Effect of Cold Plasma Surface Pre-treatment of Wheat Straw Particles on Straw Board Properties

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Effects of the plasma treatment were evaluated for particles from winter wheat stalks relative to the properties of particleboards manufactured from such treated particles. Using urea-formaldehyde adhesive, boards with a nominal density of 540 kg/m^3 and a thickness of 6 mm were manufactured. Two degrees of plasma treatment were selected: cold plasma applied at atmospheric pressure by jet system, with a generator output voltage of 26.9 V and a current of 6.9 A; and in the second treatment, a maximum voltage of 28.6 V was used with a current of 8.7 A. The physical properties (equilibrium moisture content and thickness swelling depending on relative humidity) and mechanical properties (bending strength and tensile strength perpendicular to the plane of the board) were determined. The results showed that the plasma pre-treatment of particles had a statistically significant effect on the resulting composite properties. The mechanical properties of the boards increased with both plasma treatments, but the physical properties changed negatively. Boards manufactured from particles treated with a higher degree of plasma treatment resulted in significantly higher equilibrium moisture contents and thickness swelling than the reference boards.

Keywords: Cold plasma; Wheat straw; Particleboard; Surface modification; Water uptake

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INTRODUCTION

Cellulose and lignin-based plant materials are often used in composite materials, where they can act as fillers and reinforcing materials. It is always important to ensure the thorough bonding of the plant particle and the polymer. It has been proposed that a basic prerequisite for solid particle-polymer bonding is the presence of reactive groups on the interface of both materials to provide high surface energy (Mwaikambo and Ansell 2002; Bekhta *et al.* 2013). In composites hardened by plant materials, the opposite phenomenon is usually encountered, in which the solid bond between polar cellulose and nonpolar polymer is not easily formed. The wettability of a natural fibre or a particle by polymer is further worsened by the waxy substances that natural fibres often contain. In addition, the presence of water and free hydroxyl groups, in particular in amorphous portions, reduce the possibility of creating a strong bond between plant materials and most adhesives. Furthermore, a high water and moisture uptake causes dimensional changes in plant fibres, implying a reduction in the mechanical and physical properties of the composite material (Mwaikambo and Ansell 2002; Xie *et al.* 2010; Gajdačová *et al.* 2018).

Chemical modification of the fibre not only can improve the adhesion between the surface of the fibre and the polymer, but the specific fibre strength can increase, the water absorption by the composite can be decreased, and the mechanical properties of the entire composite material can also be improved (Li *et al.* 2007). However, the disadvantage of traditional methods of chemical surface modification is the production of hazardous substances that may endanger the environment and human health. From this perspective, surface treatment using plasma is a more benign method toward the environment.

Plasma is an ionized gas containing ions, electrons, neutral and excited molecules, and photons (Baltazar-y-Jimenez *et al.* 2008). Two methods of surface treatment using plasma can be distinguished at low pressure and at atmospheric pressure. Plasma surface treatment at atmospheric pressure is less demanding for instrumentation and has been a progressive method in recent years (Cheng *et al.* 2010). The interaction of plasma with a solid surface results in varying changes in surface properties depending on the type of gas used. Surface energy may be increased or decreased, cross-linking of cellulose in the surface layer may occur, or the forming of free reactive groups may take place (Podgorski *et al.* 2000; Baltazar-y-Jimenez *et al.* 2008).

Cold plasma does not cause any changes deeper in the material, but rather only affects the surface layers (Mahlberg *et al.* 1999). The most important parameters when treating a surface with plasma are the plasma surface contact time, the distance between nozzle and surface, and the size of the current (Baltazar-y-Jimenez *et al.* 2008). Primarily the following gases are used to modify the surface of lignocellulosic materials to better bond with the polymer: oxygen (Mahlberg *et al.* 1999), air (Baltazar-y-Jimenez *et al.* 2008), and argon (Zanini *et al.* 2005).

The aim of this research was to clarify the effect of a cold plasma surface treatment of crushed winter wheat stalk particles, prior to board manufacturing, on the physical and mechanical properties of thereof produced particleboards. Specifically, this is a determination of the impact of plasma treatment on the bending strength, tensile strength perpendicular to the plane of the board (internal bonding), vertical density profile, water uptake, and thickness swelling of boards manufactured from plasma-treated wheat straw bonded with urea-formaldehyde adhesive.

EXPERIMENTAL

Materials

Straw particles

Commercially-sold chopped wheat straw particles were used to manufacture the boards (Mikó Stroh, Borota, Hungary). Using digital image analysis, the proportion of individual fractions was defined per 100 g of material sample using a particle analyzer CAMSIZER (Retsch Technology GmbH, Haan, Germany). The sample was poured into the feed chute, allowing the material to enter the measurement field through the feed guide, which prevented unwanted turbulence of the particles and gave the particles the correct orientation. The maximum range was set to 50 mm. The shortest (width) and the longest particle distance (length), measured by the Feret diameter during the projection, was assessed.

Methods

Plasma application method

The wheat straw surface was modified by atmospheric cold plasma in a mixing agent designed to treat particles and other loose materials (Fig. 1). The base consisted of an iron vessel (outer diameter of 415 mm) attached to a rotating platform. At the top, the container was covered with transparent polycarbonate (PC) to close the plasma application environment and to enable visualization of the course of the modification. Inside the container was an eccentrically-positioned cylinder (outer diameter of 110 mm) that was attached to a fixed arm that held it in a stable position relative to the bowl.

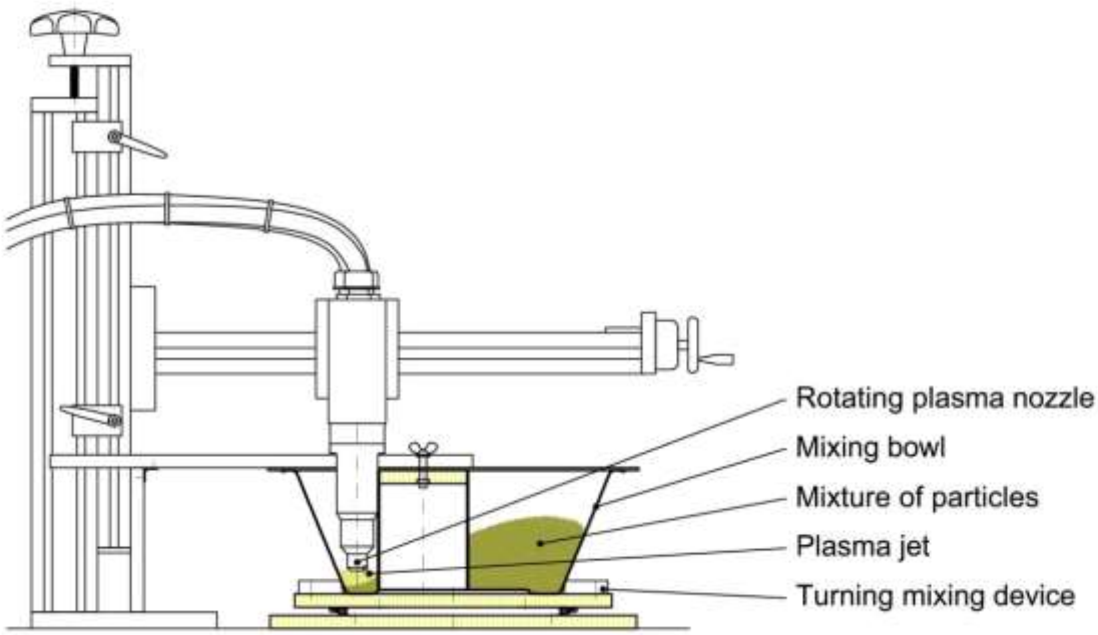

Fig. 1. Side view of the set-up of the stationary plasma aggregate with designed mixing agent

The plasma beam was generated by a high-voltage discharge from the FG 1001 generator (PlasmaTreat GmbH, Steinhagen, Germany) with a maximum output of 1000 VA, and it was distributed to the surface of the particles using compressed air (2 bar). Cold air plasma generated at atmospheric pressure was used. The rotary system of the nozzle of plasma aggregate RD1004 (PlasmaTreat GmbH, Steinhagen, Germany) with the standard AGR 131A (25°) nozzle produced a conical beam shape. To compare the effect of the treatment on wheat-straw using plasma, two variants of surface treatment and one reference variant without treatment (R) were proposed. The variants of plasma application are shown in Table 1.

Table 1. Variants of Plasma Modifications

	Voltage (Generator Set Up)	Current (Generator Set Up)
Modification A	26.9 V	6.9 A
Modification B	28.6 V	8.7 A

The plasma was applied to 100 g of wheat straw for 4 min, and during the treatment, the Alther 2590 digital thermometer (Ahlborn Mess- und Regelungstechnik GmbH, Holzkirchen, Germany) recorded the maximum temperature inside the particle blend.

The degree of plasma surface activation of particles was evaluated with the use of Arcotest (Arcotest GmbH, Moensheim, Germany) test inks designed to measure surface tension. The ink value was identified 2 s after it was applied to the surface, and then an image was recorded using the DTX 90 digital microscope (Levenhuk, Tampa, USA). Testing was conducted until the ink started to coalesce into drops. When it coalesced, a lower-value ink was used, and the boundary between the two inks was sought out.

Adhesive mixture application

After activation of the surface, the particles were resinated with a preformed urea-formaldehyde (UF), hardener (ratio solids hardener / dry adhesive was 10%), and paraffin emulsion mixture (ratio solids hydr. agent / dry particles was 1%). The solid content was 50%. A resin dosage of 10% solids on wood dry mass was applied in a planetary mixer M 301 (Bonnet, Mitry-Mory, France). The mixture was subsequently placed in a drying chamber EHR-K 15/40/20 II (Helios Ventilatoren GmbH, Villingen-Schwenningen, Germany), where it was dried at 30 °C to a moisture content of 8%. The ISI10 scale (Sartorius AG, Göttingne, Germany) was used to continuously monitor water loss, and the final moisture was determined on a moisture tester Ultra X 3011 instrument (A&P instruments, Detmold, Germany). *Via* gradual pouring, the prepared mixture (175 g) was manually layered into a mold with internal dimensions of 128 mm × 355 mm. The layer was spread evenly along the horizontal guiding lines on the inside of the mold.

Pre-pressing and hot-pressing

The mold was then closed and a cold pre-press was performed on the HLP350 hydraulic press (Höfer Presstechnik GmbH, Taiskirchen, Austria). The pressing conditions were set manually by means of the controller to an initial pressure of 4 bars for 1 min. The pre-pressed board was then removed from the mold and continued to be pressed by two heated plates set to 165 °C. The pressing plates were always separated on both sides using waxed paper to avoid adhesion of the boards to the press plate. The pressing was performed according to the pre-set program on the resulting board thickness of 6 mm. The press cycle is shown in Table 2. The total number of six boards for each variant was manufactured. The boards were then allowed to cool down and, further on, they were conditioned at 20 °C and a relative humidity (RH) of 65% for 14 days.

Table 2. Pressing Cycle

Phase No.	Thickness at the End (mm)	Moving Time (s)	Remaining Time (s)
1	40	0.1	0
2	9	4	0
3	5.9	4	6
4	6	5	5
5	6.3	3	0
6	6	3	50
7	6.5	25	0
8	500	0.1	0

Physical and mechanical properties estimation

Test samples were made from the manufactured boards with a rectangular shape for four-point bending, internal bonding, vertical density profile measurement, and samples for water uptake with thickness swelling. Before the mechanical properties measurement, the test specimens were air-conditioned at 65% RH and a temperature of 20 °C.

Strength tests (Fig. 2) were performed on a TIRA test 2850 (TIRA GmbH, Schalkau, Germany) universal testing machine. The maximum force, F_{max}, was always recorded with 1% accuracy. The maximum load was always achieved within 60 s ± 30 s. Before loading, the test specimens were placed in an air-conditioned chamber at 65% RH and a temperature of 20 °C.

The basis of the bending test was to place the test specimen (50 mm × 300 mm) flat on two parallel cylindrical supports (d = 30 mm). Another two supports (d = 30 mm) had a constant loading force, F, cantered above the axis of the board and the maximum force, F_{max}, derived by the machine was measured. The total bending strength was calculated as the arithmetic mean of the values of the following equation for each group of specimens,

$$f_m\ (N * mm^{-2}) = \frac{3 * F_{max} * l_m}{2 * b_m * t_m^2}, \quad (1)$$

where l_m is the length (mm), b_m is the width (mm), and t_m is the thickness (mm) of the test specimen for four-point bending. Tests were not performed according to EN 798 (2004) because no characteristic values of board properties were determined. Using this measuring method, it is possible to compare measured strength values with previously obtained data from the authors' research.

The internal bonding of boards was estimated according to EN 319 (1993) on samples with dimensions of 50 mm × 50 mm.

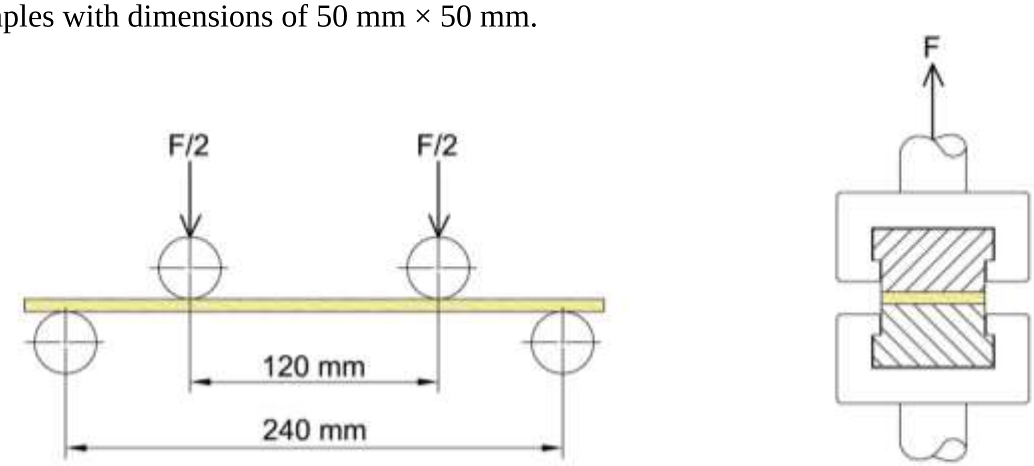

Fig. 2. Diagram of mechanical properties testing

According to Eq. 2, dimensional changes at different air-conditioning stages were determined for water uptake and thickness swelling. The test specimens were first dried to 0% board moisture at 103 °C (air-conditioning stage 0). The samples were then air-conditioned at 20 °C and 65% RH (air-conditioning stage 1) and then at 85% RH (air-conditioning stage 2).

This was reverted to desorption at 65% RH (air-conditioning stage 3), and the last phase was the reverse drying of the samples back to 0% moisture at 103 °C (air-conditioning stage 4). Weight with dimensions of test specimens at marked points was determined at each stage.

$$\beta_x(\%) = \frac{l_x - l_0}{l_0} \text{ x } 100 \quad (2)$$

Measuring points were indicated to ensure repeatability by measuring (mm) in the same position for all air-conditioning stages (x = 0 to 4). Furthermore, the moisture content of samples w_x (%) according to Eq. 3 was determined, where m_0 is the dry sample weight (g) and mass m_x is the weight of the samples at air-conditioning levels (g). The density of the samples ρ_x (kg/m^3) was again calculated from the mass m_x and the sample volume V_x according to Eq. 4:

$$w_x\ (\%) = \frac{m_x - m_0}{m_0} \text{ x } 100 \quad (3)$$

$$\rho_x\ (kg * m^{-3}) = \frac{m_x}{V_x} \quad (4)$$

The vertical density profile of boards was measured on a Compact X-ray density profile Analyser DPX300-LTE (Imal, Modena, Italy). The test samples had dimensions of 50 mm × 50 mm and were air-conditioned at 20 °C and 65% RH.

Scanning electron microscopy (SEM) of ruptured samples, after internal bonding tests 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.

Statistical methods

Besides descriptive statistics, an analysis of variance was used to determine whether any of the pairwise differences from the number of means were significant. The Tukey *post hoc* test was employed to determine the significant differences between group means. A significance level of $\alpha = 0.05$ was selected and all computations were performed using Statistica12 software (StatSoft CR s.r.o., Prague, Czech Republic).

RESULTS AND DISCUSSION

Particle size analysis

A digital imaging analysis revealed a heterogeneous proportion of the used wheat straw particles from which the boards were made. As shown in Fig. 3, there are also a number of dust particles in addition to the wheat straw stalks. Nevertheless, it was found that from 100 g of the sample, 30.9% of the particle was from 1.657 mm to 2.696 mm wide. In terms of length (Feret diameter), 29.1% of particle sizes ranged from 8.393 mm to 13.656 mm.

The projection of some deformed particles may result in inaccuracies in measurements, which may be due to the processing of, for example, broad and thin or narrow and long stalks. To avoid distortion of the results, a sufficiently large set of 100 g was chosen to cover these inaccuracies.

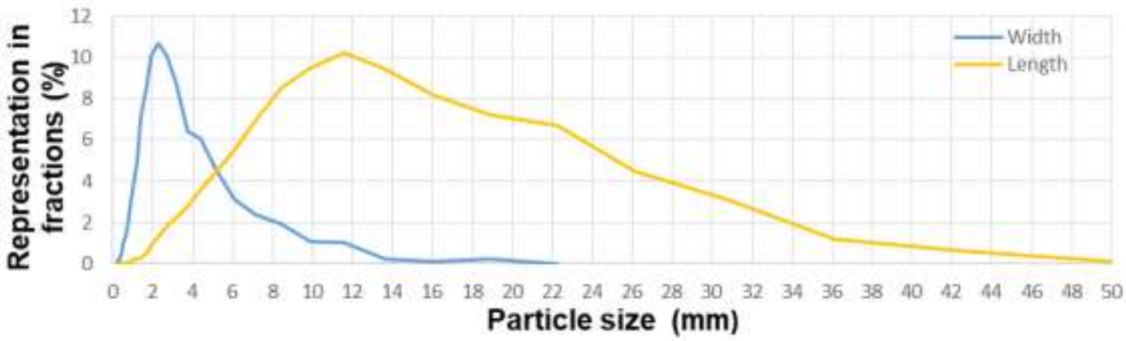

Fig. 3. Results of the particle size analysis

Plasma application and surface tension changes

Due to the designed enclosed mixer, it was possible to modify the particle mixture homogeneously using plasma. Two modification degrees of lower (26.9 V/6.9 A) and maximum power (28.6 V/8.7 A) were used to treat the straw. Despite the fact that cold plasma was used, the maximum average vessel temperature reached 81 °C at lower power, while at maximum power it increased up to 86 °C. As a result, most likely a small effect of thermal treatment has to be taken in consideration in addition to the plasma treatment of the wheat straw. This effect led to a pre-drying of the particles.

After plasma treatment, test inks on the outer sides of the straw determined a change in the surface energy of the modified particles, which were compared with the reference particles without modification. The surface tension on the outside of the reference straw covered range values from 24 mN/m to 28 mN/m. Wheat straw with a lower degree of plasma treatment (A) ranged from 28 mN/m to 30 mN/m, and at a higher degree of modification (B), the surface tension was from 30 mN/m to 32 mN/m. The variability of the unmodified particles was thus higher than that of the plasma-treated particles. Therefore, the testing inks confirmed that both degrees of cold plasma treatment increased the surface wettability and changed the surface energy. However, these results could be affected by the different moisture of particles. Plasma-treated particles in the missing agent were partly dried and then immediately examined by test inks. The treated particles were not conditioned because the effect of cold air plasma on the physical properties of modified material decreases with time after the plasma application (Klímek *et al.* 2016).

Density and vertical density profile

The average density of the reference boards was 540.0 kg/m^3. Boards with a lower degree of modification reached 524.9 kg/m^3 and boards with a higher degree of modification had an average density of 545.7 kg/m^3. Due to manual layering, the density also exhibited considerable variability (Table 3). Further, it was noticed that the boards manufactured from the reference particles had the steepest density profile (Fig. 4a). The different average density, the variability of the density, and the shape of the density profile have to be taken into consideration while interpreting the physical and mechanical properties measured. Figure 4b shows a cross-sectional cut of the board showing individual particles of winter wheat stalk.

Table 3. Density of Straw Board at 25 °C and 65% Relative Humidity

Density	Ref.	Modification	
		A	B
Mean (kg/m³)	540.0	524.9	545.7
Median (kg/m³)	540.5	528.8	553.4
Standard deviation (kg/m³)	27.9	31.5	31.0
Minimum (kg/m³)	486.2	473.8	491.8
Maximum (kg/m³)	613.2	586.4	607.1

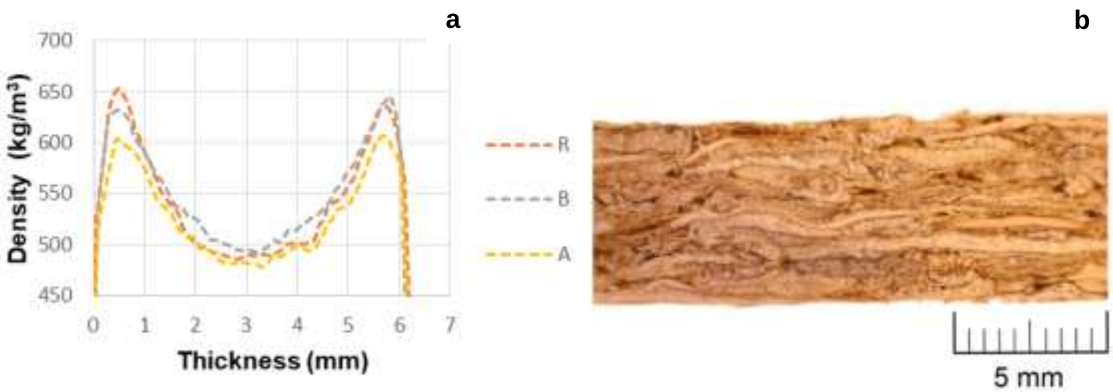

Fig. 4. (a) Vertical density profile of straw boards at 25 °C and 65% relative humidity; (b) side view of pressed board with nominal thickness of 6 mm

Equilibrium moisture content and thickness swelling

Figure 5 shows a graph of the dependency of equilibrium moisture content of the boards on relative humidity, and Fig. 6 shows a graph of the dependency of thickness swelling on relative humidity, and these average values are subsequently specified numerically in Table 4. The highest values of equilibrium moisture and thickness swelling were obtained from boards manufactured from particles modified by a higher degree of plasma treatment (type B), and all of the differences in the given moisture level were statistically significant (Table 5). Adversely, the lowest values of equilibrium moisture were obtained from the boards manufactured from unmodified particles. In terms of the equilibrium moisture of boards manufactured from particles treated with a lower degree of plasma treatment and untreated particles, a statistically significant difference only appeared in the first air-conditioning stage (20 °C/RH 65%, absorption cycle).

Type B boards once again exhibited the highest thickness swelling. As expected, the lowest values of thickness swelling were not reached by boards from the reference particles (Fig. 6). Except for in the first air-conditioning stage (this difference was not statistically significant (Table 6)), a higher thickness swelling in the reference boards was ascertained than in the boards manufactured from modified particles with a lower degree of plasma treatment. This was explained by a variation in the average density of boards, where the average density of the reference boards was 540.0 kg/m^3 and boards with a lower degree of modification reached 524.9 kg/m^3.

Table 4. Average Values of Thickness Swelling and Equilibrium Moisture of Boards on the Given Level

Board Type	Relative Humidity (%)	Thickness Swelling (%)	Moisture (%)
R	0a	0.0	0.0
R	65a	8.3 (0.7)	8.8 (0.4)
R	85	45.9 (5.7)	19.3 (0.6)
R	65d	37.9 (5.5)	11.0 (0.6)
R	0d	30.7 (4.8)	0.0
A	0a	0.0	0.0
A	65a	9.4 (1.6)	9.8 (0.8)
A	85	39.9 (4.5)	19.7 (0.7)
A	65d	33.8 (4.2)	11.4 (0.8)
A	0d	27.9 (2.6)	0.0
B	0a	0.0	0.0
B	65a	10.1 (1.3)	10.8 (0.3)
B	85	53.1 (4.2)	23.0 (0.4)
B	65d	45.3 (3.9)	13.1 (0.3)
B	0d	36.6 (3.3)	0.0

Note: a = absorption, d = desorption, values in parentheses are standard deviations

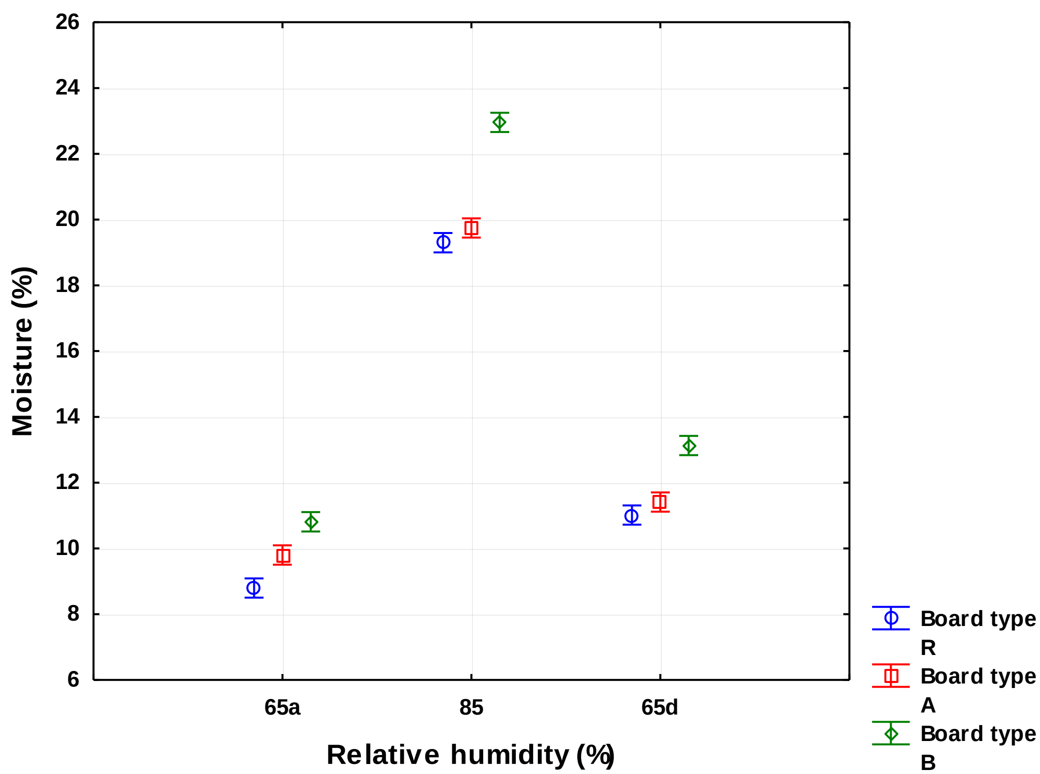

Fig. 5. Graph of the dependency of equilibrium moisture of the boards on relative humidity, a = absorption, d = desorption

Table 5. Appropriate Statistical Significances of Differences in Fig. 5

T = 20°C, RH = 65%, Absorption			
	R	A	B
R		s.	s.
A	s.		s.
B	s.	s.	

T = 20°C, RH = 85%			
	R	A	B
R		n.s.	s.
A	n.s.		s.
B	s.	s.	

T = 20°C, RH = 65%, Desorption			
	R	A	B
R		n.s.	s.
A	n.s.		s.
B	s.	s.	

Note: s. = statistically significant, n.s. = not significant, and α = 0.05

In comparison with commercially produced particleboards, non-recoverable thickness changes of produced boards reached relatively high values. This was explained by the material used. From the authors' previous study it is already known that boards produced from after harvest remains reached non-recoverable thickness changes higher than 30% (Hýsek *et al.* 2018). It was concluded that the plasma treatment of the particles had a statistically significant effect on the equilibrium moisture content of the boards and their thickness swelling; however, the thickness swelling values were negatively affected by the different average densities of the boards. In contrast, when non-recoverable changes were compared with the bending strength results, it can be assumed that modification A was the better level of plasma pre-treatment for the purpose of this study. These boards reached lower non-recoverable thickness changes as well as higher bending strength. Therefore, it was assumed that the lower level of plasma modification caused better adhesion, in comparison to the more aggressive modification B.

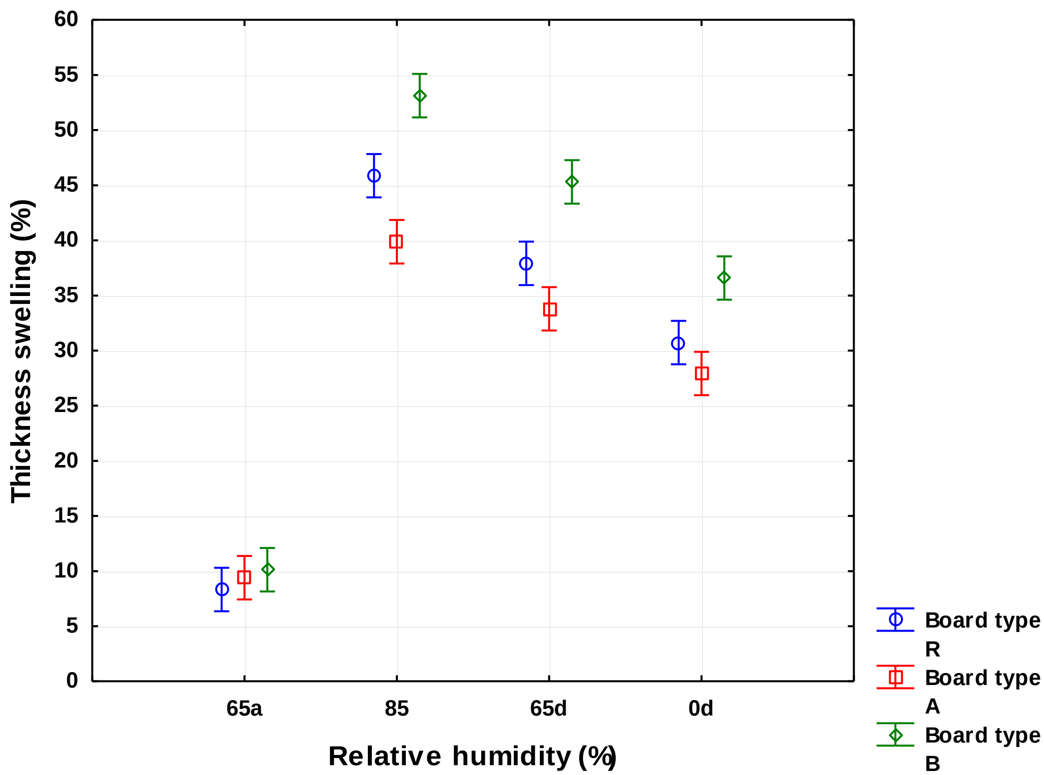

Fig. 6. Graph of the dependency of thickness swelling on relative humidity, a = absorption, d = desorption

Table 6. Appropriate Statistical Significances of Differences in Fig. 6

T = 20°C, RH = 65%, Absorption			
	R	A	B
R		n.s.	s.
A	n.s.		n.s.
B	s.	n.s.	

T = 20°C, RH = 85%			
	R	A	B
R		s.	s.
A	s.		s.
B	s.	s.	

T = 20°C, RH = 65%, Desorption			
	R	A	B
R		s.	s.
A	s.		s.
B	s.	s.	

T = 103°C, Desorption			
	R	A	B
R		n.s.	s.
A	n.s.		s.
B	s.	s.	

Note: s. = statistically significant, n.s. = not significant, and α = 0.05

Bending strength and internal bonding

Table 7 shows the average values with basic descriptive statistics for the bending strength of boards. Figure 7 shows the bending strength variation analysis and Table 8 shows data on the statistical significance of the differences. The results show that in both cases of plasma treatment of the particles there was an increase in flexural strength compared to the reference material, but the increase was only statistically significant for modification A. There was also no statistically significant difference in the flexural strength between the two different plasma treatments.

Table 7. Average Values with Basic Bending Strength Descriptive Statistics

Bending Strength	Ref.	Modification	
		A	B
Mean (MPa)	4.9	5.5	5.1
Median (MPa)	4.8	5.5	5.4
Standard Deviation (MPa)	0.5	0.3	0.7
Minimum (MPa)	4.2	5.0	3.9
Maximum (MPa)	5.6	6.0	5.9

Table 8. Appropriate Statistical Significances of Differences in Fig. 7

	R	A	B
R		s.	n.s.
A	s.		n.s.
B	n.s.	n.s.	

Note: s. = statistically significant, n.s. = not significant, and α = 0.05

Table 9 shows the average values with the basic descriptive statistics for the internal bonding of the boards. Figure 8 shows the internal bonding variation analysis and Table 10 shows data on the statistical significance of the differences. The results showed that in both cases of plasma treatment there was a statistically significant increase in internal bonding. There was no statistically significant difference between the different plasma treatments. Both observed mechanical properties reached lower values than the boards made form rapeseed stalk particles, where internal bonding was 0.34 MPa to 0.50 MPa and the bending strength was 5 MPa to 10 MPa (Hýsek *et al.* 2018). However, in previous research, boards with an average density of 600 kg/m^3 and with an inverse vertical density profile (maximal density in the middle of the board) were produced.

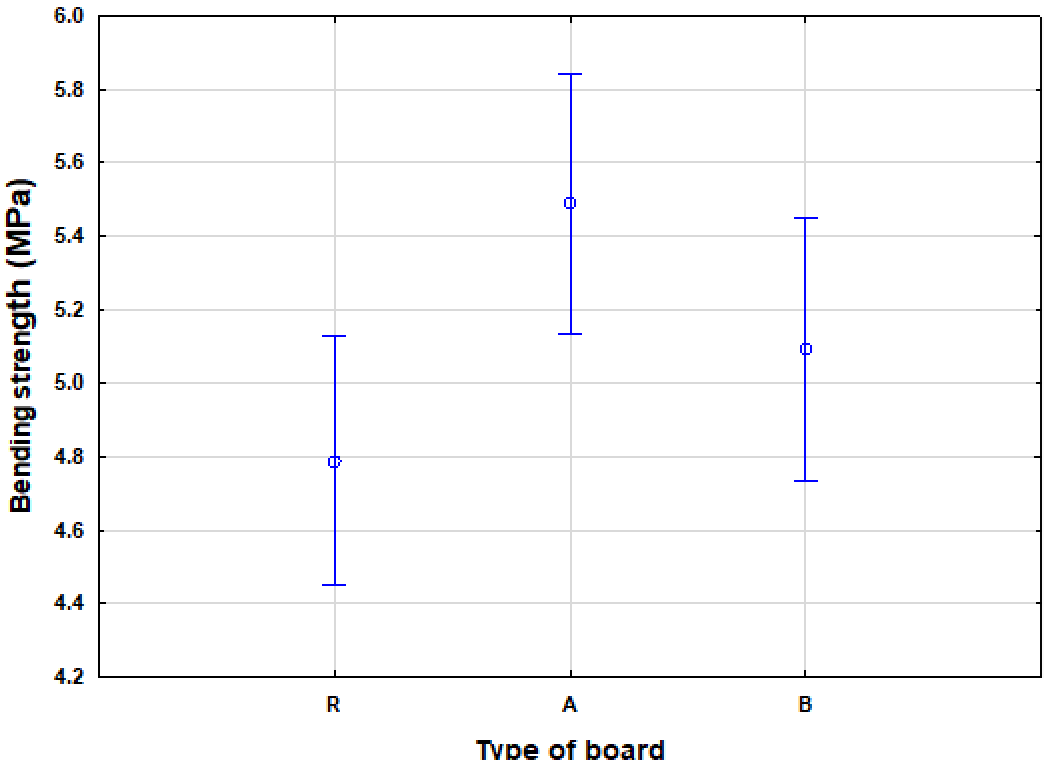

Fig. 7. Analysis of variance - dependence of flexural strength on modification of particles

Table 8. Appropriate Statistical Significances of Differences in Fig. 7

	R	A	B
R		s.	n.s.
A	s.		n.s.
B	n.s.	n.s.	

Note: s. = statistically significant, n.s. = not significant, and α = 0.05

Table 9. Average Values with Basic Internal Bonding Descriptive Statistics

Transverse Tensile Strength	Ref.	Modification	
		A	B
Mean (MPa)	0.037	0.058	0.061
Median (MPa)	0.036	0.054	0.063
Standard deviation (MPa)	0.006	0.019	0.017
Minimum (MPa)	0.028	0.033	0.039
Maximum (MPa)	0.050	0.092	0.098

Table 10. Appropriate Statistical Significances of Differences in Fig. 8

	R	A	B
R		s.	s.
A	s.		n.s.
B	s.	n.s.	

Note: s. = statistically significant, n.s. = not significant, and α = 0.05

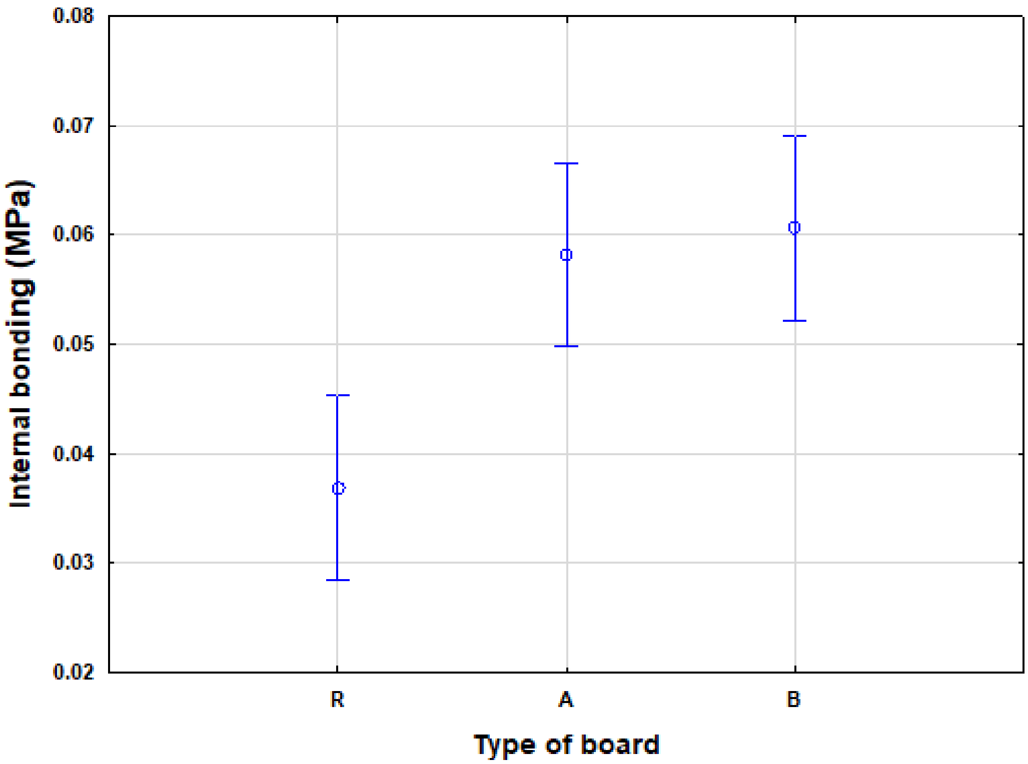

Fig. 8. Analysis of variance – dependence of internal bonding on modification of particles

Figure 9 shows a SEM microscopic image of the damaged joint from the tensile test perpendicular to the plane of the boards. The image shows the noticeable impact of the modification on the nature of the damage. In terms of boards manufactured from the reference unmodified particles (Fig. 9a), there was only adhesion damage between the adhesive and the particle surface. In terms of boards made from plasma-modified particles (Figs. 9b, 9c), cohesive breakage in the particle material was also observed, which indicated a better joint of the modified particle-adhesive.

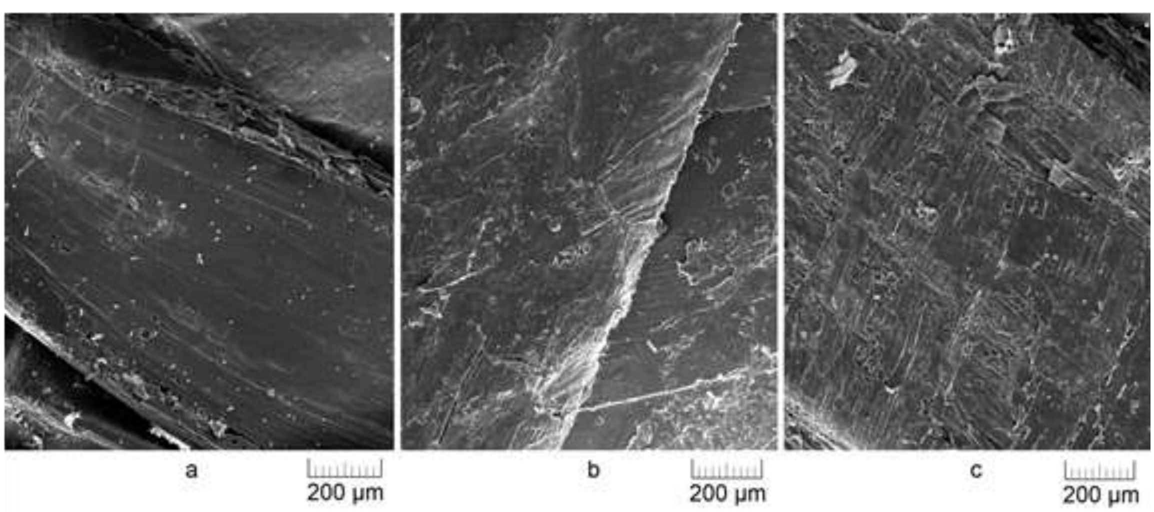

Fig. 9. SEM analysis of particle of boards after strength testing: (a) reference, (b) modification A, and (c) modification B

CONCLUSIONS

1. The effect of plasma treatment on the properties of composite material made from winter wheat stalk particles was investigated. Test inks showed an increase in surface energy and confirmed that plasma treatment influences surface properties of the particles.
2. The plasma treatment of the particles had a statistically significant effect on the equilibrium moisture content of the boards and on their thickness swelling, with increased degrees of plasma treatment the equilibrium moisture content also increased.
3. Opposite of the physical properties, the positive effect of plasma pre-treatment of the particles was observed in the mechanical properties. Both the flexural strength and internal bonding of the boards were increased. The highest increase in flexural strength was achieved by the type A plasma treatment, whereas a difference between the individual types of plasma treatment was not observed in the internal bonding.
4. A better joint of the modified particle-adhesive was reached by the plasma treatment. A noticeable impact of the modification on the nature of the damage in produced composite materials was observed through SEM analysis.

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