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The Influence of Coupling Treatment on Fungal Resistance of Wood Flour/Polypropylene Composites

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Abstract: The aim of this study was to investigate the effect of coupling agent loading on the natural durability of wood flour/polypropylene composites against white-rot fungi (*Trametes versicolor*). The blend composites were prepared through the melt mixing of wood flour/polypropylene at 50% weight ratios, with various amounts of compatibilizer (0, 2 and 4 per hundred compounds (phc)) in hake internal mixer then the samples made by injection molding. Then, the specimens were exposed to the purified fungus at 25°C and 75% relative humidity for 6 weeks. After the mentioned periods, weight loss, hardness, impact strength, flexural strength and water absorption of specimens were measured. The results indicated that the coupling treatment had significant effects on the natural durability, physical mechanical and properties of composites. The hardness, impact strength and flexural strength of composites increased with increase of coupling agent. However, the weight loss and water absorption decreased with increase of compatibilizer loading. Finally, it was found that the biggest improvement of the natural durability of composites exposed to rainbow fungus (*Trametes versicolor*) can be achieved for the coupling agent loading at 4 phc.

Key words: Composites · Fungal Resistance · Compatibilizer · Weight Loss · Hardness · Strength

INTRODUCTION

The need for materials having specific characteristics for specific purposes, while at the same time being nontoxic and environmentally friendly, is increasing, due to a lack of resources and increasing environmental pollution. Studies are ongoing to find ways to use lignocellulosic materials in place of synthetic materials as reinforcing fillers. Thus, research on the development of composites prepared using various recycled materials is being actively pursued. Among the possible alternatives, the development of composites using agro-wastes or lignocellulosic materials as reinforcing fillers and thermoplastic polymers as matrixes is currently at the center of attention. These composites would resolve environmental problems and offer the possibility of producing products having a range of different physical properties and functions.In recent years, Composite materials based on lignocellulosic materials, namely wood plastic composite (WPC), demonstrate remarkable environmental and economical advantages and they have, therefore, recently attracted much attention [1-3].

Although, wood plastic composite lumber is promoted as a low-maintenance and high-durability product, after a decade of exterior use in the construction industry, questions have arisen regarding durability. These questions are based on documented evidence of failures in the field of wood plastic composite decking products due to such impacts as polymer degradation, wood decay and susceptibility to mold which negatively impact the aesthetic qualities of the product [4].

Many different organism cause damage to wood, but fungi are among the most damaging. Decay fungi are single-celled or multicellular filamentous organisms that use wood as food. Brown-, white- and soft-rot fungi all appear to have enzymatic systems that demethoxylate lignin, produce endocellulases and with some fungi from each group, use single electron oxidation systems to modify lignin (Eaton and Hale 1993). Decay fungi need food, oxygen (air), the right temperature (10 to 35°C; optimum 24 to 32°C) and moisture (above the fiber saturation point; about 30% moisture content) to grow. White-rot fungi decompose all the structural components (cellulose, hemicelluloses and lignin) from wood. As the

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wood decays it becomes bleached (in part from the lignin removal) or white with black zone lines. White-rot fungi occur mainly on hardwoods but can be found on softwoods as well. The degraded wood does not crack across the grain until it is severely degraded. It keeps its outward dimensions but feels spongy. The strength properties decrease gradually as decay progresses, except toughness. White-rot fungi have a complete cellulose complex and also the ability to degrade lignin [5].

The wood components in wood plastic composites are responsible for its susceptibility to fungal attack and the fungal decay has a substantial effect on the physical and mechanical properties of wood plastic composites. Fungal attack in wood plastic composites has been reported in the some literatures [6-10]. The objective of this study is to evaluate the influence of coupling treatment on fungal resistance of wood plastic composites against white-rot fungi (*Trametes versicolor*).

MATERIALS AND METHODS

Material: The cellulosic material used as reinforcing filler in the composite was fresh Beech (*Fagus Orientalis*). Beech pieces were ground with a Thomas-Wiley mill to pass through a 60-mesh screen and then they were dried again and stored in sealed plastic bags prior to compounding. The polymer matrix used in this study was polypropylene (PP) with trade name EPC 40 R, was obtained from Arak Petrochemical Company, Iran. Its melt flow index was 7 g/10 min at 150°C and and a density of 0.965 g/cm^3. PP-g-MA provided by Solvay Co (Belgium); with trade name of Priex 20070 (MFI=64 gr/10min, grafted maleic anhydride 0.1 Wt. %) was used as coupling agent.

Composite Preparation: At the first, wood flour was dried in an oven at 65±2°C for 24 hours. Then polypropylene, wood flour and coupling were weighed and bagged according to formulations given in Table 1. The mixing was carried out by a hake internal mixer (HBI System 90, USA). First the PP was fed to the mixing chamber; after it was melted, the coupling agent was added. At the third minute, wood flour was added and the total mixing time was 10 min. The compounded materials were then ground using a pilot scale grinder (WIESER, WGLS 200/200 Model). The resulted granules were dried at 105°C for 4 hours. Test specimens were prepared by injection molding (Eman machine, Iran). Finally, the specimens were stored under controlled conditions (50% relative humidity and 23°C) for at least 40 hours prior to testing.

Table 1: Composition of the studied formulations

Polyethylene (Wt. %)	Wood flour (Wt. %)	Coupling agent (phc)
50	50	0
50	50	2
50	50	4

phc = per hundred compounds

Fungus Culture: Malt extract agar was used at a concentration of 48 g/L as the culture medium. Purified white-rot fungi (*Trametes versicolor*) were used in this study as the biological degradation agents. The purified white-rot fungi were transferred to Petri dishes containing malt extract agar under sterile hood using sterile pincers. The dishes were kept at 25ºC for one week until the culture medium was fully covered by the fungi. The cultured fungi were transferred into kolle-flasks containing the culture medium that were incubated for two weeks at 25ºC. Then, the test specimens were transferred into the kolle-flasks. The flasks containing the fungi and the wood plastic composite specimens were stored in an incubator for 6 weeks at 25ºC and 75% relative humidity.

Measurements: Dry weights of the specimens were measured after 24 h at 103 ± 2°C and weight losses were calculated using the following equation,

$$\text{Weight loss (\%)} = \frac{(MB - Ma)}{Mb} \times 100 \qquad (1)$$

Where *Mb* and *Ma* denote the oven-dry weights prior to and after incubation with fungi, respectively.

Hardness tests were carried out according to ASTM D-1037 specifications by an Instron hardness tester model 4486 and 10 KN load-cell and of each group, five specimens were tested. The cross-head speed was 5 mm/min (The amount of ball penetration in the specimen is 5.6 mm according to wood hardness standard, but because of the rupture of specimens at this rate, it was modified to 2 mm).

The flexural tests were measured according to ASTM D-790, using an Instron machine (Model 4486, England); the tests were performed at crosshead speeds of 5 mm/min. A Zwick impact tester (Model SIT 20 D, Iran) was used for the Izod impact test. All the samples were notched on the center of one longitudinal side according to ASTM D256. For each treatment level, five replicate samples were tested.

Water absorption tests were carried out according to ASTM D7031 specification. Specimens with a dimension of 20 × 20 × 20 mm were cut for water uptake measurements. Five replicates were used for each sample code. To ensure the same moisture content for the specimens before each test, all the specimens were oven-dried at 102±3°C. The weight and thickness of dried specimens were measured to a precision of 0.001 g and 0.001 mm, respectively. The specimens were then placed in distilled water and kept at room temperature. For each measurement, specimens were removed from the water and the surface water was wiped off using blotting paper. Weight and thicknesses of the specimens were measured at different time intervals during the long-time immersion. The measurements were terminated after the equilibrium thicknesses of the specimens were reached. The values of the water absorption in percentage were calculated using the following equation:

$$WA(t) = \frac{W(t) - W_0}{W_0} \times 100 \quad (2)$$

Where WA (t) is the water absorption at time t, W_o is the oven dried weight and W (t) is the weight of specimen at a given immersion time t.

Statistical Analysis: The statistical analysis was conducted using SPSS programming (Version 16) method in conjunction with the analysis of variance (ANOVA) techniques. Duncan multiply range test (DMRT) was used to test the statistical significance at $\alpha = 0.05$ level.

RESULT AND DISCUSSION

The results of an ANOVA indicated that the coupling agent content had significant effects ($p<0.05$) on the natural durability, physical and mechanical properties of wood flour/polypropylene composites (Table 2).

The influence of coupling agent content on the weight loss of composites was shown in Figure 1. As can be seen, the weight loss of composites decreased with increase of coupling treatment. White-rot fungi (*Trametes versicolor*) deteriorate wood by decaying the lignin. The presence of more wood flour implies more lignin in the system. Therefore, the higher weight loss of composites containing more wood flour can be explained by the fact that more lignin is available for the fungus to feed on. The presence of the coupling agent makes the wood content less accessible for the fungus through better wetting of wood particles.

Also as shown in Figure 2, the hardness of wood flour/polypropylene composites increased with increase of coupling agent. It is well established that that presence of coupling agent enhances the interface adhesion between wood flour and pp matrix and brings better

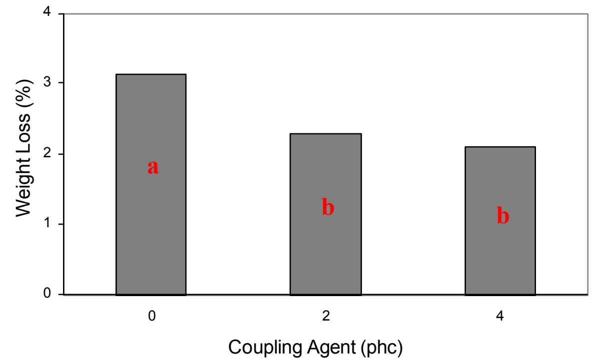

Fig. 1: Effect of coupling agent content on weight loss of composites

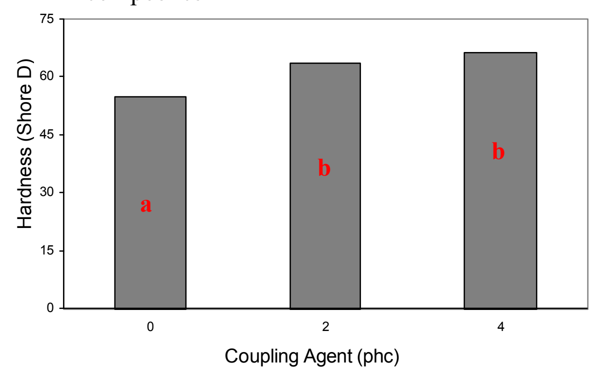

Fig. 2: Effect of coupling agent content on hardness of composites

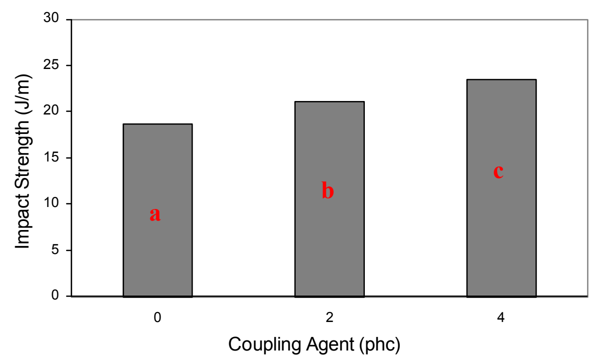

Fig. 3: Effect of coupling agent content on impact strength of composites

encapsulation of wood particles by the plastic which consequently results in higher hardness. The fungus only deteriorated the points in the specimens that have been more accessible. Because in the Shore D hardness test, a needle penetrates the specimen, it is likely to reach areas with no damage at all.

The effect of coupling agent content on the flexural strength of wood flour/polypropylene composites is shown in Figures 3. As can be seen, the flexural strength of composites increases with increase of coupling agent.

Table 2: Statistical analysis of variance (ANOVA) for the effect of coupling treatment on natural durability of composites

	Sum of Square	Mean of Square	F	P Value
Weight Loss (%)	0.001	0.0005	0.784	0.000
Hardness (Shore D)	30.330	15.1700	1.970	0.015
Impact Strength (J/m)	161.370	80.6800	3.870	0.007
Flexural Strength (MPa)	49.270	24.6300	1.050	0.003
Water Absorption (%)	4.120	2.0600	0.420	0.012

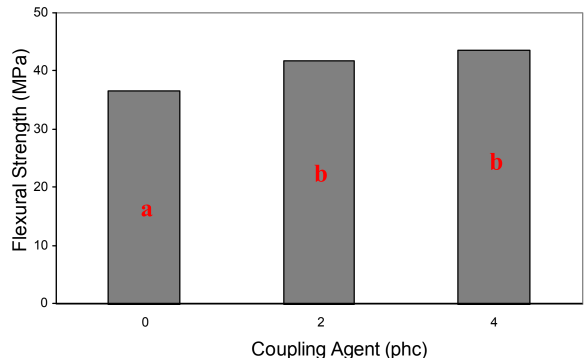

Fig. 4: Effect of coupling agent content on flexural strength of composites

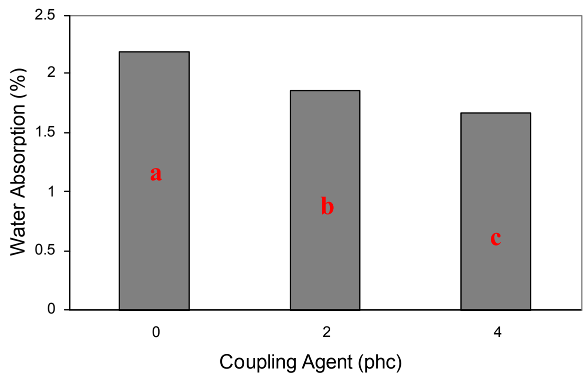

Fig. 5: Effect of coupling agent content on water absorption of composites

It is well established that, with increase of the flexural strength, indicating enhanced compatibilization between wood flour and PP matrix [11-17].

Figure 4 illustrates the effect of coupling agent on the impact strength of composites. As can be seen, the impact strength of samples increases with increase of coupling agent. The enhancement in the impact strength could be attributed to the more homogeneous dispersion of the fiber resulting from the increasing wet-ability of the fiber with increasing concentration of the coupling agent that leads to more uniform distribution of the applied stress and requires more energy for the fiber de-bonding and subsequent fiber pull out as these are the causes of impact failure of the composites.

Figure 5 also shows that the water absorption decreases by adding coupling agent. This means that it is the interfacial region which influences the water uptake of the composite. Because uncompatibilized wood flour composite has weak fiber/matrix adhesion in nature, the interface is enhanced in the presence of the coupling agent. Generally it is necessary to use compatibilizers or coupling agents in order to improve the polymer/fiber bonding and in turn to enhance water resistance. The coupling agent chemically bonds with the OH groups in the wood flour and limits the water absorption of the composites. As a result, it is important to use coupling agents to improve the quality of adhesion between plastics and fibers, to reduce the gaps in interfacial region and to block the hydrophilic groups [11-17]. According to a review of the literature the incompatibility between phases results in a poor interfacial adhesion between hydrophilic wood and the hydrophobic polymer matrix, which results in poor adhesion and therefore in poor ability to transfer stress from the matrix to the fiber reducing the composite properties. So, the use of coupling agents improves the quality of adhesion between polymer and wood flour to reduce the gaps in interfacial region and to block the hydrophilic groups.

CONCLUSION

The following conclusions could be drawn from the results of the present study:

- The results of an ANOVA indicated that the coupling treatment had significant effects on the natural durability, physical mechanical and properties of composites.
- The hardness, impact strength and flexural strength of composites increased with increase of coupling agent. However, the weight loss and water absorption decreased with increase of compatibilizer loading.
- The biggest improvement of the natural durability of wood flour/polypropylene composites exposed to rainbow fungus (*Trametes versicolor*) can be achieved for the coupling agent loading at 4 per hundred compounds.

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