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Utilization of olive mill sludge in manufacture of lignocellulosic/polypropylene composite

Nadir Ayrilmis · Umit Buyuksari

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Abstract This study evaluated some physical and mechanical properties of injection-moulded polypropylene (PP) composites reinforced with various mixtures of the wood flour (WF) and the olive mill sludge (OMS). Water resistance of the composites improved by the increasing OMS flour content. However, the flexural properties of the composites decreased with increasing OMS flour content. The addition of a coupling agent improved the compatibility between the lignocellulosic material and PP through esterification and thus reduced the water absorption and improved the stability and mechanical properties. The findings obtained in the study showed that the OMS was capable of serving as new reinforcing filler in the manufacturing of thermoplastic polymer composites.

Introduction

Olive mill sludge (OMS), wet olive pomace, is the solid and wet lignocellulosic by-product generated in massive quantities by olive-oil extraction industries which use the two-phase centrifugation method (Fig. 1). The OMS is obtained by pressing the fruit, leaving a residue of seed husks (fragmented olive stones), seed, pulp, and peel. The olive fruit consists of pulp (70–90%), stone (9–27%), and seed (2–3%) on a total weight basis [1]. The refined OMS differs from the crude OMS mainly by lower oil content and smaller water content because it has been dehydrated during the oil extraction process. The OMS oil is extracted from the crude OMS using chemical solvents, mostly hexane, and by heat. After the extraction process, the OMS has 0.1–0.3% olive oil based on the oven-dry weight of the OMS [2].

The olive oil industry is very important in Mediterranean countries, both in terms of wealth and tradition. Spain is the main world producer followed by Italy, Greece, Turkey, Syria, and Tunisia. Large amounts of the OMS are produced in the Mediterranean region, an area that accounts for 95% of the total olive oil production worldwide. Countries of the European Union (EU) like Italy, Spain, and France produce about 70% of olive in the world. Turkey, Tunisia, Syrian Arab Republic, and Morocco are the other important producers. Spain, the leading olive-oil producer, produces about 4 million tones of the OMS per year [3]. According to the data of Institute of Olive Researches, 15–22 kg of olive oil and 35–45 kg of the OMS are obtained for a 100 kg of olive [2]. This given amount of the OMS is based on wet basis. The chemical composition of OMS (two phase system) is presented in Table 1 [4].

The OMS has greatly contributed to environmental degradation for the following reasons: (a) the conversion from classic-type mills (3.25 kg of OMS per kg of the olive oil produced) to centrifuge-type mills (5 kg of the OMS per kg of the olive oil produced), (b) a huge increase in the olive oil production [5]. It was reported that the total OMS production is average 200–250 thousand/year in Turkey [6]. Although the majority of the OMS produced in evaporation ponds is disposed of in landfill sites [5], the remainder is used as a heat source because of its oil content in the Aegean and the Southeastern Anatolian region of Turkey [7]. With the continued generation of these

N. Ayrilmis (✉) · U. Buyuksari
Department of Wood Mechanics and Technology, Istanbul University, Forestry Faculty, Bahcekoy, Sariyer, 34473 Istanbul, Turkey
e-mail: nadiray@istanbul.edu.tr

U. Buyuksari
e-mail: buyuku@istanbul.edu.tr

Fig. 1 Wet olive mill sludge (OMS) generated in massive quantities by olive-oil extraction in the olive oil mill

Table 1 The chemical composition of olive mill sludge (two-phase system)

The chemical composition	Value
Moisture content (%)	56.80 ± 2.20
Fat and oils (%)	4.65 ± 1.74
Proteins (%)	2.87 ± 0.01
Total sugars (%)	0.83 ± 0.01
Cellulose (%)	14.54 ± 0.17
Hemicellulose (%)	6.63 ± 0.37
Lignin (%)	8.54 ± 0.18
Ash (%)	1.42 ± 0.09
Kjeldahl nitrogen (%)	0.43 ± 0.006
Phosphorous as P_2O_5 (%)	0.04 ± 0.003
Phenolic compounds (%)	2.43 ± 0.15
Potassium as K_2O (%)	0.32 ± 0.027
Calcium as CaO (%)	0.37 ± 0.036
Total carbon (%)	25.37 ± 2.03
C/N ratio	59.68 ± 5.25

residues, the need for proper utilization of viable management strategies is imperative. Growing demand for the lignocellulosic/plastic composite has led to continuous efforts to find new resources as an alternative to wood. Value-added lignocellulosic/plastic composites made from the industrial wastes can be considered as an alternative solution to this problem. One of these residuals is the OMS, which is produced in high quantities in the olive oil industry.

In recent years, ligno-cellulose materials and industrial wastes such as rice-husks, bagasse, palm oil, wood chips, paper sludge, waste tire have been widely used as reinforcing fillers in the reinforced polymer composite [8–16]. The reinforced polymer composites made using ligno-cellulose materials and industrial wastes as fillers have several advantages, such as their low cost, renewability, biodegradability, and absence of associated health hazards [17]. These natural fillers are lighter, cheaper, and provide much higher strength per unit mass than most inorganic fillers such as carbon black, calcium carbonate, talc, and zinc oxide [18].

An extensive literature search did not reveal any information about the OMS flour as an alternative to the wood flour in the manufacture of wood plastic composite. From the literature, we know that physical and mechanical properties of the lignocellulosic/plastic composites can be influenced by raw material characteristics [8–14]. The objective of this study was to determine some physical and mechanical properties of the polypropylene (PP) composites reinforced with various mixtures of the wood flour and the OMS flour and to evaluate the compatibilizer performance.

Experimental

Materials

The olive mill sludge (OMS) containing 0.1–0.3% oil, was supplied from a commercial olive mill manufacturer located in Edremit, Balikesir, Western Turkey. First the OMS were dried in an oven at 60 °C for 10 h to moisture content of 20–30% based on the oven-dry OMS solid weight. Following the drying, the OMS were then processed by a rotary grinder without adding additional water. Finally, the OMS flour passing through a U.S. 35-mesh screen and was retained by a U.S. 80-mesh screen. The OMS flour was then dried in a laboratory oven at 100 °C for 24 h to moisture content of 1–2%.

Wood particles (a 50:50 blend) consisting of pine (*Pinus nigra Arnold var. pallasiana*) and beech (*Fagus orientalis* Lipsky) species were obtained from a commercial particleboard plant in Turkey. The moisture content of the particles, as determined by oven-dry weight, was found to be 4–5% prior to the treatment. The wood particles were processed by a rotary grinder without adding additional water. The wood flour passing through a U.S. 35-mesh screen was retained by a U.S. 80-mesh screen and then dried in a laboratory oven at 100 °C for 15 h to moisture content of 1–2%.

The PP (T_m = 160 °C, ρ = 0.9 g/cm^3, MFI/230 °C/2.16 kg = 6.5 g/10 min) produced by *Petkim Petrochemical* Co., Turkey, was used as the polymeric material. Maleic anhydride-grafted PP (MAPP-OPTIM-415; the reactive modifier maleic anhydride (MAH) content = 1 wt%) was supplied by *Pluss Polymers* Pvt. Ltd. in India.

Lignocellulosic/polypropylene composite manufacture

The wood flour and OMS flour were dried to 1–2% moisture content using in an air dryer oven at 100 °C for 24 h and then stored in a polyethylene bag in an environmental controller. The wood flour, the OMS flour, and the PP and the MAPP granulates were processed in a 30-mm conical co-rotating twin-screw extruder (*Aysa Instrument Com*, Istanbul, Turkey) with a length-to-diameter (*L/D*) ratio of 30:1. The raw materials were fed into the main feed throat using a gravimetric feed system. The barrel temperatures of the extruder were controlled at 170, 180, 190, and 190 °C for zones 1, 2, 3, and 4, respectively. The temperature of the extruder die was held at 200 °C. The extruded strand passed through a water bath and was subsequently pelletized. These pellets were stored in a sealed container and then dried for about 3–4 h before being injection molded. The temperature used for injection molded samples was 170–190 °C from feed zone to die zone. The WPC samples were injected at injection pressure between 45 and 50 kg/m^2 with cooling time about 30 s. Finally, the samples were conditioned at a temperature of 23 ± 2 °C and relative humidity of 50 ± 5% according to ASTM D 618-08 [19]. The formulations of the composites are given in Table 2. Air-dry density values of the samples varied from 1.00 to 1.04 g/cm^3.

Determination of water resistance

The thickness swelling (TS) and water absorption (WA) tests were carried out according to ASTM D 570-05 specifications [20]. The test samples were in the form of a disk 50.8 mm in diameter and 3.2 mm in thickness. Ten replicate samples were tested for each WPC formulation. The conditioned samples were placed in a container of distilled water maintained at a temperature of 23 ± 1 °C. The weights and thicknesses of the samples were measured at different time intervals during the long period of immersion. At the end of 2-, 24-, 48-, and 72-h of submersion, the samples were removed from the water one at a time, all surface water were wiped off with a dry cloth, and weighed to the nearest 0.001 g and measured to the nearest 0.001 mm immediately. The values of the WA as percentages were calculated with Eq. 1:

$$\mathrm{WA}_{(t)} = \frac{W_{(t)} - W_0}{W_0} \times 100 \quad (1)$$

where $\mathrm{WA}_{(t)}$ is the water absorption (%) at time t, W_0 is the initial weight of the sample, and $W_{(t)}$ is the weight of the sample at a given immersion time t.

The values of the TS as percentages were calculated with Eq. 2:

$$\mathrm{TS}_{(t)} = \frac{T_{(t)} - T_0}{T_0} \times 100 \quad (2)$$

where $\mathrm{TS}_{(t)}$ is the thickness swelling (%) at time t, T_0 is the initial thickness of the sample, and $T_{(t)}$ is the thickness at time t. Density of the samples was measured on the TS samples.

Determination of flexural properties

The flexural properties, modulus of rupture (MOR) and modulus of elasticity (MOE), were measured in three-point bend tests using a standard Material Testing System (Zwick Z010 with 2.5 kN load cell) at a crosshead speed of 2.8 mm/min in accordance with ASTM D 790-03 [21]. The MOR and MOE of the samples with dimensions of 127 mm × 12.7 mm × 3.2 (thickness) mm were determined at ambient conditions of 23 ± 2 °C and 50 ± 5% relative humidity according to ASTM D 618-08 [19]. Five replicate samples were tested for each WPC formulation.

Table 2 Compositions of the evaluated formulations

WPC formulation code	Wood flour content	Olive mill sludge (OMS) content	Polypropylene (PP)	Maleic anhydride-grafted polypropylene (MAPP)
WPC1	40	0	60	0
WPC2	40	0	57	3
WPC3	30	10	60	0
WPC4	30	10	57	3
WPC5	20	20	60	0
WPC6	20	20	57	3
WPC7	10	30	60	0
WPC8	10	30	57	3
WPC9	0	40	60	0
WPC10	0	40	57	3

Results and discussion

Water resistance

The TS and WA values of the samples significantly decreased with increasing OMS flour content (Table 3). Statistical analysis found some significant differences ($p < 0.01$) among the WPC means for the TS and WA values. Significant differences were determined individually for these tests by Duncan's multiple-comparison tests. The results of Duncan's multiple range test are shown by letters in Table 3. The lowest TS value was 0.31% for the WPC10, while the highest TS value was found as 0.89% for the WPC1 type after 2-h of submersion in water. The similar trends were also observed for 24-, 48-, and 72-h of submersions. The WA values of the samples showed the similar tendency to the results of the TS. The WA values of the composites were higher than the TS values. Hardboard (density $\geq$ 800 kg/m^3) standard ANSI/AHA A135.4 [22] was used here for comparison of the TS and the WA values since there was no established minimum property for wood plastic composite. The TS and WA values of all composite types did not exceed the hardboard (3.2 mm thickness) minimum property requirements of 20% (TS) and 25% (WA) according to ANSI/AHA A135.4 Standard, respectively. The TS and WA values of the samples were also much less than those of particleboard, oriented strandboard, and medium density fiberboard because the matrix polymers are hydrophobic [23]. In a previous study, average WA and TS values of MDF panels after 24-h of submersion were found as 15.9 and 6.7%, respectively [24].

The TS and WA values of the samples significantly decreased with increasing OMS content in the composite, while they increased with increasing wood flour content (Figs. 2, 3). The TS and WA values of the samples containing the OMS flour were less than those of the samples made with wood flour (among composites without the MAPP). Table 3 also shows that the samples containing the MAPP exhibited lower TS and WA than those made without the MAPP. Among the composites containing the MAPP, the composites without the OMS exhibited the higher TS. In general, in the higher amounts of the OMS (above 20%) in the composite, there was no difference between the TS/WA of the composites without the MAPP and the TS/WA of those made with the MAPP.

The impact of wood to plastics ratio on the TS and WA properties can be explained by the differences in water absorption between wood and plastics. With increasing the wood flour content in the composite, there are more water residence sites thus more water is absorbed [8]. On the other hand, the composites made from higher plastic content had less water absorption sites and thus decreased

Table 3 Results of the water resistance of the composites

Composite code	Composite density (g/cm^3)	Water resistance								
		Water absorption (%)				Thickness swelling (%)				
		2-h	24-h	48-h	72-h	2-h	24-h	48-h	72-h	
WPC1	1.01	1.15 (0.11) A[a]	1.31 (0.23) A	1.72 (0.23) A	1.77 (0.22) A	0.89 (0.20) A	1.11 (0.23) A	1.22 (0.21) A	1.36 (0.16) A	
WPC2	1.03	0.94 (0.20) AB	1.18 (0.19) C	1.36 (0.19) BC	1.48 (0.24) BC	0.79 (0.15) ABC	0.92 (0.17) BC	1.01 (0.19) BC	1.16 (0.23) BC	
WPC3	1.02	1.02 (0.19) A	1.25 (0.21) B	1.46 (0.46) B	1.59 (0.21) AB	0.84 (0.14) AB	1.04 (0.27) AB	1.07 (0.16) B	1.24 (0.34) AB	
WPC4	1.00	0.75 (0.11) CD	0.95 (0.14) D	1.23 (0.14) D	1.31 (0.22) DEG	0.66 (0.11) CD	0.79 (0.09) D	0.88 (0.11) BC	0.96 (0.32) DE	
WPC5	1.02	0.82 (0.11) BC	1.1 (0.22) C	1.29 (0.24) C	1.38 (0.19) CD	0.72 (0.32) BC	0.86 (0.41) C	0.97 (0.32) BC	1.13 (0.19) CD	
WPC6	1.02	0.69 (0.10) DE	0.86 (0.21) D	1.16 (0.22) DE	1.23 (0.30) EFI	0.59 (0.21) DE	0.74 (0.16) DE	0.82 (0.05) CD	0.90 (0.11) EF	
WPC7	1.01	0.61 (0.16) EF	0.77 (0.01) E	0.91 (0.12) EF	1.12 (0.11) FGH	0.52 (0.13) EF	0.66 (0.10) EF	0.75 (0.12) DE	0.82 (0.15) F	
WPC8	1.01	0.52 (0.09) FG	0.67 (0.05) EF	0.82 (0.1) FG	0.98 (0.1) HI	0.45 (0.08) EF	0.61 (0.10) F	0.70 (0.06) E	0.76 (0.05) G	
WPC9	1.02	0.47 (0.3) GH	0.60 (0.14) F	0.75 (0.08) G	0.86 (0.08) HI	0.39 (0.06) EF	0.55 (0.11) FG	0.63 (0.07) E	0.69 (0.51) GH	
WPC10	1.04	0.42 (0.4) H	0.52 (0.06) G	0.68 (0.07) H	0.79 (0.05) I	0.31 (0.05) F	0.48 (0.07) G	0.54 (0.06) E	0.61 (0.13) H	

Values in parentheses are standard deviations

[a] Groups with same letters in column indicate that there was no statistical difference ($p < 0.01$) between the samples according to the Duncan's multiply range test

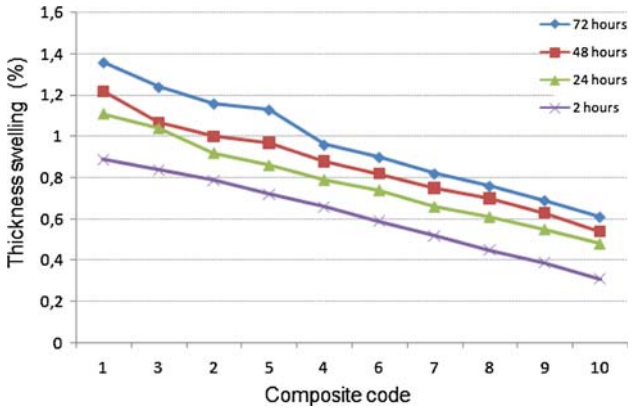

Fig. 2 Thickness swelling curves for all formulations

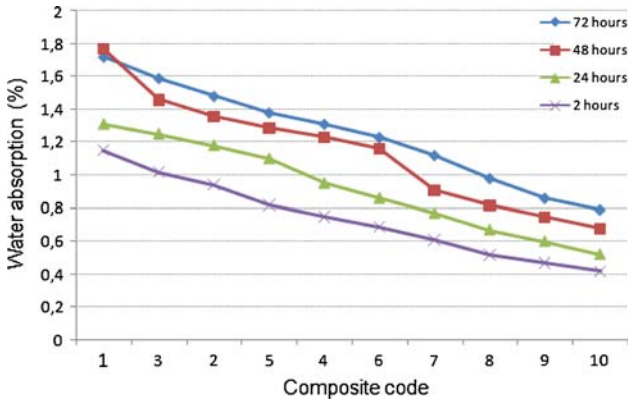

Fig. 3 Water absorption curves for all formulations

water absorption. The presence of hydroxyl and other polar groups in various constituents of the wood flour resulted in poor compatibility between the hydrophilic wood flour and the hydrophobic plastics, which increased the water absorption. The moisture absorption in the composites is mainly due to and hydrogen bonding sites in the wood flour. Water absorption by cellulose and hemicelluloses depends on the gaps and flaws at the interfaces, and the micro-cracks in the matrix formed during the compounding process, the presence of lumens, fine pores, and the number of free hydroxyl groups thus the amorphous regions are accessible by water [25]. The WPC composites have potential to take up water under humid conditions due to the presence of numerous hydroxyl groups available for interaction with water molecules via hydrogen bonding [26]. According to Stark [27], less encapsulation occurs with WPC having higher wood fine content, wood easily expose on the surface of the WPC. Therefore, higher amount of water absorption was observed in those WPC. The differences in water absorption can be also related to the chemical structures of the fillers. The lower TS in the composites containing higher amounts of the OMS flour can be related to lower amounts of hygroscopic materials, cellulose and hemicelluloses, in the cell walls of the OMS filler (Table 1). This can explain why the composites containing large amounts of the OMS absorbed lower water.

Generally, it is necessary to use compatibilizers or coupling agents to improve the filler/fiber bonding and in turn to enhance the WA. The compatibilizing agents have a positive effect on the WA [23]. The strong interfacial bonding between the filler and the polymer matrix caused by the compatibilizing agents (the MAPP chemically bonds with the OH groups in the lignocellulosic filler) limits the WA of the composites. The coupling agents improve the quality of adhesion between plastics and fibers to reduce the gaps in the interfacial region and to block the hydrophilic groups [23]. With the addition of the MAPP (3 wt%), the compatibility between the lignocellulosic material and the PP improved because the anhydride moieties in the MAPP entered into an esterification reaction with the surface hydroxyl groups of the lignocellulosic material [28]. However, a larger amount of the OMS negatively influenced the MAPP performance and caused lower bonding performance between the PP and the filler.

Flexural properties

The average MOR and MOE values of the composites measured using 3-point bending tests are shown in Table 4. Statistical analysis found some significant differences ($p < 0.01$) between some panel means for the MOR and MOE values. Significant differences among the composite types were determined individually for these tests by Duncan's multiple-comparison tests (Table 4). The MOR significantly increased with increasing wood flour content in the structure (Fig. 4). The highest MOR value was 55.8 N/mm^2 for the WPC2 while the lowest MOR value was found for the WPC9 having a value of 37.5 N/mm^2. The MOE values showed parallel tendency to the results of the MOR (Fig. 5).

The flexural properties of the composites were negatively affected by the addition of the OMS flour. The MOR values of the WPCs significantly decreased with increasing OMS flour content from 10 to 40% in the composite. For example, the average MOR value of the composite made from 40% wood flour (composite code: WPC1) was 50.7 N/mm^2 as compared to that made from 40% OMS flour (composite code: WPC9) which is about 37.5 N/mm^2. The MOR and MOE of the composites made using the MAPP were higher than those of the composites at the same plastic to wood ratio. In a previous study, MOR and MOE values were found as 44.2 and 3000 N/mm^2 for WPCs made from 60% PP and 40% hardwood fiber while the same properties were found as 72.4 and 3220 N/mm^2 for the WPCs containing 3% coupling agent, respectively [23].

Table 4 Results of the flexural properties of the composites

Composite code	Composite density (g/cm^3)	Flexural properties	
		Modulus of rupture (N/mm^2)	Modulus of elasticity (N/mm^2)
WPC1	1.01	50.7 (2.05) B[a]	4170.1 (84.9) C
WPC2	1.03	55.8 (3.51) A	4554.7 (110.6) A
WPC3	1.02	46.2 (1.62) CD	3846.5 (89.7) DE
WPC4	1.00	52.3 (1.14) B	4287.1 (105.7) BC
WPC5	1.02	44.5 (1.99) DE	3737.4 (80.5) EF
WPC6	1.02	47.4 (1.45) C	4067.8 (101.1) CD
WPC7	1.01	41.1 (1.16) FG	3433.7 (94.7) GH
WPC8	1.01	43.2 (1.26) EF	3565.4 (49.2) FG
WPC9	1.02	37.5 (0.36) H	3124.9 (109.8) I
WPC10	1.04	40.4 (0.64) G	3244.3 (145.7) HI

Values in parentheses are standard deviations

[a] Groups with same letters in column indicate that there was no statistical difference ($p < 0.01$) between the samples according to the Duncan's multiply range test

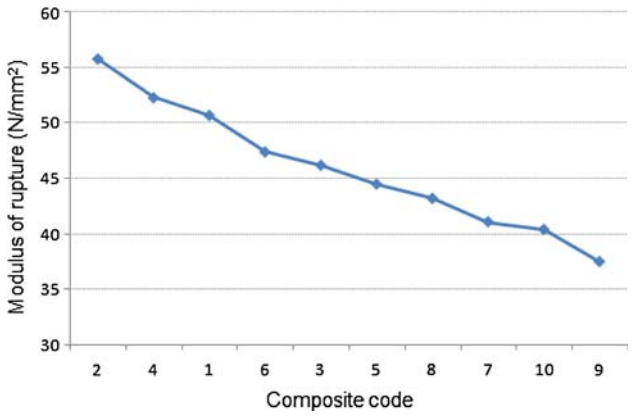

Fig. 4 Modulus of rupture curves for all formulations

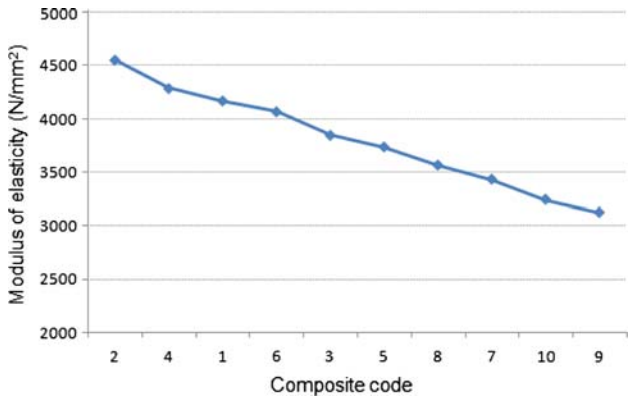

Fig. 5 Modulus of elasticity curves for all formulations

Wood is a lignocellulosic material made up of three major constituents (cellulose: 42–44%, hemicelluloses: 27–28%, and lignin: 24–28%) with some minor constituents (extractives: 3–4%) [29]. The major portion of the wood is crystalline cellulose. The aligned fibril structure of the cellulose along with strong hydrogen bond has high stiffness thus addition of the wood flour can increase the stiffness of the thermoplastic based composites. Lignin as an amorphous polymer does not greatly contribute to the mechanical properties of the wood flour but plays an important role in binding the cellulose fibrils that allows efficient stress transfer to the cellulose molecules. Hence, the wood filler increases the stiffness of the PP without excessively increasing the density [26]. The lower MOR and MOE values of the composites containing higher amounts of the OMS flour can be related to lower amounts of cellulose and lignin materials in the cell walls of the OMS filler (Table 1). The addition of the coupling agent improved the compatibility between the lignocellulosic material and the PP through esterification and thus reduced the WA and improved the dimensional stability and mechanical properties.

Conclusions

The findings obtained in the study showed that the OMS was capable of serving as new reinforcing filler in the manufacturing of thermoplastic polymer composites, which will reduce cost and give environmental benefits. The higher OMS contents exhibited lower water absorption and thickness swelling because of lower hygroscopicity of the OMS flour. However, the flexural properties of the composites decreased with the increasing OMS flour content. In general, the addition of the MAPP had not a significant effect on the water resistance and flexural properties of the composites made with higher OMS flour contents. Therefore, it can be said that at higher OMS contents, although the compatibilizer improves the water resistance by limiting maximum water absorption, it has not a significant effect on the rate of the flexural properties. Based on the findings obtained in the present study, a 40/60 formulation of the OMS flour and PP appears to a practical choice for applications requiring a higher water resistance such as outdoor deck flooring and window frames.

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