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ORIGINAL ARTICLE

Some mechanical properties of WPCs with wood flour and walnut shell flour

Bekir Cihad Bal[1]*

[1]Vocational School of Technical Sciences, Kahramanmaraş Sütçü İmam University, Kahramanmaraş, Türkiye
**bcbal@hotmail.com*

Abstract

This study used a high-density polyethylene (HDPE) polymer matrix, pine-wood flour (PWF) and walnut-shell flour (WSF) to produce wood–plastic composite (WPC) boards. The PWF and WSF filler amounts were adjusted to 20%, 30%, and 40% by weight. Some of the mechanical properties of the produced composite boards were comparatively investigated, such as the flexural strength, flexural modulus, deformation at break, tensile strength, tensile modulus, and elongation at break. Flexural tests and tensile tests were performed according to ASTM D790 and ASTM D638, respectively. According to the data obtained, the flexural strength, deformation at break, tensile strength, and elongation at break decreased as the filler content increased. In addition, the flexural modulus values of all the test groups increased with the filler content. However, the tensile modulus values of the test groups that used the WSF filler were smaller than those of the groups without filler.

Keywords: *HDPE, pine wood flour, walnut shell flour, WPC.*

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1 Introduction

Wood plastic composites (WPCs) are used in different areas for outdoor applications such as decking, cladding, fencing, and pergolas. The application areas for WPCs are increasing day by day. In parallel with this increase, scientific studies on WPCs have increased. Recycled or virgin polymers such as polyethylene (low density or high density), polypropylene, polyvinyl chloride, and polylactic acid are used for WPC production as a polymer matrix. Some lignocellulosic materials are used as fillers in the form of flour or fibre to optimize the properties of these composites[1]. In previous studies, the effects of lignocellulosic fibres on the mechanical properties of WPC boards were investigated by many researchers. For example, Wambua et al.[2] studied the mechanical properties of WPCs produced from polypropylene and natural fibres such as sisal, kenaf, hemp, jute, and coir, and they compared the results with the corresponding properties of glass mat-reinforced polypropylene composites. Leao et al.[3] produced WPCs using sugar cane bagasse and elephant grass, and determined some of their mechanical properties. Jordá–Vilaplana et al.[4] manufactured WPC boards with bio-based polyethylene and short fibres from *Cortaderia selloana*. Sobczak et al.[5] produced and tested the mechanical properties of WPCs with natural fibres (like jute, hemp, kenaf, sisal, flax). Dolza et al.[6] produced and tested WPC boards using bio-based high-density polyethylene (BioHDPE) and natural fibres such as hemp, flax, and jute short fibres. Hyvärinen and Kärki[7] produced WPC boards using barley straw fibre and picea wood flour, and compared the mechanical properties of the WPC boards. They reported that the substitution of picea wood flour with barley straw flour was found to weaken the mechanical properties of the WPC boards except impact strength. The density of picea wood flour is higher than that of natural fibres. In addition, some flours made of fruit seed shells are used as filler materials in WPC production in laboratory experiments. In previous studies, some researchers investigated the effects of shell flours such as almond, apricot, sunflower hazelnut, and walnut flours on the mechanical properties of WPC boards. For example, Essabir et al.[8] prepared WPC boards using polypropylene and almond-shell particles, and investigated some of their technological properties. Essabir et al.[9] produced WPC boards using HDPE, talc, and apricot-shell flour, and investigated some of their mechanical and thermal properties. Barczewski et al.[10] produced WPC boards with polypropylene and sunflower-husk flour, and investigated some of their mechanical properties, as well as the microstructure and surface quality of the WPC boards. Taşdemir[11], Akbaş et al.[12], and Sutivisedsak et al.[13] produced WPC boards using polymers such as HDPE, LDPE, and PP, with hazelnut-shell flour as a filler material.

In addition, walnut-shell flour (WSF) was used in some previous studies. For example, Włodarczyk–Fligier et al.[14] investigated some of the mechanical properties of WPCs produced with a polypropylene matrix and with 30, 40, and 50% WSF contents. They reported that the applied WSF increased the hardness and stiffness modulus of the WPC, and decreased the tensile strength. Dobrzyńska et al.[15] used differential scanning calorimetry, thermogravimetry, dynamic mechanical thermal analysis, and scanning

electron microscopy to investigate a WPC produced with polypropylene and WSF. Tabar et al.[16] produced WPC using HDPE, silicon dioxide, wood flour, and WSF. They reported that with the addition of up to 50 wt% WSF, the tensile and flexural properties of the composites were chanced compared to the addition of poplar wood flour. They determined that the addition of WSF to HDPE exhibited lower mechanical properties than the composite containing the same ratio of poplar wood flour (50%). In addition, the mechanical properties of a WPC produced with wood flour (40 wt%) was greater than that of a WPC produced with WSF (40 wt%). Salasinska and Ryszkowska[17] produced a WPC using polyethylene waste and WSF, and reported some positive effects of the WSF on the physical and mechanical properties. Zahedi et al.[18] investigated some of the physical and mechanical properties of a WPC produced with WSF and polypropylene compared with those of a WPC produced with wood flour and polypropylene. They reported that the mechanical properties of the WPC produced from wood flour were greater than those of the WPC produced from WSF. Zhang et al.[19] investigated some of the mechanical properties of WPCs produced from peanut husk, rice husk, and WSF, along with HDPE. The results showed that the mechanical properties of the WPC produced from walnut shell were greater than those of the WPC produced from peanut husk, but lower than those of the WPC produced from rice husk.

In previous studies, there has not been sufficient study of the effect of the WSF on the mechanical properties of WPC boards. In addition, some of the mechanical test results obtained by researchers were different from each other. For this reason, the aim of this study was to determine the effect of the WSF amount and wood flour amount on some of the mechanical properties of a WPC.

2 Materials and Methods

2.1 Materials

For this study, pine (*Pinus nigra*) wood sawdust was obtained from a saw mill in Kahramanmaraş–Türkiye. The sawdust was sieved, and 40 mesh (in the range of 455 µm and 900 µm) wood flour was separated for experiments (Figure 1A). Walnut shells were obtained from domestic usage, where the species was the *Maraş-18* walnut tree species. The walnut shells were cleaned of rot and imperfections before being ground using a grinder (Brader 1500). Then, the shell flour was sieved, and 40 mesh (in the range of 455 µm and 900 µm) shell flour was separated for experiments (Figure 1B). The pine-wood flour (PWF) and WSF were dried in an oven at 103±2 ºC for 24 hours. The HDPE matrix was obtained from Petkim Petrochemical Company in Turkey (Figure 1C). The melt flow index of the HDPE is 190 °C/2.16 kg.

The compositions of the composites are given in Table 1. The WSF and PWF were mixed with HDPE as described in Table 1, and the mixtures were transferred to a single-screw extruder machine to compound. The temperatures of the three sections of the barrel of the extruder were 170, 180, and 190 °C. The extruder speed was 40 rpm. The extruded blend was taken in a filament form from the barrel exit with a nozzle diameter of 3 mm (Figure 2A). The extruded blend in filament form was cooled in the air (Figure 2B). The cooled blend was cut into pellets (Figure 2C), and these pellets were ground (Figure 2D). The ground blend was placed in a metal mould and transferred to electrically heated plates at a temperature of 185 ± 5 °C. Non-stick baking paper was used to prevent sticking. The blend was heated and melted over a period of 15 min. No pressure was applied during this procedure. At the end of this duration, the mixture was removed from the heater with the metal mould and immediately placed in a cold press. A total of 12 kg/cm^2 of pressure was applied in the cold press. The board was taken from the metal mould, and a composite board was thus obtained with the dimensions of 4 × 180 × 220 mm^3 (thickness × width × length). Four composite boards were produced for each group. A total of 28 boards were produced for the present study. Test samples were prepared from these boards. Four test samples were cut from each board for each test. Thus, sixteen test specimens were prepared for each test. The test samples were cut using a

Figure 1. (A) PWF, (B) WSF, and (C) HDPE.

Table 1. Compositions of the composites (by wt%).

Content	Groups						
	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7
HDPE	100	80	70	60	80	70	60
PWF	0	20	30	40	0	0	0
WSF	0	0	0	0	20	30	40

Figure 2. Extruder die (A), extruded blend (B), pellets (C), and ground blend (D).

Table 2. Density test data, ANOVA P values, and Duncan test results.

Groups	1	2	3	4	5	6	7	P value
x	950D	995C	1019B	1039A	997C	1020B	1044A*	P<0.001
ss	3.64	7.06	9.11	11.87	19.67	13.34	20.29	

Means followed by the same letter are not significantly different with each other using Duncan multiple comparison test at $\alpha = 0.05$; x: arithmetic mean; ss: standard deviation. *Highest value.

laboratory band saw. The edges of each test sample prepared for the tensile test were shaped with a CNC router.

2.2 Methods

Flexural tests and tensile tests were performed according to ASTM D790-15[20] and ASTM D638-22[21], respectively. Flexural tests were conducted using a three-point bending test procedure on a universal testing machine (UTM). The span length was 60 mm. The support span-to-depth ratio was 15/1. The preload was 3 N, and the test speed was 2 mm/min. The test was ended when the load decreased to 80% of the maximum load. At the end of the test, the deformation was noted as the deformation at break.

Tensile tests were conducted on dog-bone-shaped test samples (Type I) as described in ASTM D638-22[21]. The distance between grips was 115 mm, the preload was 5 N, and the test speed was 5 mm/min. The test was ended when the test sample broke or the load decreased to 80% of the maximum load. At the end of the test, the elongation was noted as the elongation at break.

3 Results and Discussion

The density values, one-way ANOVA P values, and Duncan test results are given in Table 2. The lowest density values were found for the test samples of group 1. The highest density values were found for the test samples of group 7. The density values of groups 2, 3, and 4, which used PWF as a filler, were lower than those of groups 5, 6, and 7, which used WSF as a filler. It can be said that the reason for these differences was the press pressure and the different densities of PWF and WSF. After pressing, the thickness of the boards with PWF was slightly higher than those with WSF. As a result, the density of the PWF boards was measured to be somewhat low. In addition, the densities of all the test samples (groups 2, 3, 4, 5, 6, and 7) were measured to be higher than those of the unfilled control samples (group 1). The difference was statistically significant ($P < 0.001$). As the amount of filler in the composite board increased, the density of the board increased accordingly. Similar results related to WPCs were reported by other researchers[22-27]. This situation complied with the general composite rule, which applies especially to WPCs. According to Stark and Berger[28], and Çavuş and Mengeloğlu[29], the density increase was due to the higher cell wall density of the lignocellulosic materials used as the filler. In addition, Ayrılmış et al.[30] studied some properties of polypropylene composites filled with WSF. They determined that the density of the composites significantly increased with the WSF content compared to control samples. Similar results were reported by Salasinska and Ryszkowska[17]. Włodarczyk–Fligier et al.[14] studied some of the properties of WPCs prepared with a polypropylene matrix with filler from WSF, and they reported that a higher filler content led to a higher density. Related to the filler type, Zimmermann et al.[31] reported that a lower wood fibre-share and larger sieve-size increased the compound´s density. As a result, as can be seen in previous studies and the present study, the filler type and filler content can change the density of the composite material.

The flexural test data, one-way ANOVA P values, and Duncan test results are given in Table 3. The data analyses showed that the test samples in the control group had the highest flexural strength (39.4 N/mm^2), and the test samples of group 7 had the lowest (20 N/mm^2). The flexural strengths of all the test groups were lower than that of the control group. The differences between the control group and test groups were statistically significant ($P < 0.001$). The flexural strength decreased as the filler content increased for both PWF and WSF. Similar results related to the flexural strength of WPCs produced with wood flour were reported by some researchers[22-24,26,27,32-37]. In addition, similar results related to the flexural strength of WPCs produced with WSF were reported by Ayrılmış et al.[30]. They reported that the flexural strengths of WPCs decreased noticeably with the addition of WSF. Matuana and Stark[22], and Ayrılmış et al.[30], suggested that the reduction in the flexural strength was due to the poor compatibility between the polar WSF and nonpolar polymer, which formed weak interfacial regions.

Zahedi et al.[18] and Tabar et al.[16] studied the effects of WSF and poplar-wood flour on the properties of WPCs, and they determined that the flexural strength of the WPC produced using poplar-wood flour with a content of 50% was greater than that of WPC produced using WSF with a content of 50%. The results of this study are compatible with those of the present study.

The highest flexural modulus was found for test group 7. The lowest flexural modulus was found for the control group. Flexural modulus values of all the test groups were greater than that of the control group. In addition, the flexural modulus values of the test groups that used WSF were greater than those of the test groups that used PWF. The flexural modulus increased with the filler content. The differences between the control group and test groups were statistically significant ($P < 0.001$), as can be seen in Table 3. Similar results related to the flexural modulus values of WPCs produced with wood flour were reported in different studies[23,24,33,34,36]. In addition, similar results related to the flexural modulus values of WPCs produced with WSF were also reported by Ayrılmış et al.[30] and Zahedi et al.[18]. They reported that the flexural modulus values of WPCs that used poplar-wood flour were greater than those with WSF.

The deformation-at-break values at the end of the flexural tests are also given in Table 3. In addition, the load–deformation curves from the flexural tests of group 1, group 4, and group 7 are shown in Figure 3. The highest deformation at break was found for the control group, and the lowest was found for group 7. The deformation at break decreased as the filler content increased. The differences between groups were statistically significant ($P < 0.001$), as can be seen in Table 3.

In addition, it can easily be seen that the load–deformation curves for groups 1, 4, and 7 were different (Figure 3). The load–deformation curves for the test samples of the control group were very similar to each other. However, the curves for the test samples of groups 4 and 7 were not similar, and some test samples broke at the end of the small deformation. Deformation-at-break test data related to WPCs have been given by several researchers. Fiore et al.[38] provided the deformation-at-break test data from their study, which showed that the deformation at break decreased as the filler content increased. Similar results for the deformation at break were reported by Bal[26].

The tensile test data, one-way ANOVA P values, and Duncan test results are given in Table 4. Based on the

Table 3. Flexural test data, ANOVA P values, and Duncan test results.

Tests	Groups							P values
	1	2	3	4	5	6	7	
Flexural strength (N/mm²)	39.4**A***	29.8**C**	26.1**D**	25.9**D**	32.2**B**	25.1**D**	20.0**E**	P<0.001
	*0.83***	*2.70*	*2.71*	*3.96*	*3.34*	*4.86*	*3.98*	
Flexural modulus (N/mm²)	1077**F**	1099E**F**	1175D**E**	1323**B**	1208**CD**	1271**BC**	1412**A***	P<0.001
	100.1	*41.0*	*88.2*	*187.9*	*85.5*	*120.2*	*87.2*	
Deformation at break (mm)	19.4**A****	13.9**B**	9.6**C**	6.9**D**	14.9**B**	9.4**C**	6.2**D**	P<0.001
	0.31	*2.54*	*2.57*	*3.20*	*3.28*	*3.80*	*1.39*	

*Means followed by the same letter are not significantly different with each other using Duncan multiple comparison test at $\alpha = 0.05$. **Standard deviation in italic form. ***Highest value.

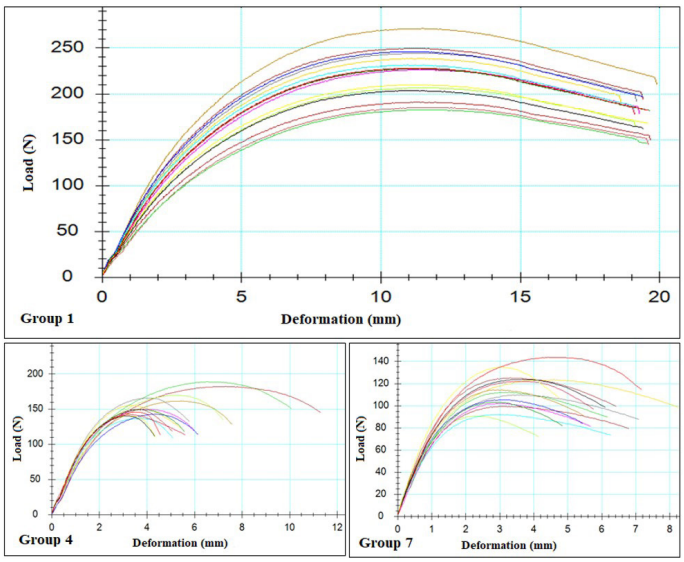

Figure 3. Load–deformation curves from flexural tests (group 1, group 4, and group 7).

data related to the tensile strength and elongation-at-break analyses, it can be said that the highest tensile strength and elongation at break were found for the test samples of group 1 (control group), and the lowest were found for the test samples of group 7. The differences between the groups were statistically significant ($P < 0.001$). The tensile strengths and elongation-at-break values of the test samples produced from PWF were greater than those of the test samples produced from WSF. In contrast to the tensile strength and elongation at break, the tensile modulus increased with the amount of filler, as can be seen in Table 4. In previous studies, similar results related to the effect of wood flour on WPCs were reported[22-24,26,27,33-37,39]. According to Matuana and Stark[22], the addition of wood flour filler to polymeric matrices decreased the ductile behaviour of the matrix by making the WPC material more brittle than that of the polymeric material. Thus, the tensile strength and elongation at break decreased compared to the unfilled polymeric matrix.

The amount of increase in the tensile modulus was greater in test samples using PWF than in those using WSF. It can be said that the most important reason for this difference was the fibrous structure of PWF compared to WSF. Similar results were reported by Zahedi et al.[18] and Tabar et al.[16]. According to Tabar et al.[16], the reason for this difference between the effects of wood flour and WSF could be the fibrous nature and different chemical composition of wood. In addition, the long fibres of the PWF compared to WSF provided more significant reinforcing effects in the WPCs produced because of their higher aspect ratio. The differences in the fibre morphologies, chemical contents, densities, cellulose–lignin contents, and aspect ratios across different filler types accounted for the varying reinforcement in lignocellulosic–plastic composites[40].

Stress–strain curves based on the tensile tests of groups 1, 4, and 7 are given in Figure 4. An analysis of the curves shows that the curves for the test samples of group 1 (unfilled group) are very similar to each other. The elongation at break of group 1 is 25.6%, as can be seen in Table 4. In contrast, the elongation-at-break values of groups 4 and 7 are 4.7% and 4.5%, respectively. The area under the curve of group 1 is larger than those for groups 4 and 7. This area is also

Table 4. Tensile test data, ANOVA P values, and Duncan test results.

Test	Groups							P values
	1	2	3	4	5	6	7	
Tensile strength (N/mm²)	23.6**A***	15.8**B**	13.3**C**	12.1**D**	15.0**B**	10.9**E**	9.7**F**	P<0.001
	*0.4***	*1.5*	*1.7*	*1.7*	*1.3*	*1.0*	*1.7*	
Tensile modulus (N/mm²)	335**BC**	344**AB**	356**AB**	367**A***	305**C**	311**C**	329**BC**	P<0.001
	38.3	*12.0*	*24.4*	*22.7*	*18.2*	*27.5*	*85.5*	
Elongation at break (%)	25.6**A****	7.3**B**	5.9**C**	4.7**C**	8.4**B**	5.4**C**	4.5**C**	P<0.001
	2.4	*1.3*	*2.5*	*0.8*	*1.4*	*2.7*	*1.2*	

*Means followed by the same letter are not significantly different with each other using Duncan multiple comparison test at $\alpha = 0.05$. **Standard deviation in italic form. ***Highest value.

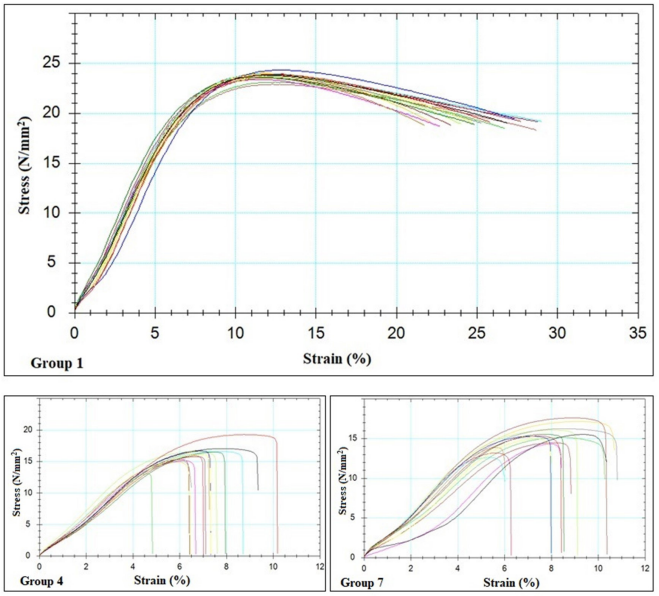

Figure 4. Stress–strain curves based on tensile tests (group 1, group 4, and group 7).

Table 5. The composition (%) of the walnut shell and black pine wood.

	Cellulose	Hemicellulose	lignin	ash	
Walnut shell	25.6	22.1	52.3	2.8	Demirbaş[46]
	25.4	21.2	49.1	3.6	Rao[41]
	26.51	21.27	49.18	2.13	Ayrılmış et al.[30]
	Holocellulose	**α Cellulose**	**lignin**	**ash**	
Black pine wood	73.5	50.2	25.4	-	Pekgözlü et al.[42]
	72.3	-	26.4	0.18	Kırcı and Ateş[43]
	71.5	50.4	26.7	0.2	Kılıç et al.[44]

an indicator of the toughness value. A larger area indicates greater ductility. The toughness of the composite material changed when the PWF or WSF filler was added to the compound. Group 1 (HDPE) exhibited necking before fracture, but this phenomenon is not observed for the filled composites (groups 4 and 7). It is known that necking is a mode of ductile flow of material under tension. It can be said that the more necking the material has, the more ductile it is.

The brittleness of the composite material increased with the amount of filler, as can be seen in Table 4 and Figure 4. According to Matuana and Stark[22], the mechanical properties of a WPC comply with the rule of mixtures. Increasing the amount of lignocellulosic filler in the polymeric matrix decreased the ductile behaviour of the WPC by making the material more brittle. Therefore, it can be said that the PWF or WSF filler material increased the brittleness of the composite material.

In this study, differences were determined between the mechanical properties of the groups filled with WSF and those filled with PWF. There are many reasons for this difference. The particle and fiber-based composites' mechanical properties depend on the particle size, fiber length or aspect ratio, degree of dispersion, interfacial adhesion, and particle loading[41-45]. In addition, according to Tabar et al.[16], the reason for this difference between the effects of wood flour and WSF could be the fibrous nature and different chemical composition of wood. Table 5 shows the data obtained as a result of chemical analyses made by different researchers. According to these data; it is clearly seen that the chemical composition of WSF and PWF are different. Walnut shell has more lignin and less cellulose than pine wood. Lignin and cellulose are quite different compounds from each other. Lignin has a brittle structure and cellulose has a ductile structure. Therefore, composites with high lignin-containing filler material have a more brittle structure[18,30,41,47]. When the values given in Table 5 are examined, it can be said that the high lignin content of the walnut shell increases the brittleness of the composite material. In addition, it can be said that another reason why the bending strength and tensile strength of composites filled with PWF are higher than those filled with WSF, is the fibrous structure of PWF.

4 Conclusions

In this study, the effects of PWF and WSF on the mechanical properties of wood–high-density polyethylene composite boards produced with different filler contents were investigated. According to the data obtained, the following conclusions can be drawn for the filled composites compared to unfilled composites.

1. WPC boards were successfully produced with PWF and WSF. The density of the WPC boards increased with the filler content;
2. The flexural and tensile strengths of test samples from the filled groups decreased as the filler content increased. The reduction effect of the WSF on these strengths was greater than that of the pine-wood flour;
3. The flexural modulus values of test samples from the filled groups increased with the filler content. The increasing effect of the WSF on this property was greater than that of the pine-wood flour. In contrast, the tensile modulus values of the groups filled with WSF were lower;
4. Contrary to previous studies, the use of WSF as a filler material in a polymer composite is not recommended considering all the test results obtained.

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