Dimensional Stability and Mechanical Properties of Bio-based Composites Produced from Hydro-thermal Treated Wheat Straw

Ümit Büyüksarı [a,*] and Ömer Özyürek [b]

Bio-composites were produced from untreated (UT) and hydro-thermally treated (HTT) wheat straw (WS) particles and wood, and their dimensional stability and mechanical properties were investigated. The HTT treatment consisted of subjecting the WS particles to a steam explosion process for 8 min at 180 °C. The HTT and UT WS particles were mixed with the wood particles at 10, 20, 30, and 40% ratios. The physical properties, including density, water absorption (WA), and thickness swelling (TS), were determined for the bio-based composites. The mechanical properties evaluated included the modulus of rupture, modulus of elasticity, and internal bond strength. Statistical analyses showed that the hydro-thermal treatment and the WS ratio had significant effects on the dimensional stability and mechanical properties of the bio-composites. The WA of the composites after 2-h and 24-h rose significantly when the HTT WS particle ratio was increased from 10 to 40%. The 2-h and 24-h WA values of HTT-10 were 6.3% and 5.3% lower than those of UT-10, respectively. Improvements in the 2-h TS value were achieved by the HTT WS particles at the 10% ratio, and in the 24-h TS value at the 10 and 40% ratios. The mechanical properties of the composites were higher in the HTT group, but decreased in both the UT and HTT groups as the WS ratio increased.

Keywords: Hydro-thermal treatment; Composite; Wheat stalks; Dimensional stability; Mechanical properties

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INTRODUCTION

The manufacture of bio-based composites such as fiberboard and particleboard is gradually increasing. Consequently, the pressure on forest sources is rising, necessitating the search for other renewable resources. Using crop residues to produce bio-based composites has become both economically and environmentally attractive (Mantanis *et al.* 2000). As a renewable material, wheat straw (WS) is plentifully available worldwide as the principal by-product of the wheat harvest (Markessini *et al.* 1997). The various applications for WS include its use as fuel, animal feed, garden mulch, and stable bedding (Sampathrajan *et al.* 1992). Turkey produces WS at a rate of roughly 27 million tons per year. Compared with wood, WS has a more complex morphology. In addition to the actual fibers, it is comprised of many elements, including parenchyma cells and vessels, and large amounts of ash and silica are found in the epidermal cells (Markessini *et al.* 1997; Hafezi and Hosseini 2014). The chemical composition of the WS is similar to wood but its structure is looser and its strength is lower. It contains less lignocellulose cells and more ash and extractive with lower molecular weights than wood (Ghaffar *et al*. 2017a).

Crop residues can serve as excellent substitutes for wood because of their abundance and general availability (Çöpür *et al*. 2007). The use of agricultural residues in particleboard production is both environmentally and socio-economically advantageous (Rowell 1995). A great body of research is available on the application of agricultural residues in the manufacture of bio-based composites such as fiberboard, particleboard, and wood-plastic composites (Turreda 1983; Kalaycıoğlu 1992; Yalınkılıç *et al.* 1998; Grigoriou *et al*. 2000; Nemli *et al.* 2001; Bektaş *et al*. 2002; Nemli *et al*. 2003; Mo *et al*. 2003; Cheng *et al*. 2004; Güler and Özen 2004; Bektaş *et al*. 2005; Güler *et al*. 2006; Çöpür *et al*. 2007; Güler *et al*. 2008; Azizi *et al.* 2011; Ayrılmış *et al*. 2013; Kaymakcı and Ayrılmış 2014; Ahmad *et al.* 2017; Sitz *et al.* 2017; Chougan *et al.* 2020; Hýsková *et al.* 2020; Shang *et al.* 2020; Tupciauskas *et al.* 2021). Most of these studies have found that these composite panels complied with the standard requirements in terms of their mechanical properties. However, their physical properties were sub-standard, as composites made from agricultural resides generally exhibit higher thickness swelling (TS) and water absorption (WA) compared to composites made from wood. The poor water resistance of these composites is the main reason limiting their use in the industry. Mo *et al.* (2003) stated that WS particleboards bonded using urea formaldehyde (UF) yielded WA and TS values of 107.2 and 56.5% for 2-h and 125.3 and 63.9% for 24-h immersion times, respectively. Kalaycıoğlu (1992) reported that particleboards manufactured from tobacco stalks and tea factory wastes yielded WA and TS values ranging from 60 to 71% and from 22 to 37%, respectively. Güler (2001) also determined that the 24-h TS values of particleboards produced from cotton stalks varied between 18.1 and 35%. Likewise, Bektaş *et al.* (2002) and Nemli *et al.* (2003), respectively, reported that for sunflower stalk and kiwi pruning-based particleboards, the values of WA and TS were higher.

Heat treatment is among the most effective methods of enhancing the dimensional stability of wood and wood-based panels. One main disadvantage of heat treatment is the loss of strength. Hydro-thermal treatment (HTT) is more protective than heat treatment because of the presence of moisture together with temperature. When wood is thermally treated in a moist environment, initially, the cleaving of particular hemicellulose acetyl groups leads to formation of carbonic acids, and in particular acetic acid (Navi and Sandberg 2012). The decrease in free hydroxyl groups resulting from dehydration causes a reduction in the moisture uptake, which is also affected by hydrophobic elements formed by reactions of the wood polymer cross-linking (Tjeerdsma and Militz 2005).

Because of its thin wax layer and high ash and silica content, WS is not a suitable raw material for composite production. These factors prevent WS from absorbing moisture from water-based adhesives and affect its bondability (Markessini *et al.* 1997; Hafezi and Hosseini 2014). Several studies investigating the properties of WS-wood composites have reported attempts at improving them by applying various treatments such as using acetic anhydride, soap solutions, hot water, and steam applications, as well as boiling in soap solution, treating with ethanol-benzene, adding ethanol to UF, and using silane as a coupling agent (Han *et al.* 2001; Bekhta *et al*. 2013; Ghaffar *et al*. 2017a,b; Bekhta *et al.* 2018). Some of these improved the physical and mechanical properties of particleboards containing WS particles. Xu *et al*. (2009) stated that the three fundamental techniques for improving bio-based composite dimensional stability were steaming, acetylation, and wax-sizing. They found that, in contrast to untreated control boards, the use of wax significantly improved 24-h WA and TS, but negatively affected internal bond strength of MDI-bonded bagasse particleboards. Han *et al.* (2010) studied the temperature of the steam and duration of the retention period in terms of their effects on WS morphology, wettability, acidity, and silicon and ash content and concluded that post steam explosion, the WS acidity had increased, whereas the silicon and ash levels, and straw contact angle had been reduced significantly. Ghaffar *et al.* (2017a) employed a pre-treatment with hot water and steam combination to understand the bonding and failure mechanisms of different anatomical sections (node and internode) of the WS. They concluded that the pre-treatment modified the surface of the WS with the removal of extractives, waxes, and silica. Similarly, another study Ghaffar *et al.* (2017b) determined that pre-treatment diminished extractives, waxes, and silica (weight %) content of the WS.

This study examined the dimensional stability and mechanical properties of bio-based composites fabricated from untreated (UT) WS and from WS that had been treated hydro-thermally (HTT). The ultimate goal was to promote the increased use of WS in the manufacture of bio-based composites.

EXPERIMENTAL

Materials

The raw materials used in this study included WS and industrial wood particles. The WS was harvested from the Düzce region and the wood particles were obtained from the Kastamonu Entegre Company in Kocaeli, Turkey. The WS was coarsely chopped and horizontal vibrating sieves were then used to classify the WS particles. The particles remaining in the 3 to 1.5 mm sieve were used in forming the core, and those remaining in the 1.5 to 0.8 mm sieve were used in the middle layer. The HTT was applied to the WS particles in a steam explosion machine for 8 min at 180 °C. The wood particles and WS were mixed at different ratios, placed in an oven, and dried at 100 ºC to 3% oven dry (o.d.), as the target moisture content. The UF resin (65% solid content) was utilized at adhesive levels of 8 and 10% (based on the o.d. weight of the particles) in the core and outer layers, respectively, with the addition of 1% ammonium chloride (NH_4Cl) as a hardener for the UF resin. The aim was to achieve a density of 0.600 g/cm^3. The panels were formed in a hot press under laboratory conditions (Fig. 1) and conditioned in a climate-controlled cabinet for a three-week period before testing. The design of the experiments and production parameters are shown in Tables 1 and 2, respectively.

(a) (b)

Fig. 1. (a) Composites produced from HTT WS and UT WS and wood; (b) trimmed composites

Table 1. Experimental Design

Board Type	Treatment	Wheat Straw Ratio (%)	Wood Particle Ratio (%)
Control	Untreated	0	100
UT-10	Untreated	10	90
HTT-10	Hydro-thermally treated	10	90
UT-20	Untreated	20	80
HTT-20	Hydro-thermally treated	20	80
UT-30	Untreated	30	70
HTT-30	Hydro-thermally treated	30	70
UT-40	Untreated	40	60
HTT-40	Hydro-thermally treated	40	60

Table 2. Production Parameters

Parameter	**Value**
Press temperature (°C)	150
Pressing time (min)	7
Peak pressure (MPa)	2.6
Thickness (mm)	18
Dimensions (mm)	500×500
Outer layer (% of whole board)	35
Middle layer (% of whole board)	65
Number of boards for each type	2

Methods

Some physical properties including density (EN 323 1993), water absorption (WA), and thickness swelling (TS) (EN 317 1993) were investigated. In addition, the mechanical properties determined for the produced panels included modulus of rupture (MOR) (EN 310 1993), modulus of elasticity (MOE) (EN 310 1993), and internal bond (IB) (EN 319 1993) strength. The 3-point bending test setup and a fractured sample are shown in Fig. 2. It was conducted at a span-to-depth ratio of 20:1. The crosshead speed was adjusted so that fracture would happen within an average of 60 s±10 s. The averages were taken of 20 samples for physical and 10 for mechanical properties.

(a) **(b)**

Fig. 2. (a) Bending test setup; (b) Fracture of bending sample

Analysis of variance (ANOVA) at $p<0.05$ was first applied to all comparisons for these properties. Duncan's multiple-range test was then used to determine the significant differences between the mean values of the groups.

RESULTS AND DISCUSSION

Figure 3 indicates the ANOVA and Duncan's test results for the density of the composites. No statistically significant difference ($p<0.05$) in the density values was found among the groups. Although a density value of 0.600 g/cm^3 was the goal, the average density values achieved ranged from 0.593 to 0.621 g/cm^3.

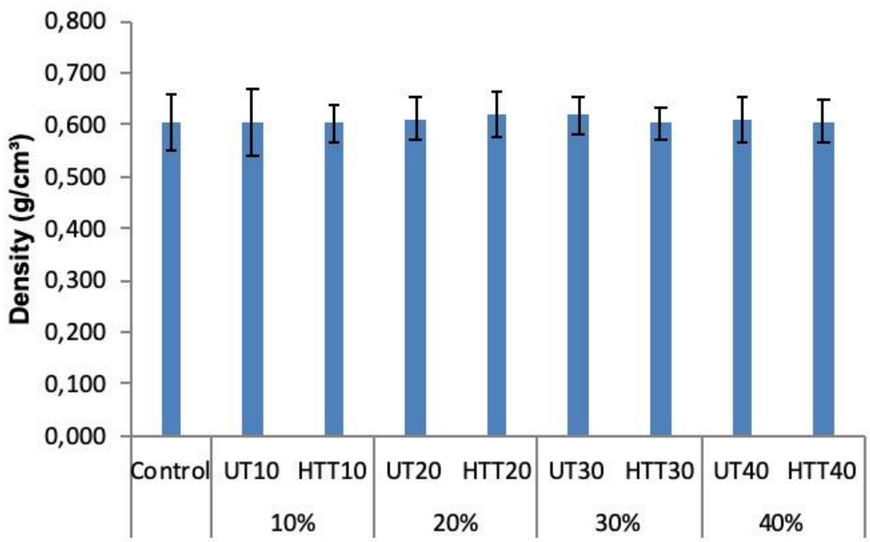

Fig. 3. Average density values of the produced composites

The WA values for 2 and 24 h and Duncan's test results for the produced panels are given in Table 3. The lowest WA after 2 h of submersion in water (53.1%) was found for the UT-20 group containing 20% untreated WS particles, and the highest (84.3%) was found for the HTT-40 group containing 40% HTT WS particles. The WA of the composites after 2 h rose significantly when the HTT WS particle ratio was increased from 10 to 40%. The 2-h WA value of HTT-10 was 6.3% lower than that of UT-10. However, this difference was not statistically significant. The average 2-h WA value of the composites containing 20% UT WS decreased by 19.4% in comparison with the 100% wood particle control panels; however, with additional amounts of the UT WS particles, the 2-h WA values increased. Hafezi and Hosseini (2014) reported that the WS ratio in particleboard significantly affected the 2-h WA value, and Mo *et al*. (2003) found that the 2-h WA value of WS particleboard bonded using UF was 107.2%.

In the case of the 24-h WA values, the control group containing 100% wood had the lowest, whereas the UT-40 (40% UT WS) exhibited the highest WA. Bekhta *et al*. (2013) reported the value for WA as 71.6% with wood-WS particleboard produced at a ratio of 60:40. In this study, the findings for the UT-40 group were 98.5%. This difference may have arisen from the use of different wood species and parameters for board production in the studies. Mo *et al.* (2003) found the 24-h WA value of WS particleboard bonded with UF as 125.3%. It was stated that the particleboard produced from the WS had higher 24-h WA value than that of particleboard containing 15% poplar wood and 85% WS (Hafezi and Hosseini 2014). The 24-h WA values of the boards increased as the HTT WS ratio was increased from 10 to 40%. However, the 24-h WA values of the HTT-10 and HTT-40 groups were 5.3% and 3.9% lower compared to the UT-10 and UT-40 groups, respectively.

Table 3. Average 2-h and 24-h WA Values and Duncan's Mean Separation Test Results of the Manufactured Panels

Immersion Time	Panel Type	N	X (%)	SD	SE	X_{min} (%)	X_{max} (%)	C_v (%)
2 h	Control	20	65.9 B	10.6	2.4	49.1	88.1	10.6
	UT-10	19	78.4 CDE	8.7	2.0	66.2	91.3	8.7
	HTT-10	20	73.5 C	11.8	2.6	51.6	91.2	11.8
	UT-20	20	53.1 A	9.8	2.2	30.4	71.1	9.8
	HTT-20	20	76.1 CD	6.8	1.5	64.7	87.8	6.8
	UT-30	18	67.5 B	8.9	2.1	55.4	87.9	8.9
	HTT-30	15	83.1 E	4.0	1.0	79.0	94.5	4.0
	UT-40	20	81.4 DE	8.5	1.9	69.5	94.8	8.5
	HTT-40	19	84.3 E	7.5	1.7	75.9	99.0	7.5
24 h	Control	20	77.7 A	8.4	1.9	64.4	95.4	10.8
	UT-10	19	87.3 DE	5.7	1.3	79.4	95.9	6.5
	HTT-10	20	82.7 BC	8.7	1.9	70.3	97.1	10.5
	UT-20	20	80.5 AB	6.9	1.5	69.1	93.7	8.6
	HTT-20	20	86.0 CD	5.5	1.2	77.5	95.9	6.3
	UT-30	18	91.3 EF	5.5	1.3	82.1	101.5	6.0
	HTT-30	15	92.1 F	3.9	1.0	86.9	102.8	4.2
	UT-40	20	98.5 G	7.8	1.7	84.3	112.2	7.9
	HTT-40	19	94.7 FG	6.1	1.4	87.9	105.5	6.5

N: number of specimens, X: average, SD: standard deviation, SE: standard error, X_{min}: minimum value, X_{max}: maximum value, C_V: coefficient of variation. Groups with identical capital letters in a column for each immersion time indicate that there is no statistical difference (p< 0.05) between the samples according to Duncan's multiple-range test.

By applying HTT to WS particles, improvements were achieved in the 2-h WA value for the 10% WS ratio, and in the 24-h WA values for the 10% and 40% WS ratios. Similar improvements were also observed by Bekhta *et al*. (2013) in the 24-h WA value of particleboards containing 40% WS particles. Their study showed that, compared to particleboard containing untreated straw particles, the particleboards exhibited a 24-h WA value lower by 9.8% when soaked in acetic anhydride solution, by 8.3% when boiled in soapy solution, by 4.8% when boiled in water, and by 11.5% when steamed at 100 °C. In addition, the use of wax was shown to significantly improve the 24-h WA value of MDI-bonded bagasse particleboards compared to control boards without wax (Xu *et al.* 2009).

Table 4 presents the TS of the particleboards after 2 and 24 h and results of the Duncan's test. The lowest 2-h TS value (10.3%) was found for the control group produced from 100% wood particles and the highest (23.4%) for the HTT-30 group (30% HTT WS particles). The 2-h WA values were increased by adding WS particles to the boards. This could have been related to the inherent characteristics of the WS and industrial wood particles. In a previous study, Hafezi and Hosseini (2014) found that the 2-h TS values of particleboards produced from WS and poplar wood ranged from 24 to 58% and that the particleboard containing 45% poplar wood yielded a higher value compared to particleboard of 100% WS. The TS value of WS particleboard has also been determined as 56.5% for 2-h immersion (Mo *et al.* 2003).

In the HTT groups, the 2-h TS value of the particleboards increased when the HTT WS particle ratio was increased from 10 to 40%. In the UT groups, the UT-20 group had the lowest 2-h TS value, whereas the UT-40 had the highest.

Table 4. Average 2-h and 24-h TS Values and Duncan's Mean Separation Test Results of the Manufactured Panels

Immersion Time	Panel Type	N	X (%)	SD	SE	X_{min} (%)	X_{max} (%)	C_v (%)
2 h	Control	20	10.3 A	0.9	0.2	9.2	12.5	8.4
	UT-10	19	14.3 B	0.9	0.2	12.4	16.2	6.6
	HTT-10	20	14.1 B	1.6	0.4	11.2	17.8	11.4
	UT-20	20	10.4 A	1.2	0.3	8.5	12.8	11.8
	HTT-20	20	18.1 C	1.2	0.3	15.7	20.2	6.9
	UT-30	18	17.5 C	2.0	0.5	14.5	24.0	11.5
	HTT-30	15	23.4 E	2.3	0.6	17.9	27.0	10.0
	UT-40	20	22.1 D	1.5	0.3	19.7	25.2	6.6
	HTT-40	19	22.5 DE	2.1	0.5	18.0	26.2	9.3
24 h	Control	20	14.5 A	1.6	0.4	11.9	17.3	10.9
	UT-10	19	20.5 BC	1.9	0.4	16.2	23.7	9.1
	HTT-10	20	19.6 B	3.0	0.7	14.2	24.0	15.5
	UT-20	20	21.6 C	1.4	0.3	18.5	23.8	6.6
	HTT-20	20	24.0 D	2.0	0.4	20.9	27.7	8.3
	UT-30	18	27.5 E	2.6	0.6	23.6	34.3	9.3
	HTT-30	15	30.0 F	3.5	0.9	23.5	35.0	11.8
	UT-40	20	32.7 G	1.9	0.4	29.5	36.0	5.8
	HTT-40	19	29.3 F	2.8	0.6	24.3	34.4	9.6

N: number of specimens, X: average, SD: standard deviation, SE: standard error, X_{min}: minimum value, X_{max}: maximum value, CV: coefficient of variation. Groups with identical capital letters in a column indicate that there is no statistical difference ($p < 0.05$) between the samples according to Duncan's multiple-range test.

The control group had the lowest 24-h TS values and the UT-40 group containing 40% UT WS particles had the highest. In the UT groups, the 24-h TS values of the particleboards increased as the WS particle ratio was increased from 10 to 40%. Bekhta *et al.* (2013) found a TS value of 40.9% for particleboard produced from 60% wood and 40% WS particles. Hafezi and Hosseini (2014) observed an increase in the 24-h TS value with increasing percentage of poplar particles and found that the boards with 45% poplar and 55% WS particles had the highest 24-h TS value. The 24-h TS value of WS particleboard was found as 63.9% by Mo *et al.* (2003).

Improvements in the 2-h TS value were achieved by the HTT WS particles at the 10% ratio, and in the 24-h TS value at the 10 and 40% ratios. Similar improvements in 24-

h TS values were also observed by Bekhta *et al.* (2013) in boards containing 40% WS particles and by Xu *et al*. (2019) via the use of wax, as previously mentioned above.

The HTT of the WS particles can have a negative effect on the WA and TS values of the particleboards because of the removal of the thin, waxy layer of the epidermal cells, silica, and non-polar extractives. However, removing this layer of silica and non-polar extractives leads to an increased interaction between the resins and WS particles. The thin waxy layer covering the WS epidermal cells contains silica and ash in high amounts (Markessini *et al.* 1997). Previous studies have stated that this waxy layer prevented absorption of water-based formaldehyde resins (Markessini *et al.* 1997; Hafezi and Hosseini 2014; Yasin *et al.* 2010). Han *et al.* (2010) increased the wettability and acidity of WS and reduced its silica content by applying a steam explosion treatment. Han *et al.* (1998) also stated that the wettability of WS was increased by treatment with ethanol/benzene to remove the waxy layer and non-polar extractives from its surface.

Figure 4 gives the average MOR values and standard deviation of the produced particleboards. The mean MOR values of the particleboards ranged from 12.6 MPa for the HTT-10 group to 5.3 MPa for the UT-40 group. The MOR value of the control group was 11.8 MPa, whereas the HTT-10 group had a 6.8% higher MOR value and the other groups had lower MOR values. Similar low MOR values have been determined in previous studies (Boquillon *et al.* 2004; Bekhta *et al.* 2013). Boquillon *et al*. (2004) found a MOR value of 3 MPa for straw panels bonded with UF and Bekhta *et al.* (2013) determined the MOR value of particleboard containing 40% WS particles as 5.58%. This value is very close to the present result for particleboard containing 40% UT WS. The EN 312 (2010) standard suggests a minimum MOR value of 11.5 MPa for particleboards manufactured for general-purpose use and both the HTT-10 and the control groups in this study met this requirement.

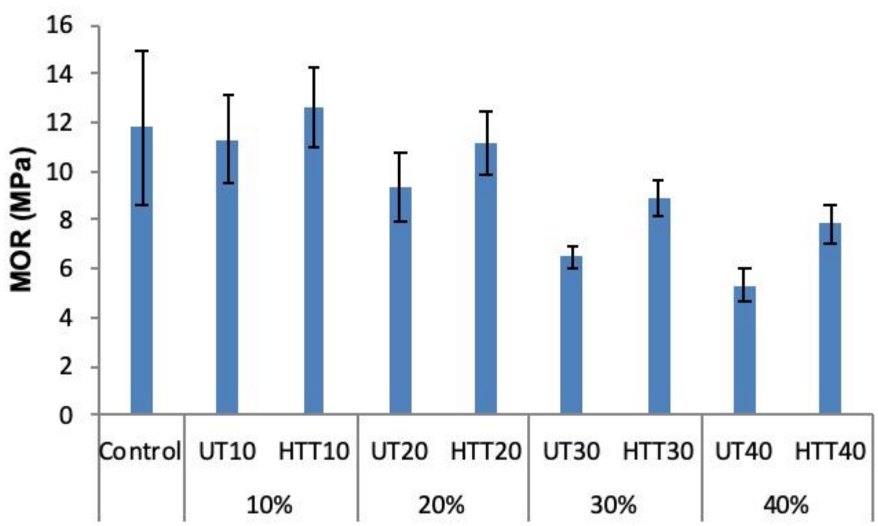

Fig. 4. Average MOR values of the produced composites

The MOR decreased with the increase of the WS particle ratio in both the UT and HTT groups. The UT-40 group (containing 40% untreated WS) had a 55.1% lower MOR value than that of the control. Likewise, lower MOR values have been reported for particleboard produced using agricultural residues (Bektaş *et al.* 2005; Nemli *et al.* 2008; Güler *et al.* 2009; Nemli *et al.* 2009; Büyüksarı *et al*. 2010). Güler *et al.* (2009) found the average MOR values of the control group and boards containing 40% hazelnut husk as 15.6 MPa and 12.7 MPa, respectively.

The application of HTT improved the MOR values of the produced particleboards. The improvements in the MOR values increased in parallel with the increasing WS ratio: 11.4% for the 10% WS groups and 48.0% for the 40% WS groups. This may have been caused by the HTT application, which removed components from the epidermal cells, including the thin waxy layer, silica, and non-polar extractives. Han *et al.* (2010) found that treatment via steam explosion decreased the silica content of WS. In a previous study, particleboards containing WS particles were soaked in a solution of acetic anhydride, boiled in a soap solution, boiled in plain water, and steamed at 100 °C. The researchers reported that these boards exhibited higher MOR values than particleboards produced from untreated WS particles (Bekhta *et al*. 2013). They observed increases of 48% and 135%, respectively, in MOR for boards steamed at 100 °C and treated with acetic anhydride.

Figure 5 presents the average MOE and standard deviation values of the UT WS and HTT WS particleboards. The highest MOE value was 2410 MPa for the HTT-20 group (20% HTT WS particles), whereas the lowest was found as 1301 MPa for the UT-40 group (40% untreated WS particles). All HTT groups exhibited higher values of MOE than the control group. Moreover, the values of the UT-10 and UT-20 groups were higher compared to the control group. With the exception of the UT-30 and UT-40 groups, the particleboards fabricated in this study were in accordance with the required MOE standard (1600 MPa) for general-purpose end-use (EN 312 2010).

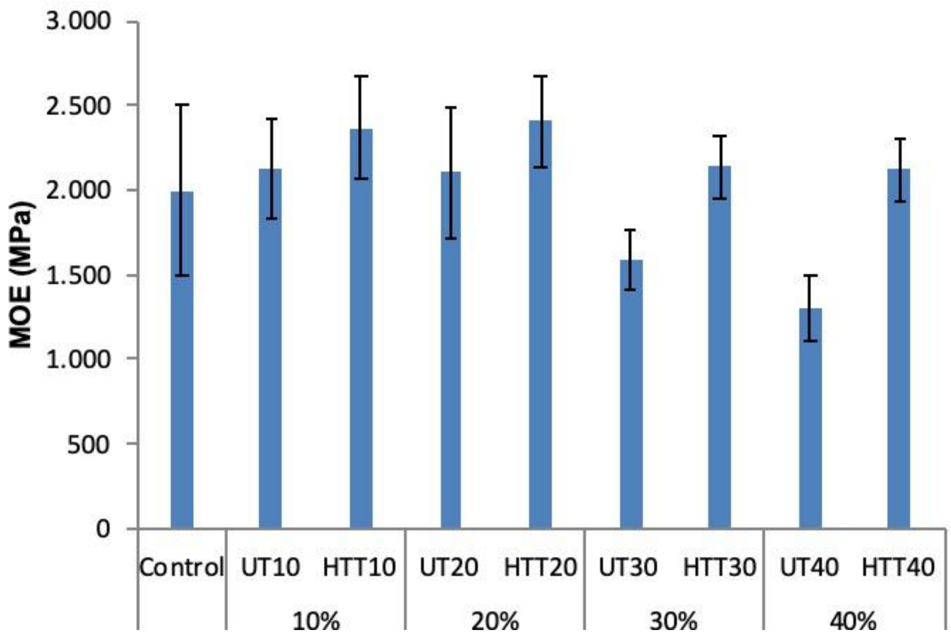

Fig. 5. Average MOE values of the produced composites

The UT group MOE value decreased with the increase of the WS ratio. The UT-10 group MOE value was 63.3% higher than that of the UT-40 group. Similarly, Güler *et al.* (2009) observed that the MOE value of particleboard containing 10% hazelnut husk was 11.1% higher compared to particleboard containing 40% hazelnut husk.

In the HTT groups, the MOE value of the particleboards rose when the HTT-WS particle ratio was increased from 10 to 20% and thereafter decreased. The HTT application to the WS particles improved the MOE value of the particleboard for all ratios: 11.4, 14.5, 34.7, and 62.9% for the groups containing 10, 20, 30, and 40%, respectively. This may have been because the HTT application resulted in the non-polar extractives, silica, and thin waxy layer being removed from the epidermal cells.

Figure 6 shows the average IB strength values and standard deviation of the particleboards containing UT and HTT WS particles. The highest IB strength value (0.37 MPa) was found in the control group. The IB strength of the particleboards significantly decreased with the addition of WS. Similarly, lower IB strength values were found for other bio-based composites containing agricultural residues (Çöpür *et al*. 2007; Nemli *et al*. 2001; Bektaş *et al*. 2002; Boquillon *et al*. 2004; Güler and Özen 2004; Bektaş *et al*. 2005). Boquillon *et al*. (2004) stated that straw panels bonded with UF showed poorer mechanical properties, with an IB value of 0.02 MPa. Bekhta *et al.* (2018) assumed that the thin wax layer of WS impeded wetting and bonding and it affected the particle adhesion, representing a significant complication for particleboard production. Another reason for the lower IB strength could have been the lower surface free energy of the WS. The surface free energy of wheat straw at 34.5 mJ/m^2 is much lower than that of redwood or Douglas fir (Boquillon *et al*. 2004). Lower surface free energy implies a lower wettability and higher contact angle (CA) of the straw outer surface. Boquillon *et al.* (2004) determined the CA values, initially and after 100 s, of UF resin on the exterior of the straw as 84 ° and 82 °. They also concluded that the poor wettability of the straw with the use of UF explained the very poor properties exhibited by the panels.

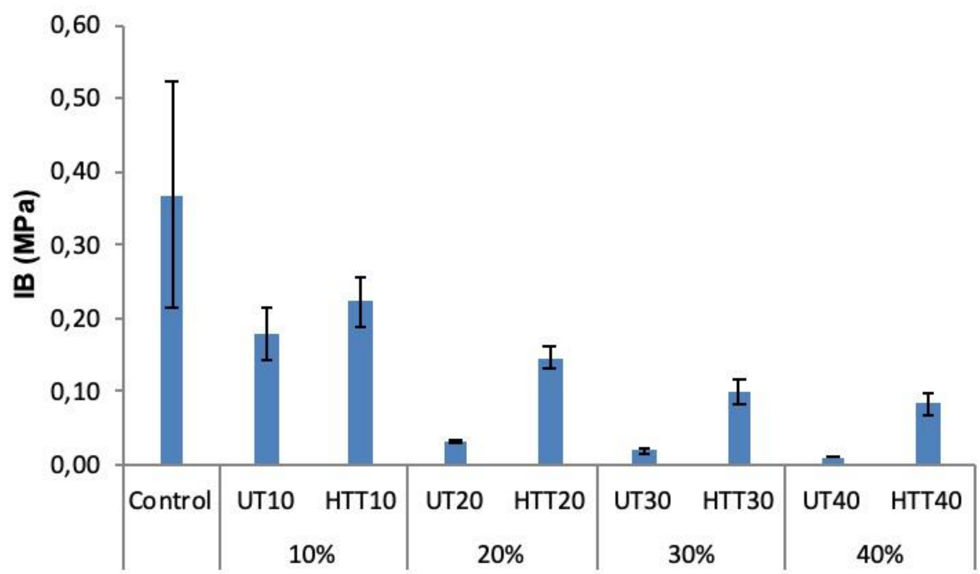

Fig. 6. Average IB values of the produced composites

All of the HTT panels demonstrated higher IB strength values than the UT groups. The IB strength of the HTT WS particleboards was improved by the HTT, which might have resulted from the withdrawal of the waxy layer, non-polar extractives, and silica by the HTT. Several studies on particleboard material have found that the extractives adversely affected its mechanical properties (Nemli *et al*. 2004; Nemli and Çolakoglu 2005; Nemli and Aydın 2007). Wasylciw (1998) improved the bonding ability of straw particleboard by eliminating the external layer of wax. Moreover, steam explosion-treated straw has been shown to exhibit increased wettability, higher acidity, and a lower amount of silica, all of which contribute to better bonding of the straw particles with the watersoluble binding agents (Han *et al*. 2010). Bekhta *et al.* (2018) reported that boiling increased the IB strength of WS particleboard. In another study, Bekhta *et al.* (2013) improved the IB value of particleboard via WS pre-treatments in which samples were soaked in a solution of acetic anhydride, boiled in a soap solution, boiled in plain water, and steamed at 100 °C. Their results showed 0.11 MPa IB strength for the untreated WS particleboard compared with 0.30 MPa for the particleboard containing WS that had been boiled in soapy solution.

The IB strength of the UT WS particleboards declined with increasing WS content. The IB strength of the UT-10 (containing 10% WS) was 0.18 MPa, but for the UT-40, it was 0.01 MPa. When the WS ratio was increased from 10 to 20%, the IB strength was reduced by 82.6%. Previous studies have shown IB strength to decrease when ratios of agricultural residue were increased in panels (Nemli *et al*. 2003; Bektaş *et al.* 2005; Güler *et al*. 2008). Similar to the UT board groups, the IB strength of the HTT board groups decreased when increasing amounts of WS were added; for example, the IB strength values of the HTT-10 and HTT-40 groups were 0.22 MPa and 0.08 MPa, respectively. None of the produced particleboards containing WS complied with the required IB standard (0.24 MPa) for general-purpose end-use.

CONCLUSIONS

1. After 2 and 24 h of immersion, the composites containing 10 and 40% hydrothermally treated wheat straw (HTT WS) exhibited improved values for water absorption (WA) and thickness swelling (TS) compared to those containing untreated wheat straw (UT WS); however, the composites containing 10% and 40% HTT WS had lower WA and TS values.
2. The WA and TS values generally rose with the increasing of the WS ratio in the composites.
3. The modulus of rupture (MOR) values were reduced with increased WS ratios in both the UT and HTT groups. The MOR values of all HTT groups were higher than those of the UT groups.
4. The composites containing HTT WS exhibited higher modulus of elasticity (MOE) values compared to the control and the groups containing UT WS.
5. The application of HTT to WS was significantly and positively effective on the MOR, MOE, and internal bond strength (IB) values at all composite WS ratios.

ACKNOWLEDGMENTS

This study received the support of the Düzce University Research Fund (Project Number: 2017.02.03.550).

REFERENCES CITED

Ahmad, Z., Asgher, M., and Iqbal, H. M. N. (2017). "Enzyme-treated wheat straw-based PVOH biocomposites: Development and characterization," *BioResources* 12(2), 2830-2845. DOI: 10.15376/biores.12.2.2830-2845

Ayrılmış, N., Kaymakcı, A., and Özdemir, F. (2013). “Sunflower seed cake as reinforcing filler in thermoplastic composites,” *Journal of Applied Polymer Science* 129(3), 1170-1178. DOI: 10.1002/app.38806

Azizi, K., Tabarsa, T., and Ashori, A. (2011). “Performance characterizations of particleboards made with wheat straw and waste veneer splinters,” *Composites Part B: Engineering* 42(7), 2085-2089. DOI: 10.1016/j.compositesb.2011.04.002

Bekhta, P., Korkut, S., and Hiziroglu, S. (2013). "Effect of pretreatment of raw material on properties of particleboard panels made from wheat straw," *BioResources* 8(3), 4766-4774. DOI: 10.15376/biores.8.3.4766-4774

Bekhta, P., Salca, E., and Kozak, R. (2018). “Properties of wood-straw composites bonded with modified UF adhesive and pre-treated straw particles,” *Pro Ligno* 14(1), 37-41.

Bektaş, İ., Güler, C. and Kalaycioglu, H. (2002). “Manufacturing of particleboard from sunflower stalks (*Helianthus annuus* L.) using urea-formaldehyde resin,” *KSU J. Science and Engineering* 5(2), 49-56. (In Turkish)

Bektaş, İ., Güler, C., Kalaycioglu, H., Mengeloglu, F., and Nacar, M. (2005). “The manufacture of particleboards using sunflower stalks (*Helianthus annuus* L.) and poplar wood (*Populus alba* L.),” *Journal of Composite Materials* 9(5), 467-473. DOI: 10.1177/0021998305047098

Boquillon, N., Elbez, G., and Schönfeld, U. (2004). “Properties of wheat straw particleboards bonded with different types of resin,” *Journal of Wood Science* 50, 230–235. DOI: 10.1007/s10086-003-0551-9

Büyüksarı, Ü., Avcı, E., Ayrılmış, N., and Akkılıç, H. (2010). “Effect of pine cone ratio on the wettability and surface roughness of particleboard," *BioResources* 5(3), 1824-1833. DOI: 10.15376/biores.5.3.1824-1833

Cheng, E., Sun, X., and Karr, G. S. (2004). “Adhesive properties of modified soybean flour in wheat straw particleboard,” *Composites Part A* 35(3), 297-302. DOI: 10.1016/j.compositesa.2003.09.008

Chougan, M., Ghaffar, S. H., Al-Kheetan, M. J., and Gecevicius, M. (2020). “Wheat straw pre-treatments using eco-friendly strategies for enhancing the tensile properties of bio-based polylactic acid composites,” *Industrial Crops and Products* 155, article no. 112836. DOI: 10.1016/j.indcrop.2020.112836

Cöpür, Y., Güler, C., Akgül, M., and Taşçıoğlu, C. (2007). “Some chemical properties of hazelnut husk and its suitability for particleboard production,” *Building and Environment* 42, 2568-2572. DOI: 10.1016/j.buildenv.2006.07.011

EN 310 (1993). “Wood-based panels: Determination of modulus of elasticity in bending and of bending strength,” European Committee for Standardization, Brussels, Belgium.

EN 317 (1993). “Particleboards and fibreboards—Determination of swelling in thickness after immersion in water,” European Committee for Standardization, Brussels, Belgium.

EN 319 (1993). “Particleboards and fibreboards—Determination of tensile strength perpendicular to the plane of the board,” European Committee for Standardization,

Brussels, Belgium.
EN 323 (1993). "Wood-based panels - Determination of density," European Committee for Standardization, Brussels, Belgium.
EN 312 (2010). "Particleboard specification," European Committee for Standardization, Brussels, Belgium.
Ghaffar, S. H., Fan, M., and McVicar, B. (2017a). "Interfacial properties with bonding and failure mechanisms of wheat straw node and internode," *Composites Part A: Applied Science and Manufacturing* 99, 102-112. DOI: 10.1016/j.compositesa.2017.04.005
Ghaffar, S. H., Fan, M., Zhou, Y., and Madyan, O. A. (2017b). "Detailed analysis of wheat straw node and internode for their prospective efficient utilization," *Journal of Agricultural and Food Chemistry* 65(41), 9069-9077. DOI: 10.1021/acs.jafc.7b03304
Grigoriou, A., Passialis, C., and Voulgradis, E. (2000). "Kenaf core and bast fiber chips as raw material in production of one-layer experimental particleboards," *Holz Roh-Werkst*. 58(4), 290-291. DOI: 10.1007/s001070050429
Güler, C. (2001). *Research on the Production Possibilities of the Particle Board from Cotton (Gossypium hirsitum L.) Stalks*, Ph. D. Dissertation, ZKÜ Institute of Science, Zonguldak. (In Turkish)
Güler, C., and Özen, R. (2004). "Some properties of particleboard made from cotton stalks (*Gossypium hirsitum* L.)," *Holz Roh-Werkst*. 62, 40-43. DOI: 10.1007/s00107-003-0439-9
Güler, C., Bektaş, I., and Kalaycioglu, H. (2006). "The experimental particleboard manufacture from sunflower stalks (*Helianthus annuus* L.) and calabrian pine (*Pinus brutia* Ten.)," *Forest Products Journal* 56(4), 56-60.
Güler, C., Cöpür, Y., and Tascioglu, C. (2008). "The manufacture of particleboards using mixture of peanut hull (*Arachis hypoqaea* L.) and European black pine (*Pinus nigra* Arnold) wood chips," *Bioresource Technology* 99(8), 2893-2897. DOI: 10.1016/j.biortech.2007.06.013
Güler, C., Çöpür, Y., and Büyüksarı, Ü. (2009). "Producing particleboards from hazelnut (*Corylus avellana* L.) husk and European black pine (*Pinus nigra* Arnold)," *Wood Research* 54(1), 125-132.
Hafezi, S., and Hosseini, K. D. (2014). "Surface characteristics and physical properties of wheat straw particleboard with UF resin," *J. Indian Acad. Wood Sci.* 11(2), 168-173. DOI: 10.1007/s13196-014-0136-9
Han, G., Zhang, C., Zhang, D., Umemura, K., and Kawai, S. (1998). "Upgrading of urea formaldehyde-bonded reed and wheat straw particle boards using silane coupling agents," *Journal of Wood Science* 44(4), 282-286. DOI: 10.1007/BF00581308
Han, G. P., Umemura, K., Zhang, M., Honda, T., and Kawai, S. (2001). "Development of high-performance UF-bonded reed and wheat straw medium-density fiberboard," *Journal of Wood Science* 47(5), 350-355. DOI: 10.1007/BF00766784
Han, G., Deng, J., Zhang, S., Bicho, P., and Wu, Q. (2010). "Effect of steam explosion treatment on characteristics of wheat straw," *Industrial Crops and Products* 31, 28-33. DOI: 10.1016/j.indcrop.2009.08.003
Hýsková, P., Hýsek, S., Schönfelder, O., Šedivka, P., Lexa, M., and Jarský, V. (2020). "Utilization of agricultural rests: Straw-based composite panels made from enzymatic modified wheat and rapeseed straw," *Industrial Crops and Products* 144, article no. 112067. DOI: 10.1016/j.indcrop.2019.112067
Kalaycioglu, H. (1992). "Utilization of crops residues on particleboard production," in:

Proc. ORENKO 92, First Forest Products Symp., Trabzon, Turkey, pp. 288-292.
Kaymakcı, A., and Ayrılmış N. (2014). "Waste chestnut shell as a source of reinforcing fillers for polypropylene composites," *Journal of Thermoplastic Composite Materials* 27(8), 1054-1064. DOI: 10.1177/0892705712461648
Mantanis, G., Nakos, P., Berns, J., and Rigal, L. (2000). "Turning agricultural straw residues into value-added composite products: A new environmentally friendly technology," in: *Proc. of the 5th International Conference on Environmental Pollution*, Thessaloniki, Greece, pp. 840-848.
Markessini, E., Roffael, E., and Rigal, L. (1997). "Panels from annual plant fibers bonded with urea-formaldehyde resins," in: *Proceedings of 31st International Particleboard/Composite Materials Symposium*, Pullman, WA, USA, pp.147-160.
Mo, X., Cheng, E., Wang, D., and Sun, X. S. (2003). "Physical properties of medium-density wheat straw particleboard using different adhesives," *Industrial Crops and Products*18, 47-53. DOI: 10.1016/S0926-6690(03)00032-3
Navi, P., and Sandberg, D. (2012). *Thermo-Hydro-Mechanical Wood Processing*, EPFL Press, Lausanne, Sweden.
Nemli, G., Kalaycioglu, H., and Alp, A. (2001). "Suitability of date palm (*Phoenix dactyliferia*) branches for particleboard production," *Holz Roh-Werkst*. 59, 411-412. DOI: 10.1007/s00107-001-0257-x
Nemli, G., Kirci, H., Serdar, B., and Ay, N. (2003). "Suitability of kiwi (*Actinidia siensis* Planch) prunings for particleboard production," *Industrial Crops and Products* 17(1), 39-46. DOI: 10.1016/S0926-6690(02)00057-2
Nemli, G., Kırcı, H., and Temiz, A. (2004). "Influence of impregnating wood particles with mimosa bark extract on some properties of particleboard," *Industrial Crops and Products* 20(3), 339-344. DOI: 10.1016/j.indcrop.2003.11.006
Nemli, G., and Çolakoglu, G. (2005). "Effects of mimosa bark usage on some properties of particleboard," *Turkish Journal of Agriculture and Forestry* 29(3), 227-230.
Nemli, G., and Aydın, A. (2007). "Evaluation of the physical and mechanical properties of particleboard made from the needle litter of *Pinus pinaster* Ait.," *Industrial Crops and Products* 26(3), 252-258. DOI:10.1016/j.indcrop.2007.03.016
Nemli, G., Yıldız, S., and Gezer, E. D. (2008). "The potential for using the needle litter of Scotch pine (*Pinus sylvestris* L.) as a raw material for particleboard manufacturing," *Bioresource Technology* 99, 6054-6058. DOI: 10.1016/j.biortech.2007.12.044
Nemli, G., Demirel, S., Gümüşkaya, E., Aslan, M., and Acar, C. (2009). "Feasibility of incorporating waste grass clippings (*Lolium perenne* L) in particleboard composites," *Waste Management* 29, 1129-1131. DOI: 10.1016/j.wasman.2008.07.011
Rowell, R. M. (1995). "A new generation of particleboard materials from agro-based fiber," in: *Proceedings of the 3rd International Conference on Frontiers of Polymers and Advanced Materials*, Kuala Lumpur, ML, pp. 659-665.
Sampathrajan, A., Vijayaraghavan, N. C., Swaminathan. K. R. (1992). "Mechanical and thermal properties of particleboards made from farm residues," *Bioresource Technology* 40(3), 249-251. DOI: 10.1016/0960-8524(92)90151-M
Sitz, E. D., Bajwa, D. S., Webster, D. C., Monono, E. M. Wiesenborn, D. P., and Bajwa, S. G. (2017). "Epoxidized sucrose soyate—A novel green resin for crop straw based low density fiberboards," *Industrial Crops and Products* 107, 400-408. DOI: 10.1016/j.indcrop.2017.04.057
Shang, X., Yang, J., Song, O., and Wang, L. (2020). "Efficacy of modified rice straw fibre on properties of cementitious composites," *Journal of Cleaner Production* 276,

124184. DOI: 10.1016/j.jclepro.2020.124184
Tjeerdsma, B. F., and Militz, H. (2005). "Chemical changes in hydrothermal treated wood: FTIR analysis of combined hydrothermal and dry heat-treated wood," *Holz Roh-Werkst.* 63, 102-111. DOI: 10.1007/s00107-004-0532-8
Tupciauskas, R., Rizhikovs, J., Brazdausks, P., Fridrihsone, V., and Andzs M. (2021). "Influence of steam explosion pre-treatment conditions on binder-less boards from hemp shives and wheat straw," *Industrial Crops and Products* 170, article no. 313717. DOI: 1016/j.indcrop.2021.113717
Turreda, L. D. (1983). "Bagasse, wood and wood-bagasse particleboards bonded with urea formaldehyde and polyvinyl acetate/isocyanate adhesives," *NSTA Technol. J.* 8(3), 66-78.
Xu, X., Yao, F., Wu, Q., and Zhou, D. (2009). "The influence of wax-sizing on dimension stability and mechanical properties of bagasse particleboard," *Industrial Crops and Products* 29(1), 80-85. DOI: 10.1016/j.indcrop.2008.04.008
Wasylciw, W. (1998). "The straw panel manufacturing process and straw's potential in flooring substrates," in: *Proceedings of the Symposium on Plastic Laminates*, Georgia, USA, pp. 291-296.
Yalınkılıç, M. K., Imamura, Y., Takahashi, M., Kalaycioglu, H., Nemli, G., Demirci, Z., and Özdemir, T. (1998). "Biological, physical and mechanical properties of particleboard manufacture from waste tea leaves," *International Biodeterioration and Biodegradation* 41(1), 75-84. DOI: 10.1016/S0964-8305(98)80010-3
Yasin, M., Bhutto, A. W., Bazmi, A. A., and Karim, S. (2010). "Efficient utilization of rice-wheat straw to produce value added composite products," *International Journal of Chemical and Environmental Engineering* 1(2), 136-143.

Article submitted: August 31, 2021; Peer review completed: September 28, 2021; Revised version received and accepted: October 6, 2021; Published: October 11, 2021.
DOI: 10.15376/biores.16.4.7901-7915