifted to the right). In turn, the presence of low-molecular extractives during the thermal ageing of the wood adversely affected the cellulose, because the molar mass was significantly reduced. As can be observed in figure 6, there was a large difference in the molar mass distribution between the cellulose in the aged wood without extractives, and the aged cellulose with the addition of the synthetic antioxidant (PG). The molar mass distribution curves of the cellulose from the SEC analysis show that, in these ageing conditions, lignin was much a better stabilizer than the synthetic antioxidants used.

![Graph showing molar mass distributions](image)

**Fig. 6.** The molar mass distributions for cellulose, cellulose with antioxidant (PG), cellulose in wood without extractives and cellulose in wood with extractives aged at 95°C (1008 h) in normal atmosphere with 65% relative air humidity

Figure 7 shows the results of the SEC analysis of the cellulose and the cellulose with antioxidants (EQ, PG and BHT) aged at 95°C in a nitrogen atmosphere in anhydrous conditions. As expected, the stabilizing effect of the synthetic antioxidants (PG, EQ and BHT) during the ageing of the cellulose in a nitrogen atmosphere was observed.

Comparing the changes in the average molar mass of the aged cellulose with the addition of propyl gallate (in a nitrogen atmosphere) with the results of this material aged in the air (figures 1 and 7) in anhydrous conditions, an interesting phenomenon was observed. The propyl gallate at an elevated temperature (95°C) in normal atmosphere (in anhydrous conditions) proved to be a catalyst for the cellulose degradation, whereas under anaerobic conditions it acted as an inhibitor.
Fig. 7. The relationship between the weight average molar mass ($M_w$) and ageing time for cellulose and cellulose with antioxidants (EQ, PG and BHT) aged at 95°C in a nitrogen atmosphere in anhydrous conditions

On the basis of the results obtained in these studies, the hypothesis is confirmed that, in certain conditions (oxygen atmosphere, high temperature), initiation of the oxidation and degradation of the polymer under the influence of antioxidants may occur. In turn, in the absence of oxygen or when there is insufficient oxygen (in a nitrogen atmosphere), this type of reaction does not occur and antioxidants in the majority of cases act as inhibitors of the thermal degradation of polymers.

Conclusions

In this paper, the thermal ageing of cellulose with natural and synthetic antioxidants under various conditions was studied. On the basis of the experiments performed, the following conclusions were drawn:

1. Among the used synthetic antioxidants, ethoxyquin was the best. In addition, BHT also had stabilizing properties and slowed down the depolymerisation of the cellulose.
2. In turn, relating to the degradation process of cellulose, propyl gallate showed inhibitory as well as catalytic properties. PG under aerobic anhydrous conditions accelerated the depolymerisation of the cellulose, while under anaerobic conditions (in a nitrogen atmosphere) and aerobic with 65% relative air humidity it slowed down the process.
3. Furthermore, in the ageing conditions applied, the smallest decrease in the average molar mass of the cellulose was observed in the wood
without extractives. The results indicate that, in this case, the lignin played the role of a hidden antioxidant. In the presence of the lignin, oxidative the cellulose depolymerisation process progressed more slowly than with the participation of synthetic antioxidants.

4. The extractives, at elevated temperature conditions, did not show stabilizing properties, and furthermore accelerated the degradation of the cellulose.

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RESEARCH REPORTS

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VARIABILITY IN STATIC BENDING STRENGTH OF THE “TABÓRZ” SCOTS PINE WOOD (PINUS SYLVESTRIS L.)

The paper presents the results of research on variability in the static bending strength of the “Tabórz” Scots pine wood (Pinus sylvestris L.). The wood samples for examination were obtained from the trunks of 260-year-old Scots pines felled in the “Sosny Taborskie” Nature Reserve. The mean value for all the tested wood samples amounted to 77.3 MPa, whereas for the individual trunks it reached 105.3 MPa, 66.4 MPa and 60.1 MPa, respectively. It is believed that the research results presented here are the first empirical data published concerning the mechanical properties of the wood of the “Tabórz” Scots pine, the trunks of which, due to their high quality, are considered in Europe to be extremely valuable timber.

Keywords: “Tabórz” Scots pine, static bending strength, wood

Introduction

Scots pine (Pinus sylvestris L.) is the predominant tree species in Polish forests, while its trunks are the most common raw material used by the wood-based industry. Regarding all of the species of trees growing in forests in Poland, the properties of pine wood are the most investigated [Paschalis 1980; Buchholz 1984; Paschalis and Staniszewski 1994; Tomczak et al. 2009, 2010; Jelonek et al. 2010, 2012; Tomczak and Jelonek 2012]. Particularly valuable specimens of Scots pine grow in Puszcza Taborska (Taborska Forest), within the Forest District of Miłomłyn, known as “Tabórz” Scots pine. The quality of this timber has been recognised in Europe since the 16th century, when it was mentioned for the first time in historical records. Despite the fact that the timber in question is

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well known and highly regarded all over the world, it is believed that no empirical data have yet been published describing its properties. This paper aimed to fill this gap, partially at least, and presents the results of studies on variability in the static bending strength of the “Tabórz” Scots pine wood.

**Materials and methods**

The research material was obtained from compartment 94a of the Forest Subdistrict of Tabórz, situated on the edge of the “Sosny Taborskie” Nature Reserve, in the Forest District of Milomlyn. According to the stand description, the pine trees in question are 260 years old [Forest Management Plan... 2014]. They grow in a fresh mixed deciduous forest, on rusty podzolic soil, mixed with beech (of 80, 120 and 200 years old) and oak trees (of 120 years old). Closure of the stand was determined as moderate, while the site index class for the pine was assessed as I. In the course of the sanitation cutting performed in this stand, three pine trees were felled. From each of them, a block with a length of 0.5 m was cut. Before cutting down the trees, their diameters at breast height were measured and the north direction was marked on their trunks, while after felling, the lengths of their stems were also measured. Due to the high monetary value of the butt-end parts of the felled trees, permission was only given for samples to be taken from the higher parts of the trunks. Therefore, the blocks were sampled from the stems at their half-length. A radial board enclosing the pith was cut out from every block in the N-S direction marked on the trunk, and then, two radial boards were taken out in the E-W direction. These boards were cut lengthwise into wood samples, the cross-sections of which had dimensions of 20 × 20 mm, according to the scheme presented in figure 1. The wood samples were then trimmed so their lengths were equal to 300 mm, and their longitudinal sections were smoothened. Within each of the geographically oriented segments (N, S, E and W), the wood samples were given respective numbers corresponding with a certain section of the trunk radius, according to the scheme in figure 1. Tests for static bending strength were performed on the
EDZ-20 universal testing machine with a measuring range of 0-200 kN. The samples were placed on the supporting elements of the machine and tests for static bending strength were performed on a three-point bending system, i.e. one pressing and two support ing elements, according to the procedure as referred to in the PN-77/D-04103:1978 standard. Following this, the moisture content of the wood was determined [PN-77/D-04100: 1978]. The value of the static bending strength of the wood obtained at a moisture content of W%, measured at the moment of the test performance, was recalculated and converted into the bending strength at a moisture content of 12%, according to the PN-77/D-04103:1978 standard. The computed values of the static bending strength at a moisture content of 12% were compared within the individual trees and the entire research material, and based on this, mean values and coefficients of variation were calculated.

**Results and discussion**

As mentioned above, the research material was obtained from the middle parts of the trunks of three felled pine trees. The diameters at breast height and lengths of their stems amounted to: 50 cm and 28 m for tree no. 1, 51 cm and 30 m for tree no. 2, 53 cm and 30 m for tree no. 3, respectively. The diameters of the 0.5-metre blocks, cut out from the trunks and measured with the bark, were as follows: tree no. 1: 37 cm, trees nos. 2 and 3: 38 cm each. The three aforementioned blocks were cut into 52 boards, which served to test the static bending strength of the wood. Tree no. 1 provided 21 boards (tab. 1): five boards from each of the northern (N), southern (S) and eastern (E) sections, and six boards from the western (W) one. From tree no. 2, a total number of 15 boards were obtained: five from the N section, four from the S one and six from the W section. Due to the fact that there was a knot in the eastern section of the block, obtaining boards from this part of the trunk proved to be impossible. The block cut out from tree no. 3 provided 17 boards: three boards from each of the N and S sections, six from the W one and five from the E section.

As shown in table 1, the mean value of the static bending strength for the entire research material amounted to 77.3 MPa. The mean values for the three examined trees were as follows: tree no. 1: 105.3 MPa, tree no. 2: 66.4 MPa and tree no. 3: 60.1 MPa. With regard to this parameter, the variability between the analysed trees amounted to 31.7%. A lower level of variability was observed for the trees that displayed similar values of the coefficient of variation: 16.1% for tree no. 1 and 19.5% for tree no. 3. The lowest static bending strength, amounting to 37.2 MPa, was recorded for a wood sample obtained from section 6, on the western side of tree no. 2, whereas the highest, 133.7 MPa, was for a sample from section 3, on the western side of tree no. 1.
Tab. 1. Presentation of the results of measurements concerning the static bending strength of the “Tabórz” pine wood

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<td>E 80.8</td>
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<td>E 99.9</td>
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<tr>
<td>mean 105.3</td>
<td>mean 66.4</td>
<td>mean 104.5</td>
<td></td>
<td>V [%] 16.1</td>
<td>V [%] 17.2</td>
<td>V [%] 15.4</td>
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<tr>
<td>In total mean 77.3</td>
<td>V [%] 31.7</td>
<td>mean 99.9</td>
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$\sigma_{12}$ – static bending strength of wood at a moisture content of 12%, V – coefficient of variation

The variance analysis revealed that there were statistically significant differences in the static bending strength between the investigated trees, while
Scheffé's test proved that this value for the wood of tree no. 1 was significantly higher when compared with the other two specimens, between which no statistically significant differences were detected (fig. 2).

![Box plot showing static bending strength of wood](image)

**Fig. 2. Static bending strength of the wood from the three investigated trees**

Due to the detection of clearly visible resinosis in 14 wood samples obtained from tree no. 1, the results of the tests performed for this specimen were subjected to the T-test, aimed at verifying whether the differences between the samples taken from the wood with and without resinosis were significant. However, it was not possible from this test to reject the hypothesis that there were no statistically significant differences ($p = 0.61276$). The mean values obtained for both of the examined groups amounted to 107.0 MPa for the wood with resinosis and 102.1 MPa for the wood without resinosis.

With regards to the geographically oriented segments, from which the wood samples were taken, no significant differences in the investigated parameter were established (Kruskal-Wallis test: $p = 0.4277$). The particular sections of the trunk radius did not differ in this respect either (variance analysis: $p = 0.7664$). Nevertheless, as presented in figure 3, an alignment of mean values for particular sections of the trunk radius took the characteristic shape of a curve, similar to a reversed parabola. The lowest mean value, 63.5 MPa, was recorded for samples from section 6, taken from the central part of the trunk, while the highest, 87.2 MPa, was observed for section 3.
Fig. 3. Static bending strength in particular sections along the radius of the trunk cross-section

The mean value of the static bending strength of the “Tabórz” pine wood, obtained in the course of this study and amounting to 77.3 MPa, falls within the very wide range given in the existing literature, comprising values between 34 and 205 MPa [Galewski and Korzeniowski 1958; Krzysik 1974, SpławaNeyman and Owczarzak 2006]. Such a wide range is most likely due to the fact that the studies were conducted on highly diversified material, taken from trees of varying ages, growing in varied habitats and under different climatic conditions. For instance, considerably lower values of static bending strength, compared with those presented here, were given by Vestøl and Hoibø [2010] for pine trees from Norway and Great Britain, where they amounted to 50.3 MPa and 53.8 MPa, respectively. Considering that in the above-mentioned research the wood from the butt-end parts of the trunks (near the nominal diameter for round wood) was examined, while the research material for this study was taken from the stems at their half-length, it can be assumed that comparing the values of the static bending strength of the butt-end parts of the trees in question would reveal even greater differences. This hypothesis is based on the results of the investigations carried out by Jelonek et al. [2011], according to which the static bending strength of wood sampled from a pine tree at its half-length is significantly lower than that of a sample taken at breast height. Such assumptions are also supported, though indirectly, by studies conducted by
Repola [2006], as well as Witkowska and Lachowicz [2012, 2013], which indicated that the density of pine wood decreases from its tip towards the trunk base, and this is the physical property that correlates with the mechanical properties of wood, the static bending and compression strength in particular [Krzysik 1974; Jelonek et al. 2005].

If it is assumed, based on the results of the investigations conducted by Jelonek et al. [2011], that the static bending strength of pine wood at a moisture content ranging between 30% (fibre saturation point) and 0% (absolutely dry wood) increases by ca. 1.5 MPa when the moisture content drops by 1%, the mean value obtained in this study is slightly lower (by ca. 3 MPa) than the one given in the above-mentioned research for a wood sample taken from the trunks of pine trees coming from the Forest District of Olesno (southern Poland), at their half-length.

The mean value of the static bending strength for tree no. 1, amounting to 105.3 MPa, was significantly higher when compared with the other two specimens. The value of the static bending strength of the wood sampled from tree no. 2 was lower by 37%, while for tree no. 3 it was lower by 43%. It is believed that such differences could primarily have been due to the resinosis recorded in 14 wood samples taken from tree no. 1. However, the statistical analyses did not confirm this assumption since the differences in the static bending strength between the samples with and without resinosis proved to be insignificant. Thus, it is possible that the significantly higher static bending strength of the wood samples from tree no. 1 was due to higher density of its wood, which may also be associated with its varied macrostructure. Indeed it was proved that the wood density of conifers is correlated with certain properties of their macrostructure, such as annual ring width and share of late wood [Wąsik 2007].

An analysis concerning the variability in the static bending strength of the “Tabórz” pine wood depending on the distance of the wood sample from the trunk pith indicated that its highest values were recorded more or less at the half-length of the radius of the trunk cross-section (section 3), whilst the lowest values were encountered in the centre, near the pith, comprising so-called juvenile wood. Similar results were obtained by Aleinikovas and Grigaliūnas [2006] for pine trees from Lithuania, as well as Tomczak and Jelonek [2013] for pine trees from western Poland. Moreover, the latter authors reported similar distributions of wood density along the trunk radius. This confirms the relationship between the mechanical properties of pine wood and its density, as mentioned above. Therefore, one might expect the wood density of the investigated “Tabórz” Scots pine to be highest at the half-length of the radius of the trunk cross-section, although this is just an assumption and should be verified at a further stage of the research.

The “Sosny Taborskie Nature Reserve”, where this research was carried out, is passively protected [Dziekoński 2004]. A discontinuation of silvicultural
treatments, supporting the main objective of establishing the nature reserve for this particular Scots pine, may result in a decrease in the highly regarded quality of its wood in the future. This concern is based on the results of comparative studies relating to the technical quality of the wood of spruce trees growing in commercial stands and nature reserves [Michalec et al. 2013]. Furthermore, Dziekoński [2004] indicated in recent years a marked decrease in the share of pine in the “Sosny Taborskie” Nature Reserve, with the pine being displaced by broadleaved species, mostly oak and beech. The above-mentioned author anticipates that maintaining the current form of protection may result in an absolute elimination of pine species from the reserve. In this context, the results of the examination of the “Tabórz” Scots pine in terms of the static bending strength of its wood, presented in this paper, may become a valuable historical record in the future. For the very same reasons, the scope of investigations into this particular pine wood should be extended to include other mechanical and physical properties, as well as its macrostructure and anatomy.

Conclusions

Based on the results obtained from studies on the variability in the static bending strength of the “Tabórz” Scots pine wood, the following statements and conclusions may be drawn:

- The mean value of the static bending strength of the wood for the entire research material amounted to 77.3 MPa, while the mean values for particular trees were as follows: 105.3 MPa, 66.4 MPa and 60.1 MPa, respectively.
- The wood from tree no. 1 was characterised by a significantly higher static bending strength, when compared with the other two specimens, which might have been due to its different macrostructure and density.
- The mean value of the static bending strength of the “Tabórz” pine wood obtained in this study was higher than those reported for pine trees from Finland and Great Britain, whereas it was similar to that of pine trees from southern Poland.
- The lowest value of the static bending strength was recorded for the wood from the central part of the trunk, located near the pith, while the highest was observed at the half-length of the radius of the trunk cross-section.
- Regarding the fact that there are no published empirical data describing the properties of the “Tabórz” pine wood, it is recommended that the scope of research is extended to include other mechanical and physical properties of this pine wood, including the variability in its macrostructure and anatomy.
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List of standards


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Tomasz Stawicki, Paweł Sędłak

STUDY OF THE IMPACT OF LUBRICANT TYPE ON SELECTED OPERATIONAL PARAMETERS OF A CHAINSAW USED IN BEECH TIMBER CUTTING

The focus of the study was to determine the influence of the lubricant used for greasing the cutting subsystem of a petrol chainsaw on selected operational characteristics. Three lubricating oils were studied, two of which were commercially available, while the other was a suitably modified rapeseed oil, prepared specifically for the study. It was shown that the application of selected lubricating components in the cutting subsystem of the chainsaw resulted in significant differences in the consumption of both the fuel and the lubricating agent.

Keywords: petrol chainsaw, chainsaw, oil consumption, fuel consumption

Introduction

The use of portable chainsaws carries the risk of environmental contamination. This is a result of the usual design of a chainsaw, featuring an open lubricating system. In a system like this, the oil is dispersed and released into the environment via a variety of channels. It is estimated that between 50 and 85% of the oil is absorbed by sawdust, while, during rough-hewing, depending on the type of work performed, anything between 10 to 35% of the oil goes directly into the soil [Skoupy 2004]. According to previous research data [Rudko et al. 2010], under Polish felling conditions, i.e. implemented working methods and the amount of harvested timber (30 million m³ per year), as much as 6 million dm³ of oil finds its way into the soil every year. However, this does not reveal the full scale of the problem, as chainsaws are now used extensively by non-professionals. The amateur application of chainsaws makes it impossible to monitor how they are used, which, with insufficient technical knowledge and a lack of environmental risk awareness, may result in dangerous practices. The existing literature on the subject identifies the danger caused by the application
of used motor oils for the lubrication of the chainsaw cutting subsystem [Giefing 1991; Rudko and Rybczyński 2010].

Considering the scale of the potential risks, it is not surprising that new solutions are being sought to minimize the negative impact of chainsaw lubricants on the natural environment. In scientific studies, it has been suggested that only bio-degradable oils are appropriate for use as chainsaw lubricants, and ones which meet specific quality standards [Lauhanen et al. 2000; Zembrowski et al. 2010], rather than mineral oils, which are still widely used in the lubrication of chainsaws. Other research has been conducted to study the influence of design solutions and the operating parameters of chainsaws on their effectiveness [Gendek 2006; Maciak 2013]. The results of such studies may be considered in terms of pro-ecological recommendations, which might provide guidelines for the reasonable management of consumables (both lubricating oils and fuels). Particularly relevant to the matter under discussion is a study on the influence of lubrication intensity and type of lubricant on the movement resistance of the chainsaw [Wojtkowiak and Tomczak 2003; Wojtkowiak 2004; Nordfjell et al. 2007; Rudko and Rybczyński 2010]. Complementary data on both the durability of the chainsaw cutting subsystem and safety of the natural environment would be a desirable outcome of such a study.

An overview of the existing literature led to the conclusion that it was worthwhile conducting experimental research on the possibilities of the application of lubricating substances for chainsaws, which are safe for the environment and, at the same time, do not cause a deterioration in the operational parameters of these machines. With that in mind, the present study took the form of a field experiment and the adopted method in which the study was conducted made it possible to transfer the results to wood cutting in forests.

In addition, it may also be applicable for the use of motor chainsaws in the preparatory processing of fuelwood, e.g. in individual households. It was considered that the use of chainsaws for the processing of fuelwood, combined with the increased interest in this method of processing wood in households, generates a problem of particular gravity, relating to the risk of environmental contamination in rural areas.

**Materials and methods**

The study intended to determine whether the application of various lubricating components of the cutting subsystem of the Husqvarna 357 XP chainsaw could significantly affect its operational parameters, and, in particular, the consumption of consumable supplies: fuel and chainsaw lubricating oil. Three oils were compared, two of which are available commercially, while the last, rapeseed oil, was modified by the authors of the study. In the case of those products dedicated to the lubrication of the chainsaw cutting subsystem, one was the result of the processing of crude oil (mineral base) and, according to the
manufacturer, it additionally contained an undisclosed percentage of vegetable oil. This oil is referred to in the study as “A”. The other lubricating component, marked with as “B”, was, according to the manufacturer's claims, composed of vegetable ingredients (the list of ingredients was not provided).

As mentioned earlier, the third lubricant was rapeseed oil modified with sulphur content at an experimentally determined level (purely for analysis). For this oil a number of measurements of lubrication parameters were conducted in a four-ball tribometer test, in accordance with the standard methodology [PN-76/C-04147]. An analysis of the results demonstrated desirable changes in the lubrication properties of the rapeseed oil, with only a 1% content of sulphur, which in turn suggested this type of oil was suitable for use as the lubricating component in the chainsaw's cutting sub-system.

The main study was performed in 2013. The wood material was beech wood, arranged in stacks, numbered accordingly and divided into research samples, 100 cylinders each. The cylinders were numbered according to their location in the stack, and their average diameter was established by measuring the smallest and the greatest diameter of the cylinders at each end of the cylinder (arithmetic average of the four measurements). The diameters established this way were used to calculate the surface area of the kerfs on each cylinder. As each cylinder was cut four times, the total cutting area for the studied sample was calculated as the ratio of the total surface areas of the kerfs and of the number of cuts made, as demonstrated below:

\[
F_c = \frac{4 \cdot \sum_{i=1}^{100} \pi d_i^2}{4} 
\]

(1)

where: \( F_c \) – total surface area of the wood cut in the studied sample [m²],

\( d_i \) – diameter of the \( i \)-th shaft [m].

In relation to thus specified cutting surface, the mass fuel consumption was calculated, as well as the volume consumption of the oil lubricating the cutting sub-system of the Husqvarna 357 XP motor chainsaw. Field tests were carried out in two stages. Each of them included the cut test of one hundred wood cylinders used for each of the three lubricating oils (6 test samples in total). The results were not examined immediately after the first stage of the study. In view of a chance to repeat the tests, the second stage was regarded being suitable for verification of the assumed methodology for assessment of the functional qualities of tested oils. Repeating the tests also made it possible to assess, by comparing the results for the same lubricants, how the random distribution of the wood cylinder diameters used in the test samples affected the measurement results.
As a result of the research being carried out in field conditions, there was no control over a number of the parameters (e.g. ambient temperature and air humidity), as well as the execution of the cutting process at constant force values (e.g. chainsaw feed speed, chainsaw motor rotational speed). It should be noted, however, that although such conditions differ significantly from the laboratory trial regime, they resemble more closely the actual way chainsaws are used.

Due to a number of technical and operational factors determining fuel and lubricating oil consumption, the research was adapted so as to make it possible to draw conclusions on the basis of a comparative assessment of the results:

- the wood cutting was carried out using the same chainsaw, after prior maintenance activities (e.g. adjusting fuel and lubrication sub-systems, replacement of filters and of the chainsaw driving wheel),
- the individual study trials were performed using new chainsaws and guides (produced by the OREGON Company),
- on the basis of preliminary studies, the size of the study sample was adopted so as to make it possible to perform the scheduled work without the need to sharpen the sawing chain (successive sharpening operations, in particular when performed manually, do not ensure repeatability of chainsaw cutting blades geometry, which would result in a change in the parameters of the performance of the cutting sub-system),
- before commencing each study trial, the chain was initially tensioned in accordance with the PN-ISO 6535 [PN-ISO 6535:1999] standard,
- the wood processing was performed by a team of three, consisting of an experienced lumberjack who made the cuts, and two assistants who loaded the wood cylinders from the stack onto the sawbuck, resulting in efficient chainsaw use and a working pace comparable between individual study trials.

Results and discussion

In order to attain the major research goals, a number of indications were used and measurements taken, the results of which are presented in table 1. In accordance with the methodologies described above, the total surface area of the cuts in the study samples was determined, and in relation to this, the mass fuel consumption of the chainsaw and the volumetric consumption of lubricating oil were defined. During the field work, the fuel and lubricating consumption were assessed by weight, and because of the different specific gravities of the oils used, their consumption was calculated in laboratory conditions following a determination of their densities using the pycnometer method. Table 1 also shows the dynamic viscosity measurements of the oils used, measured using a Bookfield viscometer, in accordance with DIN 53019 (DIN 53019-1: 2008). Parameters such as the viscosity and lubricity of oils (the tendency of the lubricating agent to form durable adsorption films on friction surfaces) can affect
the resistance of the cutting system to motion, and therefore constitute determinants of fuel and lubricating oil consumption. However, the study did not focus on the assessment of tested lubricants with respect to their viscosity and lubricity, but the aim was to determine the differences in the chainsaw’s consumption of working fluids depending on the lubricating oil used in the cutting system. It was concluded that the investigated variables directly determined the usefulness of the oils used, indirectly pointing to the differences in their rheological characteristics.

Table 1. Summary of primary test results

<table>
<thead>
<tr>
<th>Tested parameter</th>
<th>Cutting sub-system lubricating oils in the Husqvarna 357 XP chainsaw</th>
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<tbody>
<tr>
<td></td>
<td>Oil “A”</td>
</tr>
<tr>
<td></td>
<td>Sample</td>
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<tr>
<td>Surface area of the wood cut in the studied sample ( F_c ) [m²]</td>
<td>13.41</td>
</tr>
<tr>
<td>Chainsaw fuel consumption [g]</td>
<td>1553.3</td>
</tr>
<tr>
<td>Lubricating oil consumption [g]</td>
<td>626.72</td>
</tr>
<tr>
<td>Volume consumption of oil [cm³]</td>
<td>699.14</td>
</tr>
<tr>
<td>Oil consumption relative to the area of the cut [cm³·m⁻²]</td>
<td>52.13</td>
</tr>
<tr>
<td>Fuel consumption relative to the area of the cut [g·m⁻²]</td>
<td>115.82</td>
</tr>
<tr>
<td>Density at a temperature of 293 K [g·cm⁻³]</td>
<td>0.896</td>
</tr>
<tr>
<td>Dynamic viscosity at a temperature of 293 K [mPa·s]</td>
<td>209.21</td>
</tr>
</tbody>
</table>

The temperature, at which the density and viscosity of the oil (293 K) was determined, was selected from the range of the variability of the thermal environmental conditions in which the experimental tests were carried out. The assumption was also made that the variability of the ambient temperature within a range of several degrees which characterized the existing thermal conditions should not be a factor influencing the objectivity of the research. The fact that the cutting of the wood was conducted with periodically repeated short breaks, in order to refill the operation fluids, favoured a stabilization of the thermal
loads of the sawing machine. Taking into account that the oil reservoirs had relatively low volumes and the effect of heat transfer from the structural elements to the oil, this should have lead to temperature stabilization. These considerations, although not verified experimentally, became the basis for calling into question the comparability (as regards temperature) of the working conditions of the lubricating oils. On the basis of these assumptions, it was considered possible to interpret the differences in the amount of fuel and lubricating oil consumed by the saw as a result of the variability in working qualities (including rheology and lubricity) of the lubricants used in the cutting system. In the following part the study focused exclusively on identifying the impact of the lubricating agents used in the cutting system on the consumption of consumables, that is, on verifying the main postulate of the research.

As is clear from the data presented in table 1, in the case of the “B” oils and the rapeseed oil modified with sulphur, a similar level of consumption was found, expressed in cm³·m⁻² of the wood cutting area. In the 1st and 2nd study trials, a higher rapeseed oil consumption was registered, by 5.8% and 3%, respectively, in relation to the “B” oil. In light of this, oil “A” (with a mineral base) fared rather unfavourably, and its consumption in the first study trial was nearly 21% higher than that of oil “B”. The fuel consumption measurement results indicated that the rapeseed oil modified with sulphur had the best operational properties of all the lubricating agents used for the chainsaw cutting sub-system. With this oil, the fuel consumption was lowest, expressed as g·m⁻² of the cutting surface area, and in relation to oil “A”, it was lower by 24% and 15%, respectively, in the first and second study trials. In relation to oil “B”, the use of rapeseed oil as the chainsaw lubricant resulted in a reduction in fuel consumption by approx. 10%, in both the study trials performed.

The test results were statistically analyzed, with the intention of establishing whether the random variability of the cylinder diameters in the individual trials could have affected the reported differences in the consumption of the lubricating oils and the fuel consumption by the chain saw. Parametric tests for significance by t-Student were carried out to verify if there were significant differences between the diameters of the cylinders cut in individual study trials. For the adopted significance level $\alpha = 0.05$, there was no evidence to reject the null hypothesis of the equality of averages, which indicated a lack of statistically significant differences between the mean diameters of the wood cylinders used in the test samples. The test results, however, did not specify the distribution of the variable tested in the research trials, which was considered an interesting supplement to the statistical analysis. For this purpose, figure 1 shows the frequency histograms and the corresponding distributions of the density of the average diameters of the cylinders.
Fig. 1. Histograms of distribution of wood cylinders diameter frequencies in study trials: a, b – oil “A” (trials 1 and 2), c, d – oil “B” (trials 1 and 2), e, f – modified rapeseed oil (trials 1 and 2), s – standard deviation [m], $\bar{x}$ – average cylinders diameter in the study trial [m]

In the first study trial, in which oil “A” was used to lubricate the cutting sub-system (fig. 1a), the greatest asymmetry in the distribution of the variable under examination was found, tending towards values lower than the trial average $\bar{x} = 0.198$ m. The demonstrated variability of diameters resulted in a relatively higher, than in other cases, percentage of works performed with lower cutting resistance and higher chainsaw motor rotational speed. As a result, this could have translated into an increased consumption of fuel and lubricating oil, relative to the unit surface area of the saw cuts. It should be noted that in the case of the second study trial, all the histograms of the distribution frequency of the cylinders cut with the application of individual lubricants were closer to the normal distribution. In spite of this, in this case also, the application of oil “A” resulted in the highest consumption of oil and fuel relative to the surface area of
the cut. The statistical analysis performed provided the basis for the recognition of the adopted research methodology as one that enabled an objective evaluation of the operational parameters of the lubricating oils based on a comparative assessment of the results.

Conclusions

The analysis of works performed when cutting the beech wood cylinders demonstrated significant differences related to the consumption of both fuel and lubricating oil by the cutting sub-system of the Husqvarna 357 XP chainsaw, depending on the type of lubricant used. The highest consumption of oil and fuel, relative to the surface area of the wood cut, was observed when using the mineral oil, which constituted useful practical information for operation of chainsaws. This means that the use of lubricating components produced from plants may be justified not only from an economical, but also from an ecological perspective. One aspect of vegetable oil-based lubricants deserving particular attention is the reduction in the risk of environmental contamination, particularly soil and water contamination during work in the forest and when preparing wood for heating, and air pollution, which may occur when the sawdust resulting from wood processing is burned, as is often the case. The results achieved in this study should encourage further research on the suitability of rapeseed oil for all-year-round application, both for individual usage, as well as for timber extraction in forests. It would be particularly useful to confirm the desirable operational characteristics of rapeseed oil in cold temperatures, and its physico/chemical stability in storage, as this information would influence the use of chainsaws.

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List of standards


PN-76/C-04147. Badanie własności smarowych olejów i smarów (Research into the lubricating properties of oils and greases)

Maciej SYDOR, Marcin WOLPIUK

ANALYSIS OF RESISTANCE TO AXIAL WITHDRAWAL OF SCREWS EMBEDDED IN LOCALLY REINFORCED MDF

This paper presents results of the analysis on screw withdrawal strength in locally reinforced MDF. Various amounts of the agent locally reinforcing the boards were applied and universal screws and euro screws were tested. Experiments were conducted using a universal strength testing machine, with samples collected following the guidelines of the ISO 27528:2009 standard. The following parameters were measured: 1) work required for maximum force, 2) maximum force, 3) withdrawal capacity and 4) stiffness. The research showed a 100% increase in resistance to axial withdrawal of universal screws and a 50% increase in euro screws in comparison to the analogous results for the non-reinforced board. Optimal amounts of the reinforcing agents were also determined.

Keywords: screw, universal screw, euro screw, withdrawal, holding strength, MDF, fibreboard, local modification, local reinforced

Introduction

Medium-density fibreboard (MDF) is produced by binding wood fibres with a synthetic binding agent. In Europe this board is available in a thickness ranging from 1.8 to 60 mm and it is commonly used in the furniture industry and as architectural built-in elements. Structures made from MDF are frequently exposed to considerable loading and one of the methods typically used to connect MDF is to apply steel threaded fasteners. The strength of structural steel is at least 10 times greater than that of wood-based materials (in practice it is 10-15 times greater). The failure of an over-stressed joint comprising of steel threaded fasteners causes the failure of wood-based board. Thus, joint strength is determined by the strength of wood-based panel elements rather than that of screw connectors. Furniture design requires quality verification in terms of its rigidity and the strength of the applied construction solutions [Smardzewski and Kłos 2011]. In the past to improve the strength of wood-based boards, twenty or thirty percent solutions of gluten glue or urea-formaldehyde resin were applied.
inside the pilot holes. This solution provided an enhanced screw holding capacity and uniform values for different fastening directions [Ławniczak and Paprzycki 1961]. Wood based composites were also investigated after vacuum-impregnation of copper azole and chromated copper arsenate [Taşçioğlu et al. 2014]. Advances in polymer chemistry have led to the development of novel polyurethane products with potentially advantageous properties [Proszyk 2009].

The aim of the study was to investigate the effect of local reinforcement of MDF with a novel polyurethane product on resistance to axial withdrawal of screws.

The experiment described in this paper was conducted whilst taking into consideration the conclusions and observations made in the course of preliminary studies by Pohl and Wolpiuk [2011], e.g. the range of applications was increased, the experimental stand was modified to ensure the acquisition of increased measurement data, while changes were introduced in the design of the pull-out testing machine to increase its rigidity. Additionally, the strength properties were precisely determined for the MDF, from which samples were collected.

**Material and methods**

Analyses were conducted on MDF of 18 mm in thickness. The wood-based panel was composed of three layers (fig. 1) [Geimer et al. 1975; Wilczyński and Kociszewski 2007]. The averaged material properties of the two outer (face) layers (of arbitrarily assumed 2.2 mm thickness) were identical, while the core, of lower density, was characterised by markedly different material properties.

The research was used on a board produced by the same manufacturer as used in the Kociszewski’s study [Kociszewski 2014]. Table 1 presents the numerical values of the material properties for layers of MDF, from which samples were collected.

Analyses were conducted for two types of fasteners, commonly used in the assembly of furniture joints, i.e. universal screws of $4 \times 35$ and euro screws of $6.3 \times 23$ in size (fig. 1).

MDF samples had dimensions of $75 \times 75 \times 18$ mm (specified in the standard ISO 27528:2009). Fasteners were mounted in the face of test pieces to a depth of 15 mm into previously drilled pilot holes with a diameter corresponding to the minor diameter of the screw (diameter $D_2$ in fig. 2). The manner of screw mounting in the panel samples and the direction of the pull-out force is presented in figure 3.
Table 1. Physical properties of wood-based panels used in research

<table>
<thead>
<tr>
<th>Parameter</th>
<th>MDF(^1)</th>
<th>MDF with PUR application(^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness (t) (mm)</td>
<td>18.0 ±0.2</td>
<td></td>
</tr>
<tr>
<td>Moisture content (\varphi) (%)</td>
<td>7.7</td>
<td></td>
</tr>
<tr>
<td>Density (\rho) (kg/m(^3))</td>
<td>mean</td>
<td>787.7</td>
</tr>
<tr>
<td></td>
<td>outer layer</td>
<td>952.7</td>
</tr>
<tr>
<td></td>
<td>inner layer</td>
<td>732.5</td>
</tr>
<tr>
<td>Young's modulus (GPa) (EN 310)</td>
<td>(E_{x1})</td>
<td>6.08</td>
</tr>
<tr>
<td></td>
<td>(E_{y1})</td>
<td>5.88</td>
</tr>
<tr>
<td></td>
<td>(E_{z1})</td>
<td>1.16</td>
</tr>
<tr>
<td></td>
<td>(E_{x2})</td>
<td>2.68</td>
</tr>
<tr>
<td></td>
<td>(E_{y2})</td>
<td>2.65</td>
</tr>
<tr>
<td></td>
<td>(E_{z2})</td>
<td>0.50</td>
</tr>
</tbody>
</table>

\(^1\)According to Kociśzewski [2014].
\(^2\)According to the authors' study.

Fig. 1. A model of a three-layer wood-based panel: \(x\) – in the board plane perpendicular to direction of mat formation, \(y\) – direction of panel production, \(z\) – perpendicular to board plane.

The panel was locally reinforced using the PUR 555.6 nano polyurethane product by Kleiberit. The application rates of the product were determined based on the volume of fastener threads. The volume of the applied preparation is referred to as application unit \((V)\), described by the formula:

\[
V = \frac{\pi (D^2 - D_3^2) \cdot L}{4}
\]
where: $L$ – depth of fastener mounting  
$D$ – major diameter of the thread  
$D_3$ – minor diameter of thread.

![Fig. 2. Fasteners used in tests: a) universal screw; b) euro screw](image)

![Fig. 3. Manner of fastener mounting](image)

Thus the volume of the application unit depends on the dimensions of the tested screw. In the described experiment two values of the application unit were used: for universal screws $V_U = 0.12 \text{ cm}^3$, while for euro screws $V_E = 0.28 \text{ cm}^3$. Nine application rates were adopted, from zero to eight units (tab. 2).
Table 2. Volume of applied panel reinforcement agent depending on fastener type

<table>
<thead>
<tr>
<th>No. of application units</th>
<th>Volume of reinforcement agent (cm³)</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$V_U$ (universal screw)</td>
<td>$V_E$ (euro screw)</td>
</tr>
<tr>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>1</td>
<td>0.12</td>
<td>0.28</td>
</tr>
<tr>
<td>2</td>
<td>0.24</td>
<td>0.56</td>
</tr>
<tr>
<td>3</td>
<td>0.36</td>
<td>0.84</td>
</tr>
<tr>
<td>4</td>
<td>0.48</td>
<td>1.12</td>
</tr>
<tr>
<td>5</td>
<td>0.60</td>
<td>1.40</td>
</tr>
<tr>
<td>6</td>
<td>0.72</td>
<td>1.68</td>
</tr>
<tr>
<td>7</td>
<td>0.84</td>
<td>1.96</td>
</tr>
<tr>
<td>8</td>
<td>0.96</td>
<td>2.24</td>
</tr>
</tbody>
</table>

The reinforcement agent was immediately applied into the previously drilled pilot holes using a syringe. Figure 4 presents the section of a hole left after the withdrawal of a euro screw, with the darker zone representing the area penetrated by the reinforcement agent.

![Image of a cross section of a hole](image)

**Fig. 4.** A cross section of the modified MDF sample by the PUR reinforcing agent (shows the area of modification of the prebored pilot hole for euro screw)

The screw holding capacity in the locally reinforced MDF samples was tested 72 hours after the reinforcement agent application and screw mounting, i.e. after the complete curing of the preparation. The pull-out test was performed in a Zwick Z050 universal strength testing machine at the following parameters:
1. initial force: 5 N,
2. after the initial force was reached screws were pulled out at 2 mm/min.

In the course of the experiment, the holding force of screws in the panel was measured depending on the translocation (withdrawal of the pulled-out screw from the hole). This made it possible to determine the following values:

1) The amount of work needed to reach maximum force (withdrawal work denoted as $W_w$, expressed in joules) – calculated on the basis of the pull-out strength ($F$) and displacement ($s$), which is represented by the area under the curve in figure 5,

2) The amount of withdrawal strength denoted as $W_s$, expressed in newtons) – read from the testing machine as a maximum force during each experiment,

3) The amount of withdrawal capacity denoted as $W_c$, expressed in newtons per millimetre) – the screw holding value of a test piece is the quotient of maximum force ($F_{max}$) and embedded depth of the screw ($L$) – according to the recommendations of ISO 27528: 2009,

4) stiffness (denoted as $C_f$, expressed in newtons per mm and being a quotient of force to limit of proportionality and translocation to limit of proportionality – fig. 5).

![Figure 5. Method of calculating stiffness](image-url)
For each screw, a series of 9 samples were prepared for each application rate. For two types of the tested screws this yielded a total of 162 pull-out tests (9 samples in a series, 9 series – from 0 to 8 application units, 2 types of screws: 9 · 9 · 2 = 162). Based on the nine replications performed for each series, the standard deviations were calculated for each series and shown on graphs as error bars.

**Results and discussion**

The results of the experiment are presented in four graphs (figs. 6-9). The bar graphs present the test results of the holding capacity of universal screws and euro screws mounted in the face of MDF. Figure 6 presents work to maximum force – withdrawal work ($W_w$), figure 7 – recorded values of maximum force – withdrawal strength ($W_s$), while figure 8 presents the holding force of screws per unit of screw penetration length (i.e. values from fig. 7 divided by the depth of screw mounting of 1.5 cm – withdrawal capacity) ($W_c$). Figure 9 presents changes in stiffness ($C_i$) depending on the number of application units of the reinforcement agent.

![Graph showing work to failure depending on the number of application units of reinforcement agent](image)

*Fig. 6. Work to failure depending on the number of application units of reinforcement agent*
Fig. 7. Maximum holding force depending on the number of application units of reinforcement agent

Fig. 8. Screw withdrawal capacity depending on the number of application units of reinforcement agent
Conclusions

- The polyurethane agent at the rate of 6 application units (0.72 cm³) improved withdrawal capacity of universal screws by over 100% (from the initial approx. 780 N/cm to approx. 1650 N/cm – fig. 8). A further increase in the amount of the reinforcement agent did not result in an enhancement of withdrawal capacity of universal screws.
- In the case of euro screws the capacity increased by approx. 50% already at two application units (0.56 cm³). A further increase in the number of application units of the curing agent resulted in a slight reduction of withdrawal capacity of euro screws, thus it is not justified (fig. 8).
- Stiffness, i.e. the ratio of force to the limit of proportionality, and translocation to the limit of proportionality in the case of universal and euro screws reached its maximum value at four application units (for 0.48 and 1.12 cm³, respectively). In the case of universal screws stiffness increased by a maximum of approximately forty percent, while in the case of euro screws – by approximately sixty percent (fig. 9).

Based on the recorded results it may be stated that the use of local modification of MDF is justified in the case of strongly over-stressed joints. Screws mounted within locally reinforced panels exhibit an increased withdrawal capacity of fifty to one hundred percent in comparison to screws mounted within boards, which are not locally reinforced. Screws have to be mounted within a short time after the application of the reinforcement agent.
(upon its complete curing it is practically impossible to mount the fasteners due to the significant increase in resistance of screws being screwed in).

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Ján Parobek, Hubert Paluš, Martina Kalamárová, Erika Loučanová, Anna Križanová, Katarína Repková Štofková

COMPARATIVE ANALYSIS OF WOOD AND SEMI-FINISHED WOOD PRODUCT TRADE OF SLOVAKIA AND ITS CENTRAL EUROPEAN TRADING PARTNERS

The aim of this paper was analyse the competitiveness of wood and semi-finished wood products in Slovakia in comparison with selected Central European countries. The research applied the commonly used competitiveness index, the Revealed Comparative Advantage (RCA) index, to analyse the position and changes in trade competitiveness of Slovakia during the period 2009-2013. Additionally, the Comparative Price Level (CPL) index was used to evaluate the differences in foreign trade prices for industrial roundwood and selected semi-finished wood products in order to examine the position of the Slovak wood and wood product trade. The results showed that Slovakia had the strongest comparative advantage in the trade in industrial roundwood due to its sufficient wood resources and competitive prices.

Keywords: CE countries, competitiveness, revealed comparative advantage, wood and semi-finished wood product trade, comparative price level

Introduction

The use of domestic renewable resources is significantly influenced by political, social and economic changes. Wood production has a long tradition in the Slovak Republic and wood as a significant renewable resource is closely linked to many other sectors of the national economy. The competitiveness of each sector depends on the process of restructurisation and modernization of production facilities, as well as the process of the specialization of production [Šupín 2013]. The most recent theory of the competitiveness of countries on...
world markets is based on international trade and economic growth. This theory represents new aspects of innovation, the real utilization of resources and economic development.

Wolff et al. [2007] state that the concept of competitiveness is rather complex as the term is used at different levels of aggregation with different meanings. The concept of competitiveness can be distinguished at product level, business unit or firm level, at industry level and at regional or national level. In a broader context, Latruffe [2010] defines competitiveness from two perspectives: (i) as the ability to face competition and to be successful when facing competition, and (ii) as the ability to sell products that meet demand while at the same time ensuring profits over time which enable the firm to thrive.

Forest industry companies must continually strive to improve or at least maintain their market share [Oblak and Glavonjić 2014]. One phenomenon of the development of the forest industry is the placement of products on the global market, increasing added value and more efficient utilization of wood [Paluš et al. 2015; Ambrušová and Šulek 2014]. The forest industry has several clear comparative advantages in comparison with other sectors: for instance, sufficient input based on renewable resources, or the possibility of using recycled material. Forest wood of poor quality and dimensional parameters, side products (residues) from wood processing, post-consumer wood, and wood from fast-growing tree plantations are the most accepted energy carriers both politically and socially, the use of which contributes to a reduction in the share of coal in electric and thermal energy generation [Ratajczak et al. 2012]. In the case of environmentally sensitive markets, the competitiveness of forest products can be influenced by factors related to the origin of the wood material from sustainable and renewable sources [Kaputa 2013]. From the perspective of the national economy, this sector is able to utilize a high proportion of input based on domestic resources [Lagaña et al. 2008].

Due to a growing global demand for wood and wood products, it is crucial to be competitive on the international market in order to make use of the potential gains of increased demand. A country that best utilises its given resources within a particular sector may enjoy a significant comparative advantage. According to Noor et al. [2008], the concept of comparative advantage is derived from the traditional theory of international trade, while the term competitiveness goes beyond comparative advantage, as no country can be competitive in every economic activity. Porter [1990] claims that productivity is the only meaningful concept of competitiveness. According to Kagochi [2007] some of the underlying factors that influence competitiveness include technology, human capital, product quality and differentiation, exchange rate, and other external factors.

Traditional trade theory explains international competitiveness in terms of the comparative advantage of nations: a nation engages in trade and gains a comparative advantage not because it can produce a good or service much
more cheaply, but because it is relatively more efficient than other nations in producing this good or service [Ricardo 1911]. The theory proves that each nation would benefit from specializing in the product in which it enjoys a comparative advantage, that way raising the total global output of each product and improving the situation of all participating nations [Carvalho et al. 2009]. Several indicators have been developed to measure the competitive situation of a specific sector or country. According to Gries and Hentschel [1994], these can be classified into result-oriented indicators and determinant-oriented indicators. Many studies using the result-oriented indicators to evaluate the competitiveness of forest-based sectors and related agricultural sectors have been elaborated in different countries [Prasad 2004; Dieter and Englert 2007; Gonuguntla 2007; Noor et al. 2008; Carvalho et al. 2009; Zhang et al. 2012; Song and Gazo 2013, Loučanová et al. 2014; Paluš et al. 2014]. The application of generally applicable rules for measuring the competitiveness of the wood processing industry at meso and macro level provides new insights to support the sector in order to maximize the optimal use of domestic renewable resources.

The forest industry is one of the sectors in which the Slovak economy may at least partly influence European markets with the maximum utilization of its own resources. There is an effort to increase added value and to support the domestic consumption of wood commodities. This effort and the development of the forest industry depends in the broader context on society-wide interest, and in the narrow context on different stakeholders entering the wood product chain. The wood market in Slovakia is constantly developing, and the demand for roundwood changes depending on the possibilities of its use. There are many factors influencing production and consumption patterns. On the one hand, timber production is subject to the available resources, which are the result of long-term forest management and long-term planning. On the other hand, timber production tries to adapt to rapidly changing market conditions and the requirements of wood processing sectors which vary over a relatively short period of time. According to Parobek et al. [2014a], besides the pulp and paper industry, the forest industry producing final higher added value wood commodities such as furniture, wood construction, etc. is, in many cases, still unable to compete on the European market. Therefore, the production and export of wood and semi-finished products is an important part of the Slovak forest industry’s revenue.

The aim of this paper was analyse the competitiveness of the wood and semi-finished wood product sectors compared to other sectors in the national economies of Slovakia and selected trading partners. The research applied the commonly used RCA index to analyse the position and changes in competitiveness in selected Central European countries during the period 2009-2013. Additionally, it evaluated the differences in foreign trade prices for industrial roundwood and selected semi-finished wood products in order to
examine the position of the Slovak wood and semi-finished wood product trade in comparison to traditional trading partners.

**Research methodology**

Result-oriented-indicators revealing the competitive situation in the sector from an ex-post perspective were used to determine the competitiveness of the forest-based sector, to compare the development of export performance and competitiveness in international markets. The most commonly used method is the calculation of the Revealed Comparative Advantage (RCA) index. A modified version of the RCA index using a mathematical logarithmic function [Bobáková and Hečková 2007] was used in this paper in order to analyse the competitiveness of the forest-based sector and trade in wood and semi-finished wood products compared to other sectors in a specified period, 2009-2013, within the national economy of the Slovak Republic.

\[
RCA_{it} = \ln \left( \frac{\sum_{i=1}^{n} \frac{EX_{it}}{IM_{it}}}{\sum_{i=1}^{n} \frac{EX_{it}}{IM_{it}}} \right)
\]

where: 
- \(EX_{it}\) – exports (in USD) of wood and semi-finished wood products from the selected country in the period \(t\),
- \(IM_{it}\) – imports (in USD) of wood and semi-finished wood products to the selected country during the period \(t\),
- \(\sum_{i=1}^{n} EX_{it}\) – total exports (in USD) from the selected country during the period \(t\),
- \(\sum_{i=1}^{n} IM_{it}\) – total imports (in USD) to the selected country during the period \(t\).

Another tool used in comparative analysis is the Comparative Price Level (CPL) index, which is defined as the ratio of purchasing power parities of the market in each of the analysed countries [Parobek et al. 2014a]. The comparative price level is calculated as:

\[
CPL = \frac{P_{f}}{P_{d}}
\]

where: 
- \(P_{d}\) – expresses the price of wood and semi-finished wood products produced in the selected country,
- \(P_{f}\) – expresses the price of wood and semi-finished wood products produced in a country other than the selected country.
In this case, the following categories of wood and semi-finished wood products were analysed:

1. total industrial roundwood
2. coniferous industrial roundwood
3. non-coniferous industrial roundwood
4. coniferous sawnwood
5. non-coniferous sawnwood
6. wood-based panels
7. paper and paperboard

Slovakia’s main trade partners in Central Europe are Austria, Poland, Germany, Hungary and the Czech Republic. The research was therefore focused on these countries. The CPL indices for the Slovak Republic trade partners were expressed relative to the price level for the Slovak Republic. If the price level index of a given country was above 100%, then the prices in that analysed country were, on average, higher than in the Slovak Republic as a whole. Conversely, if a price level index was below 100%, then the prices were, on average, lower than in the Slovak Republic as a whole:

- if CPL ≤ 1 (or 100%) the competitiveness of a partner country was higher than the competitiveness of the Slovak Republic
- if CPL ≥ 1 (or 100%) the competitiveness of the Slovak Republic was higher than the competitiveness of a partner country.

Data from Comtrade [2015] and Faostat [2015] were used as background data for the analysis of the current state of the wood processing industry and competitiveness index calculation.

**Results and discussion**

In this research the RCA competitiveness index was calculated to reveal the competitive advantage of the different semi-finished wood products traded internationally compared to the other sectors of the national economy. The calculated RCA values for industrial roundwood during the period 2009-2013 are shown in table 1. The positive RCA values for Slovakia, Hungary and the Czech Republic clearly revealed a competitive advantage for the industrial roundwood trade, showing only slight changes over the examined time period.

In general, Slovakia had one of the highest RCA values (2.23 in 2010) thanks to the strong export of coniferous industrial roundwood. According to Faostat [2015], an export volume amounting to 1.97 Mio. m³ was approximately 53% of the total coniferous industrial roundwood production. There was a notable difference between the international trade of coniferous and non-coniferous industrial roundwood. In 2013, the RCA index for coniferous roundwood was 20 times higher than for non-coniferous roundwood. This situation reflected the high demand for coniferous industrial roundwood, especially sawlogs, from surrounding countries, in particular from Austria.
Slovakia was followed by Hungary in showing a strong competitive advantage. According to data from the Comtrade [2015] database, there were significant exports of non-coniferous industrial roundwood compared to other sectors, reaching a total of 46.07 Mio. USD. Imports of industrial roundwood were approximately 4.6 times lower.

Table 1. RCA competitiveness indices for industrial roundwood (coniferous and non-coniferous) for Central European countries in period 2009-2013

<table>
<thead>
<tr>
<th></th>
<th>2009</th>
<th>2010</th>
<th>2011</th>
<th>2012</th>
<th>2013</th>
</tr>
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<tr>
<td><strong>Industrial Roundwood</strong></td>
<td></td>
<td></td>
<td></td>
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</tr>
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</tr>
<tr>
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</tr>
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<tr>
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</tr>
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<td>1.77</td>
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</tr>
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<td>0.50</td>
<td>0.32</td>
<td>0.43</td>
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</tr>
</tbody>
</table>

Germany and Austria were dependent on major imports of industrial roundwood and their RCA values were negative. The analyses confirmed a negative development of the RCA indicators except for Hungary and Poland. The RCA values for the former increased by 11% during the monitored period.

Table 2 shows the results of the competitiveness analyses of trade in semi-finished wood products. Austria had the strongest competitive advantage from among the Central European countries in all products except for the production of non-coniferous sawnwood. This country obviously had a low production of non-coniferous sawnwood due to a lack of non-coniferous wood resources. Coniferous forests cover ca 74.8% of the total forest area [Faostat 2015]. In spite of sufficient coniferous wood resources, imports of industrial coniferous roundwood were relatively high (6.8 Mio. m³) and thus the comparative advantage was weak. However, the RCA index for coniferous sawnwood was 0.98.
Table 2. RCA competitiveness indices for sawnwood (coniferous and non-coniferous), paper and wood-based panels for Central European countries in period 2009-2013

<table>
<thead>
<tr>
<th></th>
<th>2009</th>
<th>2010</th>
<th>2011</th>
<th>2012</th>
<th>2013</th>
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<tr>
<td>Germany</td>
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<td>0.16</td>
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<td>-0.34</td>
<td>-0.06</td>
<td>-0.22</td>
</tr>
<tr>
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</tr>
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<tr>
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<td>-0.06</td>
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<td>Hungary</td>
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<td>0.82</td>
<td>0.64</td>
<td>1.03</td>
<td>0.90</td>
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<td>Poland</td>
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<td>-0.15</td>
<td>-0.32</td>
<td>-0.28</td>
<td>-0.30</td>
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<tr>
<td>Slovakia</td>
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<td>1.52</td>
<td>0.78</td>
<td>0.50</td>
<td>1.13</td>
</tr>
<tr>
<td><strong>Wood-based panels</strong></td>
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<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Austria</td>
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<td>1.06</td>
<td>1.00</td>
<td>1.07</td>
<td>1.08</td>
</tr>
<tr>
<td>Czech Republic</td>
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<td>0.43</td>
<td>0.50</td>
<td>0.46</td>
<td>0.36</td>
</tr>
<tr>
<td>Germany</td>
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<td>0.10</td>
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</tr>
<tr>
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<td>0.33</td>
<td>0.39</td>
<td>0.54</td>
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</tr>
<tr>
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<td><strong>Paper and paperboard</strong></td>
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<td>Germany</td>
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<td>0.12</td>
<td>0.09</td>
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</tr>
<tr>
<td>Hungary</td>
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<td>-0.41</td>
<td>-0.38</td>
<td>-0.35</td>
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<tr>
<td>Poland</td>
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<td>-0.34</td>
<td>-0.30</td>
<td>-0.30</td>
<td>-0.35</td>
</tr>
<tr>
<td>Slovakia</td>
<td>0.57</td>
<td>0.66</td>
<td>0.21</td>
<td>0.22</td>
<td>0.15</td>
</tr>
</tbody>
</table>

The situation in sawnwood competitiveness was very similar in Germany and the Czech Republic, even if the values of the competitiveness indices were several times lower compared to Austria, and the RCA indices for non-coniferous sawnwood showed negative trends. In terms of time, the development of the competitiveness indices was relatively stable and the variability of the values depended on the country and the product.
Among the analysed countries, Slovakia recorded the highest RCA values in sawnwood (1.03) in 2013, in particular for non-coniferous sawnwood (1.13). This country had positive RCA values during the analysed time period in all commodities except for wood-based panels. The wood-based panel trade in Slovakia was characterized by a revealed comparative disadvantage and a significant intra-industry specialization with imports prevailing over exports as a result of specialisation in domestic production. According to Faostat [2015], the value of wood-based panel imports (210 Mio. USD) were twice as high as the exports.

Negative values of the RCA competitiveness indices were revealed for coniferous and non-coniferous sawnwood as well as paper traded by Poland.

The results show that Slovakia had the strongest comparative advantage in the trade of industrial roundwood (coniferous in particular) due to its sufficient wood resources and competitive prices. A similar situation, however with non-coniferous roundwood, was found for Hungary. These results confirmed the Heckscher-Ohlin theorem [Ohlin 1933] which assumes that it is mainly the relative allocation of production factors, such as the natural resources, that determines a nation’s comparative advantage. Austria used much more industrial roundwood than it produced domestically. Its domestic industry depended on imports of industrial roundwood mostly from neighbouring countries. However, the trade in products such as sawnwood, wood-based panels and paper and paperboard was competitive. There were significant differences between these two countries in terms of export competitiveness. While in Austria the forest industry was competitive in the export of value added products, comparative advantages were revealed for Slovakia in low value products (roundwood and coniferous sawnwood).

In addition, the foreign trade price level of the analysed semi-finished wood product categories were used to calculate the CPL indices based on the Faostat [2015] data (tab. 3). The price levels of Slovakia’s main trade partners in the Central European region were expressed relative to Slovakia’s price level. The prices of the Slovak products thus represented the average price levels, in this case the absolute number “1” (or 100%).

<table>
<thead>
<tr>
<th>Commodity</th>
<th>Austria</th>
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<th>Germany</th>
<th>Hungary</th>
<th>Poland</th>
</tr>
</thead>
<tbody>
<tr>
<td>Industrial roundwood</td>
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<tr>
<td>Industrial roundwood (C)</td>
<td>1.56</td>
<td>1.54</td>
<td>1.42</td>
<td>0.84</td>
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</tr>
<tr>
<td>Industrial roundwood (NC)</td>
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<td>1.87</td>
<td>1.09</td>
<td>2.50</td>
</tr>
<tr>
<td>Sawnwood (C)</td>
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<td>0.70</td>
<td>0.79</td>
<td>0.66</td>
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<td>Sawnwood (NC)</td>
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<td>1.45</td>
<td>0.66</td>
<td>0.39</td>
<td>0.95</td>
</tr>
<tr>
<td>Wood-based panels</td>
<td>0.88</td>
<td>1.24</td>
<td>1.05</td>
<td>0.95</td>
<td>1.02</td>
</tr>
<tr>
<td>Paper and paperboard</td>
<td>1.34</td>
<td>0.73</td>
<td>1.36</td>
<td>1.15</td>
<td>0.95</td>
</tr>
</tbody>
</table>
The values greater than 1 indicated that the export prices of the given commodities in the respective country were higher than the Slovak export prices and vice versa. With the exception of non-coniferous industrial roundwood and paper products, Hungary had the lowest prices amongst all the other exported commodities compared to Slovakia. In line with the RCA results revealing a comparative disadvantage in the trade of industrial roundwood for Austria, the CPL indices also indicated the highest prices for this commodity. This was one of the reasons for the high imported volumes of industrial roundwood to this country with a significant share also from Slovakia. On the other hand, the prices of coniferous sawnwood, which were 15% lower than the Slovak prices may indicate a higher technological efficiency in the sawmilling industry in Austria. Similarly, coniferous sawnwood prices were lower than in Slovakia in all the other analysed countries.

The results of this single CPL analysis clearly supplemented the RCA analysis and confirmed that the Slovak Republic had the lowest prices of raw wood material (roundwood) compared to most of the analysed countries, which supports the export of these commodities and the higher prices of higher value added products such as sawnwood and, to a certain extent, paper products.

An understanding of the differences in price levels is important in connection with other economic indicators, such as economic growth and gross domestic product, because higher relative prices can make the economy look healthier than it really is and, therefore, the prices alone are not sufficient for competitiveness analyses. A combination of RCA and CPL indices can help to clarify the competitiveness of the analysed sector. In Austria, for instance, the prices of wood-based panels were 1.5 times greater than the prices in Slovakia but the RCA index had the greatest value (the strongest competitiveness). These differences occurred for the following three reasons. Firstly, in spite of the higher prices of the wood-based panels, foreign trade was stronger and there was strong competition in this sector compared to other industries within the Austrian economy. Secondly, the greater value of the CPL index reflected a better economic situation in this country and thus a higher price level. Finally, there were significant differences in the structure of the wood-based panel qualities (and thus prices) which both countries traded internationally.

The above-mentioned results emphasise the complexity involved in understanding the term competitiveness as defined by Wolff et al. [2007] and the necessity to understand it in a broader context as mentioned by Latruffe [2010]. Therefore, if the competitiveness of the forestry and forest-based sector in Slovakia is linked to trade with industrial coniferous roundwood, it is important to understand it, in a broader context, in connection with the low level of wood processing.
Conclusions

A nation’s competitiveness can be evaluated through the ability of a nation to produce goods and services which meet the requirements of international markets. Such requirements include, for example, technology, human capital, the quality of the product and other factors.

Development of the forest-based sector depends on the production and utilisation of raw wood material. Raw wood material is an important renewable and sustainable source and can be considered one of the competitive factors of products placed on environmentally sensitive markets. This research applied the commonly used competitiveness indicators, RCA and CPL, to analyse the competitiveness of the wood and semi-finished wood product sectors compared to other sectors in the national economies of the selected countries and among the countries.

Slovakia had the strongest comparative advantage in the trade of industrial coniferous roundwood due to its sufficient wood resources and competitive prices. The competitiveness of the forestry and forest-based sector reflects the low level of wood processing, which is generally not appropriate from the viewpoint of long-term sustainable development.

The latter part of the research highlighted the comparative foreign trade prices for industrial roundwood and selected semi-finished wood products. In general, the development of competitiveness indices depends on the resources and trends in the analysed sector, as well as on trends in the other sectors of the national economy. Therefore, the results of the comparative price analysis should be understood in a broader context.

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