

TENSILE AND IMPACT PROPERTIES OF STEAM-EXPLODED WOOD-POLYPROPYLENE COMPOSITES

*Qingzheng Cheng**†

Postdoctoral Research Associate
Division of Forestry and Natural Resources
West Virginia University
Morgantown, WV 26506

Trairat Neimsuwan†

Assistant Professor
Department of Forest Products
Kasetsart University
Bangkok, 10903 Thailand

Siqun Wang†

Associate Professor
Tennessee Forest Products Center
University of Tennessee
Knoxville, TN 37996

Jingxin Wang†

Associate Professor
Division of Forestry and Natural Resources
West Virginia University
Morgantown, WV 26506

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Abstract. Wood-plastic composites were made from polypropylene and steam-exploded (SE) flour from small-diameter loblolly pine. SE wood content, maleic anhydride grafted polypropylene (MAPP) content, and extruder screw speed were investigated using an orthogonal test design to examine their effects on tensile and impact properties of the composites. The results showed that as SE wood-flour content was increased, the modulus of elasticity (MOE) of the composites also increased, whereas the tensile strength decreased. The MAPP content had a distinct effect on the tensile strength and no such effect on MOE; however, it had no clear trend for the impact properties of the composites. The wood content had distinct effects on both the tensile and impact properties of the composites. The screw speed also affected the tensile and impact properties of the composites.

Keywords: Impact, steam-exploded wood flour, tension, wood-plastic composites, SE, MAPP.

INTRODUCTION

The use of wood-plastic composites (WPCs) has grown greatly in the US in recent years, mainly in the residential decking market after the removal of chromated copper arsenate treated wood decking from residential markets (Rowell 2007). WPCs are intended for exterior use and

are typically subjected to many loads, both static and dynamic, during installation and in service. The typical method for producing WPCs is with a twin screw extruder, which accomplishes several tasks: feeding, melting, mixing, venting, and developing the material (Killough 1995; Machado and Martin 1997). Other methods used to fabricate WPCs include injection molding and mat forming followed by compression molding (Yin et al 2007; Cheng et al 2009b).

* Corresponding author: q.cheng@mail.wvu.edu

† SWST member

A common WPC-producing process in research includes compounding and pellet production followed by injection or compression molding. The polymer and wood particles or fibers are mixed together by extrusion or compounding in the compounding process. The compound is then chopped into pellets. The process parameters, materials characteristics, and application conditions all affect WPC properties (Rangaraj and Smith 2000; Kim et al 2008; Cheng et al 2009a, 2009b). In extrusion compounding, parameters that control the extruder performance include the screw rotational speed, feeding rate, temperature along the die and barrel, and vacuum level for the devolatilization process (Machado and Martin 1997).

Material characteristics can also affect WPC properties. For example, fiber damage or fiber attrition can occur from high shear stresses typical of the injection process (Osswald 1999). Also, because raw material properties can affect WPC performance, increasing wood content and particle size can help increase the mechanical properties (eg tension, flexure, compression, and impact) of WPCs if the wood particles are well dispersed in the polymer matrix (Stark and Berger 1997; Stark and Rowlands 2003). Because of the differences in raw material properties, a compatibilizer or copolymer can be used to help combine raw materials. Maleic anhydride grafted polypropylene (MAPP) is used to improve the compatibility between wood fibers and polypropylene (Snijder et al 1997).

The properties and treatment of the raw material also affect WPCs. For example, steam explosion (SE) treatment has been shown to be an effective method of producing wood flour/fibers. Steam explosion wood flour has reduced hygroscopicity and a higher content of crystalline and/or paracrystalline cellulose and has been shown to increase the mechanical properties and water resistance of polymer composites (Angles et al 1999; Yin et al 2007). The modulus of elasticity (MOE) of SE wood flour and polypropylene (PP) composites has been increased remarkably by using 50% flour (Yin et al 2007). The bending MOE of SE/PP composites made by a wet

process followed by compression molding was increased, whereas the modulus of rupture (MOR) decreased with increasing SE wood–flour content, and both MOE and MOR of the composites increased with an increase in MAPP content (Cheng et al 2009b). Apparently, there is a lack of information of how the raw material composition/ratio and processing affect the mechanical properties of SE wood-flour polymer composites. Therefore, the objective of this study was to investigate the effects of wood content, MAPP content, and extruder screw speed on the tensile and impact properties of the composites.

MATERIALS AND METHODS

Materials

Wood flour from SE small-diameter loblolly pine (*Pinus taeda* L.) was used as a filler material to make WPCs. Steam-exploded wood flour was produced in a batch reactor at 228°C with a retention time of 25 min (Yin et al 2007). Steam-exploded wood flour is a mixture of wood elements of different sizes, including particles, single fibers, and fiber fragments. To make a homogeneous distribution in the plastic matrix, the flour must be fine, and oversized particles must be eliminated by screen filtering. After the large particles were refined by using a Wiley mill, a #20 screen (0.84 mm) was used to separate the oversized particles. All of the wood flour that passed through the screen was used. The wood flour was dried to constant weight before compounding. Polypropylene (MFI = 4 g/min, Hercules) was used as a matrix and MAPP (EPOLENE G-3003, acid number: 8, average molecular weight = 52,000) was used as a coupling agent.

Experimental Design and Composite Processing

The experimental design was an orthogonal test with 4 factors with each factor at 3 levels ($L_9[3^4]$; Table 1) to investigate the effects of three factors (wood fiber content, MAPP content, and extrusion screw speed) on the mechanical properties of

the composites (Wu et al 1995). The blank column was used as error column for data analysis. A corotating twin-screw extruder was used to mix the composite components for each sample. The temperatures of the three zones (feed, transition, and metering) in the extruder were 165, 195, and 210°C. The compounded material was immediately cooled in a water bath and pelletized. After being dried, the pellets were used to make injection-molded tensile specimens (“dog bone”) in accordance with ASTM D 1708 (ASTM 2002a) and Izod impact bars in accordance with ASTM D 256 (ASTM 2002b) using an injection machine. The injection mold temperature was 210°C. Pellets were heated in the cylinder until melt (190°C) and then injected to the heated mold for 5 s. The mold was opened and samples were cooled under room conditions. The thickness of the molds was 4.76 mm.

Testing and Data Analysis

Tensile tests were conducted according to ASTM D 1708 (ASTM 2002a) using an Instron testing machine (model 5567) with a crosshead speed of 3.9 mm/min. The notched Izod impact strength was tested according to ASTM D 256 (ASTM 2002b) using an Izod impact tester (Tinius Olsen model 92T Impact Tester). Both tensile and notched Izod impact tests had five replications. Maximum difference and analysis of variance (ANOVA) for the orthogonal test were used to find the effects of the different

factors on the mechanical properties (Wu et al 1995). Maximum difference analysis is defined as $R_j = \text{Max}(I_j, II_j, III_j) - \text{Min}(I_j, II_j, III_j)$, where $j = A, B, C$, or D in Table 1. $I_j = \text{SUM}(y_1)$, $II_j = \text{SUM}(y_2)$, $III_j = \text{SUM}(y_3)$, where y is each property and 1, 2, 3 are the three levels in Table 1. A pair comparison by the Statistical Analysis System (t-tests [least significant difference]) was also used to check the significance of the effects on the properties between levels of each factor or between factors ($\alpha = 0.05$).

RESULTS AND DISCUSSION

Tensile Properties

The wood and MAPP contents significantly affected the tensile properties of the composites, and screw speed also influenced those properties in some cases (Figs 1-3). With no addition of MAPP, the tensile strength (TS) decreased when wood content increased, whereas MAPP addition improved the TS of WPCs. Normally, adding wood fiber can increase the tensile MOE of WPCs (Stark and Berger 1997). Adding SE wood flour to WPCs sharply decreased the elongation at break (EB) of PP as a result of the lower EB of wood particles (Fig 1) (Tasdemir et al 2008). The MAPP did not significantly influence the EB of pure PP but dramatically

Table 1. Orthogonal test design with 4 factors and each factor at 3 levels ($L_9[3^4]$).

| Sample no. | Wood content A (%) | MAPP content B (%) | Screw speed C (rpm) | Error column D ^a |
|------------|--------------------|--------------------|---------------------|-----------------------------|
| 1 | 1 (0) | 1 (0) | 1 (9) | 1 |
| 2 | 1 (0) | 2 (5) | 2 (18) | 2 |
| 3 | 1 (0) | 3 (10) | 3 (27) | 3 |
| 4 | 2 (25) | 1 (0) | 2 (18) | 3 |
| 5 | 2 (25) | 2 (5) | 3 (27) | 1 |
| 6 | 2 (25) | 3 (10) | 1 (9) | 2 |
| 7 | 3 (50) | 1 (0) | 3 (27) | 2 |
| 8 | 3 (50) | 2 (5) | 1 (9) | 3 |
| 9 | 3 (50) | 3 (10) | 2 (18) | 1 |

^a This blank column (no factor) is used as error column for the analysis of variance as shown in Table 3.

MAPP, maleic anhydride grafted polypropylene.

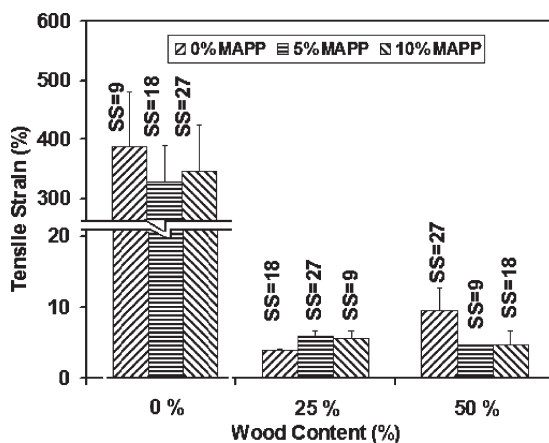


Figure 1. Tensile strain of composites with different wood content, maleic anhydride grafted polypropylene (MAPP) content, and screw speed (SS).

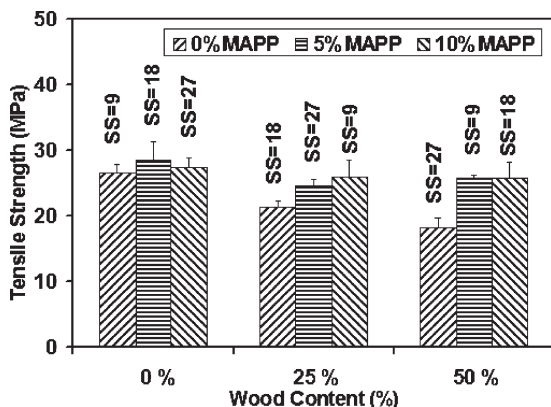


Figure 2. Tensile stress of composites with different wood content, maleic anhydride grafted polypropylene (MAPP) content, and screw speed (SS).

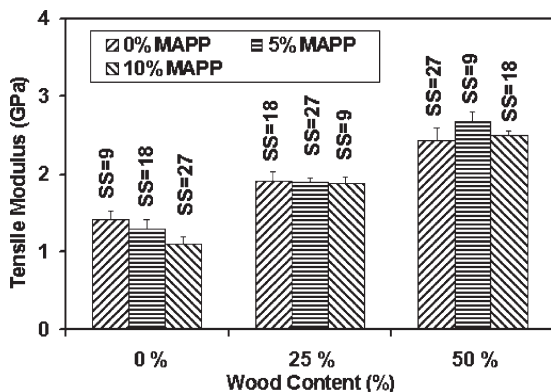


Figure 3. Tensile modulus of composites with different wood content, maleic anhydride grafted polypropylene (MAPP) content, and screw speed (SS).

affected the EB of 25% wood composites. Adding MAPP decreased the EB of 50% wood composites mainly because the screw speeds of 9 and 18 rpm were not high enough for this wood content in that the wood and MAPP did not disperse well in the composites. There were no differences of EB between 5 and 10% MAPP additions for both 25 and 50% wood composites (Fig 1).

MAPP influenced the tensile strength of the PP matrix before blending, but not statistically (Fig 2). The strength of wood composites without MAPP decreased dramatically, but adding MAPP

increased the TS of wood composites, which had comparable TS as pure PP. The highest screw speed of 27 rpm for the 50% wood composite without MAPP still had much lower TS, which indicated that MAPP had much more influence on TS than screw speed and that TS of wood composites with MAPP could increase if greater screw speed had been used (Fig 2). These results had the same trend of wood fiber modified with MAPP that can increase the TS because MAPP can form new covalent bonds and hydrogen bonds with the hydroxyl groups on the fiber surface (Kazayawoko and Balatinecz 1997; Snijder et al 1997).

The wood addition significantly increased the tensile MOE, whereas the addition of MAPP did not affect tensile stiffness (Fig 3). Similar results were obtained showing that MAPP did not significantly affect tensile or flexural moduli (Stark and Rowlands 2003). The screw speed also did not influence tensile MOE in the 25 and 50% wood content. Compared with commercial wood flour (Stark and Rowlands 2003), SE wood flour had a greater improvement for tensile MOE but was lower in TS without MAPP. After MAPP was added, SE wood flour could have a comparable influence on TS as commercial wood flour.

For different properties, a group of the most indicative factors can be chosen from the results using the maximum difference. The changing of each property according to the changing of the levels of the different factors may also be observed by data analysis (Wu et al 1995). Table 2 shows the maximum difference (R) analysis on the tensile and impact properties of the composites in the orthogonal test. The R values indicated that the effects of the factors for MOE were ranked as follows: wood content > screw speed > MAPP content, but there was no significant difference between screw speed and MAPP content. For TS, the effects of the factors were ranked as follows: MAPP content > wood content > screw speed. The effects of factors for maximum strain were ranked in the following order: wood content > MAPP content > screw speed, but again, there

Table 2. Maximum difference analysis (R_j) for the properties of the composites.

| Items | Wood content A (%) | MAPP content B (%) | Screw speed C (rpm) | Error column D |
|------------------|--------------------|--------------------|---------------------|----------------|
| Tensile MOE | 3.81 | 0.38 | 0.55 | 0.20 |
| Tensile strength | 11.50 | 14.25 | 8.02 | 6.01 |
| Tensile strain | 1046.0 | 63.2 | 62.3 | 55.1 |
| Impact strength | 0.96 | 0.16 | 0.14 | 0.12 |

MAPP, maleic anhydride grafted polypropylene; MOE, modulus of elasticity.

was no significant difference between the effects of MAPP content and screw speed. Generally, the stiffness of the composites was increased by adding wood fillers, but the composite strength also decreased because there was little or no adhesion between the filler and the matrix. The addition of MAPP might be helpful for both stiffness and strength (Stark and Berger 1997; Cheng et al 2009b). The ANOVA showed the same results as the maximum difference analysis. Table 3 shows the ANOVA on tensile MOEs. The wood content factor had a significant influence on MOE, whereas MAPP only slightly influenced the MOE presumably because the MAPP may be not completely dispersed in the WPCs (Cheng et al 2009b).

Impact Strength

Impact strength was decreased by adding wood to the PP matrix, although the addition of MAPP and screw speed did not provide clear trends. The pure PP with 5% MAPP had the highest impact strength. Compounds made by adding MAPP to the pure PP have higher impact strengths than compounds without MAPP. However, the compounds with 25 and 50% wood content addition showed lower impact strength (Fig 4).

The tests of notched Izod specimens reflected the energy required for crack propagation. Crack propagation occurred at the PP-wood flour interface because of the poor interface between the hydrophilic wood flour and the hydrophobic PP. The higher SE wood-flour content significantly decreased the composite impact strength (Fig 4). This result was different compared with

Table 3. Analysis of variance on tensile modulus of elasticity.

| Sources | S_j | f_j | \bar{S}_j | F_j^A | $F_{(1-\alpha)}$ | Significant |
|--------------|--------|-------|-------------|---------|------------------|-------------|
| Wood content | 2.4152 | 1 | 2.4152 | 349.35 | 161 | * |
| MAPP content | 0.0245 | 1 | 0.0245 | 3.54 | 161 | |
| Screw speed | 0.0498 | 1 | 0.0498 | 7.20 | 161 | |
| Error | 0.0069 | 1 | 0.0069 | | | |
| Total error | 0.0069 | 1 | 0.0069 | | | |

S_j is the sum of the squared deviation of the j column, here $S_j = 1/3(I_j^2 + II_j^2 + III_j^2) - T^2/9$, where $T = \text{SUM}(y_i)$, f_j is the degree of freedom, $\bar{S}_j = S_j/f_j$, $F_j^A = \bar{S}_j/(S_e^A/f_e^A)$, where S_e^A is total error of error items and f_e^A is the total degree of freedom of error items. $F_{(1-\alpha)}$ is F critical value, when $\alpha = 0.05$, $F_{0.95}(f_j, f_e^A)$.

MAPP, maleic anhydride grafted polypropylene.

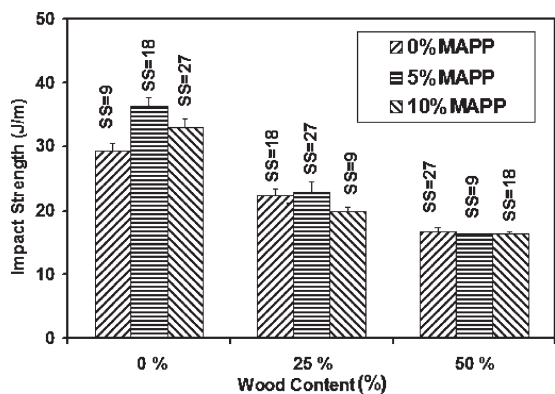


Figure 4. Impact strength of composites with different wood, maleic anhydride grafted polypropylene (MAPP) contents, and screw speed (SS).

commercial wood flour-reinforced PP composites, in which higher wood content slightly increased the notched impact strength of the composites (Stark and Rowlands 2003). This may be from the difference between the SE wood flour and commercially mechanically milled wood flour such as surface chemistry, intrinsic wood particle properties, and geometric differences (Angles et al 1999; Stark and Rowlands 2003; Yin et al 2007).

With the addition of 25% wood flour to WPCs, the impact strength was significantly reduced. The impact strength of the 25% wood composite with 10% MAPP was significantly lower than both 0 and 5% MAPP composites mainly because of its lower screw speed (9 rpm), which influenced the dispersion of wood flour and MAPP. With the addition of 50% wood content,

the impact strength continuously decreased, but no differences were detected among the MAPP contents and screw speeds (Fig 4). The addition of MAPP to the WPCs did not affect notched impact strengths, but it could improve unnotched impact energy because interfacial bonding lowers the stress concentrations, which can increase unnotched impact energy (Stark and Rowlands 2003).

The maximum differences of impact strength are also shown in Table 2. The R values indicate that the effects of factors were ranked as follows: wood content > MAPP content > screw speed. There was no significant difference between screw speed and MAPP content. The ANOVA on impact strength indicated that only the effect of the wood-flour content on the impact strength was significant at $\alpha = 0.05$.

CONCLUSIONS

Steam-exploded wood flour and PP with and without MAPP were used to fabricate WPCs. The processing and material variables were shown to have an influence on the composite characteristics. An orthogonal test design was used to compare the influences of wood content, MAPP content, and extruder screw speed on the tensile and impact properties of the composites. The SE wood content had significant effects on both the tensile and impact properties of the composites. MAPP content had a distinct effect on the tensile strength but no significant effect on the tensile modulus and no clear trend on the impact properties. Screw speed had a significant effect on some of the tensile and impact properties. The effects of factors on these properties were ranked as follows: wood content > MAPP content > screw speed.

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