Preparation and Characterization of Wood-(Nylon 12) Composites

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Received 20 February 2006; accepted 17 August 2006 DOI 10.1002/app.25274 Published online in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: The melting temperature of nylon 12 is lower than the degradation temperature of wood, which makes the preparation of wood-(nylon 12) composites through a regular compounding/compression molding process possible. Results indicated that wood-(nylon 12) composites had higher modulus of rupture, higher modulus of elasticity, and higher tensile strength than nylon 12, wood-polypropylene (PP) composites, and wood-high density polyethylene (HDPE) composites, respectively. Wood-(nylon 12) composites also had higher thermal stability than wood-PP composites and wood-HDPE composites. Acting as a nucleating agent, wood increased the crystallization temperature and the degree of crystallinity of nylon 12 in wood-(nylon 12) composites. The superior mechanical properties of wood-(nylon 12) composites were ascribed to the good interfacial adhesion between wood and nylon 12 and the increased transcrystallinity of nylon 12 by wood. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 103: 270–276, 2007

Key words: composites; compounding; compression molding; nylon 12; wood

INTRODUCTION

Nylons (or polyamide materials) are a series of thermoplastic polymers with a repeating amide group (-CONH-) in their chains. The polar amide group provides nylon materials with many superior properties such as high melting temperature and good toughness.¹ Commonly used nylons include nylon 6, nylon 6-6, nylon 10, and nylon 12. Nylon 12 has a lower melting temperature ($\sim 175^{\circ}$ C) than nylon 6 $(\sim 215^{\circ}C)$ and nylon 6-6 $(\sim 264^{\circ}C)$.² The melting temperature of nylon 12 is also lower than the degradation temperature of wood ($\sim 240^{\circ}$ C).³ Of all commercially available nylons, nylon 12 has the lowest water sorption rate.^{1,2} Moreover, nylon 12 has higher impact strength and better permeability to gases and liquids at ambient temperature than nylon 6, nylon 6-6, and nylon 10.¹ Nylon 12 has high tensile strengths and excellent scratch and wear resistance as well.² Nylon 12 has been extensively used for tubing, piping, packaging, food processing, clothing, marine products, and many other applications.^{1,2}

Several attempts have been made to mix nylon 6 and nylon 6-6 with wood fibers and pure cellulose.^{4–6} It is difficult to thoroughly and uniformly mix one of

these two nylons with wood fibers without significant degradation of wood because the melting temperatures for these two nylons are close to or higher than the degradation temperature of wood.^{3,7} Nylon 12 can potentially be used to make wood-(nylon 12) composites because of its low melting temperature. However, none has been published so far on the preparation and characterization of wood-(nylon 12) composites.

In this study, we prepared wood-(nylon 12) composites and compared them with wood-PP (polypropylene) composites and wood-high density polyethylene (HDPE) composites in terms of mechanical properties, thermal properties, and interfacial morphology.

EXPERIMENTAL

Materials

Pine flour (40 meshes in size) was supplied by American Wood Fibers Company (Schofield, WI). Wood flour was oven-dried at 103°C for 24 h prior to use. The moisture content of the oven-dried wood flour was 0.50%. Three thermoplastics (nylon 12, polypropylene (PP) and high density polyethylene (HDPE)) were obtained from commercial sources: Nylon 12 pellets (Grilamid[®] L20G, density: 1.01 g/cm³ at 20°C) from EMS-CHEMIE North America (Sumter, SC), HDPE (high density polyethylene, melt flow index: 0.55 g/10 min), and PP (melt flow index: 1.5 g/ 10 min) from BP Solvay Polyethylene North America (Houston, TX).

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Contract grant sponsor: USDA (National Research Initiative Competitive Grants Program; contract grant number: 2003-35103-13864).

Journal of Applied Polymer Science, Vol. 103, 270–276 (2007) © 2006 Wiley Periodicals, Inc.

Compounding wood flour with thermoplastics

Wood was mixed with a thermoplastic in a Brabender Plasticorder with a mixing bowl (60 mL) and two roller blades (C. W. Brabender Instruments, South Hackensack, NJ). A one-step compounding process was used for various wood-thermoplastic composites. Wood/thermoplastic weight ratios were 0/100, 40/60, 50/50, and 60/40.

Wood and a thermoplastic (the total weight of wood and a thermoplastic: 46 g) with one of the previously described wood/thermoplastic weight ratios were compounded in the Brabender bowl at 45 rpm for 10 min at a predetermined temperature. The compounding temperature was 190°C for wood-(nylon 12) blends, 180°C for wood-PP blends, and 160°C for wood-HDPE blends. After compounding, all blends were removed from the Brabender bowl, cut into small pieces with chisel while the blends were still hot, and then stored for compression molding.

Preparation of wood-thermoplastic composites

A stainless-steel mold with dimensions of 101.6 \times 101.6 \times 2.0 mm³ was used for preparation of woodthermoplastic composites via a compression molding process. The platens of Carver press (Model 3891, Carver, Wabash, IN) were preheated to a designated pressing temperature: 200°C for wood-(nylon 12) composites and 185°C for wood-HDPE and wood-PP composites. Each wood-thermoplastic blend was put into the mold. The mold with a lid plate on top of the blend was placed onto the hot press and preheated for 10 min. The press was closed slowly allowing the wood-thermoplastic blend to flow into the mold shape. The mold was pressed at 344.8 kPa for 10 min, removed from the hot press, and then cooled in a separate cold press (Carver, Summit, NJ) at 344.8 kPa under ambient conditions. Some of the resulting wood-thermoplastic composites were cut into test specimens (55.0 mm \times 13.5 mm \times 2.0 mm) for evaluation of modulus of rupture (MOR) and modulus of elasticity (MOE). Other composites were cut into test specimens (63.5 mm \times 9.53 mm \times 2.0 mm with a neck width of 3.18 mm in the center section) for evaluation of tensile strength.

Measurement for mechanical properties of the resultant wood-thermoplastic composites

Ten specimens of each wood-thermoplastic composite were evaluated for their strength and stiffness with a three-point bending test in accordance with ASTM D790-02. The specimens were tested on a Sintech machine (Model Sintech 1/G, MTS Systems, Enumclaw, WA). The support span was 43 mm, and the crosshead speed was 1.0 mm/min. The MOR and MOE were calculated from the load-deflection data. The MOR was determined at the first point on the load-deflection curve where a slope was zero. The MOE was determined from the slope in the initial elastic region of the load-deflection curve.

The Sintech machine was also used for the measurement of tensile strength. The dumbbell specimens were prepared from wood-thermoplastic boards in accordance with ASTM D638. The crosshead speed was 0.25 mm/min. Five specimens of each woodthermoplastic composite were used for the determination of the tensile strength.

Thermal analysis

Wood-thermoplastic composites were characterized with a differential scanning calorimetry (DSC) system (Model DSC 2920, TA Instruments, New Castle, DE). Each composite specimen (~ 10 mg) was sealed in an aluminum pan and flushed with a nitrogen stream under a pressure of 379 kPa. The specimen was first heated from 20 to 200°C at a heating rate of 10°C/min and held at 200°C for 5 min. The specimen was then cooled to 20°C at a cooling rate of 10°C/min, and heated again to 200°C at a heating rate of 10°C/min. The crystallization temperature (T_c) and melting temperature (T_m) were determined from the first cooling curve and the second heating curve, respectively. The degree of crystallinity (X_c) is calculated based on the following equation⁸:

$$X_c = \frac{\Delta H_c}{W_m \cdot \Delta H_f^0} \tag{1}$$

where ΔH_c is the heat of crystallization of a woodfilled thermoplastic, ΔH_f^0 is the heat of crystallization of the theoretically perfect crystalline thermoplastic, and W_m is the weight fraction of the thermoplastic in a wood-thermoplastic composite. For a thermoplastic, W_m is equal to one. The ΔH_f^0 values for nylon 12, PP, and HDPE are 233.6, 209, and 289 J/g, respectively.^{9–11}

A thermogravimetric analyzer (Model TGA 2950, TA Instruments, New Castle, DE) was used for analyzing the thermal stability of wood-thermoplastic composites. Each composite specimen ($\sim 10 \text{ mg}$) was heated from room temperature to 600°C at a heating rate of 10°C/min. During heating, the specimen was flushed with a nitrogen stream under a pressure between 276 and 414 kPa.

Analysis of interfacial adhesion

The fractured surfaces of test specimens after the tensile test were analyzed with scanning electron microscopy (SEM). The fractured surfaces were coated with a 15-nm layer of gold. Micrographs from the fractured



Figure 1 Effect of wood flour content on MOR of woodthermoplastic composites. Data are the mean of ten replicates and the error bars represent two standard deviations.

surfaces were obtained with an AmRay SEM (Model 3600FE, AmRay Company, Bedford MA) at an accelerating voltage of 5.0 kV.

Statistical analysis of experimental results

Experimental results were statistically analyzed with a Duncan's grouping test by means of SAS software for comparing the effects of the thermoplastic type and the wood flour content on MOR, MOE, and the tensile strength of wood-thermoplastic composites.

RESULTS

Mechanical properties

The MOR of nylon 12 was higher than that of PP or HDPE (Fig. 1 and Table I). When compared with the

TABLE I Duncan's Grouping Tests for Strength Properties of Wood-Thermoplastic Composites

Wood flour content	0 wt %	40 wt %	50 wt %	60 wt %
MOR				
Wood-(nylon 12)	C (α)	Β (α)	Β (α)	Α (α)
Wood-PP	Α (β)	Β (β)	C (β)	D (β)
Wood-HDPE	Β (γ)	$A(\gamma)$	Β (γ)	C (γ)
MOE				
Wood-(nylon 12)	D (β)	C (α)	Β (α)	Α (α)
Wood-PP	C (α)	Β (β)	$A/B(\beta)$	Α (α)
Wood-HDPE	Β (γ)	$A(\gamma)$	Α (γ)	Α (β)
Tensile strength	(1)		(1)	(17)
Wood-(nylon 12)	Β (α)	Α (α)	Α (α)	Α (α)
Wood-PP	Α (β)	Β (β)	$B/C(\beta)$	C (β)
Wood-HDPE	Α (γ)	Β (γ)	C (γ)	D (γ)

The English letters are used for comparing each strength property in the same row and the Greek letters are used for comparing each strength property in the same column. The same letter indicates no significant difference at a 95% confidence level. same wood flour content (40, 50, or 60 wt %), wood-(nylon 12) composites had a higher MOR than wood-PP or wood-HDPE composites (Fig. 1 and Table I). At each wood flour content, wood-PP composites had a higher MOR than wood-HDPE composites.

Wood-(nylon 12) composites at 40 wt % wood flour content had a higher MOR than nylon 12 (Fig. 1 and Table I). The MOR of wood-(nylon 12) composites remained statistically the same when the wood flour content was increased from 40 to 50 wt %, and significantly increased when the wood flour content was further increased from 50 to 60 wt %. Wood-PP composites at 40 wt % wood flour content had a lower MOR than PP. The MOR of wood-PP composites gradually decreased when the wood flour content was increased from 40 to 60 wt %. Wood-HDPE composites at 40 wt % wood flour content had a higher MOR than HDPE. However, the MOR of the wood-HDPE composites significantly decreased when the wood flour content was increased from 40 to 60 wt % (Fig. 1 and Table I).

Nylon 12 had a lower MOE than PP, but had a higher MOE than HDPE (Fig. 2 and Table I). At 40 or 50 wt % wood flour content, the MOE of wood-thermoplastic composites had the following order: wood-(nylon 12) > wood-PP > wood-HDPE (Fig. 2 and Table I). At 60 wt % wood flour content, however, the MOE of wood-(nylon 12) composites was statistically the same as that of wood-PP composites, but was higher than that of wood-HDPE composites (Fig. 2 and Table I). At each wood flour content, the MOE of wood-PP composites was higher than that of wood-HDPE composites (Fig. 2 and Table I). At each wood flour content, the MOE of wood-PP composites was higher than that of wood-HDPE composites (Fig. 2 and Table I).

Wood-(nylon 12) composites at 40 wt % wood flour content had a higher MOE than nylon 12. The increase in the wood flour content from 40 to 60 wt % significantly increased the MOE of wood-(nylon 12) composites (Fig. 2 and Table I). Wood-PP composites



Figure 2 Effect of wood flour content on MOE of woodthermoplastic composites. Data are the mean of ten replicates and the error bars represent two standard deviations.



Figure 3 Effect of wood flour content on tensile strength of wood-thermoplastic composites. Data are the mean of five replicates and the error bars represent two standard deviations.

at 40 wt % wood flour content had a higher MOE than PP. The MOE of wood-PP composites significantly increased when the wood flour content was increased from 40 to 60 wt % (Fig. 2 and Table I). Wood-HDPE composites at 40 wt % wood flour content had a higher MOE than neat HDPE. However, the MOE of wood-HDPE composites remains statistically the same when the wood flour content was increased from 40 to 60 wt % (Fig. 2 and Table I).

Nylon 12 had higher tensile strength than PP, and PP had higher tensile strength than HDPE (Fig. 3 and Table I). At each wood flour content (40, 50, or 60 wt %), the tensile strength of wood-(nylon 12) composites was significantly higher than that of wood-PP composites, and the MOE of wood-PP composites was higher than that of wood-HDPE composites (Fig. 3 and Table I).

The tensile strength of wood-(nylon 12) composites at 40 wt % wood flour content was higher than that of nylon 12, but the tensile strength remained statistically the same when the wood flour content was increased from 40 to 60 wt % (Fig. 3 and Table I). The tensile strength of wood-PP composites at 40% wood flour content was lower than that of PP, and the tensile strength gradually decreased when the wood flour content was increased from 40 to 60 wt % (Fig. 3 and Table I). The tensile strength of wood-HDPE composites at 40 wt % wood flour content was lower than that of HDPE. The tensile strength of wood-HDPE composites significantly decreased when the wood flour content was increased from 40 to 60 wt % (Fig. 3 and Table I).

Thermal stability

There was small weight loss of wood before 100°C, which was due to the evaporation of water from

wood (Fig. 4).³ The weight loss of wood from 100 to 225°C was negligible (Fig. 4). The rate of weight loss for wood gradually increased from 225°C onward and there was a sharp weight loss between 250 and 400°C. These results are consistent with published results that hemicelluloses, lignin, and cellulose start to degrade at 225, 250, and 325°C, respectively.¹² About 75 wt % of wood had been degraded at 400°C (Fig. 4).

Nylon 12 and PP had a very similar thermal stability and had negligible degradation until 400°C (Fig. 4). HDPE even did not start to degrade until about 450°C (Fig. 4). Therefore, all three thermoplastics had a much higher thermostability than wood. All three thermoplastics were almost completely decomposed at about 500°C, which means that all three thermoplastics had a similar thermostability and had a similar thermal degradation pattern (Fig. 4).

All three wood-thermoplastic composites had a higher thermal stability than wood (Fig. 5). Wood-PP and wood-HDPE composites had two distinct rapid degradation stages, one at 300-400°C and another at 450-520°C (Fig. 5). The degradation stage at 300-400°C resulted from the degradation of wood, and the degradation stage at 450-520°C resulted from the degradation of PP or HDPE. The weight loss rate at 400-450°C for wood-PP or wood-HDPE composites was much lower than that at the two previously mentioned stages. However, wood-(nylon 12) composites had a more uniform degradation rate at 300-500°C than wood-PP or wood-HDPE composites (Fig. 5). Wood-(nylon 12) composites lost lower weight than wood-PP or wood-HDPE composites at any given temperature between 300 and 400°C where the weight loss resulted from the degradation of wood in the composites. This implied that nylon 12 slowed down



Figure 4 Thermogravimetric curves of wood, PP, HDPE and nylon 12.



Figure 5 Thermogravimetric curves of wood-PP, wood-HDPE and wood-(nylon 12) composites.

the degradation of wood. Wood-(nylon 12) composites were thus more thermally stable than wood-PP or wood-HDPE composites below 450°C.

The effect of the wood flour content on the thermal stability of wood-(nylon 12) composites is shown in Figure 6. The weight loss percentage of a wood-(nylon 12) composite was higher than that of nylon 12 and lower than that of wood at any given temperature between 300 and 480°C. At any given temperature between 300 and 480°C, the lower the wood/(nylon 12) weight ratio in the wood-(nylon 12) composites the lower the weight loss percentage (Fig. 6).

Interfacial morphology

SEM graphs of fractured surfaces of the wood-thermoplastic composites are shown in Figure 7. For wood-HDPE composites [Fig. 7(a,b)], most of wood was pulled out and the fractured surfaces were very rough. Wood-PP composites had readily visible gaps between wood and PP [Fig. 7(c)]. The rough grooved surface that resulted from wood pullout was also readily visible at high magnification for wood-PP composites [Fig. 7(d)]. The fractured surfaces of wood-(nylon 12) composites were smoother than those of wood-PP and wood-HDPE composites [Fig. 7(e,f)]. The fractured surfaces also show little wood pullout. It appeared that most of wood was embedded in the nylon matrix even after tensile failure [Fig. 7(e,f)]. The cavities or gaps in Figure 7(f) appeared to result from broken wood rather than wood pullout. There were few gaps between wood and nylon 12.

Crystallization and melting behaviors

The results of DSC analyses are listed in Table II. Determined from DSC cooling curves, the crystallization temperatures (T_c) of HDPE, PP, and nylon 12 were 115.4°C, 119.5°C, and 134.8°C, respectively. The T_c of HDPE in wood-HDPE composites or PP in wood-PP composites was not significantly different from that of HDPE or PP, which implied that incorporation of wood in HDPE or PP did not significantly change T_c of HDPE or PP even when the wood flour content was as high as 60 wt % (Table II). However, the T_c of nylon 12 in wood-(nylon 12) composites was atleast 16.8°C higher than that of nylon 12 (Table II). The degree of crystallinity (X_c) of HDPE in wood-HDPE composites or PP in wood-PP composites was the same as that of HDPE or PP, whereas the X_c of nylon 12 in wood-(nylon 12) composites was 1-12.1% higher than that of nylon 12 when the wood flour content was increased from 40 to 60 wt % (Table II).

All three wood-thermoplastic composites had a melting temperature (T_m) close to that of corresponding thermoplastic (Table II). PP, HDPE, wood-PP, and wood-HDPE composites all had one melting temperature peak, whereas wood-(nylon 12) composites had two melting temperature peaks at 180 and 175°C, respectively, (Fig. 8). It is still poorly understood why wood-(nylon 12) composites had two melting temperature peaks.

DISCUSSION

The interfacial adhesion between wood and HDPE or between wood and PP is very poor because hydrophilic wood is not compatible with hydrophobic polyolefins. The poor interfacial adhesion does not allow the effective transfer of stress from PP or HDPE matrix to wood. Wood-HDPE and wood-PP composites thus typically have lower MOR and lower tensile strength than the corresponding HDPE and PP.^{13,14}



Figure 6 Thermogravimetric curves of wood-(nylon 12) composites with different wood/(nylon 12) weight ratios.



Figure 7 SEM micrographs of tensile-fractured surfaces of wood-thermoplastic composites with 40 wt % wood. (a) wood-HDPE composites $(100 \times)$, (b) wood-HDPE composites $(505 \times)$, (c) wood-PP composites $(100 \times)$, (d) wood-PP composites $(100 \times)$, (e) wood-(nylon 12) composites $(100 \times)$, and (f) wood-(nylon 12) composites $(100 \times)$.

Nylon 12 has numerous amide groups that can form strong hydrogen bonding with hydroxyl groups of wood. The hydrogen bonding will improve the interfacial adhesion between wood and nylon 12, thus improving the strength of the wood-(nylon 12) composites. The SEM graphs of fractured composite specimens indeed demonstrated that the interfacial adhesion between wood and nylon 12 was stronger than those between wood and HDPE or between wood and PP. These explain why wood-(nylon 12) composites

	$-\Delta H_c$			ΔH_m				
Composite specimen	T_c (°C)	(J/g)	T_m (°C)	(J/g)	X_c (%)			
Nylon 12	134.8	52.0	180.2	45.7	22.3			
40 wt % wood + 60 wt % nylon 12	152.4	32.7	179.1	25.8	23.3			
50 wt % wood + 50 wt % nylon 12	152.7	29.3	178.6	25.9	25.1			
60 wt % wood + 40 wt % nylon 12	152.4	32.1	178.6	27.5	34.4			
PP	119.5	98.1	164.2	95.0	46.7			
40 wt % wood + 60 wt % PP	116.3	58.6	163.0	51.1	46.8			
60 wt % wood + 40 wt % PP	116.5	38.9	162.2	33.2	46.6			
HDPE	115.4	181.9	133.1	173.0	62.9			
40 wt % wood + 60 wt % HDPE	116.4	107.7	133.4	106.4	62.1			
60 wt % wood + 40 wt % HDPE	116.3	72.6	133.0	68.7	62.6			

 TABLE II

 Thermal Properties of Wood-Thermoplastic Composites

в C D Heat flow (W/g) E A- 40 wt % Wood + 60 wt % Nvlon 12 B- Nylon 12 C-40 wt % Wood + 60 wt % PP D- PP E- 40 wt % Wood + 60 wt % HDPE F- HDPE 40 60 100 120 140 160 180 200 20 80 0 Temperature (°C)

Figure 8 Melting curves of thermoplastics and woodthermoplastic composites from the second heating procedure of the DSC characterization.

have higher MOR and higher tensile strength than wood-HDPE or wood-PP composites (Fig. 1).

Wood significantly promoted the crystallization of nylon 12 in wood-(nylon 12) composites, but had limited effects on promoting the crystallization of HDPE in wood-HDPE composites or of PP in wood-PP composites (Table II). The strong hydrogen bonding between wood and the nylon 12 matrix may facilitate wood to act as a nucleating agent. Presumably, the promoted crystallization was mainly initiated from wood, which resulted in transcrystallization. The transcrystalline structure can facilitate stress transfer at the interface, thus effectively improving the mechanical properties of the resultant composites.8,15 The required energy for breaking the composites also increases along with increasing the degree of crystallinity of the thermoplastic matrix. Therefore, the increased degree of crystallinity of nylon 12 in wood-(nylon 12) composites may be a part of reasons why wood-(nylon 12) composites had higher strength and stiffness than wood-HDPE or wood-PP composites.

It is still not fully understood why wood-(nylon 12) composites were more thermally stable than wood-HDPE and wood-PP composites. One of the speculations is that strong hydrogen bonding between wood and nylon 12 increased the energy required for thermal degradation of wood, thus slowing down the thermal degradation of wood.

CONCLUSIONS

Wood-(nylon 12) composites had much higher MOR, MOE, and tensile strength than nylon 12. Wood-(nylon 12) composites also had higher mechanical properties than wood-PP and wood-HDPE composites. Wood significantly increased the crystallization temperature and the degree of crystallinity of nylon 12. The strong interfacial adhesion between wood and nylon 12 and the increased degree of crystallinity of nylon 12 in wood-(nylon 12) composites are proposed to be responsible for the superior mechanical properties of wood-(nylon 12) composites. Wood-(nylon 12) composites were more thermally stable than wood-HDPE and wood-PP composites. The strong hydrogen bonding between nylon 12 and wood may account for the improved thermal stability of wood-(nylon 12) composites.

We thank EMS-CHEMIE North America and BP Solvay Polyethylene North America for providing us with thermoplastics. We are grateful to Dr. Willie E. Skip Rochefort and Mr. Kevin Harris for use of TGA and DSC instruments. We also appreciate Dr. Lech Muszynski for his support on the preparation of tensile specimens of wood-thermoplastic composites.

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