

DEVELOPMENT OF STRAW PARTICLEBOARD USING CITRIC ACID AS BIO-BASED ADHESIVE

Amin Khatiri¹

<https://orcid.org/0000-0002-8634-6437>



Ali Shalbafan^{1,*}

<https://orcid.org/0000-0002-7355-5977>

Saeed Kazemi Najafi¹

<https://orcid.org/0000-0003-4434-0018>

ABSTRACT

The intensive utilization of agricultural residues, particularly wheat straw (WS), in particleboard production as an environmentally sustainable alternative to wood-based panels remains limited due to their poor bonding performance with conventional adhesives. Citric acid (CA) has recently gained increasing attention as a bio-based adhesive and appears to be a promising solution to overcome the bonding challenges associated with wheat straw. However, there is limited information available on the combined effects of the moisture content (MC) of the mat and the adhesive dosage on the behavior of WS particleboard bonded with citric acid. Therefore, the objective of this study was to determine the optimal moisture content (MC) and the optimal CA content required to achieve satisfactory physical and mechanical properties without pretreating the straw particles. To this end, the resinized straw particles were oven-dried at different times intervals (2 h to 14 h) and temperatures (80 °C and 100 °C) to obtain mat with various moisture content (0,5 % - 20 %). The effects of adhesive content of 15 %, 20 %, 25 %, and 30 % (based on the dry mass of the straw) on the properties of the panels were also studied. The optimum moisture content of the mat before hot pressing was about 11%, and the corresponding panel exhibited flexural strength, modulus of elasticity and internal bond of about 12 MPa, 3700 MPa and 0,26 MPa, respectively, while the thickness swelling and water absorption after 24-hour immersion were 6,5% and 46 %, respectively. Increasing the adhesive content from 15 % to 30 % led to an increase in flexural strength from 9 MPa to 13 MPa and 117% in internal bond. However, a further increase in the adhesive content above 20 % did not lead to any statistically significant differences in the physical properties. In general, the results showed that due to the acidic nature of citric acid, it is possible to produce particleboard from wheat straw without pretreating the straw particles before use.

Keywords: Agricultural residues, bio-based adhesive, citric acid, particleboard, wheat straw lignocellulosic materials.

INTRODUCTION

The increase in global population have increased the demand for wood-based panels (WBP), with global production of more than doubling from about 178 million m³ in 2000 to about 381 million m³ in 2023 (FAO 2025). On the other hand, competition for raw wood supply has intensified, leading to an increase in wood prices, mainly due to increased demand for raw wood from both WBP producers and the bioenergy sector (Shalbafan *et al.* 2017). In countries like Iran, the wood supply is much worse due to the tripling of production volume in the last decade (more than 3,5 million m³) and ongoing forest conservation plans (Beygi Heidarlou *et al.* 2023). The use of agricultural waste as a renewable and abundant material in the world and also in Iran is considered one of the most reliable solutions to overcome this challenge (De Melo *et al.* 2015, Nazerian *et al.* 2016). Most agricultural residues are quite similar to wood in terms of the chemical and morphological composition of the fibers. Researchers have shown the potential of various agricultural residues such as bagasse stalks (Adefris *et al.* 2022), rice straw (Li *et al.* 2010), cotton stalk (Nazerian *et al.* 2016), rice husk (De Melo *et al.* 2015) and oil palm fronds (Zakaria *et al.* 2021) for particleboard production.

¹Tarbiat Modares University. Faculty of Natural Resources and Marine Sciences. Department of Wood and Paper Science and Technology. Noor, Iran.

*Corresponding author: ali.shalbafan@modares.ac.ir

Received: 01.02.2024 Accepted: 23.06.2026

Wheat straw has significant potential to replace wood for the production of WBP as it is much more widespread and abundant than other lignocellulosic sources in the world. Global wheat production is estimated at about 808 million tons (FAO 2025). It is stated that for every ton of wheat, about 1,3 to 1,6 tons of straw remain in the field (Neitzel *et al.* 2022). Thus, it is estimated that at least 1 billion tons of wheat straw remains in the fields every year as residue, most of which is burned, which has a negative impact on global warming potential. Previous research has shown that although wheat straw can be used as a raw material for the production of WBP, the composition and hydrophobic structure of the straw surface leads to incompatibility between the straw and the conventional resins (e.g., urea formaldehyde) used for WBP production (Boquillon *et al.* 2004, Hematabadi and Behrooz 2012). In other words, although straw is one of the most abundant agricultural byproducts worldwide, its use in the production of particleboard remains limited compared to conventional wood resources. The poor adhesion between straw particles and common adhesives has restricted the production of high-performance straw-based panels and diminished the industrial appeal of this renewable raw material. Overcoming this challenge is therefore crucial for expanding the utilization of agricultural residues, particularly wheat straw, for reducing dependence on wood resources, and for promoting the development of more sustainable and circular bio-based material systems.

To overcome the above challenge, the wheat straw was pretreated with various materials before being used for board production. Pretreatments of wheat straw with enzymes (Zhang *et al.* 2003), steam, hot water, sulfuric acid (Harlvarsson *et al.* 2009), acetic anhydride and soap solution (Bekhta *et al.* 2013), silane coupling agents (Hafezi *et al.* 2016), and plasma (Hýsek *et al.* 2018) have been carried out to improve the adhesion of wheat straw with urea formaldehyde adhesives. Other studies have also suggested the use of alternative adhesive systems such as isocyanate (p-MDI) (Boquillon *et al.* 2004, Cao *et al.* 2017), acrylated epoxidised soybean oil (Tasooji *et al.* 2010) and tannin adhesive (Tabarsa *et al.* 2011) to make panels from wheat straw. Although these approaches have improved the bonding performance of WS particleboard, many of them require additional pretreatment steps, special processing conditions, or adhesive systems that increase manufacturing complexity, energy consumption, and production costs. Furthermore, some adhesive systems raise environmental and health concerns, while others have demonstrated satisfactory performance only under specific laboratory conditions, limiting their broader industrial applicability. Consequently, the development of simpler, more sustainable, and industrially feasible bonding strategies for WS particleboard remains a major challenge. However, there is interest in green or environmentally friendly adhesives for WBP production (Shalbafan *et al.* 2017, Sutiawan *et al.* 2022).

Citric acid (CA) is an organic polycarboxylic acid with three carboxyl groups that is commercially produced from the fermentation of glucose or materials containing glucose and sucrose (Widyorini *et al.* 2016). CA can also be extracted naturally from various fruits and vegetables, especially citrus fruits such as oranges, tangerines, lemons, limes, etc. CA has been used for decades as a modifier to improve the dimensional stability of wood and WBP (Umemura *et al.* 2012). Recently, Menezzi *et al.* (2018) and Ando and Umemura (2021) studied the chemical bonding between the functional groups of wood carbohydrates and lignin with CA using sophisticated analytical techniques. They found that an ester bond forms between the carboxyl groups of CA and the hydroxyl groups (aromatic and aliphatic) in lignin and wood carbohydrates. So far, researchers have investigated the potential of CA as a bio-adhesive to produce different types of WBP, especially from agricultural residues (Widyorini *et al.* 2016, Kusumah *et al.* 2016, Syamani *et al.* 2018). The effective processing conditions for CA-bonded particleboard were summarized by Lee *et al.* (2020) as follows: pressing temperature higher than 180 °C, panel density of 800 kg/m³, CA content of 20 wt.% or more and pre-drying of 12 h at 80 °C. Although promising results have been reported for a variety of lignocellulosic materials bonded with CA, it remains unclear whether the same bonding mechanisms and processing conditions can be directly applied to WS particleboards. WS differs from many conventional lignocellulosic raw materials in terms of its surface chemistry, silica-rich waxy layer, and limited compatibility with adhesive systems. These characteristics may influence both ester bond formation and the curing behavior of CA during hot pressing. Furthermore, the interaction between mat MC and CA loading, which are critical parameters for adhesive curing and bond development, has not been systematically investigated for WS particleboards. Therefore, additional research is required to determine the processing conditions necessary to achieve satisfactory bonding performance in CA-bonded WS panels. The pre-drying of the resinized particles influences the MC of the mat before pressing, which is known to have a significant effect on the degree of the adhesive curing and the properties of the panels (Böhm 2009, Halligan and Schniewind 1974). Although pre-drying of the resinized particles with CA adhesives has been proposed (Kusumah *et al.* 2016, Syamani *et al.* 2018), the influence of a different time and temperature on the pre-drying of the particles needs to be further investigated.

To date, no studies have investigated the production of wheat straw (WS) particleboards using CA as a bio-based adhesive, which constitutes the primary objective of the present study. More specifically, the central research gap addressed is whether CA can overcome the inherent adhesion limitations of WS without requiring additional pretreatment steps. Owing to its acidic nature, CA may partially remove or modify the waxy surface layer of straw and form ester linkages with hydroxyl groups, thereby enabling effective bonding.

Demonstrating the ability of CA to achieve satisfactory bonding performance without additional pretreat-

ment would contribute to addressing one of the major challenges associated with the utilization of WS for particleboard production. Therefore, it is necessary to investigate not only the bonding potential of CA, but also the processing conditions required to achieve effective bond development in WS particleboards.

To evaluate and optimize this adhesive system, key processing parameters were considered as supporting variables. In this context, the effects of adhesive content and mat moisture content on board performance were investigated. Additionally, different pre-drying protocols, involving varying drying times and temperatures, were applied to control the moisture content of the resin-treated particles.

MATERIALS AND METHODS

Materials preparation

The wheat straw was obtained from the farms in Golestan province, Iran. A hammermill was used to produce the particles. The straw particles were then sieved to obtain particles of uniform size and the particles that passed mesh size 8 and remained at mesh size 50 were used as raw material. The particles were oven-dried at 80 °C for 12 h to achieve a MC of less than 5 % before resination. CA in commercial grade and powder form was obtained from Hugestone China. CA was used without further purification and dissolved in water. To reduce the toughness and brittleness of the CA (Zakaria *et al.* 2021), 2 % corn starch powder (per dry weight of straw) was also added to the CA solution. The final concentration of the adhesive was constant at 62 % throughout the study. Urea formaldehyde (UF) adhesive supplied from Amol Resin was also used as a reference adhesive.

Adhesive characterization

The properties of CA adhesives before and after the addition of starch were characterized. The viscosity of the adhesive was measured according to DIN 53211 (1987), whereby the flow time of the adhesive was measured with a DIN flow cup of 4 mm. To measure the gel time, 10 g of adhesive was poured into a glass tube and stirred at 100 °C in a water bath. The time that passes until the adhesive can no longer be stirred (becomes a gel) was recorded as the gel time. The pH value of the adhesive was measured at a temperature of $23 \pm 0,5$ according to ASTM E70-19 (2019). It should be noted that the instrument was calibrated before the measurement. The pycnometer method according to the ISO 2811-1 (2023) was used to measure the density of the adhesive. The following Equation 1 was used to calculate the density of the adhesive.

$$D = \frac{M_p - M_a}{V_p} \quad (1)$$

Where D is the adhesive density (g/ml), M_p is the pycnometer weight (g), M_a is the adhesive weight (g) and V_p is the pycnometer volume (ml). To obtain each of the above properties of the CA adhesive, five replicates were performed, of which the average was reported.

Fourier transform infrared (FTIR) spectroscopy

The FTIR spectrum was recorded to analyze and investigate the mechanism of the bonds between CA and wheat straw. For this purpose, resinized wheat straw was taken from the edge of the panel. The particles were ground into a powder, and the obtained powder was dried in a vacuum drying oven at 70 °C for 10 h. Infrared (IR) spectral data were obtained with an FTIR spectrophotometer (FT/IR-6300, JASCO Corporation, Japan) using the KBr disk technique and recorded with an average of 32 scans at a resolution of 4 cm^{-1} in the range 4000-400 cm^{-1} .

Wheat straw particleboard production

Single-layer particleboards with a thickness of 6 mm were made from resinized wheat straw particles. According to the literature (Syamani *et al.* 2018, Kusumah *et al.* 2016), the straw particles were placed in the laboratory oven (at different times and temperatures) to reduce and control the MC of the resinized particles.

After the oven, the particles were poured into a 400 x 300 mm² box to form the mat. The prepared mat was placed in a hot press for 360 seconds at a temperature of 200 °C. In addition, 6 mm thick steel bars were used to control the panel thickness during hot pressing. The maximum specific pressure was 3,5 MPa when the press reached the final panel thickness. The density of the panel was kept constant at 800 kg/m³.

In addition, a particleboard glued with 20 wt.% UF resin was produced, which had a similar thickness and density to the CA-glued particleboard and served as a reference. However, these reference boards were delaminated after hot pressing and no tests could be performed on those specimens. All CA-glued panels were taken to the laboratory for conditioning for two days. The schematic process for producing wheat straw particleboard with CA as a bio-adhesive is shown in Figure 1.

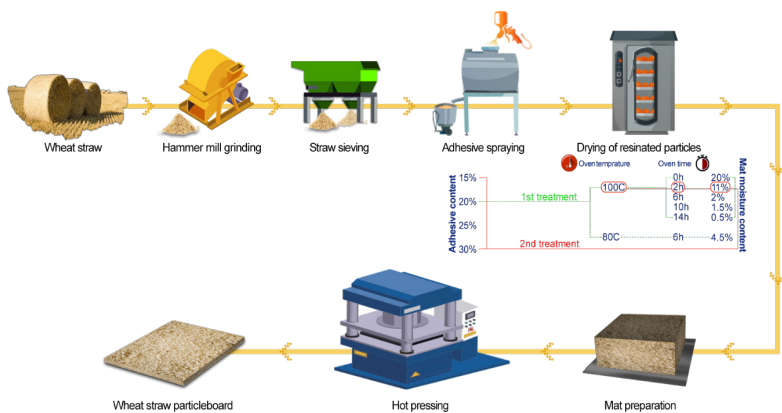


Figure 1: Process for producing wheat straw particleboard with CA as adhesive.

The MC of the resinized particles and the adhesive content were considered as research treatments. To control the MC of the mat, the resinized straw particles were placed in a laboratory oven at different time intervals (2 h, 6 h, 10 h, and 14 h) and temperatures (80 °C and 100 °C). In addition, particleboard was also produced with resinized straw particles that were not stored in the oven. Depending on the duration and temperature of the oven, the mat had a MC between 0,5 % and 20 % before hot pressing. The effect of the adhesive content on the properties of the wheat straw particleboard was also investigated at four levels of 15, 20, 25, and 30 % (based on the dry weight of the straw particles). The research treatments are listed in Table 1. Three boards were prepared for each treatment, and three test specimens were made from each board. The specimens were conditioned at 20 ± 2 °C and 65 ± 5 % relative humidity before the tests.

Table 1: Research treatments for the production of wheat straw particleboard using CA as an adhesive.

Samples code	Oven time (h)	Oven temperature (°C)	Mat moisture content (%)*	Adhesive content (%)
A	0	100	20	20
B	2		11	
C	6		2	
D	10		1,5	
E	14		0,5	
F	6	100	2	
G		80	4,5	
H	2	100	11	15
K				20
L				25
M				30
R	Reference sample with UF adhesive			20

* Moisture content was measured as a function of the corresponding drying time and temperature of the resinized particles in the oven (numbers were rounded for simplicity). Codes C and F are similar.

Panel characteristics

The evaluation of the physical and mechanical properties of wheat straw particleboards was carried out according to European standards. The modulus of elasticity and flexural strength were measured in accordance with EN 310 (1993). The internal bond strength and moisture content were measured according to EN 319 (1993) and EN 322 (1993) respectively. Thickness swelling was also tested according to EN 317 (1993) and water absorption according to Khakzad *et al.* (2020).

A displacement speed of 2 and 1 mm/min was used to measure the bending properties and internal bond strength, respectively. In addition, a digital scale with an accuracy of 0,01 gram was used to measure the weight of the specimens, and a caliper with an accuracy of 0,01 mm was used to measure the dimensions of the specimens. Nine replicates were tested for each panel variant. Differences between panel characteristics were analyzed with a one-way ANOVA using SPSS software (IBM SPSS Statistics 26). Statistical significance was set at $P<0,05$.

RESULTS AND DISCUSSION

Adhesive characteristics

The specifications of the CA adhesive including density, pH, viscosity, and gel time with and without starch as an additive are shown in Table 2. The CA adhesive with and without starch had a similar density of about 1,28 g/ml. The results also show that the pH of the CA adhesive with and without starch was 1,5 and 2,5, respectively. In other words, the pH increased with the addition of starch due to its alkalinity (Lee *et al.* 2019). Other researchers have also reported that the pH of CA adhesive without additives is less than 2 (Sutiawan *et al.* 2022), which is consistent with this result. The viscosity of the CA adhesive, with and without starch, was 38 and 45 centipoises, respectively, which requires the resinated particles to be pre-dried (Kusumah *et al.* 2016, Syamani *et al.* 2018).

The gel time of the CA adhesive without starch was about 50 seconds. The addition of starch to the CA shortened the gelation time to 42 seconds, a reduction of 16 %. The reason for this could be the formation of cross-links between CA and starch and the acceleration of adhesive curing. The lower the gel time, the shorter the production time which is industrially required.

Table 2: Characteristics of CA adhesives with and without starch

Adhesive type	Density (g/ml)	Gel time (s)	Viscosity (cP)	pH
Citric acid solution	1,28 ± 0,02	50 ± 3	38 ± 2	1,5 ± 0,1
Citric acid solution with starch*	1,27 ± 0,01	42 ± 2	45 ± 3	2,5 ± 0,2

* The starch content was 2 %, based on the dry mass of the straw.

Infrared spectroscopy (FTIR)

FTIR analysis was performed to evaluate the formation of chemical bonds between the wheat straw and the carboxylic acid groups of CA, and the results are shown in Figure 2. The peak in the range of 1722 and 1733 cm^{-1} in CA is due to C=O stretching originating from the carboxyl group or the ester group. Peaks in the range of 1208 cm^{-1} in CA have also been associated with C-O stretching and -OH bending (Umemura *et al.* 2012). The peaks at wavenumbers 1159, 1082, and 1014 cm^{-1} in corn starch were also attributed to C-O stretching (Goheen and Wool 1991). The absorption peaks at wavenumbers 992, 929, 861, 765, and 575 cm^{-1} are also attributed to the stretching vibrations of the anhydrous glucose ring in starch. A broad peak was observed at 3421 cm^{-1} which is attributed to the hydroxyl groups present in starch (Goheen and Wool 1991). The strong broad peak in 3330 cm^{-1} of wheat straw can be associated with the O-H bond. The peak in the wave-number of 1031 cm^{-1} is also caused by the stretching vibration of the C-O bond (Asghar *et al.* 2015). Peaks in the range of 1400-1200 cm^{-1} in wheat straw also correspond to the bending frequencies of C-H, O-H, or CH_2 .

FTIR spectroscopy of resinized straw with CA also revealed a peak at 1732 cm^{-1} associated with the car-

bonyl group of the stretching C=O double bond (Menezzi *et al.* 2018, Ando and Umemura 2021). Previous studies have shown that this peak is related to the adhesion between the carboxylic acid group of CA and the hydroxyl group of lignocellulosic materials, the so-called ester bond (Umemura *et al.* 2012). CA led to the formation of ester bonds (R-COO-R) by esterification between the carboxyl groups of CA and the hydroxyl groups of wood (Sutiawan *et al.* 2022). Particular attention should be paid to the appearance of the absorption band at approximately 1732 cm⁻¹ in the spectrum of the resin-treated WS. This spectral feature serves as direct evidence for the formation of ester bonds between CA and the hydroxyl groups of the WS under the applied pressing conditions. The observed chemical interaction supports the proposed bonding mechanism of the adhesive system and provides a molecular-level explanation for the satisfactory performance of the manufactured particleboard. Esterification of wood is one of the most common chemical reactions that occurs at the level of the cell structure of woody materials to improve the dimensional stability and biological durability of wood and wood products. Esterification is a chemical reaction between an acid (carboxylic acid) and an alcohol (or other -OH) to form an ester and water (Lee *et al.* 2020).

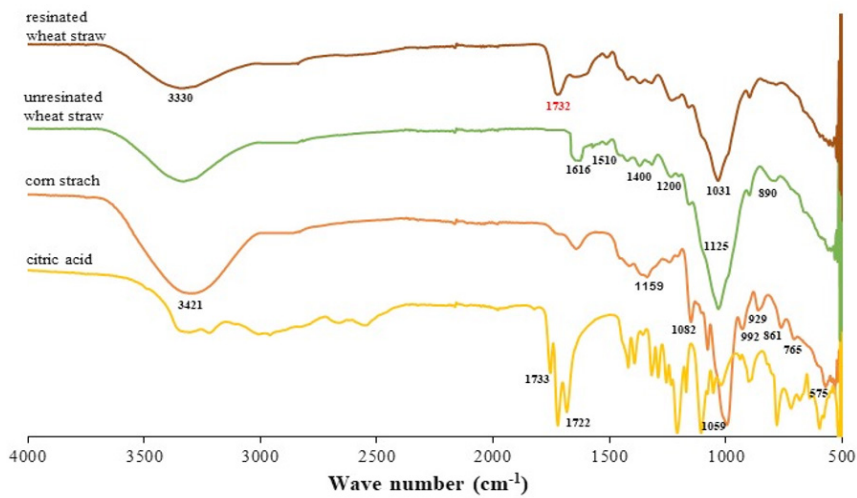


Figure 2: Fourier transform infrared spectra of CA, starch, and wheat straw with and without adhesive.

Influence of moisture content (MC)

MC of straw

It was seen that when the MC of the mat is increased up to 22 %, the mechanical properties of the boards decrease, and their physical properties increase (Böhm 2009). Halliga and Schniewind (1974) also reported a significant decrease in the flexural properties and internal bond strength of particleboard made with urea-formaldehyde resin when the mat MC was increased by more than 5 %. Therefore, the precise control of the mat MC before pressing, depending on the type of adhesive used, is an important step to achieve the desired panels properties (Shalbafan and Thoemen 2022). For panels produced with CA as the adhesive, drying of the resinized particles is required, as the CA adhesive gives the mat a higher water content and water is also generated during the esterification reaction (Lee *et al.* 2020).

The effects of time (at a constant drying temperature of 100 °C) and temperature (at a constant drying time of 6 h) in the oven on the MC of the straw mat are shown in Figure 3. As expected, increasing the temperature and drying time of the resinized particles in the oven resulted in a significant decrease in the MC of the particles. The resinized particles (20 wt.% CA) had an MC of about 20 % without drying. The MC of the mat fell by almost half and reached around 10,6 % after 2 h of drying. With increasing drying time, the MC decreased further and reached about 0,5 % after 14 h.

At a fixed drying time (6 h), the oven temperature also had a significant effect on halving the MC of the resinized particles, so that the MC decreased from about 4 % (6 h at 80 °C) to about 1,9 % (6 h at 100 °C). In the literature, a temperature of 80 °C is recommended for drying resinized particles with CA (Kusumah *et al.* 2016, Syamani *et al.* 2018, Lee *et al.* 2020). However, the results of this study show that the drying temperature of 100 °C has a greater influence on the internal bond strength, which is explained in more detail in the

corresponding section on internal bond strength. This could also have a significant impact on shortening the drying time, which is desirable in the industry.

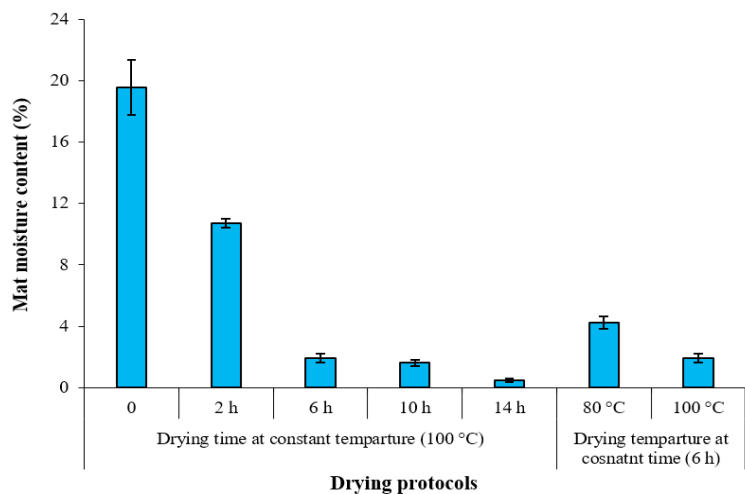


Figure 3: Influence of drying temperature and time on the MC of the mat before hot pressing.

Flexural properties

The Figure 4 shows the effect of the MC of the straw mats on the flexural properties of the wheat straw particleboard (for simplicity, the MC of the mat is rounded in Figures). The lowest flexural strength and modulus of elasticity were found for the sample with MC of 20 % (without drying the particles). By decreasing the MC of the particles, the flexural strength and elastic modulus increased significantly. The highest flexural strength and modulus of elasticity were obtained for specimens with MC of 11 % (dried at 2 h and 100 °C) at 12 and 3700 MPa, respectively. The mat always needs a certain amount of moisture, especially in the surface layers, to accelerate the heat transfer from the press plates to the core layer of the mat so that the adhesive can cure there. While a higher MC content can accelerate heat transfer to the core, it leads to an undesirable increase in vapor pressure in the core (risk of blister), a negative effect on adhesive curing, and negative effects on panels properties (Shalbafan and Thoemen 2022). Researchers also found that MC plays an important role in the quality of CA bonding with lignocellulosic materials (Kusumah *et al.* 2016, Syamani *et al.* 2018). It is reported that the optimum MC value for the surface and core layers of the particleboard mat should not exceed 15 % and 10 %, respectively, as the accumulation of steam leads to a delay in resin curing (Thoemen *et al.* 2010). Therefore, the highest flexural properties of samples with about 11 % MC can be attributed to improved bonding of the carboxylic acid groups of CA with the hydroxyl groups of wheat straw.

When the MC content of the mat was further reduced from about 11 % to about 0,5 % (dried at 100 °C for 14 h), the flexural strength and elastic modulus decreased by 36 % and 28 %, respectively, reaching about 7,5 and 2700 MPa. To further reduce MC, the straw particles were left in the oven at a temperature of about 100 °C for a longer period of time. During this time, pre-curing of the adhesive or even excessive penetration of the adhesive into the straw particles may have occurred, resulting in the formation of weak spots between the straw particles. Of course, it should also be noted that excessive reduction of the mat MC reduces the process of heat transfer to the core during pressing, which can also have a negative effect on the intensity of resin curing (Böhm 2009, Shalbafan and Thoemen 2022).

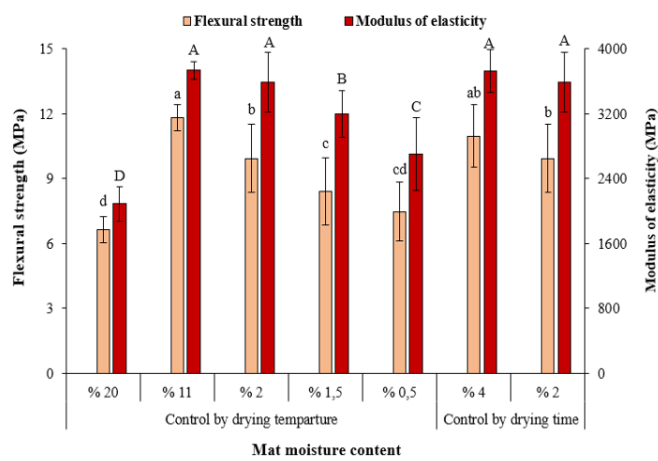


Figure 4: The effect of mat MC on the flexural properties of wheat straw particleboard.

Figure 4 also shows that although the flexural properties of panels made with straw dried at 80 °C (MC of about 4 percent) were slightly higher than those of samples dried at 100 °C (MC of about 1,9 percent), this difference was not statistically significant. In other words, a change in MC in the range of 2 to 4 percent had no significant effect on the flexural properties of the panels.

Internal bond strength (IB)

The effect of mat MC on IB was also studied and the results are shown in Figure 5. The samples prepared with undried straw particles (MC of 20 %) had the lowest IB. Reducing the MC of the particles from about 20 % to 11 % (dried at 2 h and 100 °C) increased the IB by more than 280 %. The carboxyl groups of CA were found to react with the hydroxyl groups of lignocellulosic materials to form ester bonds (Liao *et al.* 2016, Umemura *et al.* 2012, Kusumah *et al.* 2016). The results observed in this study show that the MC of the mat drastically affects the intensity and quality of CA adhesion. Excessive MC content led to an excessive increase in vapor mass in the core layer and negatively affected the adhesive bonds formed (Maloney 1977). Further reduction of MC from about 11 % to 0,5 % (dried at 14 h and 100 °C) reduced the IB values by about 23 %, which could be due to the negative effects on the heat transfer mechanism in the pressing cycle (Thoemen *et al.* 2010). Also, the prevention or excessive penetration of the adhesive into the straw particles during longer drying period seems to affect the IB.

Figure 5 also shows that reducing MC by varying temperatures (80 °C and 100 °C) leads to an increase in IB of about 15 %. It is possible that the CA had better flowability on the surfaces of the particles during the drying process at 100 °C and therefore could form better and more ester bonds.

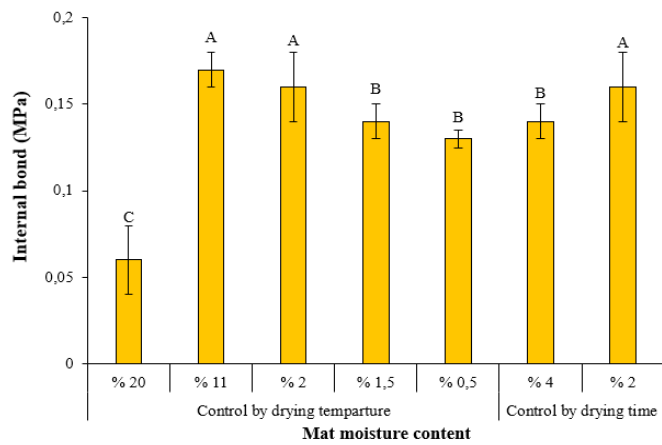


Figure 5: The effect of mat MC on the internal bond strength of wheat straw particleboard.

Physical properties

The effects of mat MC on thickness swelling (TS) after 2 h and 24 h of water immersion are shown in Figure 6. According to the results, the TS of the panels did not change significantly with the change in MC of the mat (by varying the drying temperature and time of the particles). Kusumah *et al.* (2016) observed in their study that pre-drying the resinized particles with CA reduced the TS of the samples, which was not consistent with the results of this study. Of course, in this study, the TS for all samples after 2 and 24 h of immersion in water was less than 6 % and 8 %, respectively, which is much lower than the values for conventional particleboard or for boards made with CA, which were 9,02 % (Thoemen *et al.* 2010, Kusumah *et al.* 2016).

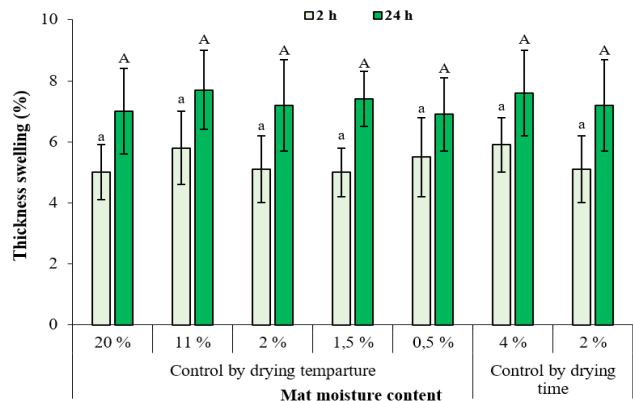


Figure 6: The effect of mat MC on the thickness swelling of wheat straw particleboard.

Figure 7 shows that the WA of the panels increased with decreasing MC of the mat. The lowest WA was observed for the panels made of undried particles having an MC of about 20 %. In this sample, the WA after 2 h and 24 h of immersion was about 17 % and 43 %, respectively. Reducing the MC of the mat to about 0,5 % (drying for 14 h at 100 °C) increased the WA to about 39 % and 56 % after 2 h and 24 h of immersion, respectively. When the MC of the mat was reduced by changing the oven temperature from 80 °C to 100 °C, the WA increased by about 20 % after 24 h of immersion. Previous studies have also shown that deviation from the optimal MC in the mat has a negative effect on the physical properties of the WBP (Böhm 2009, Thoemen *et al.* 2010). The accumulation of vapor in the core layer (high MC of the mat) and the weakening of the heat transfer process in the core (low MC of the mat) were the main factors affecting the intensity of adhesive curing, which also affects the WA of the specimens.

The WA values of the specimens in this study were less than 60 % after 24 h immersion in water, which is much lower than conventional particleboard or board made of wheat straw (Azizi *et al.* 2011, Tabarsa *et al.* 2011, Bekhta *et al.* 2013).

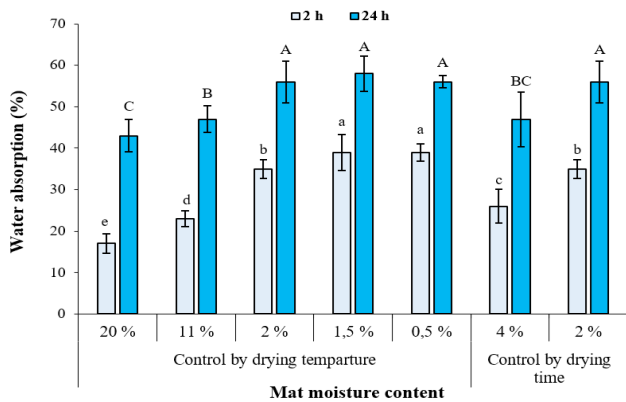


Figure 7: The effect of mat MC on the water absorption of wheat straw particleboard.

Influence of adhesive content

Flexural properties

The effects of adhesive content on the flexural properties of wheat straw particleboard are shown in Figure 8. Increasing the adhesive content from 15 to 30 % increased the flexural strength and modulus of elasticity by 30 % and 40 %, respectively, from 9 and 2385 MPa to 13 and 3934 MPa. The increase in adhesive content resulted in more bonding sites between the carboxylic acid groups of CA and the hydroxyl groups of wheat straw, which had a positive effect on the mechanical properties, especially the flexural properties (Kusumah *et al.* 2016, Lee *et al.* 2020). Further increasing the adhesive content from 20 to 30 percent showed no significant effect on the flexural properties of the panels. Azeredo *et al.* (2015) also found that flexural properties increased with the increase of CA content by up to 20 %, and the further increase had no significant effect on flexural properties. At a fixed board density, a further increase in the adhesive content is associated with a drastic reduction in the consumption of lignocellulosic material in the board. This leads to a reduction in the compressibility ratio of the mat in the panel structure, especially on the panel surfaces, which has a drastic effect on the bending properties of the panel (Khakzad *et al.* 2020, Shalbafan and Thoemen 2022).

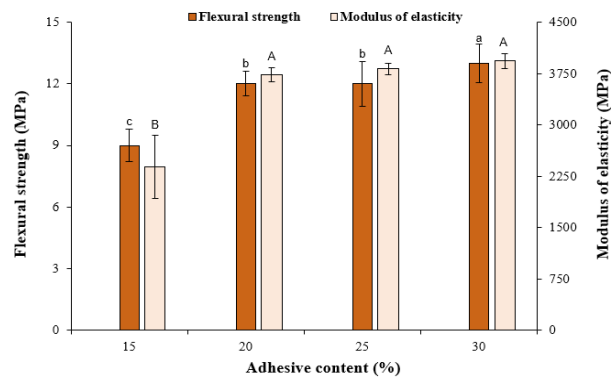


Figure 8: The effect of adhesive content on the flexural properties of wheat straw particleboard.

Internal bond strength

The effect of the different adhesive content on the IB is shown in Figure 9. The lowest IB value was about 0,12 MPa for the sample with 15 % CA, and the highest value was about 0,26 MPa for the sample with 30 % CA. In other words, the IB increased by 217 % when the adhesive content was increased from 15 % to 30 %. The reason for this could be the increased bonding sites between the hydroxyl groups of the straw and the carboxylic acid groups of the CA. In their study, Kusumah *et al.* (2016) also reported the highest IB for panels made of bagasse particles and CA at an adhesive content of 30 %. Increasing the CA content increased the ester bonds formed with bagasse. The formation of ester bonds improved the adhesion of particleboard made with CA (Syamani *et al.* 2018).

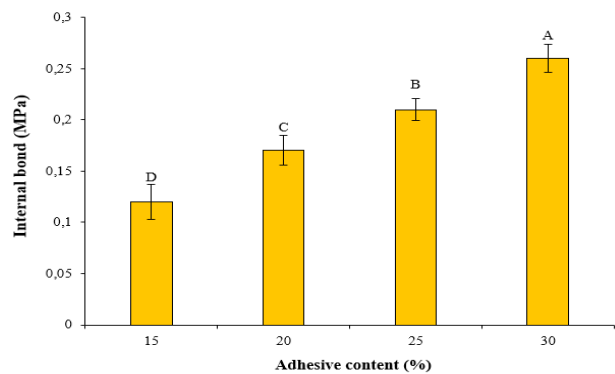


Figure 9: The effect of adhesive content on the internal bond strength of wheat straw particleboard.

Physical properties

The TS decreased significantly when the adhesive content was increased from 15 % to 20 % (Figure 10). The TS after 24 h immersion decreased from 17 % to less than 6,5 % when the adhesive content was increased from 15 % to 20 %. It has long been known that CA improves the dimensional stability of wood and wood products by reacting with the hydroxyl groups of lignocellulosic materials to form ester bonds (Umemura *et al.* 2012, Sutiawana *et al.* 2022). Therefore, the higher the CA content, the lower the TS swelling.

However, further increasing the adhesive content from 20 % to 30 % had no significant effect on the TS, which is important from an economic point of view to reduce adhesive consumption. The reason was probably the lower content of wheat straw on the board structure when a higher content of adhesive was used. Reducing the consumption of lignocellulosic material creates more voids in the board structure, which facilitates the access of water molecules to free hydroxyl groups of the wheat straw (Khakzad *et al.* 2020). In other words, it seems that the positive effect of increasing the adhesive content to improve the reaction with the hydroxyl groups of wheat straw was compensated by the accessibility of more hydroxyl groups to water. Another possible reason is that almost all hydroxyl groups present in the wheat straw were bound with up to 20 % CA and a further increase had no effect on the formation of new bonds. The researchers also found that the binding capacity of the cell wall becomes saturated with increasing amounts of CA and the cell wall molecules are no longer able to form new ester groups (Liao *et al.* 2016).

A similar trend was observed for the WA as for the TS (Figure 11). An increase of the adhesive content from 15 % to 20 % led to a significant decrease of the WA. For the samples with 15 % and 20 % adhesive content, the WA after 24 h of water soaking was 65 % and 47 %, respectively. With increasing adhesive content, more ester bonds appear to have formed between the hydroxyl groups of the straw and the CA, leading to a decrease in WA. Kusumah *et al.* (2016) also reported similar results by increasing CA content to decrease WA. However, the change in WA was not statistically significant when the adhesive content was further increased from 20 % to 30 %. The reason for this, as mentioned earlier, could be the additional reduction in the amount of wheat straw in the panel or even the saturation of all hydroxyl groups in the straw by CA, although more detailed analyses are needed to support this assumption.

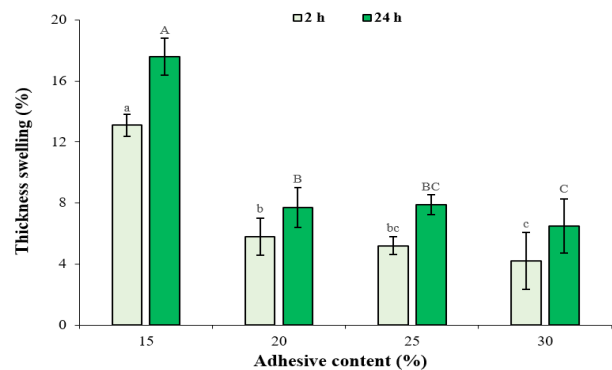


Figure 10: The effect of adhesive content on the thickness swelling of wheat straw particleboard.

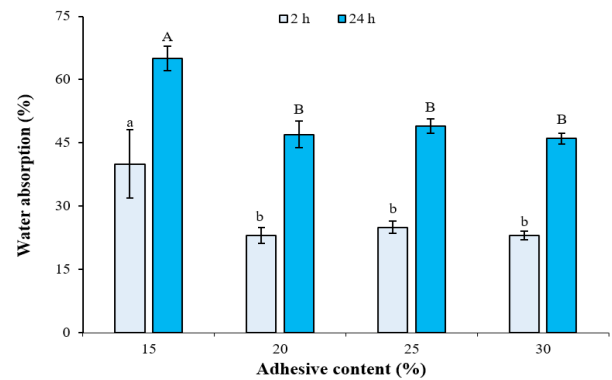


Figure 11: The effect of adhesive content on the water absorption of wheat straw particleboard.

Comparison of the properties with the literature

One of the major challenges in using wheat straw to produce WBP is the inappropriate bonding with urea-formaldehyde adhesive due to the composition and hydrophobic structure of the straw surface. This resulted in an increase in the TS and WA content of the manufactured panels with UF adhesion (Tabarsa *et al.* 2011, Bekhta *et al.* 2013). The reference panel produced in this study with 20 % UF adhesive could not be used for the physical and mechanical tests due to its insufficient adhesive quality and delamination after hot pressing. The comparison of the physical and mechanical properties of the panels produced in this study with the corresponding values from the literature (Table 3) shows that the CA successfully overcame the challenge of the surface structure of the wheat straw. CA could form a bond with the hydroxyl groups in the inner layers of the straw. Compared to other studies on wheat straw particleboard, the lowest percentage of TS and WA after 24 h was obtained in this study. However, optimization of the parameters of the production process still seems to be necessary, especially to increase the IB strength.

Table 3: Comparison of the properties of wheat straw particleboard with CA as an adhesive with the properties of the corresponding panels reported in the literature.

Literature	Adhesive type*	Straw treatment or additive used	Flexural strength (MPa)	Modulus of elasticity (MPa)	Internal bond (MPa)	Thickness swelling (24 h)	Water absorption (24 h)
Han <i>et al.</i> (1998)	UF	Amino silanes	22	-	0,11	40	-
Zhang <i>et al.</i> (2003)	UF	Enzyme treatment	16	-	0,42	8,6	-
Tasooji <i>et al.</i> (2010)	Soybean oil	Ammonium chloride	18	2447	0,55	16	-
Azizi <i>et al.</i> (2011)	UF	Ammonium chloride	27	1463	0,49	24	70
Tabarsa <i>et al.</i> (2011)	PF	Tannin and zin acetate	17	3022	0,70	11	55
Hematabadi and Behrooz, (2012)	UF	Urea treatment	13	4000	0,18	34	93
Bekhta <i>et al.</i> (2013)	UF	Acetic anhydride, steam & boiling in soapy water	13	-	0,30	27	66
Cao <i>et al.</i> (2017)	pMDI	Ammonia	26	2880	0,55	21	-
Current research	CA	-	13	3900	0,26	6,5	46

* The abbreviations stand for urea formaldehyde (UF), phenol formaldehyde (PF), polymeric methyl diphenyl diisocyanate (pMDI), citric acid (CA).

CONCLUSIONS

In this study, wheat straw, an abundant agricultural residue, was successfully used to produce one-layer particleboards using citric acid (CA) as a bio-based adhesive. The results demonstrate that CA can effectively overcome the inherent adhesion limitations of wheat straw without requiring additional pretreatment steps such as thermal, chemical, or enzymatic modification. Owing to its acidic nature, CA is capable of modifying the hydrophobic surface characteristics of wheat straw and forming ester bonds with its hydroxyl groups, which represents the central contribution of this work toward the development of sustainable adhesive systems.

The FTIR analysis confirmed the formation of ester linkages between the carboxyl groups of CA and the hydroxyl groups of wheat straw (peak at 1732 cm⁻¹ in resin-treated straw). The results further showed that mat moisture content (MC) plays a critical role in bonding performance: excessively high MC negatively affected bond formation, while excessively low MC limited heat transfer to the core layer. An optimal MC of approximately 11 % resulted in the best flexural properties and internal bonding.

In addition, a drying temperature of 100 °C was more favorable than 80 °C, likely due to improved flowability and distribution of CA on the straw surface. Increasing adhesive content generally enhanced bonding and improved the physical and mechanical properties of the panels; however, increasing CA content from 20 % to 30 % did not lead to further improvements. This may be attributed to a reduced compressibility ratio of the mat and potential saturation of available hydroxyl bonding sites in the straw.

Overall, the findings highlight the potential of citric acid as a sustainable adhesive for manufacturing particleboards from agricultural residues such as wheat straw. The study also emphasizes the importance of optimizing key processing parameters - namely moisture content, drying conditions, and adhesive loading - to fully realize the bonding capability of this bio-based system.

Authorship contributions

A. Kh.: Methodology; Investigation; Experimental work; Data curation; Formal analysis; Writing – original draft preparation. A. Sh.: Conceptualization; Methodology; Validation; Supervision; Project administration; Funding acquisition; Writing – review & editing. S. K-N.: Methodology; Scientific consultation; Validation; Advisor; Writing – review & editing; Resources.

Acknowledgements

This project was funded by the Tarbiat Modares University, Research and Development Office under project entitled “Evaluation of particle board made from wheat straw using citric acid as adhesive” with the number of 1738903.

Conflict interest

The authors declare no conflicts of interest. The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

REFERENCES

- Adefris, L. A.; Desalegn, D.; Selvaraj, S. K.; Paramasivam, V.; Chadha, U. 2022.** Experimental investigation of sorghum stalk and sugarcane bagasse hybrid composite for particleboard. *Advances in Materials Science and Engineering* 17 (1): 1844004. <https://doi.org/10.1155/2022/1844004>
- Asghar, U.; Irfan, M.; Iram, M.; Huma, Z.; Nelofer, R.; Nadeem, M.; Syed, Q. 2015.** Effect of alkaline pretreatment on delignification of wheat straw. *Natural Product Research* 29(2): 125–131. <https://doi.org/10.1080/14786419.2014.964712>
- ASTM International. 2019.** Standard test method for pH of aqueous solutions with the glass electrode. ASTM E70-19. ASTM International: West Conshohocken, PA, USA.
- Ando, D.; Umemura, K. 2021.** Bond structures between wood components and citric acid in wood-based molding. *Polymers* 13(1). e58. <https://doi.org/10.3390/polym13010058>
- Azeredo, H.M.C.; Kontou-Vrettou, C.; Moates, G.K.; Wellner, N.; Cross, K.; Pereira, P.H.F.; Waldron, K.W. 2015.** Wheat straw hemicellulose films as affected by citric acid. *Food Hydrocolloids* 50: 1–6. <https://doi.org/10.1016/j.foodhyd.2015.04.005>
- Azizi, K.; Tabarsa, T.; Ashori, A. 2011.** Performance characterizations of particleboards made with wheat straw and waste veneer splinters. *Composites Part B: Engineering* 42(7): 2085–2089. <https://doi.org/10.1016/j.compositesb.2011.04.002>
- Beygi Heidarlou, H.; Banj Shafiei, A.; Nasiri, V.; Niță, M.D.; Borz, S.A.; Lopez-Carr, D. 2023.** Impact of Iran's forest nationalization law on forest cover changes over six decades: A case study of a Zagros sparse coppice Oak Forest. *Sensors* 23(2). e871. <https://doi.org/10.3390/s23020871>
- Böhm, M. 2009.** The influence of moisture content on thickness swelling and modulus of elasticity in oriented strand board bending. *Wood Research* 54(4): 79–90. <http://www.woodresearch.sk/wr/200904/08.pdf>

- Boquillon, N.; Elbez, G.; Schonfeld, U. 2004.** Properties of wheat straw particle boards bonded with different types of resin. *Journal of Wood Science* 50: 230–235. <https://doi.org/10.1007/s10086-003-0551-9>
- Bekhta, P.; Korkut, S.; Hiziroglu, S. 2013.** Effect of pretreatment of raw material on properties of particleboard panels made from wheat straw. *BioResources* 8(3): 4766–4774. <https://doi.org/10.15376/biores.8.3.4766-4774>
- Cao, Y.; Song, W.; Yang, Z.; Chen, Z.; Zhang, S. 2017.** The properties of particleboard made from alkaline-treated wheat straw and methylene diphenyl diisocyanate binder. *BioResources* 12(2): 3265–3276. <https://doi.org/10.15376/biores.12.2.3265-3276>
- De Melo, R.R.; Stangerlin, D.M.; Campomanes Santana, R.R.; Pedrosa, T.D. 2015.** Decay and termite resistance of particleboard manufactured from wood, bamboo and rice husk. *Maderas. Ciencia y Tecnología* 17(1): 55–62. <https://doi.org/10.4067/S0718-221X2015005000006>
- Deutsche Institute fur Normung – German Institute for Standardization. 1987.** Determination of flow time using the DIN flow cup, DIN 53211 (Cup No. 4 mm). Lacke, Anstrichstoffe und ähnliche Beschichtungsstoffe; Bestimmung der Auslaufzeit mit dem DIN-Becher
- European Committee for Standardization. 1993.** Wood-based panels – Determination of moisture content. EN 322. European Committee for Standardization: Brussels, Belgium.
- European Committee for Standardization. 1993.** Wood-based panels – Determination of modulus of elasticity in bending and of bending strength. EN 310. European Committee for Standardization: Brussels, Belgium.
- European Committee for Standardization. 1993.** Particleboards and fibreboards – Determination of swelling in thickness after immersion in water. EN 317. European Committee for Standardization: Brussels, Belgium.
- European Committee for Standardization. 1993.** Particleboards and fibreboards – Determination of tensile strength perpendicular to the plane of the board. EN 319. European Committee for Standardization: Brussels, Belgium.
- Food and Agriculture Organization. 2023.** Data for forestry production and trade. FAOSTAT. United Nations. <https://www.fao.org/faostat/en/#data/FO>.
- Goheen, S.M.; Wool, R.P. 1991.** Degradation of polyethylene-starch blends in soil. *Journal of Applied Polymer Science* 42(10): 2691–2701. <https://doi.org/10.1002/app.1991.070421007>
- Halligan, A.F.; Schniewind, A.P. 1974.** Prediction of particleboard mechanical properties at various moisture contents. *Wood Science and Technology* 8(1): 68–78. <https://doi.org/10.1007/BF00350644>
- Harlvarsson, S.; Edlund, H.; Norgren, M. 2009.** Manufacture of non-resin wheat straw fiberboards. *Industrial Crops and Products* 29(2–3): 437–445. <https://doi.org/10.1016/j.indcrop.2008.08.007>
- Han, G.; Zhang, C.; Zhang, D.; Umemura, K.; Kawai, S. 1998.** Upgrading of urea formaldehyde-bonded reed and wheat straw particleboards using silane coupling agents. *Journal of Wood Science* 44: 282–286. <https://doi.org/10.1007/BF00581308>
- Hafezi, S.M.; Enayati, A.; Hosseini, K.D.; Tarmian, A.; Mirshokraii, S.A. 2016.** Use of amino silane coupling agent to improve physical and mechanical properties of UF-bonded wheat straw (*Triticum aestivum* L.) poplar wood particleboard. *Journal of Forestry Research* 27(2): 427–431. <https://doi.org/10.1007/s11676-015-0135-8>
- Hematabadi, H.; Behrooz, R. 2012.** The effect of urea pretreatment on the formaldehyde emission and properties of straw particleboard. *Journal of Forestry Research* 23: 497–502. <https://doi.org/10.1007/s11676-012-0290-0>
- Hýsek, Š.; Podlena, M.; Böhm, M.; Bartsch, H.; Wenderdel, C. 2018.** Effect of cold plasma surface pre-treatment of wheat straw particles on straw board properties. *BioResources* 13(3): 5065–5079. <https://doi.org/10.15376/biores.13.3.5065-5079>

Li, X.; Cai, Z.; Winandy, J. E.; Basta, A. H. 2010. Selected properties of particleboard panels manufactured from rice straws of different geometries. *Bioresource Technology* 101(12): 4662-4666. <https://doi.org/10.1016/j.biortech.2010.01.053>

International Organization for Standardization. 2023. Paints and varnishes – Determination of density – Part 1: Pycnometer method. ISO 2811-1. 2023. International Organization for Standardization: Geneva, Switzerland.

Khakzad, J.; Shalbafan, A.; Kazemi-Najafi, S. 2020. Lightweight tubular fiberboard: effect of hole diameters and number on panel properties. *Maderas. Ciencia y Tecnología* 22(3): 311–324. <https://doi.org/10.4067/S0718-221X2020005000306>

Kusumah, S.S.; Umemura, K.; Yoshioka, K.; Miyafuji, H.; Kanayama, K. 2016. Utilization of sweet sorghum bagasse and citric acid for manufacturing of particleboard I: Effects of pre-drying treatment and citric acid content on the board properties. *Industrial Crops and Products* 84: 34–42. <https://doi.org/10.1016/j.indcrop.2016.01.042>

Lee, C.J.; Na, J.H.; Park, J.Y.; Chang, P.S. 2019. Structural characteristics and in vitro digestibility of malic acid-treated corn starch with different pH conditions. *Molecules* 24(10). <https://doi.org/10.3390/molecules24101900>

Lee, S.H.; Md Tahir, P.; Lum, W.C.; Tan, L.P.; Bawon, P.; Park, B.D.; Osman Al Edrus, S.S.; Abdullah, U.H. 2020. A review on citric acid as green modifying agent and binder for wood. *Polymers* 12(8). e1692. <https://doi.org/10.3390/polym12081692>

Liao, R.; Xu, J.; Umemura, K. 2016. Low density sugarcane bagasse particleboard bonded with citric acid and sucrose: Effect of board density and additive content. *BioResources* 11(1): 2174–2185. <https://doi.org/10.15376/biores.11.1.2174-2185>

Menezzi, C.; Amirou, S.; Pizzi, A.; Xi, X.; Delmotte, L. 2018. Reactions with wood carbohydrates and lignin of citric acid as a bond promoter of wood veneer panels. *Polymers* 10(8). e833. <https://doi.org/10.3390/polym10080833>

Nazerian, M.; Beyki, Z.; Gargarii, R.M.; Kool, F. 2016. The effect of some technological production variables on mechanical and physical properties of particleboard manufactured from cotton (*Gossypium hirsutum*) stalks. *Maderas. Ciencia y Tecnología* 18(1): 167–178. <https://doi.org/10.4067/S0718-221X2016005000017>

Neitzel, N.; Hosseinpourpia, R.; Walther, T.; Adamopoulos, S. 2022. Alternative materials from agro-industry for wood panel manufacturing - A review. *Materials* 15(13). e4542. <https://doi.org/10.3390/ma15134542>

Shalbafan, A.; Rhême, M.; Thoemen, H. 2017. Ultra-light particleboard: characterization of foam core layer by digital image correlation. *European Journal of Wood and Wood Products* 75: 43–45. <https://doi.org/10.1007/s00107-016-1088-0>

Shalbafan, A.; Welling, J.; Hasch, J. 2017. Effect of aluminosilicate powders on the applicability of innovative geopolymer binders for wood-based composites. *European Journal of Wood and Wood Products* 75: 893–902. <https://doi.org/10.1007/s00107-017-1172-0>

Shalbafan, A.; Thoemen, H. 2022. Influence of pressing schedule and adhesive content on the rheological behavior of wood fiber-furnish mats. *Materials* 15(4). e1413. <https://doi.org/10.3390/ma15041413>

Sutiawan, J.; Hermawan, D.; Massijaya, M.Y.; Kusumah, S.S.; Lubis, M.A.R.; Marlina, R.; Purnomo, D.; Sulastiningsih, I.M. 2022. Influence of different hot-pressing conditions on the performance of eco-friendly jabon plywood bonded with citric acid adhesive. *Wood Material Science and Engineering* 17(6): 400–409. <https://doi.org/10.1080/17480272.2021.1884898>

Syamani, A.; Kusumah, S.; Astari, L.; Prasetyo, W.; Wibowo, E.S.; Subyakto, E.S. 2018. Effect of pre-drying time and citric acid content on *Imperata cylindrica* particleboards properties. *IOP Conference Series: Earth and Environmental Science* 209(1). e012034. <https://doi.org/10.1088/1755-1315/209/1/012034>

Tabarsa, T.; Jahanshahi, S.; Ashori, A. 2011. Mechanical and physical properties of wheat straw boards bonded with a tannin modified phenol-formaldehyde adhesive. *Composites Part B: Engineering* 42(2): 176–180. <https://doi.org/10.1016/j.compositesb.2010.09.012>

Tasooji, M.; Tabarsa, T.; Khazaeian, A.; Wool, R.P. 2010. Acrylated epoxidized soy oil as an alternative to urea-formaldehyde in making wheat straw particleboards. *Journal of Adhesion Science and Technology* 24(8–10): 1717–1727. <https://doi.org/10.1163/016942410X507786>

Thoemen, H.; Irle, M.; Sernek, M. 2010. Wood-based Panels: An Introduction for Specialists. Brunel University Press: London, UK. ISBN 978-1-902316-82-6. 152 pp.

Maloney, T. M. 1977. Modern particleboard & dry-process fiberboard manufacturing. San Francisco: USA, Miller Freeman Publications. ISBN 0-87930-063-9. 672 pp.

Umemura, K.; Ueda, T.; Munawar, S.; Kawai, S. 2012. Application of citric acid as natural adhesive for wood. *Journal of Applied Polymer Science* 123(4): 1991–1996. <https://doi.org/10.1002/app.34708>

Widyorini, R.; Umemura, K.; Isnain, R.; Putra, D.R.; Awaludin, A.; Prayitno, T.A. 2016. Manufacture and properties of citric acid-bonded particleboard made from bamboo materials. *European Journal of Wood and Wood Products* 74(1): 57–65. <https://doi.org/10.1007/s00107-015-0967-0>

Zakaria, R.; Bawon, P.; Lee, S.H.; Salim, S.; Lum, W.C.; Al-Edrus, S.S.O.; Ibrahim, Z. 2021. Properties of particleboard from oil palm biomasses bonded with citric acid and tapioca starch. *Polymers* 13(20). e3494. <https://doi.org/10.3390/polym13203494>

Zhang, Y.; Lu, X.; Pizzi, A.; Delmotte, L. 2003. Wheat straw particleboard bonding improvements by enzyme pretreatment. *European Journal of Wood and Wood Products* 61(1): 49–54. <https://doi.org/10.1007/s00107-002-0349-2>