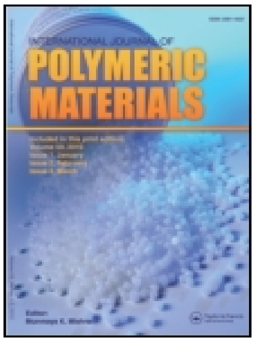

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International Journal of Polymeric Materials

ISSN: 0091-4037 (Print) 1563-535X (Online) Journal homepage: https://www.tandfonline.com/loi/gpom20

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**To cite this article:** Yali Li (2012) Effect of Coupling Agent Concentration, Fiber Content, and Size on Mechanical Properties of Wood/HDPE Composites, International Journal of Polymeric Materials, 61:11, 882-890, DOI: 10.1080/00914037.2011.617338

**To link to this article:** https://doi.org/10.1080/00914037.2011.617338

*International Journal of Polymeric Materials*, 61:882–890, 2012

ISSN: 0091-4037 print/1563-535X online
DOI: 10.1080/00914037.2011.617338

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# Effect of Coupling Agent Concentration, Fiber Content, and Size on Mechanical Properties of Wood/HDPE Composites

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In this study, the influence of coupling agent concentration (0 and 3 wt%), wood fiber content (50, 60, 70, and 80 wt%), and size (40–60, 80–100, and 160–180 mesh) on the mechanical properties of wood/high-density-polyethylene (HDPE) composites (WPCs) was investigated. WPC samples were prepared with poplar wood-flour, HDPE, and polyethylene maleic anhydride copolymer (MAPE) as coupling agent. It was found that the tensile properties and the flexural properties of the composites were improved by the addition of 3 wt% MAPE, and the improved interfacial adhesion was well confirmed by SEM micrographs. It was also observed that the best mechanical properties of wood/HDPE composites can be reached with larger particle size in the range studied, while too-small particle size was adverse for the mechanical properties of wood/HDPE composites. Moreover, the tensile modulus, tensile strength, and flexural strength of WPCs decreased with the increase in fiber content from 50 to 80 wt%; the flexural modulus of WPCs increased with the increase in fiber content from 50 to 70 wt% and then decreased as the fiber content reached 80 wt%. The variances in property performance are helpful for the end-user to choose an appropriate coupling agent (MAPE) concentration, wood fiber content, and particle size based on performance needs and cost considerations.

**Keywords** composites, coupling agent, HDPE, mechanical properties, wood flour

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Received 1 May 2011; accepted 21 August 2011.
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## INTRODUCTION

In recent years, wood fiber-reinforced plastic composites or so-called wood-plastic composites (WPCs) have attracted significant interest and they are replacing wood in many fields, including a variety of building products, automotive, infrastructure, and other industrial applications, because of their specific advantages such as high strength, low cost, biodegradability, low water absorption and thickness swelling, and better recyclability [1–4]. In general, the properties of WPCs depend on various factors, including the inherent properties of the constituent materials, interactions among these materials, and processing methods [5–7]. Since the fiber-matrix interface between the hydrophilic wood fiber and hydrophobic polyolefin plays a key role in transferring stress between the two components, the incompatibility of polar wood fiber and non-polar polyolefin leads to difficulties in obtaining uniform dispersion of wood fibers in the matrix, which in turn reduces the efficiency with which the fibers reinforce the polymer; many efforts are being made to improve the compatibility between them [8–14]. One effective method is applying a coupling agent to the system [8–9, 12–14].

Fiber content is an influential factor in WPC processing and properties. Valle and coworkers [15] investigated the effect of wood content on the thermal behavior and the molecular dynamics of PVC/wood composites. They concluded that the increasing addition of wood flour caused a small but progressive improvement of the decomposition temperature of the composites, whereas the glass transition temperature remained practically unchanged, and a gradual decrease in $T_1H$ values was observed with increasing wood content, indicating that the composites became less rigid. Nourbakhsh and Ashori [16] investigated four levels of fiber loading (10, 20, 30, and 40 wt%) on the mechanical properties of poplar/PP composites. They found that the fiber loading of 30 and 40 wt% at 190°C provided adequate reinforcement to increase the tensile and flexural strength of the PP power, the modulus increased, and the elongation and impact properties of the composites decreased with increasing the fiber content. Zhang et al. [17] reported that increased wood fiber content resulted in increased steady-state torque and viscosity.

A variety of natural materials have been used in WPCs [18–21]. Among the various natural materials, wood fibers have attracted considerable attention in the fields of both fundamental research and applications due to their ease of handling and widespread availability [22]. The quality of wood fibers depends upon many factors such as the distribution of the particles, their shape and porosity, chemical nature of the surface, and content of impurities. One of the critical parameters influencing the strength properties of WPCs is the particle size of the fibers. However, results of fiber sizes on the mechanical behavior of WPCs are contradictory. Maiti and Singh [23] examined different fiber sizes of wood flour compounded with HDPE without coupling agents.

Despite poor adhesion between filler and plastic, the extruded samples showed increasing modulus of elasticity with decreasing particle size (180–425 μm). In contrast, Chen et al. [24] investigated the effect of wood particle size on wood/HDPE composites and found that plastic composites made of larger wood particles had higher strength. Nourbakhsh et al. [25] discussed the effect of wood particle size on wood-flour/PP composites and found that the tensile modulus was not significantly affected by particle size, the tensile strength increased, and the notched izod decreased with decreasing particle size.

Here, the objects of this study were to investigate the effects of coupling agent (MAPE) concentration, wood fiber content, and particle size on the mechanical properties of poplar wood-flour/HDPE composites. The results were compared with the contradictory results of the previous work. The variances in property performance with the coupling agent (MAPE) concentration, wood fiber content, and particle size are helpful for the end-user to choose an appropriate coupling agent (MAPE) concentration, wood fiber content, and particle size based on performance needs and cost considerations.

## EXPERIMENTAL

### Materials

High-density-polyethylene (HDPE) was obtained from PetroChina Company Ltd. (Beijing, China) under the trade name T60-800 with a density of 0.963 g/cm$^3$ and a melt index 6–8 g/10 min at 190°C.

The poplar wood fibers used in this study were collected from a local sawmill. Subsequently, they were manually screened on a sieve and classified into three fractions, namely 40–60, 80–100, 160–180 mesh particles, and then were dried in an oven at 105°C for 24 h to a moisture content of less than 3%.

Polyethylene maleic anhydride copolymer (MAPE) with acid number of 7 mg/KOH and melting point 110°C was provided by Zibo Entong Plastic Powder Company Ltd. (Shandong, China).

### Sample Preparation

The wood fibers, HDPE pellets, and MAPE were blended together in an M-101 high-intensity laboratory mixer for about 3 min. The weight fraction of MAPE coupling agent was set at 0 and 3% of that of the wood fibers. A series of formulations having 50, 60, 70, and 80 wt% of 40–60 mesh wood fibers and 70% wood fibers with three different fractions were prepared. The mixture was processed with a Giant SHJ-30 co-rotating twin-screw extruder to make composite pellets. The temperatures of the first to the last chambers were 130, 135, 140, 140, and 130°C, respectively. The rotational speed was 30 rpm.

Then the composite pellets were extruded in an XSS-300 single-screw extruder with a die. The temperatures of the first to the last chambers were 150, 160, and 165°C, and the die was 150°C. The screw rotational rate was 20 rpm. The test specimens were conditioned before testing at $23 \pm 2$°C, $50 \pm 5$% RH for at least 40 h according to ASTM D 618.

## Mechanical Properties Test

The mechanical behavior of the samples was characterized via tensile and flexural tests in accordance with ASTM Standards D 638 and D 790, respectively. Strength measurements of the samples were conducted using a computer-controlled INSTRON testing machine (Model 4486). The crosshead speed of tension test was set at 10 mm/min. Six specimens were tested in each experiment to obtain a reliable average value.

## Fracture Surface Analysis

The fractured surfaces of the flexural test specimens with 70 wt% 40-60 mesh wood-flour were sputter-coated with gold and characterized with a HITACHI S-3000 N SEM at an accelerating voltage of 5 kV and emission current of 95 μA.

# RESULTS AND DISCUSSION

## Tensile Properties

The effects of coupling agent concentration, wood fiber content, and size (40–60, 60–80, and 160–180 mesh) on the mechanical properties of the composites are shown in Table 1. As can be seen, the tensile modulus is not significantly affected by particle size in the range studied; similar results have been reported by Nourbakhsh et al. [25], but the tensile strength is affected by particle size. For larger particle size, there is a moderate increase in strength. It rises from 10.2 MPa at particle size of 160–180 mesh to 12.4 MPa at particle size of 40 to 60 mesh. These results are in good agreement with the previous data [26–28]. This behavior may be due to the cutting of wood fibers during grinding, which affects the strength of wood fibers.

In order to evaluate the effectiveness of coupling agent (MAPE) on the strength behavior of the composites, they have been compared with and without coupling agent. From Table 1, we can see that the effect of coupling agent (MAPE) loading on the tensile properties of composites is significant. Whatever the particle size is, both the tensile modulus and strength are greatly improved by the addition of 3% coupling agent (MAPE). This is because of

**Table 1:** Mechanical properties of wood-flour/HDPE composites

| Particle size | | | | | | | |
|---|---|---|---|---|---|---|---|
| Mesh | mm | MAPE (%) | Fiber content (%) | Tensile modulus[a] (GPa) | Tensile strength[a] (MPa) | Flexural modulus[a] (GPa) | Flexural strength[a] (MPa) |
| 60–40 | 0.25–0.42 | 0 | 70 | 3.23 ± 0.21 | 12.4 ± 0.7 | 2.85 ± 0.23 | 27.2 ± 1.5 |
| 60–40 | 0.25–0.42 | 3 | 70 | 4.35 ± 0.30 | 24.4 ± 1.9 | 3.37 ± 0.15 | 51.1 ± 1.3 |
| 100–80 | 0.15–0.18 | 0 | 70 | 3.21 ± 0.16 | 11.1 ± 0.6 | 2.69 ± 0.20 | 25.0 ± 0.8 |
| 100–80 | 0.15–0.18 | 3 | 70 | 4.24 ± 0.40 | 23.5 ± 2.1 | 3.30 ± 0.26 | 49.3 ± 0.9 |
| 180–160 | 0.08–0.096 | 0 | 70 | 3.13 ± 0.38 | 10.2 ± 0.9 | 2.47 ± 0.12 | 22.0 ± 1.2 |
| 180–160 | 0.08–0.096 | 3 | 70 | 4.11 ± 0.33 | 22.8 ± 1.4 | 3.19 ± 0.16 | 42.5 ± 0.5 |
| 60–40 | 0.25–0.42 | 3 | 50 | 4.60 ± 0.29 | 30.2 ± 1.4 | 2.71 ± 0.20 | 58.8 ± 1.2 |
| 60–40 | 0.25–0.42 | 3 | 60 | 4.48 ± 0.17 | 26.3 ± 1.5 | 2.94 ± 0.28 | 54.3 ± 1.5 |
| 60–40 | 0.25–0.42 | 3 | 70 | 4.35 ± 0.30 | 24.4 ± 1.9 | 3.37 ± 0.15 | 51.1 ± 1.3 |
| 60–40 | 0.25–0.42 | 3 | 80 | 3.97 ± 0.14 | 18.6 ± 1.4 | 2.68 ± 0.22 | 45.5 ± 1.3 |

[a]All values are reported as mean (N = 6).

the improved interfacial adhesion between wood fibers and the matrix and stress transfer at the interface [29]. The improved interfacial adhesion was well confirmed by the SEM micrographs in Figure 1.

As shown in Table 1, the effect of wood fiber content on the tensile modulus is not significant, whereas that on the tensile strength is highly significant. With increased wood fiber content, there is a moderate decrease in the tensile modulus; it falls from 4.60 GPa to 3.97 GPa when fiber content increases from 50 to 80 wt%. However, the tensile strength decreases greatly with fiber content increasing from 50 to 80 wt%, which corresponds well with the reported data. Zaini et al. [28] reported decreasing tensile strength with increasing oil palm wood-flour filler loading from 20 to 50 wt%. However, Bouafif et al. [26] concluded that increasing maximum tensile strength increased eastern white cedar wood particle content from 0 to 45 wt%.

## Flexural Properties

Table 1 represents the result of flexural modulus and strength measurements of poplar wood-flour/HDPE composites. Apparently, flexural modulus

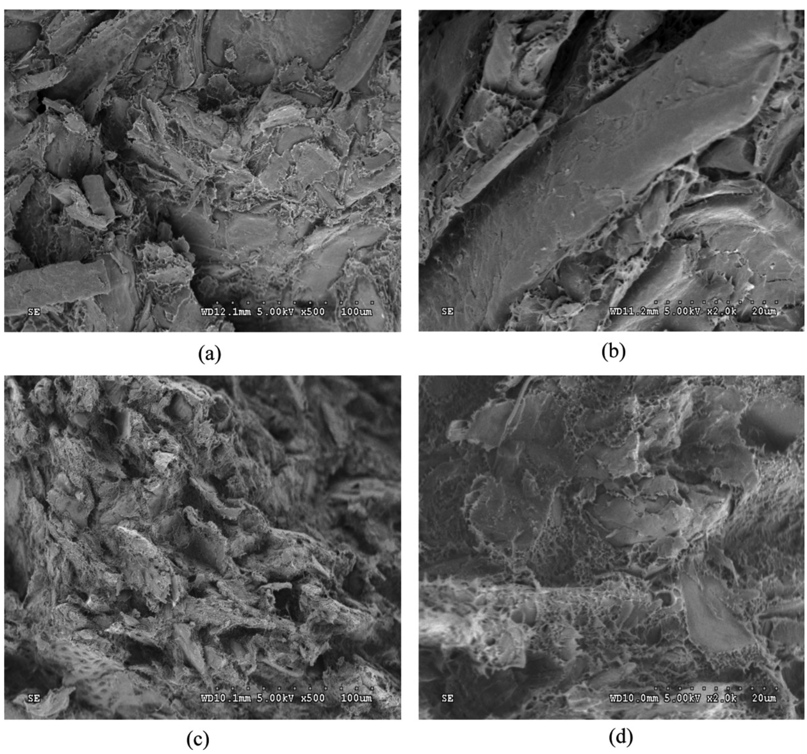


**Figure 1:** Flexural fracture surfaces of WPCs with and without MAPE as coupling agent.

and strength increase with increasing particle size. Similar results have been published by H. C. Chen et al. [24], who studied the effects of wood particle size and mixing ratios of HDPE on the properties of the composites.

Compared to wood particle size, coupling agent is a more important factor to the flexural properties. As shown in Table 1, both flexural modulus and strength of WPCs are greatly improved by the addition of 3% MAPE. It has been hypothesized that the maleic anhydride units in MAPE bond with the lignocellulosic fibers, while the polymer chain (polyethylene) in MAPE entangles with the polymer chains of the plastic matrix, leading to the improvement in the mechanical properties [30].

It can be observed that the flexural modulus of WPCs increases with the increase in fiber content from 50 to 70 wt% and then decreases as the fiber content reaches 80 wt%. The modulus of natural fibers was higher than that of HDPE [30]. Hence, when the fiber content of WPCs increased from 50 to 70 wt%, the modulus of the composite materials increased. However, in wood-plastic composites with higher levels of fiber content, plastics are utilized as adhesives for bonding wood particles together [31]. When the fiber content increased from 70 to 80 wt%, no sufficient adhesive bonding was present to achieve higher modulus values and WPC samples were easily bent under the load. The flexural strengths of WPCs significantly decrease with the increase in fiber content from 50 to 80 wt%. Chaharmahali et al. [31] have reported similar results, indicating a steady decrease of flexural strength with increase in fiber content from 60 to 80 wt%.

## Fracture Surface Observation

As reported in the literature, the morphology of polymer composites is a very important characteristic because it determines the physico-mechanical properties [32]. In this study, the state of the matrix/filler interface with and without MAPE as coupling agent was investigated by SEM.

Figure 1 shows the SEM micrographs of the fracture surfaces of WPC samples with 70 wt% 40–60 mesh wood-flour. As shown in Figure 1 (a) and (b), the surfaces of the wood fibers are quite smooth and cavities can be seen between wood fibers and the matrix polymer, which clearly indicates the poor interfacial adhesion between wood fibers and the matrix polymer. This result contributes to the poor stress transfer from matrix polymer to wood fibers, leading to poor mechanical properties. Figure 1 (c) and (d) shows the SEM micrographs taken from the fracture surface of the WPC sample with MAPE as coupling agent. It can be observed that there is better polymer/filler interfacial adhesion than that in the WPC sample without MAPE as coupling agent, which results in a reduction of the interfacial tension between wood fibers and the matrix polymer. This would be expected to increase the mechanical properties of the composites. This conclusion can be supported by the mechanical property values of WPCs shown in Table 1.

## CONCLUSIONS

This study focused on the effects of coupling agent concentration, wood fiber content, and discrete, non-overlapping particle size distributions on the mechanical properties of poplar wood-flour/HDPE composites. Based on the results above, the following conclusions can be drawn:

1. For poplar wood fiber, larger particle size showed superior mechanical properties in the range studied. Too-small particle size was adverse for the mechanical properties of wood/HDPE composites.
2. The tensile and flexural properties of wood/HDPE composites with MAPE as coupling agent were much higher than those of the ones without coupling agent. MAPE is a very effective coupling agent that can improve the interfacial adhesion of wood/HDPE composites.
3. Wood fiber content is an important factor in the properties of wood/HDPE composites. The tensile modulus, tensile strength, and flexural strength of WPCs decreased with the increase in fiber content from 50 to 80 wt%; the flexural modulus of WPCs increased with the increase in fiber content from 50 to 70 wt% and then decreased as the fiber content reached 80 wt%.

Therefore, we should choose an appropriate coupling agent concentration, wood fiber content, and particle size in making WPCs based on performance needs and cost considerations.

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