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THE INFLUENCE OF SURFACE MODIFICATION OF WOOD PARTICLES WITH CARBON NANOTUBES ON PROPERTIES OF PARTICLEBOARD GLUED WITH PHENOL-FORMALDEHYDE RESIN

Research was carried out on a newly manufactured particleboard (PB) containing carbon nanotubes (CNTs) to determine the effect of the CNTs on physical, mechanical and combustion properties of the board. The experiment consisted of two stages. In the first, wood particles were treated with an aqueous suspension of CNTs (0.2% w/w) and sodium dodecylbenzenesulfonate (0.2% w/w) as a dispersant. After drying to constant weight, a modified form of the ASTM E69 method was used to determine the effectiveness of fire protection provided by CNT-modified wood chips. The rate of wood decomposition decreased significantly, and the time to complete combustion increased from 18 to 22.5 min for the reference and CNT-modified wood particles respectively. In the second stage of the experiment a particleboard bonded with phenyl-formaldehyde resin was produced, in which the particles were modified with CNTs using the method described in the first stage. Selected physical and mechanical properties of the final board were determined. Samples of the board were tested using a mass loss calorimeter in accordance with the ISO 13927 standard, and mechanical tests were performed by applicable standard methods. However, no significant improvement in the properties of the PB were observed.

Keywords: carbon nanotubes, wood chips, particleboard, flammability, mechanical properties

Introduction

Nanotechnology is applied nowadays in many fields, including energy storage, nanoelectronics and biotechnology, as well as materials science. Recent studies

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have resulted in applications of nanomaterial composites, including wood-based panels.

Studies to date have shown that the inclusion of small quantities of various types of nanomaterials, such as nano-SiO₂, nano-ZnO, nano-Al₂O₃, nano-CuO, nano-TiO₂, nano-Ag, nanoclay, nanocellulose, etc., can significantly change the properties of both adhesive resins and joints bonded with those resins. The addition of nanoparticles helps to control the viscosity of resin solutions, increases the strength and thermal and water resistance of adhesive-bonded joints, and improves their barrier properties [Romueli et al. 2012; Dukarska and Bartkowiak 2016; Dukarska and Czarnecki 2016]. Reinforcement of adhesive-bonded joints by the addition of nanoparticles results in improved strength parameters, water resistance and hygienic properties of wood-based materials [Zhang and Smith 2010; Taghiyari et al. 2011; Veigel et al. 2012; Salari et al. 2013; Taghiyari and Farajpour Bibalan 2013; Liu and Zhu 2014; Candan and Akbulut 2015; Dukarska and Czarnecki 2016]. The use of nanomaterials also enables the production of wood-based boards with required physical and mechanical properties from alternative raw materials, most often of inferior quality, such as date or paulownia wood or annual plant waste [Salari et al. 2013; Rangavar and Fard 2015]. As regards technological processes, the use of nanoparticles enables optimization of the manufacture of particleboards (PBs). The introduction of such particles into the adhesive resin solution increases the thermal conductivity of the pressed mat and enhances heat transfer from the external layers to the core layer. This in turn improves the conditions of condensation of the adhesive resin. As a consequence, the pressing time of the boards may be shortened without impairing their physical or mechanical properties [Lei et al. 2008; Zhang and Smith 2010; Taghiyari et al. 2011, 2013; Kumar et al. 2013]. Promising results have also been obtained for the biological resistance of materials modified with nanoparticles, as they have been found to be more resistant to various types of molds and termites [Taghiyari and Farajpour Bibalan 2013; Gao and Du 2015; Marzbani et al. 2015; Dukarska et al. 2017].

When PBs are used in construction, great importance attaches not only to their mechanical properties, but also to their fire resistance, in view of the flammability of boards of this type. To reduce the fire risk, a wide range of flame retardants is used to improve the properties of flammable materials [Boruszewski et al. 2011]. It has already been proposed that recent progress in nanotechnology may be of use in producing fire retardant coatings or additives. The most promising nanomaterials include carbon nanotubes (CNTs). Their unique chemical [Lin et al. 2003; Tasis et al. 2006], thermal [Berber et al. 2000; Kim et al. 2001] and mechanical properties [Treacy et al. 1996] have been widely researched in recent years. The development of chemical vapor deposition enabled the manufacture of cheap mass-produced CNTs [Cassell et al. 1999; Chhowalla et al. 2001], which may be used *inter alia* in polymer

composites and coatings. Currently, CNTs are a popular additive to polymer composites to improve their mechanical properties and to act as a flame retardant.

CNTs dispersed in polystyrene and poly(methyl methacrylate) (PMMA) matrices may serve as a flame retardant, reducing the two key parameters of mass loss rate (MLR) and peak heat release rate (PHRR) [Kashiwagi et al. 2005; Cipiriano et al. 2007]. Not only CNTs dispersed in the polymer matrix, but also CNT-based coatings, reduced the flammability of composites. PETI330/T650 covered by thin CNT film exhibited decreases PHRR and total mass loss, and increased time to ignition (TTI) [Fu et al. 2010]. Anderson et al. [2010] made a multifunctional cellulose/CNT composite paper, which demonstrated greatly reduced flammability [Anderson et al. 2010]. Thermogravimetric measurements on a phenol-formaldehyde/cellulose composite showed that thermal stability increased slightly on the addition of 1.0 wt% of multiwall (MW) CNTs [Park and Kadla 2012]. Cotton fabrics cross-linked with polyvinylphosphonic acid presented delayed ignition and a decreased burning rate after the incorporation of MWCNTs [Gashti and Almasian 2013]. Although research on cellulose/CNT composites is not a novel area, there are few studies describing composites containing bulk wood or wood-based materials, not only cellulose, mixed with carbon nanotubes. Some current studies on the use of carbon nanomaterials and wood-related products are focused on wood-plastic composites (WPC) made of wood flour and polymer. It has been reported that the addition of CNTs to the composite lowers flammability and increases mechanical strength. Polypropylene/wood flour with 2 wt% of CNTs showed a 25% fall in PHRR and total heat released (THR) and a significant decrease in CO yield [Farsheh et al. 2011].

Unfortunately, all nanomaterials tend to aggregate in such applications. The aggregates not only diminish the positive effect, but may also clog the nozzles used for spraying resin during PB production. Therefore, we propose using CNTs as coating materials for wood particles instead of incorporating them into resins. The main aim of this research is to investigate the flammability of wood particles coated with MWCNTs. Moreover, CNT-modified wood particles were used to manufacture a CNT-modified particleboard, and the flammability, water resistance and mechanical properties of that board were then investigated.

Materials and methods

Materials

Industrial-grade NC7000 CNTs were obtained from Nanocyl SA. The dispersant used to prepare the aqueous dispersion of CNTs was sodium dodecylbenzenesulfonate (SDBS) supplied by Sigma Aldrich. The wood particles used were industrial Scots pine (*Pinus sylvestris* L.) chips with average dimensions of $15.91 \times 1.81 \times 0.91$ mm and a moisture content of 4%. The binder

used to manufacture the PB was phenol-formaldehyde resin (PF) with viscosity 679 mPas (at 25°C), density 1.215 g/cm³, solids content 48.5%, pH 8.45 and gel time 271 s (at 130°C).

Sample preparation

In the first stage of the study, wood particles were coated with CNTs. An aqueous dispersion of CNTs (0.2% w/w) and SDBS (0.2% w/w) was sonicated using a Hielscher UP400 ultrasonic homogenizer for 20 min. CNTs were deposited onto wood particles in a simple dip-coating process. A CNT suspension (4 l) was used to obtain 2 kg of CNT-modified wood particles, so that the final ratio of CNTs to wood was approximately 0.4% (w/w). Afterwards, the wood particles were dried at 105°C to obtain the humidity of the reference chips (4%).

Single-layer PBs were produced in laboratory conditions with the following parameters: thickness 12 mm, resination 8%, and density 650 kg/m³. The pressing process was carried out at 200°C, with a unit pressure of 2.5 N/mm² and for a time of 23 s per mm of board thickness. The PBs were prepared according to the same procedure: the first contained pure wood particles, and the second contained wood particles modified with CNTs.

Flammability

The study was conducted in several stages. In the first stage, a modified form of the ASTM E69 method was used to determine the effectiveness of the protection of pine wood particles with an aqueous CNT suspension. The next step was to produce particleboard from protected chips and to test it for flammability using a mass loss calorimeter (MLC), and to carry out strength tests based on current standards.

The test of the flaming properties of wood particles was performed according to a modified form of the ASTM E69 method, by burning protected and control (unprotected) samples in a tubular furnace. The modification consisted in the making of a special basket of approximately one-liter volume, made of 2 × 2 mm steel mesh, with diameter 5 cm and length 50 cm. The empty basket was weighed before the test. The test pieces were poured into portions of the basket, being successively whipped using a wooden plunger. When the basket was filled, the whole was weighed to calculate the bulk density. Then the filled basket was suspended in the tube. A burner was placed under the suspended sample, with a flame height of approximately 25 cm. The flame acted on the sample for 3 minutes. The percentage mass loss and the exhaust gas temperature at the outlet of the pipe were recorded every 0.5 minutes. Further observations and recordings were made from the moment the burner was extinguished until three weight loss measurements returned the same value. At

the same time measurements of the flue gas temperature at the outlet of the tube were made using a thermocouple.

The following types of board were investigated: pure PB (control) and PB modified with CNTs. Flammability tests were carried out on three samples for each type. MLC is a benchmark that enables the measurement of mass loss rate and HRR (heat release rate) for any heat flux used for full-scale cone calorimetry (ISO 5660). The tests were carried out in accordance with ISO 13927, with a radiant heat of 35 kW/m². The applied radiant intensity corresponds to fire protection targets, especially for polymers, and corresponds to the intensity of heat flux in a small-scale fire [Schartel and Hull 2007]. In the tests we used 100 × 100 mm particleboard samples with a thickness of 12 mm. Using the standard calorimeter software, the following combustion properties of the examined materials were determined: heat release rate (HRR), mass loss rate (MLR), THR, effective heat of combustion (HOC), final sample weight (FSW) and TTI. Average results are reported here for HRR, max. PHRR, mass loss [%] and TTI [s].

Particleboard properties

Before testing, all samples of the manufactured boards were subjected to conditioning to achieve a constant mass at 20°C and relative humidity 65%. The moisture content of the samples was about 8%. The mechanical properties of the experimental boards and their water resistance were tested according to relevant standards. We determined the modulus of rupture (MOR) and modulus of elasticity (MOE) according to EN 310 and the internal bond (IB) according to EN 319. The water resistance of the boards was investigated by measuring the internal bond after a boil test (V-100) according to EN 1087-1, and the thickness swelling (TS) after 2 and 24 h of soaking in water according to EN 317. As well as thickness swelling the water absorption (WA) was also calculated, using equation (1):

$$WA = \frac{m_2 - m_1}{m_1} \cdot 100 \quad (1)$$

where WA is the water absorption (%), and m_1 and m_2 are the sample mass before and after soaking (g).

Each test was performed in 15 replicates. The results obtained for the physical and mechanical properties of the experimental boards were statistically analyzed using Statistica software (version 13.1). To compare the average values of the physical and mechanical properties of the tested boards, we carried out a single-factor analysis of variance and Tukey's post hoc test, on the basis of which uniform average values were determined for each of the investigated properties, with the boundary significance level equal to 0.05.

Results and discussion

Fire retardant properties of CNT-modified wood particles

The burning of wood is a very complex physicochemical process. Flammable gases such as ethane, methane, and hydrogen are the main causes of flammability. As the temperature rises, the intensity of evolution of the gases increases. In the wood, as a result of low heat permeability, it is mostly the top layer that is burned. Incineration is slow, and a layer of charred wood effectively slows down the process, thanks to the very low thermal conductivity of char [Kozłowski and Władysław-Przybylak 2001; Hankalin et al. 2009]. The thermal conductivity of char is several times smaller than that of wood. After the formation of this layer and removal of the heat source, the process is suppressed, and self-extinguishing can even occur.

The highest temperatures during this part of the experiment were $177 \pm 23^\circ\text{C}$ and $191 \pm 10^\circ\text{C}$ respectively for the reference and CNT-modified wood particles. Despite the slightly higher temperature of the exhaust gases, the mass loss following exposure to the flame for 3 minutes was lower for the CNT-modified wood particles ($12 \pm 1\%$) than for the reference samples ($16 \pm 3\%$).

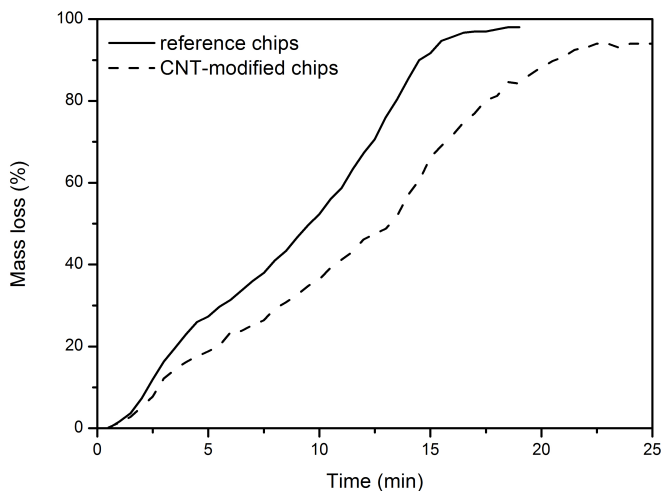


Fig. 1. Mass loss of reference and CNT-modified wood chips

Removal of the flame caused a rapid decrease in temperature followed by smoldering of the samples. After 10 minutes, the average mass loss was $52 \pm 4\%$ and $36 \pm 2\%$ for the reference and CNT-modified samples respectively. Therefore, the presence of CNTs caused a significant reduction in the rate of decomposition. Afterwards, from the beginning of the experiment, the process of combustion of the reference wood particles was accelerated and the temperature increased to $347 \pm 52^\circ\text{C}$ in 14.0 ± 1.0 minutes. Similar behavior was observed for

the CNT-modified wood particles, for which the temperature reached $323 \pm 47^\circ\text{C}$ after 17.0 ± 1.5 minutes. Such temperatures are the result of the re-emergence of flames in the higher layers of chips placed in the basket. These results suggest that the CNT coating produces a clear retardation of wood particle combustion. The whole process lasted for 18.0 ± 1.0 minutes and 22.5 ± 1.5 minutes, and the mass loss was $98.0 \pm 0.5\%$ and $94.5 \pm 1.5\%$, for the reference and CNT-modified samples respectively. The results suggest that covering wood particles with CNTs produces a fire retardant effect. The retardation of combustion is clearly visible on the dynamic mass loss graph (fig. 1), which shows that the combustion process was much slower after modification with CNTs. However, the observed smoke temperatures were not significantly changed, which may be explained by the lack of an active mechanism of flame suppression.

Several possible mechanisms have been proposed to explain the flame retardant properties of composites containing CNTs in the polymer matrix. The thermal conductivity of polymers increases after the incorporation of CNTs, particularly above 160°C [Kashiwagi et al. 2004]. The presence of CNTs in the matrix may also help to create a thermally insulating char layer [Kashiwagi et al. 2005]. Moreover, the change in the viscoelastic properties of polymers when CNTs are added may lead to an anti-dripping effect when the polymers burn [Capiriani et al. 2007].

In the case of the CNT-modified wood particles, CNTs were located only on the surface of the samples. Therefore, the CNTs did not change any physical properties of the wood, and only surface action could be observed. Among the proposed mechanisms, the most probable is the influence of CNTs on char formation. Char may either be formed more rapidly or be mechanically enhanced to become more durable. As no significant differences were recorded in the first minutes of combustion between the reference and CNT-modified particles (fig. 2), the faster formation of char is unlikely. Therefore, reinforcement of the char layer is a more probable cause. The other mechanism which may be responsible for the fire retardant action is faster heat dissipation during slow burning, caused by the high surface area of CNTs and their high conductivity.

Fire retardant properties of CNT-modified particleboards

Although the mechanism of fire retardation in CNT-modified wood particles may be of interest, it is important to investigate the flammability of complete particleboards. Therefore, a CNT-modified PB was prepared (as described in the Sample Preparation section) and tested by MLC. The average TTI was 4 s shorter for the CNT-modified PB, which may be due to the stronger infrared absorption of black CNTs. The kinetics of HRR after ignition were similar for the reference and the modified PB. The first peak value of HRR was 102 kW/m^2 ; then, after a slight decrease in HRR, its value rapidly increased. The PHRR was recorded 25 s earlier for CNT-modified PB. As in the case of TTI, the

explanation of the earlier PHRR may be the stronger infrared absorption. The values of time to PHRR and THR were slightly lower for the CNT-modified PB than for the reference, but the difference lies within the limits of measurement uncertainty. Also, no significant change was observed for total mass loss.

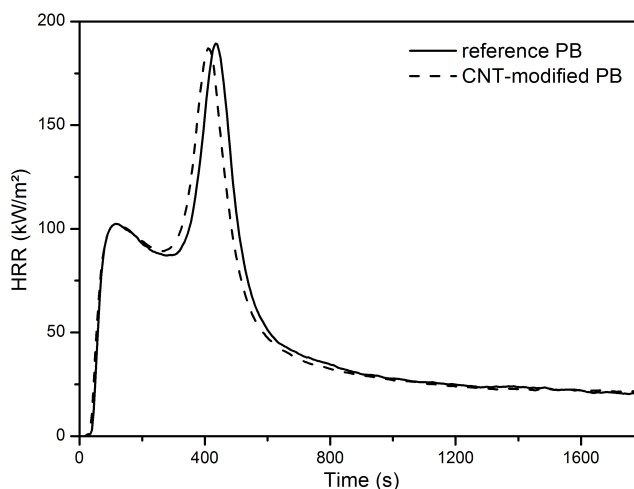


Fig. 2. Heat release rate measured by MLC during combustion of reference and CNT-modified PBs

Ineffective fire protection may be explained by the fact that the protective charred layer loses its properties during radiant heat flux. The rapid thermal decomposition causes the formation of large amounts of volatile components which, when mixed with air, ignite and influence the rate of fire spread and the rate of combustion [Grześkowiak and Wiśniewski 2010; Mačiulaitis and Praniauskas 2010]. As explained in the previous section, CNTs may either dissipate heat or enhance the char layer. Neither mechanism involves active flame suppression. The slightly decreased time to PHRR for CNT-modified PB is probably caused by stronger IR absorption and faster heating due to the optical properties of CNTs. In general, it may be claimed that although CNTs retard burning at the initial stage of combustion, there are no active flame suppressive mechanisms which might decrease HRR as observed in high-energy cone calorimetry.

Strength properties and water resistance of CNT-modified PB

Because the CNTs were located only on the surface of wood particles, they should not be expected to influence the physical properties of a single wood particle. However, the interaction between phenol-formaldehyde resin and wood particles might significantly change after CNT deposition. Another important

Table 1. Combustion properties, investigated by MLC, of reference and CNT-modified PB

	TTI (s)	THR (MJ/m ²)	PHRR (kW/m ²)	Time to PHRR (s)	Total mass loss (%)
Reference PB	38.5 (3.0)*	88 (4)	191 (5)	437 (6)	87.3 (0.7)
CNT-modified PB	34.7 (3.2)	86 (2)	197 (13)	412 (15)	88.3 (2.0)

*Standard deviations in parentheses.

factor was the presence of SDBS, used as a stabilizing agent for CNT dispersion. The properties of the reference and CNT-modified PBs are presented in table 1. A comparison of mean values obtained for each tested parameter shows that both the strength and swelling of boards made from CNT surface-modified wood chips were slightly higher than those of the reference boards. However, on consideration of the standard deviations associated with the mean values, and above all the results of post hoc analysis used for the determination of homogeneous groups, the differences between the CNT-modified and reference boards were found to be insignificant. Tukey's homogeneous group tests performed for the majority of the investigated properties identified the same homogeneous group, for which the statistical significance level p took values above the assumed value of 0.05. Only in the case of bending strength and swelling after two hours of water soaking did one-way analysis of variance enable the identification of two different homogeneous groups (a, b), indicating a statistically significant effect of CNT-modified wood chips on these two parameters. Modification of wood chips with carbon nanotubes resulted in a slight increase in board bending strength by ca. 15% (intergroup MS = 2.3665, df = 18.000, $p < 0.05$) and slightly increased swelling after short-term water treatment (by ca. 13%, intergroup MS = 4.0367, df = 18.000, $p < 0.05$). However, the lower water resistance of the CNT board after short-term water exposure was of little importance, as its swelling after 24 hours of soaking in water was similar to that of the reference boards. A general conclusion is that the modification of wood chips by the method of CNT dispersion investigated in this study does not significantly affect the strength and water resistance of the resulting PBs. This is probably due to the relatively low CNT concentration and the presence of hydrophilic SDBS. This assumption has been confirmed by other researchers studying the effects of surface modification of wood with various types of nanoparticles, who have reported significant changes in strength, hydrophobicity, color durability, fire resistance and bioresistance only at higher concentrations of nanoparticle suspensions [Nowaczyk-Organista 2009; Dong et al. 2015; Soltani et al. 2016; Taghiyari et al. 2016]. Nevertheless, preliminary results, particularly those concerning fire resistance, are promising and encourage further research in this area.

Table 2. Strength properties and water resistance of the experimental boards

Property	Reference PB	CNT-modified PB
MOR [N/mm ²]	12.9 ^a (1.8)*	14.8 ^b (1.0)
MOE [N/mm ²]	2250 ^a (260)	2400 ^a (180)
IB [N/mm ²]	0.69 ^a (0.11)	0.64 ^a (0.06)
V-100 [N/mm ²]	0.37 ^a (0.04)	0.40 ^a (0.047)
TS – 2 h [%]	22.3 ^a (1.4)	25.1 ^b (2.7)
TS – 24 h [%]	25.9 ^a (2.4)	27.5 ^a (2.5)
WA – 2 h [%]	72.1 ^a (2.6)	72.8 ^a (3.1)
WA – 24 h [%]	85.8 ^a (3.2)	87.2 ^a (2.7)

*Standard deviations in parentheses, a, b – homogeneous groups, CNT – carbon nanotubes, MOR – modulus of rigidity, MOE – modulus of elasticity, TS – thickness swelling, WA – water absorption.

Conclusions

In this study, wood particles were coated with a 0.2% (w/w) aqueous dispersion of carbon nanotubes, stabilized with SDBS. The prepared CNT-modified particles were tested using a modified form of the ASTM E69 method. The modification of wood particles with CNTs significantly influenced their combustion properties, and thermal decomposition significantly decreased. We have proposed two models describing the fire retardancy. First, due to their high surface area and thermal conductivity, the CNTs might increase heat dissipation; and second, the CNTs may mechanically enhance the char layer, which thermally insulates the wood from fire. However, although fire retardant properties were observed for CNT-modified wood particles, no significant decrease in the flammability of particleboards was observed after the addition of CNTs. A possible explanation is that during cone calorimetry the combustion was too rapid, and the fire suppressive properties of CNTs are significant only during low-energy thermal degradation. Moreover, tests of strength and water resistance in boards manufactured from both unmodified and CNT-modified wood chips did not show any significant changes in these properties, apart from a slight increase in bending strength. This may be because of the relatively low quantity of CNTs, the presence of SDBS, or the method of modification. However, the preliminary research results presented in this paper may provide a basis for further investigation into the potential of carbon nanotubes in the production of wood-based products.

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List of standards

- ASTM E69** Standard test method for combustible properties of treated wood by the fire-tube apparatus
- EN 310:1993** Wood based panels. Determination of modulus of elasticity in bending and of bending strength. European Committee for Standardization, Brussels
- EN 317:1993** Particleboards and fiberboards. Determination of swelling in thickness after immersion in water. European Committee for Standardization, Brussels
- EN 319:1993** Determination of tensile strength perpendicular to the plane of the board. European Committee for Standardization, Brussels
- EN 1087-1:1995** Particleboards – Determination of moisture resistance – Boil test
- ISO 13927:2015** Plastics – Simple heat release test using a conical radiant heater and a thermopile detector
- ISO 5660** Cone calorimeter, heat release and smoke production

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