Thermal and Mechanical Properties of Polypropylene/ Wood-Flour Composites

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ABSTRACT: In this research, polypropylene/wood-flour composites (WPCs) were blended with different contents of wood and/or maleated polypropylene (MAPP) and clay. We found that the addition of MAPP or clay in the formulation greatly improved the dispersion of the wood fibers in the composite; this suggested that MAPP or clay may have played the role of an adhesion promoter in the WPCs. The results obtained with clay indicate that it also acted as a flame retardant. The thermal tests carried out with the produced samples showed an increased crystallization temperature (T_c), crystallinity, and melting temperature (T_m) with wood loading. The increase of the two former parameters was explained by the incorporation of wood flour, which played the role of nucleating agent and induced the crystallization of the matrix polymer. On the other hand, the T_m increase was ascribed to the insulating properties of wood, which hindered the movement of heat conduction. The effects of UV irradiation on T_m and T_c were also examined. T_c increased with UV exposure time; this implied that UV degradation generated short chains with low molecular weight that could move easily in the bulk of the sample and, thus, catalyze early crystallization. The flexural strength and modulus increased with increasing wood-flour content. In contrast, the impact strength and tensile strength and strain decreased with increasing wood-flour content. All of these changes were related to the level of dispersion of the wood flour in the polymeric matrix. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 119: 3321–3328, 2011

Key words: composites; crystallization; mechanical properties; nucleation; thermal properties

INTRODUCTION

Composites made from blends of thermoplastics and natural fibers have gained popularity in a variety of applications because they combine the desirable durability of plastics with the cost effectiveness of natural fibers as fillers or reinforcing agents.[1–3] The addition of natural fibers, such as wood fibers, into the plastic matrix leads to an improvement in the stiffness of the composite and decreases in the abrasiveness on processing equipment and density of the product compared to mineral fillers. Because of these attributes, polypropylene/wood-flour composites (WPCs) are used in a variety of innovative applications, such as decking, docks, landscaping timbers, fencing, playground equipment, and window and door frames.[1–5] Despite these attributes, the compatibility between the wood fibers and polymeric matrix constitutes one important factor in the production of WPCs with improved mechanical properties.[5–10] Wood is hydrophilic in nature (with a high surface tension), which lowers its compatibility with hydrophobic polymeric materials (with a low surface tension) during composite preparation; this leads to WPCs with poor dispersions of wood fibers.[5,7–10]

Substantial research has been carried out on the surface modification of wood fibers with coupling agents to improve the strength properties of WPCs.[3,5–10] Among these, the addition of maleated polypropylene (MAPP) in polypropylene (PP)-based WPCs has been shown to appreciably improve the dispersion of fibers in the matrix and the mechanical properties of WPCs because of the formation of linkages between the OH groups of wood and maleic anhydride.[5,10] Many in-depth studies have elucidated the mechanisms of adhesion between MAPP-treated wood fibers and the PP matrix that cause the improvement.[5–10] Recently, investigations have

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shown that the mechanical properties of WPCs can also be significantly improved by the addition of nanoclay to the composites.[3,11] The use of clay instead of MAPP is interesting in terms of the fire retardancy of the WPCs, which tend to burn quite easily; this is a heavy drawback.

Although the influence of the surface treatment of wood flour with MAPP on the strength properties of WPCs has been extensively investigated,[5–10] little information is available in the open literature on the effect of combining MAPP with nanoclay on the thermal and mechanical properties of WPCs. Therefore, in this study, we examined the effects of combining MAPP and nanoclay, as a coupling agent, and the wood-flour content on the mechanical properties and thermal behaviors of the WPCs. In this study, we aimed to identify the synergistic effect between MAPP and nanoclay in enhancing the properties of the WPCs.

Emphasis was also placed on the study of the photoaging behavior of WPC specimens because, in several of their applications (dashboards or screen doors in automobiles, floor covering in the construction industry, profiles for doors and windows, ornamental panels, etc.), they are used under conditions where they experience environmental degradation.[1,2,4,12] Because WPCs are very sensitive to photoaging, another goal of this study was to examine the evolution of the aforementioned mechanical and thermal properties in UV-degraded WPCs.

EXPERIMENTAL

Materials

Pine wood particles of 425 mm (40-mesh size) supplied by American Wood Fibers (Schofield, WI) were used as wood flour. PP from the Eastman Chemical Co. (Kingsport, TN) was used as the matrix. It had a melt flow index of 5.2 g/10 min (at 190°C and a 2.16 kg load) and a density of 0.910 g/cm^3. MAPP (G-2010), supplied by Eastman Chemical Co., was used as the coupling agent. In some samples, nanoclay (Cloisite 10A, Texas) from Southern Clay was added to the formulation. This nanoclay was organically modified with dimethyl, benzyl, hydrogenated tallow, and quaternary ammonium (with modifier concentrations of 125 mequiv/100 g of clay). All ingredients were used as received.

Compounding and processing

Before compounding, the wood flour was dried in an oven for at least 48 h at 105°C to a moisture content of less than 1%. The dried wood flours were stored in a sealed plastic container to prevent the absorption of water vapor. The PP matrix, dried wood flour, MAPP, and/or clay were added to a high-intensity mixer (Papenmeier, TGAHK20, Germany) and dry-blended at room temperature for 10 min. After blending, the compounded materials were stored in a sealed plastic container. Several formulations were produced with various contents of wood flour, MAPP, and clay (Table I). For the mechanical property experiments, test specimens were molded in a 33-Cincinnati Milacron reciprocating screw-injection molder (Batavia, OH). The nozzle temperature was set to 204°C. The extrudate, in the form of strands, was cooled in the air and pelletized. The resulting pellets were dried at 105°C for 24 h before they were injection-molded into the ASTM test specimens for flexural, tensile (Type I, ASTM D 638), and Izod impact strength testing. The dimensions of the specimens for the flexural tests were 120 × 3 × 12 mm^3 (Length × Thickness × Width).

TABLE I
Compositions of the WPCs

	Composition			
	PP (%)[a]	Wood flour (%)[a]	MAPP (%)[a]	Clay (%)[a]
WPPC0	100	0		
WPPC1	95	5		
WPPC3	75	25		
WPPC4	50	50		
WPPG1	70	25	5	
WPPG2	65	25	10	
WPPG3	60	25	15	
WPPG4	55	25	20	
WPPCC	45	50	0	5
WPPCG	45	50	5	0
WPPCCG	45	50	2.5	2.5

WPPG = wood polypropylene maleated polypropylene; WPPG1 = sample n° 1 (70°/°PP, 25°/°wood and 5°/° MAPP); WPPG2 = sample n° 2 (65°/°PP, 25°/°wood and 10°/° MAPP); WPPG3 = sample n° 3 (60°/°PP, 25°/°wood and 15°/° MAPP).

[a] The percentage is based on the total weight of the composite.

Property evaluation

Scanning electron microscopy (SEM)

The state of dispersion of the wood flour in the polymeric matrix was analyzed with SEM. A FEI Quanta 400 microscope (NE Dawson Creek Drive, Hillsboro, Oregon) working at 30 kV was used to obtain microphotographs of the fractured surfaces of the composites. Samples were cut in liquid nitrogen to avoid any deformation of the surfaces.

Differential scanning calorimetry (DSC)

DSC is widely used to characterize the thermal properties of WPCs. DSC can measure important

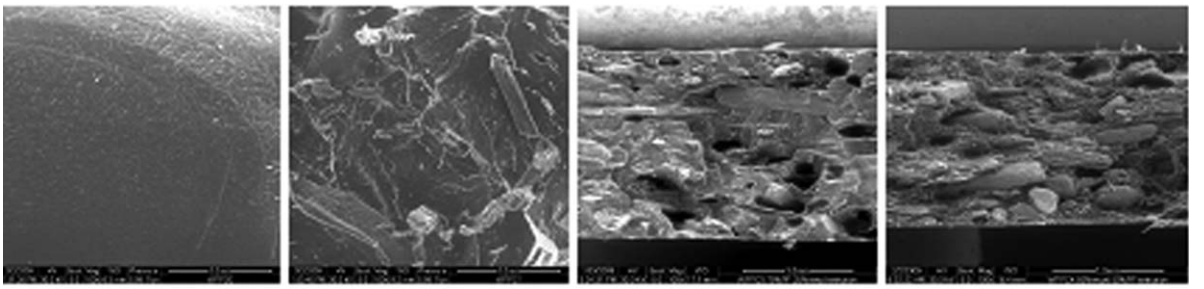

Figure 1 SEM micrographs of (a) pure PP and composites containing (b) 5, (c) 25, and (d) 50 wt % wood flour.

thermoplastic properties, including the melting temperature (T_m), heat of melting, degree of crystallinity [χ (%)], crystallization, and presence of recyclates/regrinds, nucleating agents, plasticizers, and polymer blends (the presence, composition, and compatibility). Thermal analysis of the WPC samples was carried out on a differential scanning calorimeter (PerkinElmer Instruments, Pyris Diamond DSC, Shelton, Connecticut) with the temperature calibrated with indium. All DSC measurements were performed with powdered samples of about 9.5 ± 0.1 mg under a nitrogen atmosphere with a flow rate of 20 mL/min. Three replicates were run for each specimen. All samples were subjected to the same thermal history with the following thermal protocol, which was slightly modified from the one reported by Valentini et al.[13]:

1. First, the samples were heated from 40 to 180°C at a heating rate of 20°C/min to erase the thermal history.
2. Second, the samples were cooled from 180 to 40.00°C at a cooling rate of 10°C/min to detect the crystallization temperature (T_c).
3. Finally, the samples were heated from 40 to 180°C at a heating rate of 10°C/min to determine T_m.

T_m and the heat of fusion (ΔH_m) were calculated from the thermograms obtained during the second heating. The values of ΔH_m were used to estimate χ, which was adjusted for each sample.

Mechanical properties

Tensile tests (tensile strength and tensile strain) and three-point flexural tests (flexural modulus and flexural strength) were carried out on an Instron 5585H testing machine (Norwood, MA) with crosshead rates of 12.5 and 1.35 mm/min according to the procedures outlined in ASTM standards D 638 and D 790, respectively. Eight replicates were run to obtain an average value for each formulation. Before each test, the films were conditioned in a 50% relative humidity chamber at 23°C for 48 h.

The Charpy impact strength was measured with an Instron impact pendulum tester (model PW5) according to ASTM D 256 with acutely notched specimens (notch depth = 2 mm) at room temperature. Each mean value represented an average of eight tests. The *impact strength* is defined as the ability of a material to resist the fracture under stress applied at a high speed. The impact properties of composite materials are directly related to their overall toughness. In the Charpy standard test, the only measured variable is the total energy required to break a notched sample.

UV irradiation

Irradiation in conditions relevant to outdoor exposure ($\lambda > 300$ nm) in the presence of air was carried out in a SEPAP (Service d'Etudes du Photovieillissement Accéléré des Polymères, Clermont-Ferrand, France) 12/24 unit working at a temperature of 60°C for 200 h. Details on this artificial accelerated photoaging device were given elsewhere.[14]

RESULTS AND DISCUSSION

Characterization of the initial properties of the WPC samples

Micrographs taken from the fractured surface of the specimens showed different organization of the fibers in the composites, depending on the content of wood flour and/or the presence of MAPP or clay (Figs. 1 and 2). The surface of pure PP was homogeneous [Fig. 1(a)], whereas microparticles appeared when wood flour was added to the formulations [Figs. 1(b–d)]. The incorporation of filler into the polymer matrix disrupted the homogeneity of the matrix. At a 5% wood-flour content, the matrix encapsulated the fiber surface quite well [Fig. 1(b)]. The composites exhibited interfacial debonding with

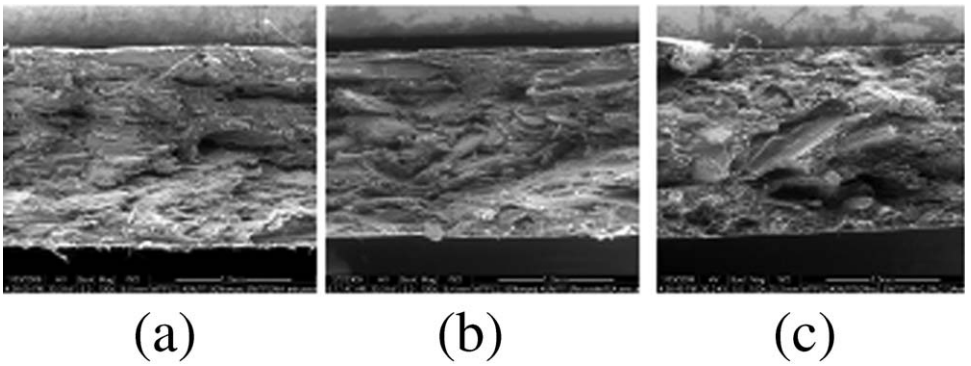

Figure 2 SEM micrographs for WPCs containing (a) 5 wt % MAPP, (b) 5 wt % clay, and (c) 2.5 wt % MAPP and 2.5 wt % clay. All of the samples contain 50 wt % wood flour.

the appearance of voids and fiber pullout with further increases in the wood-flour content [Fig. 1(c,d)]. This suggests poor interfacial adhesion between the filler and the matrix when the wood content was increased.

The addition of MAPP to the formulation greatly improved the fiber dispersion. Comparing Figure 1(d) and Figure 2(a), one can see that the presence of MAPP greatly improved the homogeneity of the blends. Strong interfacial shear strength between the filler and the matrix was observed; this indicated that the presence of MAPP helped to bind the two phases together. Some of the OH groups of wood flour reacted with maleic anhydride to form linkages and, thereby, improved the dispersion of wood in the composites.[5,7–10] Then, the incorporation of MAPP provided covalent bonds across the interface, which permitted segmental crystallization and, thus, cohesive coupling between the fiber and matrix.[12,14] The following mechanisms (Fig. 3) were suggested by Kazayawoko et al.[5] and other researchers[7–10] to describe the ability of MAPP not only to wet and disperse the wood fiber efficiently into the polymer matrix but also to form an adhesive bridge between maleic anhydride and the OH groups of wood. First, the anhydride of MAPP reacted with hydroxyl groups of the wood surface to form ester bonds. The polymer of MAPP and the anhydride of MAPP bonded with molten thermoplastic; this generated a mechanical link between the hydrophilic lignocellulose and the hydrophobic thermoplastic during processing.

The addition of clay in the formulation also led to the improvement of the fiber dispersion, as shown in Figure 2(b,c). This dispersion was even better when clay replaced the MAPP [comparison of Fig. 2(a) with Fig. 2(b)]. When both clay and MAPP were added, no synergetic effect was observed. Studies have shown that the presence of wood flour favors the separation of clay platelets.[11] Additionally, the clay platelets could be separated with the forced orientation of the extruder, which accounted for the good dispersion obtained when MAPP was replaced by clay. On the other hand, when both were present, an antagonistic effect prevailed. More investigations are needed to clarify this antagonistic effect. Nevertheless, the possibility of replacing MAPP with clay appears to be interesting because the combination of wood flour with the polymeric matrix made the composite more sensitive to flame.[15] So far, halogenated flame retardants, such as organic brominated compounds, are generally used to improve the flammability of composites, but they usually increase both smoke and carbon monoxide yield rates, which is catastrophic for the environment. Clay, when used instead of MAPP, may play the role of flame retardant and also improve the mechanical properties.

Thermal properties

T_m, T_c, crystallization enthalpy (ΔH_c), enthalpy of fusion (ΔH_f), and χ are reported in Table II for WPCs containing different percentages of wood flour. An increase in T_c was observed when wood flour was loaded into the polymer matrix. The addition of wood flour had the effect of shifting T_m to higher values. This increase was accompanied by an

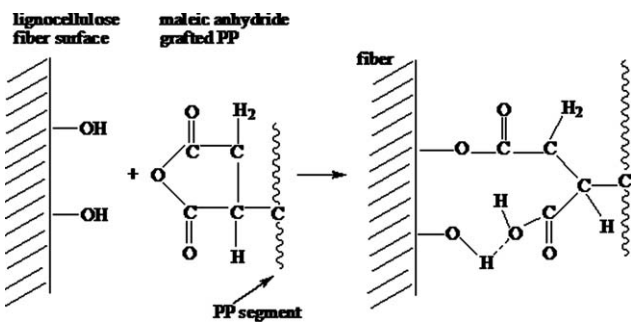

Figure 3 Reaction mechanisms of MAPP with a hydroxyl group on the surface of the wood flour.

TABLE II
Thermal Properties of the WPCs Containing Different Concentrations of Wood Flour

Sample	T_c (°C)	$-\Delta H_c$ (J/g)	T_m (°C)	ΔH_f (J/g)	χ (%)	χ_{cor} (%)
WPPC0	120.8	87.9	160.1	79.3	37.9	37.9
WPPC1	123.4	89.0	160.9	71.9	34.7	38.2
WPPC3	124.80	92.8	162.5	62.8	30.0	40.0
WPPC4	125.0	98.9	162.7	43.0	20.6	41.1

increased χ. We corrected the degree of crystallinity of the composites [χ_{cor} (%)] by taking into account the wood-flour concentration:[16,17]

$$\chi_{cor}\,(\%) = \frac{\Delta H_m\,(\mathrm{J/g})}{[1 - \mathrm{MF}]_{\Delta H_0}\,(\mathrm{J/g})}$$

where ΔH_m and ΔH_0 are the enthalpies of fusion of the composite and polymer, respectively, with 100% crystallinity and MF is the mass fraction of the filler in the composite.

These results suggest that crystallization occurred with the incorporation of wood flour, which played the role of a nucleating agent. Wood flour provided sites for heterogeneous nucleation; this induced crystallization of the polymeric matrix. As for the change in χ, it seemed to be independent of the degree of compatibility between the matrix and the filler.

T_m showed a similar trend with the addition of wood flour. This was ascribed to the poor thermal conductivity of wood. In the composite, wood flour acted as an insulating material, hindering the heat conductivity. As a result, the WPC compounds needed more heat to melt. Similar findings were previously reported by Matuana and Kim[18] for PVC-based wood–plastic composites.[18] They found that the addition of wood flour to the PVC resin caused significant increases in the temperature and energy at which fusion between the particles started. The delayed fusion time observed in rigid PVC/wood-flour composites was attributed to the poor thermal conductivity of the wood flour; this decreased the transfer of heat and shear throughout the PVC grains. These phenomena were consistent with the results of this study.

The influence of the MAPP content on the T_c values of the WPCs was also examined. A shift in T_c toward higher temperatures in the presence of MAPP was observed in the WPCs (Fig. 4). This indicated that the phenomenon of crystallization during the cooling occurred more rapidly in WPCs containing MAPP than in the pure PP. The effect of rising crystallization rates was clear for all of the composites containing MAPP. The results imply that MAPP acted as a precursor and increased crystallization. Our results contradicted a previous study, which found that good interaction between MAPP and wood flour limited molecular mobility near the wood surface and eroded the ability of nucleation.[19]

As previously mentioned, in many applications, WPC products are subjected to UV-light exposure, which causes photodegradation. The consequences of this degradation were the loss of chemical and physical properties, which reduced the lifetime of the WPC products.[4] Then, some attention was paid to the effect of the UV-light-induced degradation on the thermal characteristics (Fig. 5). Specimens of the WPCs were UV irradiated with a SEPAP 12/24 unit working at 60°C in the presence of air. Figure 5 illustrates that T_c increased with UV exposure time. It increased from 124°C for the nonirradiated composite (WPPC3-0H, wood polypropylene composite – irradiated for 0 h) to 131°C for the composite that was UV-irradiated for 200 h.

Most of the products generated by the photooxidation of PP have been identified and quantified.[20] On the basis of the characterization of the chemical structure of the many photoproducts formed, a simplified mechanism was proposed to account for the main routes of the photoinitiated oxidation of PP.[21] It was shown that the primary oxidation products formed are mainly tertiary hydroperoxides, with secondary ones representing less than 10%. The photochemical or thermal decomposition of hydroperoxides, even at relatively low temperatures, produces alkoxy macroradicals, which can rearrange by β scission. This can provoke scissions of the

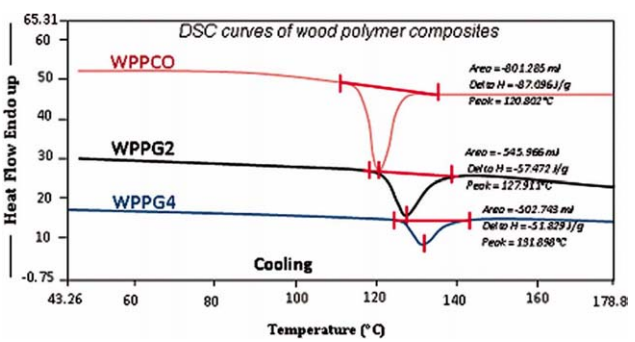

Figure 4 DSC curves of WPCs with different MAPP contents (the curves were vertically moved for clarity). The compositions of the samples are described in Table I. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

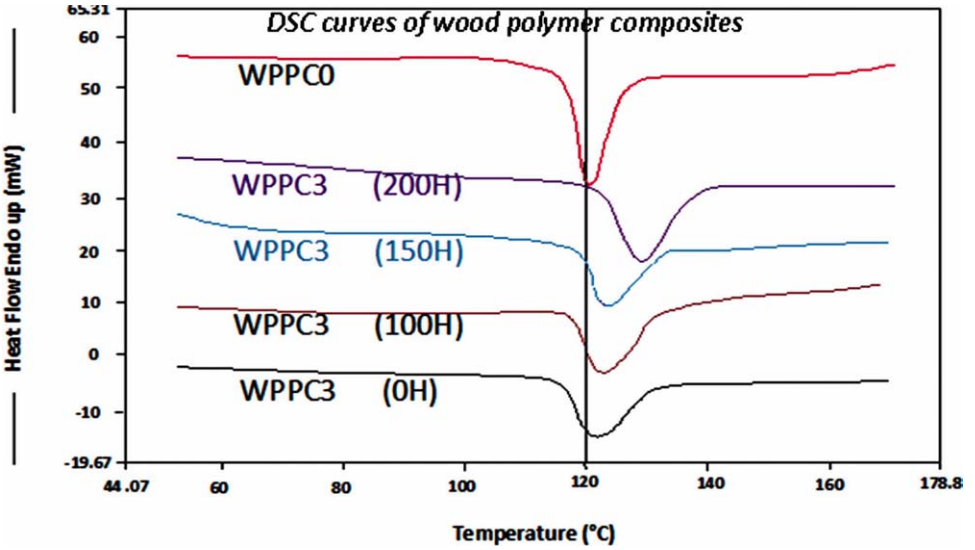

Figure 5 Thermograms of the pure PP and irradiated WPPC3 (75 wt % PP + 25 wt % wood flour). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

macromolecular chains.[22] The chain scissions generate short chains with low molecular weights that can easily move in the bulk of the composites. This suggests that the chemical process of chains scissions, the reduction of molecular weight, and the formation of oxidized groups increase the free energy crystals of the composite, which catalyzes the early crystallization.

Mechanical properties of the WPCs

Flexural and Izod impact properties

The flexural properties and notched Izod impact strength of WPCs containing different percentages of wood flour and/or clay and MAPP are summarized in Table III. The flexural strength and flexural modulus increased with wood flour content. It is accepted that the strength and modulus of a composite system depend on the properties of the blend components, the filler and the matrix. The incorporation of wood with a high modulus in WPCs increased the modulus. On the other hand, the addition of 5% clay in the formulation did not affect the flexural strength; conversely, the flexural modulus decreased dramatically [cf. the wood polypropylene composite (sample n°4:50°/°wood+50°/°PP) (WPPC4) and wood polypropylene clay composite (WPPCC) values]. In contrast, the incorporation of the same percentage (5 wt %) of MAPP in the formulation significantly increased the flexural modulus (from 3944 to 4396 MPa) because of the good dispersion of the wood fibers in the PP matrix containing MAPP. The addition of both, MAPP and clay, showed an antagonistic effect.

The effect of the wood-flour content on the notched Izod impact energy for the WPCs is also listed in Table III. The impact test machine used in this study did not provide enough energy to break the neat PP because of the high flexibility of the PP matrix. By contrast, all of the composites broke completely. The impact energy decreased with increasing wood flour loading in the composites. Increasing the wood content in the composites led to an increased stress concentration because of the poor bonding between the wood flour and the polymer. Although crack propagation became difficult in the polymeric matrix reinforced with filler, the decrease

TABLE III
Flexural Properties and Notched Izod Impact Strength of WPCs Containing Different Percentages of Wood Flour and/or Clay and MAPP

Sample[a]	Flexural strength (MPa)	Flexural modulus (MPa)	Impact strength (J/m)
WPPC0	46.75	1345.6	31.90
WPPC1	50.50	1623.2	22.72
WPPC3	54.40	2438.0	18.71
WPPC4	48.78	3944.3	14.78
WPPCC	49.90	1606.7	13.62
WPPCG	83.35	4396.3	15.38
WPPCCG	75.15	4219.7	15.88

WPPCG = wood polypropylene composite with maleated polypropylene (45°/°PP + 50°/°wood+5°/°MAPP); WPPCCG = wood polypropylene composite with clay and maleated polypropylene (45°/°PP + 50°/°Wood +2.5°/°Clay + 2.5°/°MAPP).

[a] The compositions of the samples are described in Table I.

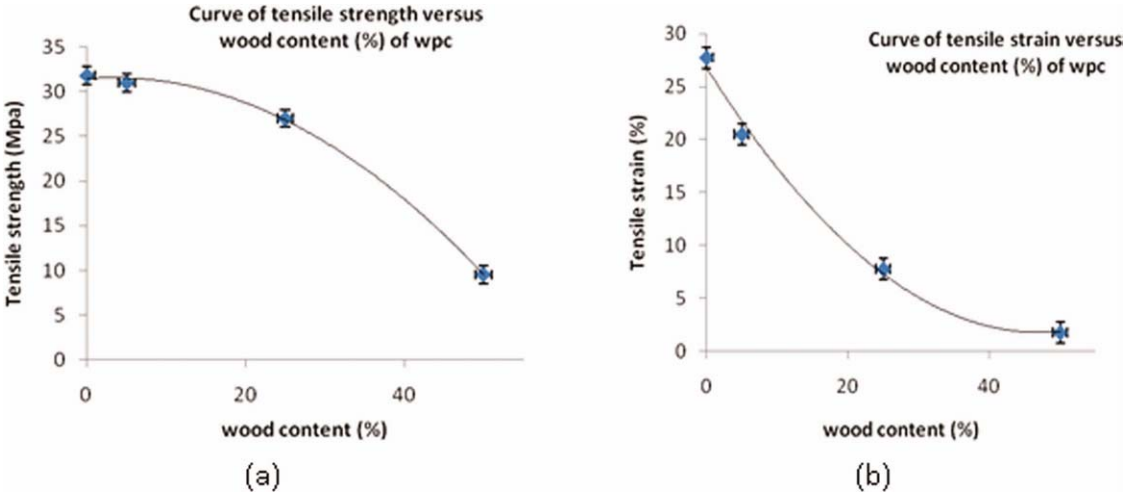

Figure 6 Effect of the wood flour content on the tensile strength and tensile strain of the WPCs. No coupling agent or clay was used in the composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

in the impact energy observed was ascribed to fiber ends, at which microcracks formed and fibers debonded from the matrix. These microcracks were a potential point of composite fractures. Another reason for the decreased impact strength may have been the stiffening of polymer chains due to the bonding between the wood fibers and the matrix. For high-impact properties, in fact, a slightly weaker adhesion between the fiber and polymer is desirable, as it results in a higher degradation of impact energy and supports the so-called fiber pullout.[23]

Tensile strength and tensile strain

Figure 6 shows the tensile strength and tensile strain of WPCs containing different amounts of wood flour. The tensile strength did not change significantly up to 25% wood in the composite. At the 50% content, a drastic decreased was observed. On the other hand, the tensile strain progressively decreased with wood content. When the wood content exceeded 25%, the matrix coverage was insufficient, and many voids appeared; this led to poor interfacial adhesion. Any break initiated spread easily, and this reduced the strength of the material. The decrease in both properties may have then been due to the poor dispersion of the wood fibers in the polymer bulk and moisture pickup. The reduced flexibility of the polymer matrix could also have been an additional reason for the decreased strengths. Some authors[24] explained this loss in strength when wood increases by moisture pickup. As the wood fiber absorbed moisture, it expands; this results in the formation of microcracks in the matrix and the degradation of the wood-flour/PP interface. The addition of wood to a nonpolar polymer matrix decreases the tensile strength of the composites. With a certain treatment that makes the polymer matrix polar, the addition of wood enhances the tensile strength according to some authors.[25]

CONCLUSIONS

The effects of the wood-flour and other compatibilizer (MAPP and clay) contents on the interfacial adhesion in the polymer matrix, the melting and crystallization behaviors, and the mechanical properties of the WPCs were studied. The characterization of samples with SEM showed different behaviors, which depended on the percentage of wood flour or MAPP incorporated into the samples. Poor dispersion was observed in the blends containing only wood flour and PP, but the addition of MAPP in the WPCs led to significant dispersion; this might have been due to the formation of linkages between the OH groups of the wood flour and maleic anhydride. The use of MAPP improved the interaction and adhesion between the fibers and matrix; this led to better stress transfer from the matrix to the fiber. The addition of MAPP greatly improved the dispersion when the content was low (5%). Increases in the wood content led to interfacial debonding with the appearance of voids and fiber pullout. An attempt to replace MAPP in the composites with clay gave unexpected results. Good adhesion was observed, and the addition of clay seemed very important in terms of the flammability of the WPCs.

The thermal properties revealed the strong nucleation ability of the wood flour and MAPP on PP crystallization. Crystallization of the WPCs and composites with the coupling agent MAPP began earlier compared to that of pure PP. This suggested that the wood fiber and MAPP compatibilizer acted as

nucleation agents and were responsible of the shift of crystallinity toward higher temperatures. The presence of wood in the composite generated imperfect adhesion between the components of the composite; this increased the concentration of stress and decreased the impact strength. The results show that the presence of wood flour in the composite was accompanied by an evolution of the crystallization.

UV irradiation of these composites increased T_m and T_c. The short chains issuing from the polymer degradation were supposed to catalyze the crystallization. The tensile strength and strain of all of the composites decreased as the percentage of wood flour increased, probably because of either the decrease in the amount of matrix material and/or the increased irregularities in the matrix. The impact strength of the composites also decreased with the addition of wood flour or other additives to the matrix.

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