Some Studies on Mechanical Properties of Wood Flour/Continuous Glass Mat/Polypropylene Composite

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ABSTRACT: The mechanical properties and the surface property of wood flour/continuous glass mat/polypropylene composites have been investigated. The suitability of wood flour as a filler for continuous glass mat–reinforced polypropylene has been tested using different mesh sizes (e.g., 20 and 40 mesh), as well as by varying the weight percentage of wood flour from 0%–30%. Moreover, different treatments such as coupling agent A-1100 and functionalized polypropylene grafting with maleic anhydride, and so forth, have also been used to improve the compatibility of wood flour and glass fiber with the polymer resin. In addition, the effects of the surface weight of glass mat and matrix resin have been studied. The extent of the improvement in mechanical properties depends on the wood flour content and size, the surface weight of the glass mat, the matrix resin, and the surface treatment of wood flour. After adding wood flour, the contact angle of distilled water on the composite surface decreases and the polar component of surface tension increases. © 2002 Wiley Periodicals, Inc. J Appl Polym Sci 85: 536–544, 2002

Key words: wood flour; polypropylene; glass fiber mat; thermoplastic composite; interface; surfaces; matrix; fillers; reinforcement; modification

INTRODUCTION

In recent years, cheap agro-wastes and agro-forest materials, for example, wood flour and nutshells, and so forth, have been widely used as reinforcements of thermoplastic composites. Wood-fiber thermoplastic composites have received considerable attention from wood and plastic industries.[1,2] Compared with inorganic fillers, the organic fillers impart added benefits such as weight reduction. Glass mat–reinforced thermoplastic-based polypropylene (GMT-PP) has been used in automotive applications and exhibits constantly rapid development as a result of its attractive advantages such as recyclability, faster processing, and higher toughness.[3] GMT using polypropylene filled with wood flour as a matrix will be cheaper than GMT-PP. In "traditional" GMT-PP, exposed fiber resulting from the significantly higher volume shrinkage of PP will irritate human skin. Because the presence of wood flour limits the shrinkage of PP during the cooling process, the wood flour–filled GMT has a better surface quality and a more comfortable feeling. Moreover, with the increase in polarity of the sheet surface after adding wood flour, the sheet will be easier to adhere to other materials, which makes the composite useful for automobile interior parts. However, the poor compatibility of hydrophilic wood flour and hydrophobic polypropylene results in a poor interfacial adhesion and strong moisture absorption. Hence, wood flour–

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Table I

Liquid	γ (dyne/cm)	γ^p (dyne/cm)	γ^d (dyne/cm)
Distilled water	27.8	50.7	22.1
Diiodomethane	50.8	6.7	44.1

reinforced polypropylene composites have poor mechanical properties and hydrothermal stability, which restricts the use of such composites. To improve the interfacial adhesion and reduce the moisture absorption, several methods have been reported for modifying the surface of wood flour.[4–7] The interaction between natural fiber and resin matrix has also been studied.[8–10] The simultaneous incorporation of wood flour and glass mat in PP has been investigated to a lesser extent.

In this work, wood flour was used as the filler for continuous glass mat–reinforced polypropylene. The effect of wood flour content and size, surface weight of glass mat, matrix resin, and interfacial treatment on the mechanical properties of composites was investigated.

EXPERIMENTAL

Materials

Polypropylene (types Y1600, Y2600, and Y3500) was purchased from the Plastics of Shanghai Petrochemical Complex (China). The melt flow index of the three polypropylenes was 16, 26, and 35g/10 min, respectively. Maleic anhydride grafted polypropylene (MPP) was prepared by the solid method in our laboratory; its grafting degree was 1.2%. The random continuous swirled bundle (50 fibers with a fiber diameter of 21 μm) mat, came from Nanjing Glass Fiber Research and Design Institute (China). They were needled to hold together, and the needling density was 28 needles/cm^2. Glass mats having three surface weights (330, 720, and 950 g/m^2) were used in our experiments. Abandoned wood flour was a mixture of various kinds of sawdust. The silane coupling agent A-1100, γ-anilinopropyltrimethoxysilane H2N(CH2)3Si(OCH2CH5)3, was supplied by Shanghai Yaohua Company (China). Antioxidants tetrakis(methylene-β-[3,5-di-tert-butyl-4-hydroxyphenyl]-propionate)methane (1010) and distearyl thiodipropionate (DSTP) were the products of Ciba-Geigy Company (Sweden). Toluene was chemically pure and used without further purification.

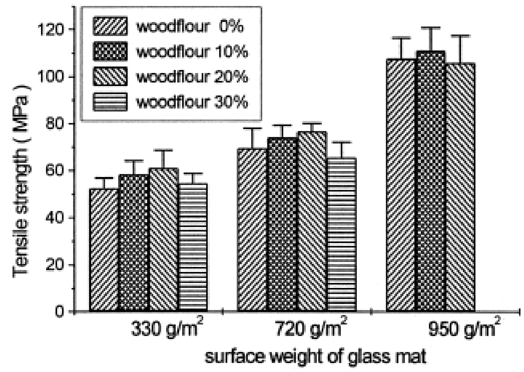

Figure 1 Effect of wood flour content and mat surface weight on tensile strength of composites. Wood flour mesh size: 40 mesh; polypropylene type: Y1600; interfacial treatment: 2%A-1100 (with respect to wood flour)+5%MPP (with respect to PP).

Surface Treatment of Wood Flour

Wood flour was heat treated at 120°C for 5 hr after being ground into different mesh sizes (20 and 40 mesh). Then it was placed in a GH-100Y high-speed mixer (made in China), followed by the addition of 2 wt % (with respect to wood flour weight) coupling agent solution in water. After mixing for about 5 min, the wood flour was removed and dried at 80°C in an oven for 1 hr. When most water was evaporated, the temperature was raised to 120°C, at which point the wood flour reacted with the coupling agent for 30 min.

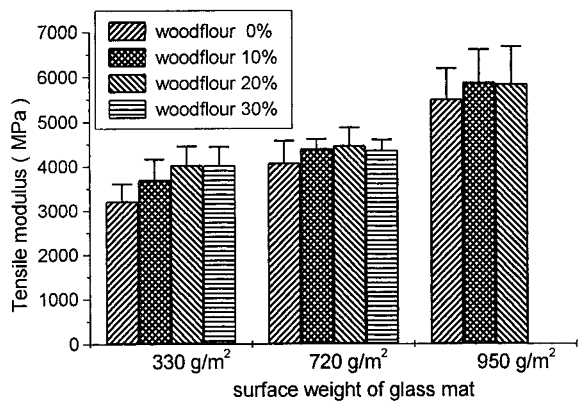

Figure 2 Effect of wood flour content and mat surface weight on tensile modulus of composites. Wood flour mesh size, polypropylene type and interfacial treatment as in Fig. 1.

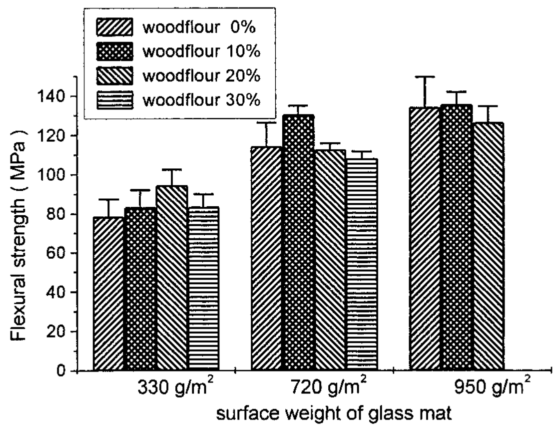

Figure 3 Effect of wood flour content and mat surface weight on flexural strength of composites. Wood flour mesh size, polypropylene type and interfacial treatment as in Fig. 1.

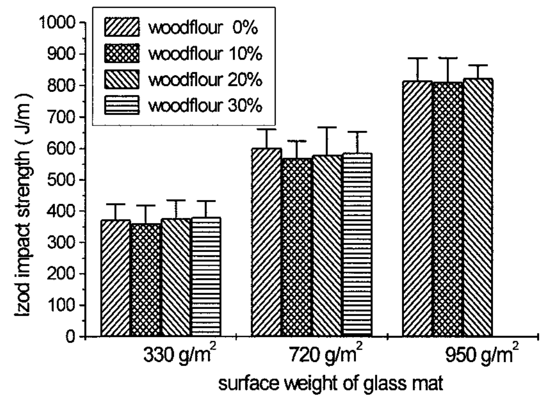

Figure 5 Effect of wood flour content and mat surface weight on Izod impact strength of composites. Wood flour mesh size, polypropylene type and interfacial treatment as in Fig. 1.

Preparation of Polypropylene Filled with Wood Flour

Wood flour was mixed with PP and additives such as the antioxidant and MPP in different weight proportions, then extruded with a GE2.8.30-41 twin-screw extruder (Luxembourg) at a screw speed of 160 rev/min and cut into pellets.

Preparation of Wood Flour/Continuous Glass Mat/Polypropylene Composite Sheets

The pellets produced in the previous step were rolled out in films with a thickness of about 1 mm. Three films and two mats sandwiched together were molded in a double-belt press of our laboratory by continuous melt impregnation.

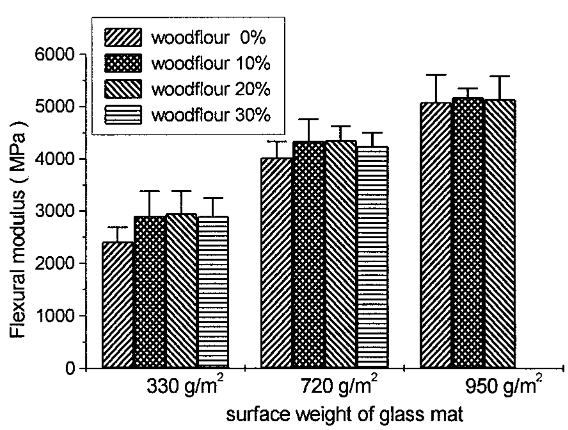

Figure 4 Effect of wood flour content and mat surface weight on flexural modulus of composites. Wood flour mesh size, polypropylene type and interfacial treatment as in Fig. 1.

Mechanical Testing

The pellet of wood flour–filled PP was injection molded with a TTI-80 plastic injection machine (China) in accordance with Chinese Standard for Test Methods GB1043-79. Then the tensile and flexural properties were measured on a CMT 4204 universal testing machine (China) at a crosshead speed of 2 mm/min, and notched charpy impact strength was tested with a WPM-charpy impact tester (Germany).

The notched Izod specimens were cut from the sheet of wood flour/continuous glass mat/polypropylene composites and tested according to ASTM D256 on a Cantilever Beam impact machine. The tensile samples were prepared and tested according to ASTM D638, type I specification. An extensometer with gauge length of 50 mm was used for modulus measurements. The flexural specimen was conducted using the universal testing machine in the three-point loading mode. The procedure ASTM D 790 was followed, an 80-mm support span and a 2 mm/min crosshead speed were used. At least five specimens were tested for each sample. All mechanical tests were performed at 23°C.

Surface Properties of Wood Flour/Continuous Glass Mat/Polypropylene Composite

The sheet surface was washed with toluene and then dried in a vacuum oven. The dynamic contact angle analyzer JY-82 (made in China) was

Table II Effect of Wood Flour Content on Mechanical Properties of PP Filled with Wood Flour

Woodflour Content	0	10%	20%	30%
Tensile strength (s.d.), MPa	32.00 (0.00)	33.81 (0.58)	35.31 (0.00)	38.16 (0.58)
Flexural strength (s.d.), MPa	52.54 (0.11)	57.32 (2.91)	66.97 (1.25)	71.55 (0.66)
Flexural modulus (s.d.), MPa	1404 (12)	1632 (84)	2097 (168)	2531 (121)
Charpy impact strength (s.d.), KJ/m^2	3.51 (0.50)	3.55 (0.21)	3.52 (0.50)	3.48 (0.62)

The composition of matrix is polypropylene Y1600 + 5% MPP.

used to measure the contact angles, and the value given is the average value of 10 measurements. The contact liquids were distilled water and diiodomethane, and the surface energy components were calculated by using the harmonic mean equation:[11]

$$\begin{cases} (1 + \mathrm{COS}\ \theta_1)\gamma_1 = 4\left(\dfrac{\gamma_1^d\gamma_s^d}{\gamma_1^d + \gamma_s^d} + \dfrac{\gamma_1^p\gamma_s^p}{\gamma_1^p + \gamma_s^p}\right) \\ (1 + \mathrm{COS}\ \theta_2)\gamma_2 = 4\left(\dfrac{\gamma_2^d\gamma_s^d}{\gamma_2^d + \gamma_s^d} + \dfrac{\gamma_2^p\gamma_s^p}{\gamma_2^p + \gamma_s^p}\right) \end{cases}$$

Where γ_s is the surface tension and γ_s^p and γ_s^d refer to the polar and dispersion components of surface tension. The subscripts 1 and 2 refer to the testing liquids distilled water and diiodomethane, respectively.

The values of surface tension and its components for water and diiodomethane were used for the calculation of surface tension of the composites from the contact angles, as can be seen in Table I.

Scanning Electron Microscopy

The tensile fracture surfaces of the composite samples were studied with a S-250 scanning electron microscope (UK), operated at 15 KV.

RESULTS AND DISCUSSION

Effect of Wood Flour Content and Glass Mat on Mechanical Properties

Several types of composite sheets were prepared with wood flours of different contents, continuous glass mats of various surface weights, and the same polypropylene; the mechanical properties of sheets were shown in Figures 1–5. The results revealed that the mechanical properties increased with the increment of mat surface weight at the same wood flour content. Both glass fiber and wood fiber are reinforcements in such a system. But the strength and modulus of glass fiber is superior to that of wood flour; the glass mat mainly determines the mechanical properties of the composites. Glass fiber ends are fewer in a continuous glass fiber mat, which prevents some crack initiations because of lower stress concentration. As glass fiber content increases, failure processes such as interfacial debonding, fiber pull-out, and local plastic deformation increase.[12–18] Thus, the enhancement of glass fiber content can also significantly improve the impact strength of composite.

When wood flour with a certain aspect ratio fills the polypropylene, the mechanical properties of the systems can be significantly improved. Ta-

Table III Effect of Woodflour Size on Mechanical Properties of Wood Flour/Continuous Glass Mat/Polypropylene Composites

Woodflour Size	20 Mesh	50% 20 Mesh + 50% 40 Mesh	40 Mesh
Tensile strength (s.d.), MPa	79.67 (11.99)	78.60 (18.76)	76.78 (3.22)
Tensile modulus (s.d.), MPa	4914 (333)	4789 (491)	4471 (396)
Flexural strength (s.d.), MPa	115.93 (11.33)	112.71 (4.96)	112.62 (3.21)
Flexural modulus (s.d.), MPa	4410 (788)	4361 (570)	4350 (269)
Izod impact strength (s.d.), J/m	550.18 (58.69)	571.71 (67.68)	579.71 (87.78)

The wood flour content is 20%; the composition of matrix is polypropylene Y1600 + 5% MPP; the surface weight of glass mat is 720 g/m^2.

Table IV Effect of Matrix Resin on Mechanical Properties of Wood Flour/Continuous Glass Mat/Polypropylene Composites

Matrix Resin	Y1600	Y2600	Y3500
Tensile strength (s.d.), MPa	73.56 (7.62)	78.62 (7.92)	77.82 (8.14)
Tensile modulus (s.d.), MPa	4385 (403)	4894 (471)	4865 (415)
Flexural strength (s.d.), MPa	109.29 (8.17)	118.28 (11.26)	124.00 (12.46)
Flexural modulus (s.d.), MPa	4289 (373)	4459 (406)	4629 (258)
Izod impact strength (s.d.), J/m	563.32 (55.29)	578.25 (34.49)	559.68 (54.32)

The wood flour content is 20%; the woodflour mesh size is 40 mesh; the surface weight of glass mat is 720 g/m^2.

ble II showed the effect of wood flour content on the mechanical properties of wood flour–filled PP. With the rise of wood flour content in polypropylene, both the tensile and flexural properties could be increased, but high wood flour content would increase melt viscosity and decrease the melt flow ability.

The flow length over the thickness direction of the melt penetrating into the mat would increase with the increasing of the mat surface weight. The effect of the melt fluidity on the system impregnation became more obvious in the composites containing a high surface weight mat. In the case of a 720 g/m^2 glass mat, the flexural strength began decreasing when the wood flour content rose above 20 wt%. At such a high wood flour content, both the tensile and flexural properties decreased in the system using a 950 g/m^2 glass mat. When adding 30 wt% wood flour, the mechanical properties of the composite decreased rapidly in the case of the 950 g/m^2 glass mat because of the bad impregnation.

For the wood flour/glass mat/PP system, the tensile and flexural properties as a function of wood flour content depended on the mat surface weight. In the case of the 330 g/m^2 mat, the flow length of the melt was short during the compounding process, so the melt easily made contact with the glass fibers in mat, and a better interfacial adhesion between the glass fiber and PP could be obtained. With the rise of wood flour content from 0% to 20%, both the tensile and flexural properties increased because of the wood flour reinforcement. However, the impregnation of melt on the glass fiber became relatively worse when the wood flour level was at 30 wt%, as the melt viscosity obviously increased. The melt flow became so difficult that some glass fibers could not make contact with the resin and play their role. Thus, the tensile and flexural properties decreased instead. Moreover, the decrease of strength was larger than that of modulus.

The result indicated that the impact strength of the composites decreased at up to 10 wt% wood flour and that above this level, the strength increased. This might be because of the fact that additional ways of energy absorption, such as wood fiber pull-out and wood fiber–PP interfacial debonding, were achieved.[19–20]

Effect of Wood Flour Size on Mechanical Properties

PP filled with different mesh sizes of wood flour was compounded with the same glass mat; the mechanical properties of the composites were presented in Table III. It was evident from Table III that the impact strength increased with the reduction in mesh size. This might be a result of the fact that the interfacial area decreased with the increase in particle size after compounding with the resin. The absorption energy of the system accordingly decreased by means of interfacial debonding, and so forth. The contrary was true for the tensile and flexural strength and modulus. Small wood flours offer a larger specific surface area in composites than larger ones in the same

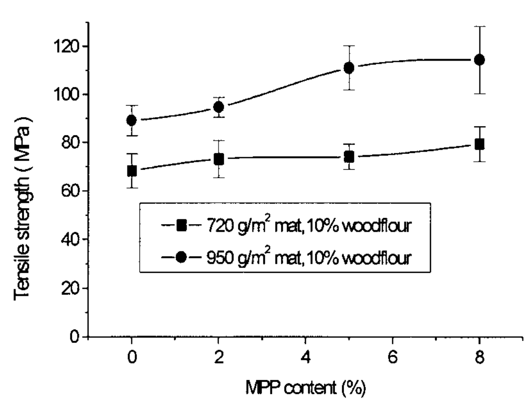

Figure 6 MPP content vs. tensile strength. Wood flour mesh size: 40 mesh; polypropylene type: Y1600.

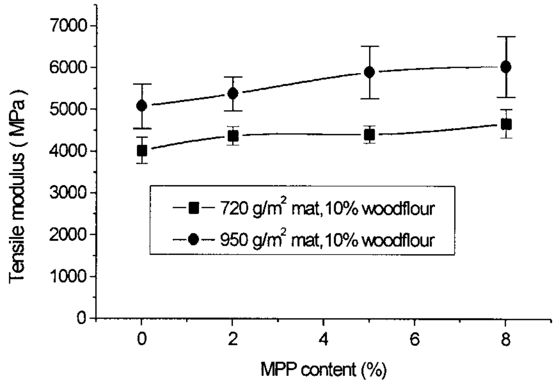

Figure 7 MPP content vs. tensile modulus. Wood flour mesh size and polypropylene type as in Fig. 6.

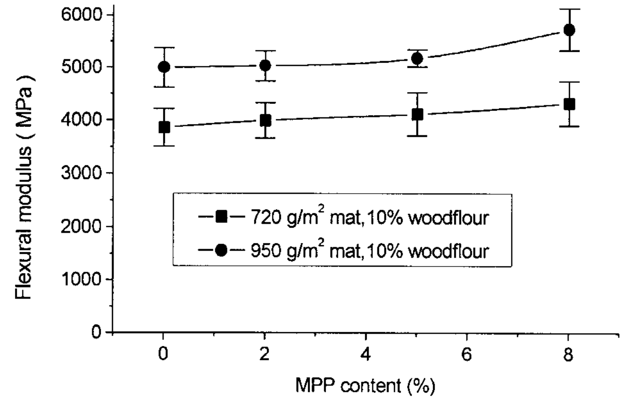

Figure 9 MPP content vs. flexural modulus. Wood flour mesh size and polypropylene type as in Fig. 6.

weight fraction. Wood flours in the 40 mesh size have a greater interaction with PP matrix; they can give a greater rise in the melt viscosity than the 20 mesh wood flours. The impregnation of the melt containing 20 mesh wood flour on the glass mat was better than that using 40 mesh wood flour because of better fluidity, which leads to a better tensile and flexural properties of the composite.

Effect of Matrix Resin on Mechanical Properties

Three types of polypropylene (Y1600, Y2600, and Y3500) that had similar molecular structures and various melt indexes were used as matrix resin. Their tensile and flexural properties were similar, and the range of impact strength was Y1600 > Y2600 > Y3500. The mechanical properties of obtained composites are shown in Table IV. The higher the melt indexes, the better the flowability. With the increment of melt index, the impregnation of resin on glass fiber and wood flour was improved, which was beneficial to fully utilize the reinforcements. Thus, the tensile and flexural properties increased slightly. Because of the improved impregnation, the ability to absorb impact energy was enhanced by interfacial debonding, fiber pull-out, and friction between fiber and PP, which could compensate for the impact strength of matrix resin. Thus, the impact strength of composite containing Y3500 had no detectable reduction than that using Y1600.

Effect of Interfacial Treatment on Mechanical Properties

Figures 6–10 showed the effect of MPP on the mechanical properties of composites.

After MPP was added to PP, the good compatibility between MPP and PP resin lead to their

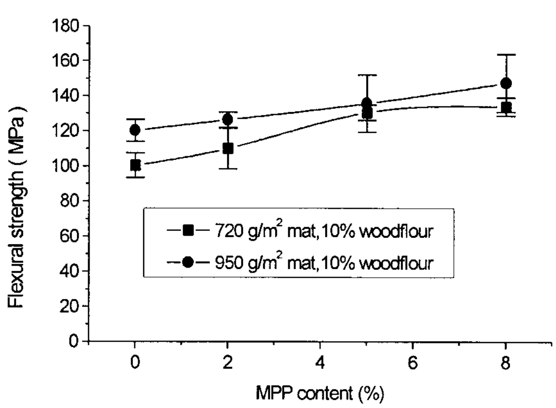

Figure 8 MPP content vs. flexural strength. Wood flour mesh size and polypropylene type as in Fig. 6.

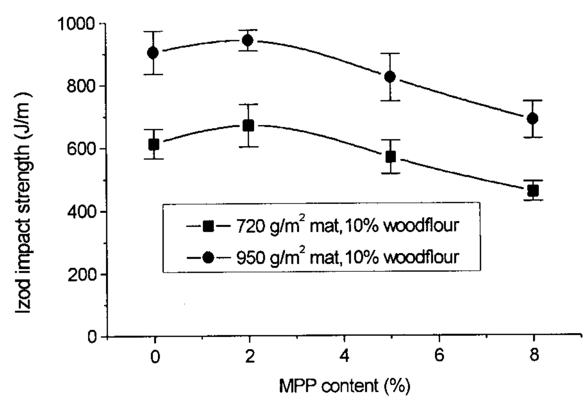

Figure 10 MPP content vs. Izod impact strength. Wood flour mesh size and polypropylene type as in Fig. 6.

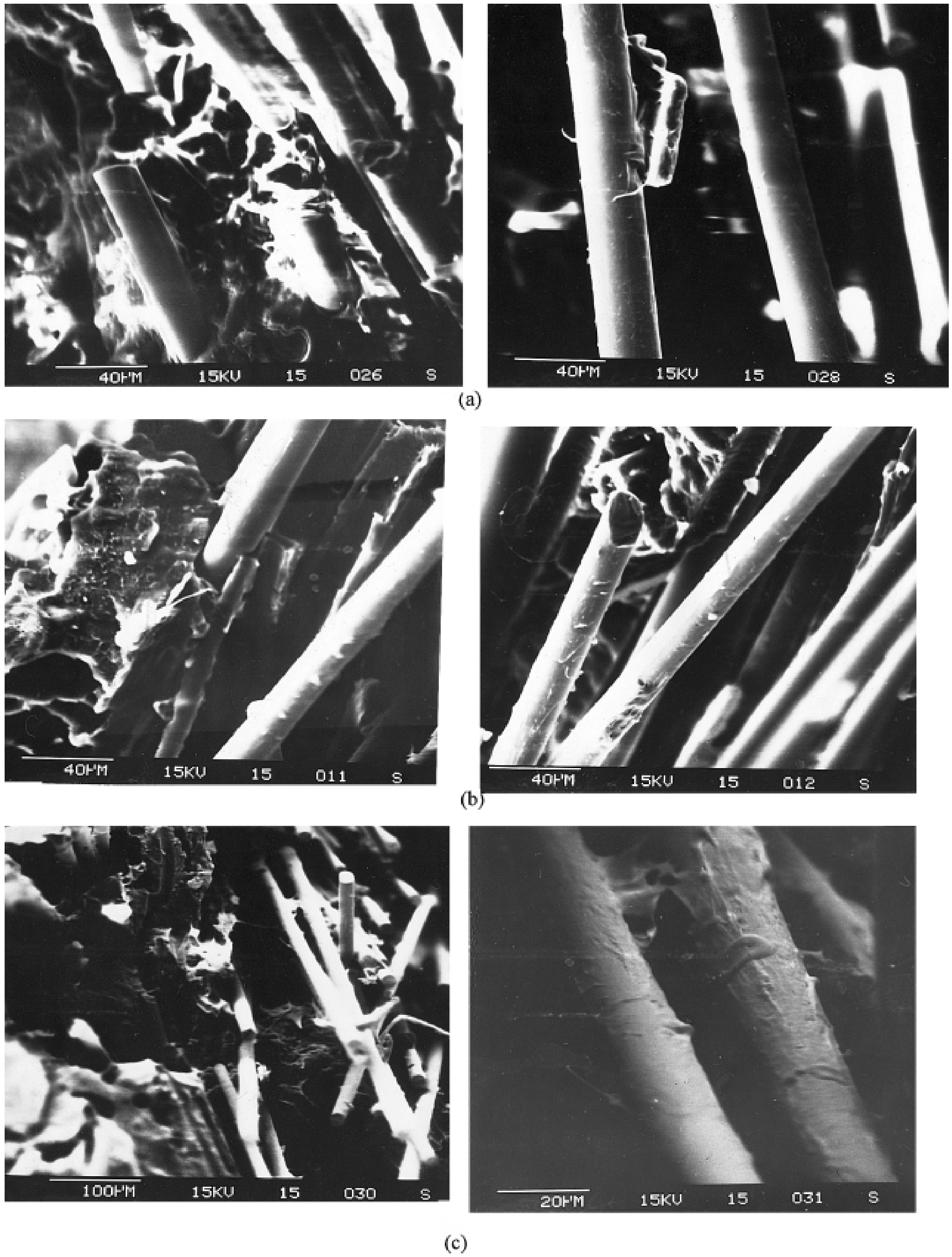

Figure 11 Scanning electron micrograph of tensile fracture surface of wood flour/glass mat/PP composites (a) no adding MPP in matrix resin; (b) adding 5% MPP in matrix resin; (c) adding 8% MPP in matrix resin.

firm chain entanglement. When making a compound with wood flour, the polar groups grafted on MPP can form a strong interaction; for example, ester linkages and hydrogen bonds with the −OH groups of wood flour.[6,8,9] The influence of functionalized polypropylene on the interfacial adhesion of glass fiber with polypropylene had been studied in our previous work.[21] In this way, MPP improved the interfacial adhesion between wood flour, glass fiber, and PP. Moreover, a strong wood flour–wood flour interaction caused by intermolecular hydrogen bonding has also been di-

Table V Effect of Surface Treatment of Woodflour on Mechanical Properties of Wood Flour/Continuous Glass Mat/Polypropylene Composites

Method	Untreated	A-1100
Tensile strength (s.d.), MPa	73.56 (7.62)	76.78 (3.22)
Tensile modulus (s.d.), MPa	4385 (403)	4471 (396)
Flexural strength (s.d.), MPa	109.29 (8.17)	112.62 (3.21)
Flexural modulus (s.d.), MPa	4289 (373)	4350 (269)
Izod impact strength (s.d.), J/m	563.32 (55.29)	579.71 (87.78)

The wood flour content is 20%; the composition of matrix is polypropylene Y1600 + 5% MPP; the woodflour mesh size is 40 mesh; the surface weight of glass mat is 720 g/m^2.

luted, which leads to the better dispersion of wood flour. So the addition of MPP can improve the tensile and flexural properties.

Figure 11 shows the scanning electron micrograph of tensile fracture surfaces of wood flour/glass mat/PP composites. The micrograph illustrates that the resin did not adhere to the fiber surface when MPP was not added into system. On the contrary, the fiber surfaces adsorbed matrix resin in the systems adding MPP. Moreover, the resin adsorbed from the system containing 8% MPP was more than of that using 5% MPP. This indicates that adding MPP improved the interfacial adhesion of system.

With the rise in MPP content, the reactive sites of polymer chains was enhanced. Accordingly, the interfacial adhesion strength increased. Thus, the tensile and flexural properties increased with increasing MPP content. However, when the interfacial adhesion strength reached a certain level, the ability of the system to absorb the impact energy would decrease because the interfacial debonding and fiber pull-out became difficult. This leads to the decrease in impact strength.

After being treated with A-1100, wood flour was compounded with a glass mat and PP in which MPP was added. Table V presents the effects of surface treatment on the mechanical properties of composites.

Compared with wood flour untreated with a coupling agent, wood flour treated with A-1100 shows some improvement in its mechanical properties.

When wood flour was treated with the coupling agent A-1100, hydroxy groups could be produced by the hydrolysis of silanes with adsorbed water. They were supposed to link to the wood flour through the formation of hydrogen bonds with the hydroxy groups at the surface of the wood flour. The remaining chain of silanes could adhere to PP with the help of a Van der Waals type of weak interaction. In this way, silane made a link between wood flour and PP. As a result, the mechanical properties were improved, compared with those of uncoated fiber composites. But the hydrogen bonds and Van der Waals weak forces are insufficient to make a strong bridge between

Table VI Surface Properties of Woodflour/Continuous Glass Mat/Polypropylene Composite

Matrix and Woodflour Concent	PP	PP + 5% MPP	PP + 5% MPP + 10% Woodflour	PP + 5% MPP + 30% Woodflour
θ_1(°)	96.01	95.57	92.40	86.83
$\sigma_{\theta 1}$(°)	1.90	0.60	2.10	0.21
θ_2(°)	54.44	53.52	51.6	53.13
$\sigma_{\theta 2}$(°)	1.60	2.50	1.70	2.73
γ (dyne/cm)	33.24	33.69	34.68	34.95
γ^p (dyne/cm)	4.00	4.08	5.34	8.28
γ^d (dyne/cm)	29.24	29.60	29.33	26.67

The wood flour content is 20%; the composition of matrix is polypropylene Y1600 + 5% MPP; the woodflour mesh size is 40 mesh; the surface weight of glass mat is 720 g/m^2.

the fiber and the polymer, and the mechanical property improves only slightly.[7]

Surface Properties of Wood Flour/Continuous Glass Mat/Polypropylene Composite

The results of contact angle measurement and the calculated surface tension are listed in Table VI. As can be seen, there was no significant difference between the contact angles of PP, either untreated or treated with MPP. But the addition of wood flour resulted in the decreasing of the contact angles of the distilled water on the surface of the wood flour/glass mat/polypropylene composite and in the polar component of surface tension increasing. Moreover, with the rise in wood flour content, the changes in contact angle and the polar component of surface tension were greater. This is mainly because the higher wood flour content can contribute more −OH groups. The increase in the polar component of surface tension of the composite makes it easy to adhere to other materials.

CONCLUSIONS

Using wood flour as the filler for continuous glass mat–reinforced polypropylene reduced the material cost without decreasing the desired properties. With the rise in wood flour content, the tensile and flexural properties increased to some extent, whereas the impact property had no detectable change. Finer particles (40 mesh) were advantageous for the tensile and flexural properties, but the contrary was true for impact strength. The mechanical properties of composites increased with an increase in the surface weight of the glass mat. However, a high surface weight made it difficult for the melt to impregnate the glass mat, especially with a high wood flour content. A matrix resin with a high melt index was beneficial to the impregnation of system. In addition, the modification of the interfacial adhesion between reinforcements and matrix resin by using functionalized polypropylene, and so forth, played a significant role in improving the mechanical properties of the composites. But the impact strength decreased above 5% of MPP level. After adding wood flour, the contact angles of distilled water on the composite surface decreased and the polar component of surface tension increased.

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