Physical and Mechanical Properties of Boards Made from Modified Rapeseed Straw Particles

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Composites made from annual plants, such as winter rapeseed, can be used as an alternative to wood-based composites because of their ecological character, low price, as well as physical and mechanical properties. The goal of this study was to prepare such boards and evaluate their properties. Unmodified material and chemically and hydrothermally modified rapeseed particles glued by powder polyester glue were used. The characteristics measured were the internal bonding, swelling and moisture uptake over time, and surface soundness. The results showed that modification of the raw material influenced the properties of the resulting composites. The highest swelling values were exhibited by the boards with particles that were alkaline-modified. Boards made from hydrothermally modified particles achieved the highest strengths compared with the commercially produced boards. The alkaline modification of the particles resulted in a stronger adhesive bond between the particle and adhesive. During the internal bonding test, the boards made from the alkaline-modified particles exhibited cohesive failures in the particles. Therefore, the modification of the particles increased the mechanical properties of the boards, but the physical properties deteriorated.

Keywords: Rapeseed; Particleboard; Internal bonding; Surface soundness; Non-recoverable thickness changes; Thickness swelling

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INTRODUCTION

Raw wood is the main component used in the production of composite materials. With increasing demands in the woodworking industry, the importance of alternative sources for the production of wood-based materials is increasing and becoming more topical (Dziurka *et al.* 2015). Various materials based on cellulose and lignin are currently used for the production of agglomerated materials (Ye *et al.* 2007). Agricultural products that are used to produce composite materials include rice stalk, coconut fiber (de Melo *et al.* 2010; Zhang and Hu 2014), corn stalk (Akinyemi *et al.* 2016), jute (Goswami *et al.* 2008), bagasse (Silva *et al.* 2016), bamboo (Widyorini *et al.* 2016), wheat straw (Zhang *et al.* 2011), and rapeseed (Dziurka *et al.* 2005). This study primarily focused on the production of composite materials made from rapeseed. Using post-harvest rapeseed residues, which have not yet been industrially used as the main component in composite materials, has the potential to contribute to solving the shortage of wood in the woodworking industry in the European Union.

The yield per hectare of rapeseed stalk is 3 t to 10 t, which means there is a production of 42 million t of post-harvest rapeseed residue in the European Union (Eurostat 2015). The chemical composition of rapeseed stalk is slightly different from that of wood. Rapeseed particles contain less cellulose and lignin, and more hemicellulose and extractive substances. The cellulose contained in stalks positively affects the resulting mechanical and physical properties of composite boards (Dziurka and Mirski 2013; Dukarska *et al.* 2017). There are different requirements for the mechanical and physical properties of composite materials, depending on their use. The change in the shape of a material caused by moisture is one of the most discussed physical properties. The effects of moisture have a major impact on the resulting properties and use of a composite material (Halligan 1970).

These effects mostly manifest themselves as a reduction in the strength of the boards, their durability, and changes in their shape (Gaff 2014). Dimensional changes can be prevented by the type of adhesive used, which can also affect the resulting usability of a material in the furniture and construction industries (Mirski *et al.* 2012; Gaff and Gašparík 2013). In terms of practical utilization, the internal bonding and surface soundness of materials are tested.

The objectives of this study were to evaluate selected mechanical and physical properties of composite materials made from rapeseed particles and to assess the effects of surface modification on these properties. The monitored mechanical properties were surface soundness and internal bonding. Because it is believed that the modification of particles disrupts the waxy and siliceous substances on the particle surface, the swelling and moisture of the boards over time were also observed. The resulting properties were compared with the properties of oriented strand board (OSB) and particleboard.

EXPERIMENTAL

Materials

Test specimens based on rapeseed stalk particles were used in this research. For the particle characteristics, a screen analysis was performed; the percentages of individual fractions are shown in Table 1. DAKOTEX2600 (Dakota Coatings N.V., Nazareth, Belgium), a powder adhesive made of polyester and epoxy resin, was used for board production. Table 2 lists the markings of the manufactured and commercial boards that were further used in the study.

Table 1. Representation of the Fractions in the Chopped Rapeseed Stalk

Fraction (mm)	0 - 0.25	0.25 - 0.5	0.5 - 0.8	0.8 - 1.6	1.6 - 2	2 - 3.15	3.15 - 8
Frequency (%)	1.2	2.8	4.8	39.4	20.1	23.1	8.6

Table 2. Marking of the Manufactured Boards and Commercial Boards

Mark	Type of Board
1	Board made from unmodified rapeseed particles
2	Board made from hydrothermally modified rapeseed particles
3	Board made from rapeseed particles modified in an alkaline environment
4	Particleboard
5	OSB

Methods

Boards made from raw and modified rapeseed particles were compared with commercially available wood-based materials, namely OSB (load-bearing board for use in humid conditions with thickness of 12 mm and density of 620 kg/m^3) and particleboard (panel for interior use in dry conditions with thickness of 12 mm and density of 670 kg/m^3). In order to disrupt the waxy layer on the surface of stems that could inhibit high-quality bonding between the particle and the adhesive, the particles were pre-treated (Částková *et al*. 2018). To achieve a better particle-adhesive bond, the rapeseed particles were hydrothermally (submerged in boiling water for 45 min) and chemically (soaked in a 2% NaOH solution at a water temperature of 25 °C) modified. To remove residual substances from the NaOH solution, the modified particles were thoroughly rinsed and dried.

Boards with a thickness of 12 mm and nominal density of 620 kg/m^3 were made. The adhesive was applied to the particles using a laboratory adhesive applicator (Imal, Modena, Italy). A resin dosage of 10% on straw dry mass was applied. After the adhesive was applied, a uniform particle sheet with the uniform area density was created on steel panels. This particle sheet was placed in a laboratory press (Strozatech, Brno, Czech Republic). The pressing was performed at a pressure of 2.3 MPa and board temperature of 185 °C for 10 min. At the end of the press cycle, a temperature of 170 °C was reached in the center of the boards. The closing speed of the press was 150 s. After the press was opened, the boards were removed and allowed to cool slowly.

The thickness of the boards was levelled to 12 mm, and then 50-mm × 50-mm × 12-mm test specimens were randomly cut from the boards. The vertical density profile of the boards was determined for these specimens. A Compact X-ray density profile Analyser DPX300-LTE (Imal, Modena, Italy) was employed.

Specimens with the dimensions 50 mm × 50 mm × 12 mm intended for the internal bonding and surface soundness testing were conditioned to an equilibrium moisture content in a climatic chamber at a relative humidity of 65% and temperature of 20 °C for 14 d. The specimens for monitoring the swelling over time were dried in an oven at 103 °C down to 0% moisture content. A total of 15 pieces of each type of board were made to monitor the swelling, 15 pieces were made for the internal bonding testing, and 15 pieces were made for the surface soundness testing.

The first monitored property was the swelling of a specimen over time. Rapeseed, OSB, and particleboard test specimens were dried to a 0% moisture content, and their dimensions and weight were measured in their oven-dry state. The monitored characteristics were the dimensions increase over time in all board directions and the moisture increase. The swelling and absorption were determined by measuring the dimensions and weight; the time intervals between measurements were 10 min, 20 min, 30 min, 1 h, 2 h, 4 h, 6 h, 12 h, 1 d, 2 d, and 3 d. The change in the dimensions was determined according to Eq. 1,

$$\beta_t = \frac{\beta_{wt} - \beta_o}{\beta_o} \cdot 100 \ [\%] \qquad (1)$$

where β_t is the increase in individual directions depending on the monitored time (%), β_o is the dimensions (mm) of the specimen in the oven dry state, and β_{wt} is the dimensions (mm) of the specimen in individual directions after each interval.

After testing of the swelling, the test specimens were oven-dried again and the non-recoverable thickness changes in the test specimens were determined according to Eq. 2,

$$NRC = \frac{t_2 - t_1}{t_1}.100\ [\%] \qquad (2)$$

where *NRC* is the non-recoverable thickness change (%), t_2 is the thickness of the specimen after the second drying (mm), and t_1 is the thickness of the specimen after the first drying (mm).

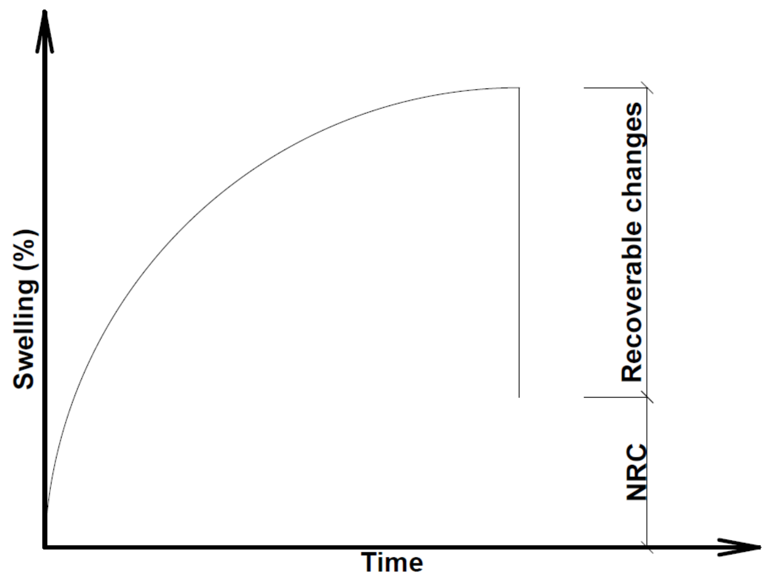

Fig. 1. Graphical representation of the calculation of the *NRC* according to Eq. 2

Internal bonding is the tensile strength of a composite material perpendicular to the plane of a board, where the board breaks down its center in the applied adhesive or material that forms the composite. The internal bonding was determined by following the standard EN 311 (2002). The test blocks to which the test specimens were glued were made of hard T-shaped ash wood, where the base of the block was identical to the dimensions of the test specimen (50 mm × 50 mm) and the dimensions of the top of the block were the same as those of the jaws of the universal testing machine (TIRA test 2850, TIRA GmbH, Schalkau, Germany). Epoxy glue was applied to the surface of a test specimen, which was glued to the wooden blocks and weighed down. After the glue hardened, the test specimens were returned to the climatic room to achieve an equilibrium moisture content. The tensile strength perpendicular to the plane of a board was determined according to Eq. 3,

$$\sigma = \frac{F_{max}}{a \cdot b}[MPa] \qquad (3)$$

where $F_{\max}$ is the maximum tensile force applied to the specimen at the moment of failure (N), and *a* and *b* are the dimensions of the test specimen (mm).

Surface soundness is another examined mechanical property. Surface soundness is the tensile strength when the surface layer is torn from the surface of a wood-based board. It is determined by measuring the force required to tear a steel rivet from a test specimen (EN 319 1993). The surface soundness test was performed according to the relevant standard EN 319 (1993). The test was performed on test specimens with the dimensions

(100 × 100) mm, and these specimens were randomly selected from the board. Test pads were glued to the test specimens with the required dimensions using epoxy adhesive. The prepared test specimens were then climatized in a climatic chamber at 65% relative humidity and 20 °C. The climatized specimens were then used to test the surface soundness with a Comtest adhesion tester (Coming Plus a.s., Prague, Czech Republic), and each specimen was tested in at least 12 places.

Scanning electron microscopy (SEM) of the broken bonds after tensile testing perpendicular to the plane of the boards were performed using a MIRA 3 electron microscope (Tescan Orsay Holding, Brno, Czech Republic) with a secondary electron detector, operated at 15 kV acceleration voltage.

An analysis of variance (ANOVA) (Fisher's F–test) and Tukey’s honest significant difference (HSD) test were used to evaluate the statistical significance of the individual factors (type of boards). A level of significance (α) of 0.05 was used for all of the statistical analyses. The statistical analyses were performed using STATISTICA 12 software (StatSoft CR s.r.o., Prague, Czech Republic).

RESULTS AND DISCUSSION

Figure 2 shows the course of water absorption by the specimens immersed in water. As was expected, the amount of water absorbed rapidly increased after immersion (Huner 2015). It is clear from the graph that the moisture increase was the steepest up to approximately 1000 min, then from 1000 min to 3000 min the moisture was almost constant, and afterwards the absorption slightly increased. Similar results were reported by Cruz *et al.* (2011). This trend did not apply to the specimens made from OSB; these specimens exhibited a continuous increase in the moisture over the measured time. The highest moisture values were achieved in the rapeseed boards made from the hydrothermally modified particles. In contrast, the lowest moisture values were achieved with the specimens made from the particleboard. Although all of the differences in the moisture content of the boards after the experiment were statistically significant, the effect of particle modification on the water absorption over time was not as great as the effect on the swelling of the boards, which is shown in Fig. 3.

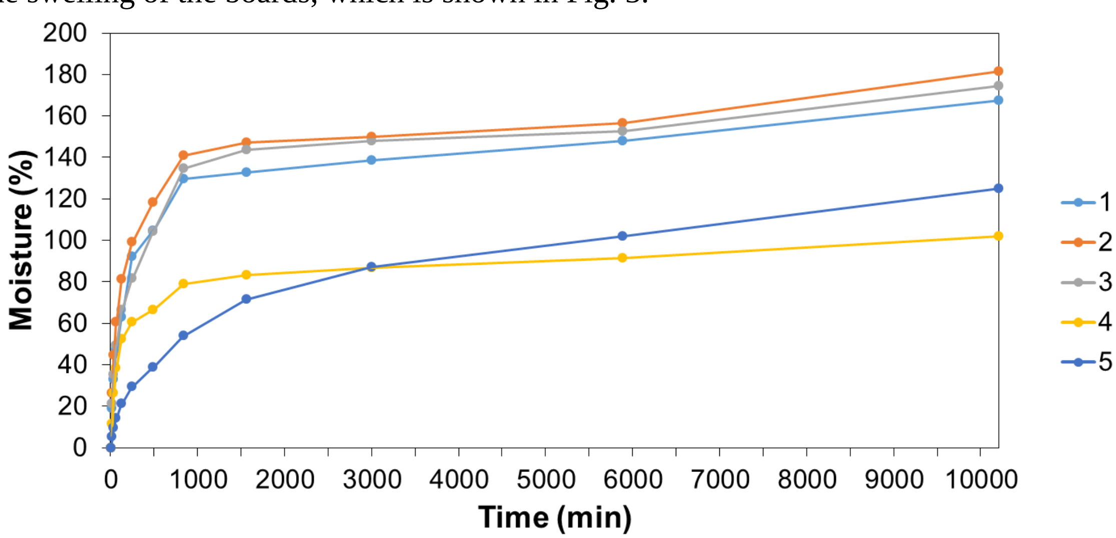

Fig. 2. Water absorption of the specimens immersed in water

Based on these results, it was concluded that boards made from rapeseed particles with the use of powder polyester adhesive can be used only for interior use, and a negative effect of particle modification on water absorption was found. The higher moisture values in the rapeseed-based particleboards were because of the absence of paraffin and other hydrophobic additives in the adhesive mixture. The figure also showed a clear effect from the particle modification on the higher resulting moisture content of the boards; the disruption of surface layer of particles led to higher water uptake (Hýsek *et al.* 2018). The differences between the moisture content of the boards at the end of the experiment were statistically significant ($p < 0.01$). A higher water absorption for hydrothermally modified materials was also reported by Huner (2015).

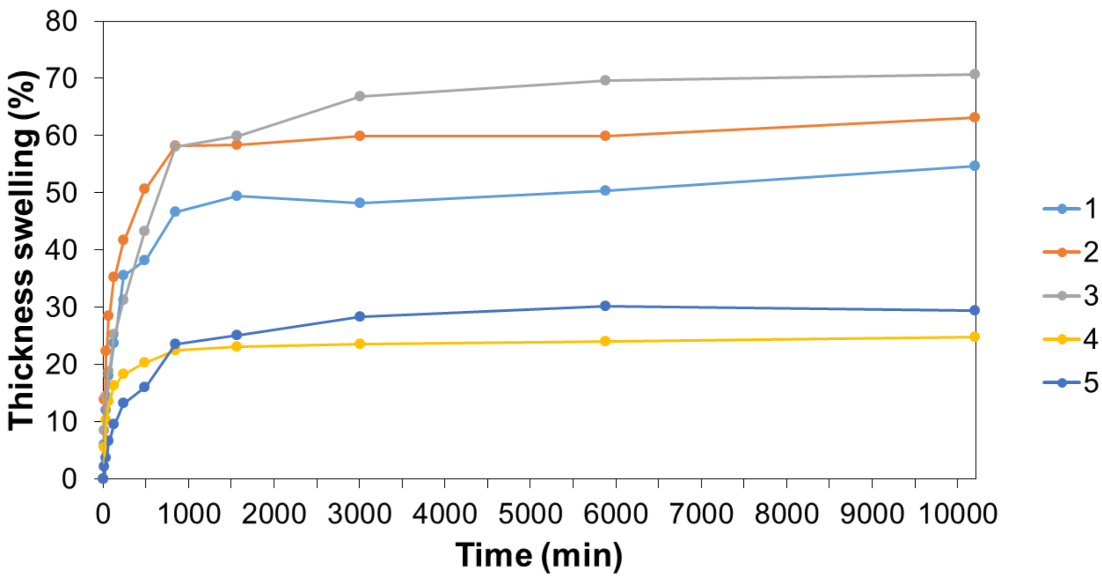

Fig. 3. Thickness swelling of the test specimens over time

Figure 3 shows the thickness swelling of the test specimens over time. The thickness swelling was increased steadily to 1000 min, as was the case with the moisture absorption over time. After this time, the thickness swelling was almost constant. The highest thickness swelling values were achieved by the test specimens made from the chemically modified particles, and the lowest swelling values were achieved by the specimens made from the particleboard, as was the case when the moisture content was monitored. The highest swelling values were achieved by the boards made with rapeseed particles. Both the hydrothermal and alkaline modifications increased the thickness swelling of the boards. Although these modifications caused a higher strength particle-adhesive bond, removing the waxy and siliceous substances from the particle surface resulted in greater particle swelling. The effect of the hydrothermal and alkaline modifications on removing waxy substances from particle surfaces was reported on in a previous study (Gajdačová *et al.* 2018). Furthermore, the alkaline modification caused a more heterogenic surface on the modified particles, which ensured better adhesive-particle bonding (Mohanty *et al.* 2000; Kim *et al.* 2006). Despite the better adhesion, removing hydrophobic substances caused a high thickness swelling. In contrast to this research, where the thickness swelling of the boards made from rapeseed particles achieved a swelling of 45% to 65% after 24 h, Dukarska *et al.* (2017) reported that the thickness swelling of rapeseed boards after 24 h increased by only 14% when a mixture of MDI (4.4′-

methylenediphenyl Isocyanate) and PF (phenol–formaldehyde) adhesive was used. The same values for rapeseed board swelling achieved by Dziurka and Mirski (2013) and Dukarska *et al.* (2017) were also achieved by Dziurka and Mirski (2013), but with the use of pMDI adhesive. Mirski *et al.* (2012) used OSB to achieve a thickness swelling of 8.9% after 24 h.

The figure also shows a clear effect of the particle modification on the resulting thickness swelling of the boards. The differences between the moisture content of the boards at the end of the experiment were statistically significant ($p < 0.01$).

Table 3 depicts the *NRC* and the thickness swelling of the boards. Boards made from the alkaline-modified particles had the highest thickness swelling values (70.7%), while the boards made from the hydrothermally modified particles achieved the highest *NRC* (37.8%). The OSB specimens exhibited the highest variability in the measured data.

Table 3. Thickness Swelling and *NRC* of the Boards

Type of Board	Thickness Swelling (%)	*NRC* (%)
Reference	54.7 (7.7)	30.5 (8.9)
Hydrothermal Treatment	63.1 (7.1)	37.8 (12.2)
Alkaline Treatment	70.7 (9.8)	29.8 (8.7)
Particleboard	24.8 (4.0)	8.6 (9.3)
OSB	29.4 (15.0)	12.1 (16.5)

Values in parentheses are the coefficients of variation in %

Figure 4 shows the volumetric swelling of the test specimens. The volumetric swelling pattern was identical to that of the thickness swelling (see Fig. 3). The differences between the achieved volumetric swellings of the boards at the end of the experiment were also statistically significant ($p < 0.01$).

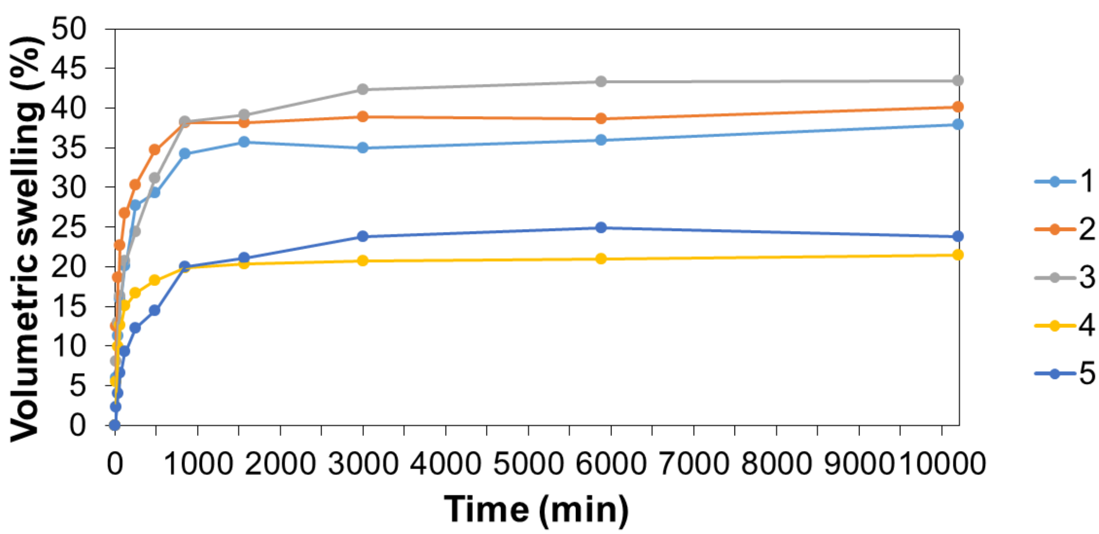

Fig. 4. Volumetric swelling of the test specimens

Figure 5 shows that the highest internal bonding (0.505 MPa) was achieved with hydrothermally modified rapeseed board specimens (bars show 95% confidence intervals). Similar results were obtained by Dukarska *et al.* (2017), who examined the internal bonding of rapeseed particleboards using a mixture of MDI and PF adhesive, with a result of 0.48 MPa. Similar values were obtained by Dziurka *et al.* (2005), who used a UF adhesive constituting 14% of the total particle volume and reported a resulting strength of 0.51 MPa. In contrast, the highest strength values were achieved by Dziurka and Mirski (2013), who reported internal bonding values of 0.82 MPa with the use of a pMDI adhesive.

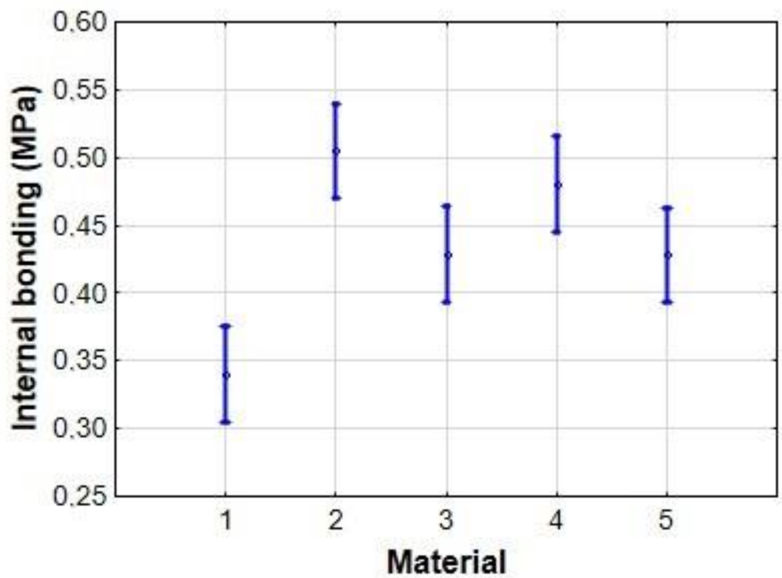

Fig. 5. Internal bonding of the boards

The results in this study indicated that the modification of the rapeseed particles improved the internal bonding of the boards in comparison with the reference set of rapeseed specimens, which achieved the lowest internal bonding values (0.340 MPa). By comparing the internal bonding results of the commercially produced OSB specimens with the results obtained by Mirski *et al.* (2012), lower internal bonding values were found in our study. The high internal bonding values may have been caused by the unusual vertical density profile of the boards made from rapeseed stalks. Because of the long press closing time, the highest density of the produced boards was reached in the middle of the vertical profile. An evaluation of the statistical significance of the differences in the tensile strength perpendicular to the plane of a board, corresponding with Fig. 5, is shown in Table 4.

Table 4. Statistical Significance of the Internal Bonding Differences

Type of Board	1	2	3	4	5
1	-	s.	s.	s.	s.
2	s.	-	s.	n.s.	s.
3	s.	s.	-	s.	n.s.
4	s.	n.s.	s.	-	n.s.
5	s.	s.	n.s.	n.s.	-

Statistical significance done according to Tukey's HSD test; s.- statistically significant; n.s. – no significant difference

Figure 6 shows a graphical representation of the results of the surface soundness tests (vertical columns represents 95% confidence intervals). The surface soundness test was not performed on the OSB specimens because this characteristic is not usually controlled in OSB. It is clear from Fig. 6 that the best results were achieved with material 4, which was a commercially available particleboard (0.325 MPa), and the worst results were achieved with material 3, *i.e.* a reference board developed by the authors without any modifications (0.119 MPa). When the modified particles were used in the materials developed by the authors, there was a trend of improvement in the tested properties with respect to the reference board; the properties of the water-modified boards (Material 2) improved by 16.7%, and the properties of the alkaline-modified boards (Material 1) improved by 33.5%. Although higher values were achieved by modifying the raw materials, the boards developed by the authors were still far behind the particleboards in terms of their values. In the article by Shalbafan *et al.* (2016), which also dealt with the surface soundness of particleboards, it was demonstrated that the results of this test were significantly affected by the board density. Shalbafan *et al.* (2016) tested boards with an approximate density of 600 kg/m^3, and in the case of the current study the density of the particleboards with a 1-mm thickness was approximately 862 kg/m^3. This density was significantly greater than the density of the boards developed in this study at a corresponding thickness (450 kg/m^3 to 500 kg/m^3), which explained the reason for the large difference in the surface soundness.

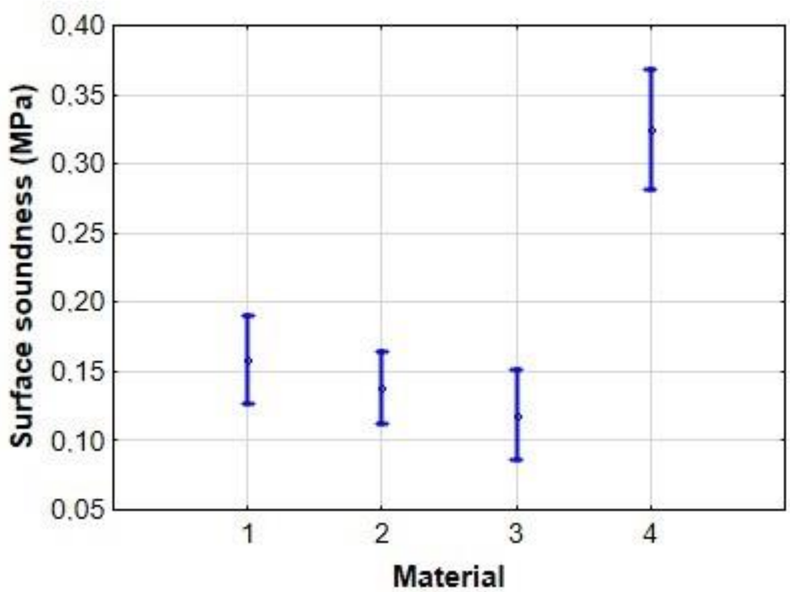

Fig. 6. Surface soundness of the boards

A statistical analysis according to Tukey's HSD test was also performed. The result of this analysis, which is shown in Table 5, was that no significant difference was observed between the developed boards (*i.e.* 1, 2, and 3). A significant difference was observed between all of the developed boards and particleboards.

Figure 7 shows SEM microscopic images of the broken bonds after tensile testing perpendicular to the plane of the boards. The effect of the modification on the nature of the failure was clear from the images. In the case of the boards made from untreated particles (Fig. 7b), there was only a failure between the adhesive and surface of the particle. In the case of the boards made from the alkaline-modified particles (Fig. 7a), a cohesive failure of the particle material was observed, which indicated that the modified particle-adhesive bond was stronger.

Table 5. Statistical Significance of the Surface Soundness Differences

Type of Board	1	2	3	4
1	-	n.s.	n.s.	s.
2	n.s.	-	n.s.	s.
3	n.s.	n.s.	-	s.
4	s.	s.	s.	-

Statistical significance determined according to Tukey's HSD test

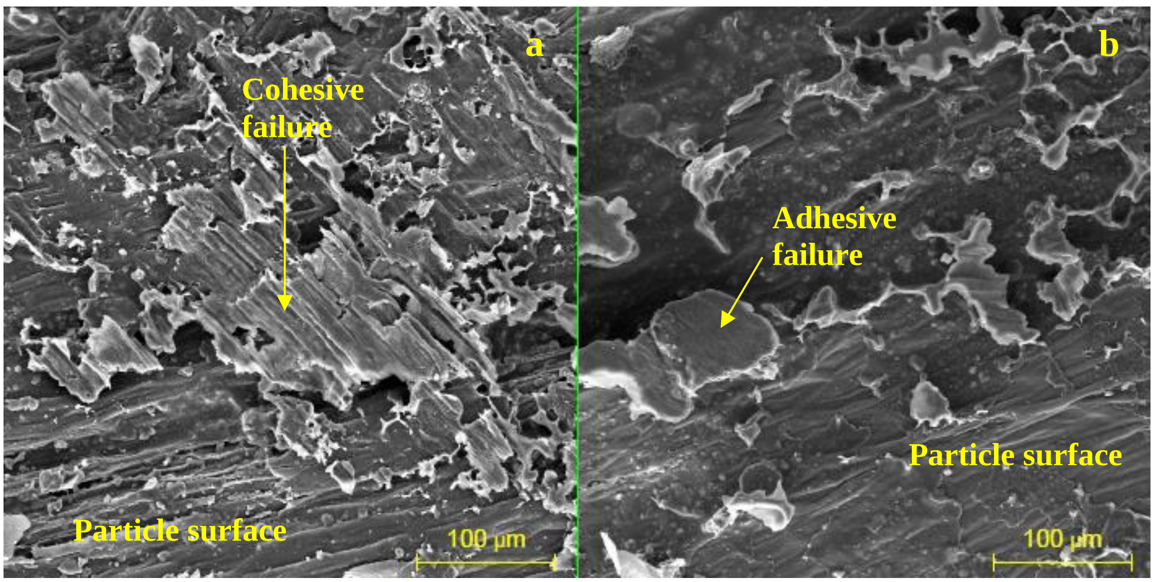

Fig. 7. SEM pictures of the ruptured internal bonding samples: particles treated in NaOH – cohesive failure in the particle (a), and untreated particles – adhesive failure (b)

CONCLUSIONS

1. The results indicated that the modification of the rapeseed particles had a statistically significant effect on the moisture absorption, thickness, and volumetric swelling. The highest swelling values were exhibited by boards with alkaline-modified particles. Conversely, the lowest swelling values were achieved by the boards with unmodified particles.
2. An effect from the modification of the rapeseed particles on the internal bonding was shown to occur. Boards made from the hydrothermally modified particles achieved the highest strength values compared with the commercially produced boards.
3. Compared with the particleboard, the surface soundness was not as high, but the highest mean internal bonding value was achieved with the boards containing unmodified rapeseed particles.

4. The alkaline modification of the particles resulted in a stronger adhesive bond between the particles and adhesive. During an internal bonding test, boards made from these particles exhibited cohesive failures in the particles. Therefore, the modification of the particles increased the mechanical properties of the boards, but the physical properties deteriorated.
5. Modification of rapeseed particles before particleboard production could improve some properties of the boards. However further research needs to be carried out in order to eliminate negative effects of modification that were observed in this study.

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