

Wood–High-Density Polyethylene Composites: Water Absorption and Mechanical Properties

Mahroo Jahadi,¹ Saied Nouri Khorasani,² Mahyar Palhang¹

¹*Alvar Plast Sepahan Company, Isfahan Science and Technology Town, Isfahan, Iran*

²*Department of Chemical Engineering, Isfahan University of Technology, Isfahan 84156-83111, Iran*

Received 22 February 2009; accepted 12 May 2010

DOI 10.1002/app.32851

Published online 9 September 2010 in Wiley Online Library (wileyonlinelibrary.com).

ABSTRACT: In this research, the effect of water absorption on the mechanical properties of wood/high-density polyethylene (HDPE) composites were investigated. HDPE (44005ARPC) was used as the polymer matrix, and spruce sawdust was used as the filler at a maximum loading of 50 wt % of the total weight of each compound. All compounds contained 5 wt % magnesium stearate as a lubricant and 0.5 wt % Irgafos 168 as a heat stabilizer. Four factors in two levels were chosen [talc (filler) at levels of 5 and 15 wt %, zinc borate (fungicide) at levels of 0 and 1 wt %, maleic anhydride polyethylene (coupling agent) at levels of 4 and 6 wt %, and method of mixing (one-step vs. two-step mixing)], and eight compounds were prepared with an L8 Taguchi orthogonal array which has 8 combinations of levels. The effects of each factor at two levels on the diffusion constant and the tensile and bend-

ing strengths (under wet and dry conditions) were investigated by the analysis of variance of means with 90% confidence. The optimum level for each factor is reported. The results show that there was a linear correlation between the diffusion constant and tensile and bending strengths when the samples were immersed in distilled water. A higher diffusion constant resulted in much lower tensile and bending strengths with immersion in distilled water until saturation was reached. Scanning electron microscopy images confirmed good mixing when two-steps mixing was used. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 119: 2627–2634, 2011

Key words: composites; compounding; mechanical properties

INTRODUCTION

New applications and end uses of wood–plastic composites include decking, flooring, and outdoor facilities. The exposure of natural fiber–thermoplastic composites to the atmosphere or their contact with aqueous media has made it necessary to evaluate their water-uptake characteristics. Water absorption can lead to a decrease in some of the properties and needs to be considered in the selection for a particular application.¹

Moisture is known to have a significant effect on the mechanical properties of polymers and their composites. Transport phenomena in composites under wet conditions can be modeled with Fick's law of diffusion, which is the simplest model for the diffusion of a solvent into a solid. Three-dimensional anisotropic Fick's diffusion models were developed by Shen and Springer.²

Moisture sorption studies in wood–thermoplastic composites were performed to compare the moisture diffusion process in three candidate materials. Rangaraj³ assessed damage due to moisture absorp-

tion by measuring coupon stiffness and strength during and after saturation. The correlation between the diffusion data and a Fickian diffusion model was reported.

Tajvidi et al.¹ investigated the long-term water absorption behavior of various natural fiber–polypropylene (PP) composites and studied the effects of different natural fiber types and contents on the water absorption behavior. They reported that the fiber type affected the amount of water absorbed and that the fiber content had a significant effect on the water absorption. Higher contents of fiber resulted in higher water absorption.

Stark⁴ investigated the influence of moisture absorption on the mechanical properties of wood flour (WF)–PP composites. The exposure environments were at 30, 65, and 90% relative humidity at 26.7°C. The specimens were placed in a water bath at room temperature. Samples were removed periodically, and their tensile, flexural, and impact properties were tested. The composites filled with 20 wt % WF absorbed moisture in all of the exposure environments, but no significant degradation of the properties was observed. The 40 wt % WF composites absorbed more moisture than the 20 wt % WF composites. The flexural properties of the composites placed in a water bath with 90% relative humidity were lower than the flexural properties of the

Correspondence to: M. Jahadi (mahroojahadi@yahoo.com).

other composites. The tensile properties and notched impact strengths decreased only for composite samples that were placed in the water bath.⁴

In this study, the effects of three additives [talc, zinc borate, and maleic anhydride polyethylene (MAPE)] and the method of mixing on the diffusion constant and mechanical properties (tensile and bending strengths) were investigated.

MECHANISM OF WATER TRANSPORTATION

The transportation of water through composite materials may theoretically follow different mechanisms; this depends on factors such as the chemical nature of the polymer, the dimensions, the morphology of the filler, and polymer–filler interfacial adhesion. In general, the water transportation behavior in polymer matrix composites can be Fickian diffusion, relaxation-controlled diffusion, or non-Fickian or anomalous diffusion. These three cases of water transportation phenomena can be distinguished theoretically by the shape of the sorption curve, which is represented by the following equation:⁵

$$\log\left(\frac{M_t}{M_{\text{sat}}}\right) = \log k + n \times \log t \quad (1)$$

where M_t is the moisture content at time t , M_{sat} is the equilibrium moisture content, and k and n are constants. The value of coefficient n varies in the three cases of water transportation: for Fickian diffusion, $n = 0.5$; for relaxation-controlled diffusion, $n > 1$; and for anomalous transportation, $0.5 < n < 1$. The coefficients (n and k) can be determined from the slope and intercept of M_t/M_{sat} versus t in the logarithm plot, which can be drawn from the experimental data.⁵

The most commonly used method for determining the Fickian mass diffusivity in polymer composites was developed by Shen and Springer.² This method assumes one-dimensional unsteady diffusion through the thickness of the composite panel, where the flat face is much greater than the thickness. According to Fick's law, the percentage weight gain at saturation and $\%M$ is initially linear with the square root of time t .²

$$\%M = \frac{4 \times \%M_{\text{sat}} \sqrt{D_A}}{h\sqrt{\pi}} \sqrt{t} \quad (2)$$

The diffusion constant is calculated from the slope of the linear part of the curves (shown in Fig. 1) with eq. (1):^{2,6}

$$D_A = \frac{\pi h^2}{16 \times (\%M_{\text{sat}})^2} \times \left[\frac{(\%M_2 - \%M_1)}{(\sqrt{t_2} - \sqrt{t_1})} \right]^2 \quad (3)$$

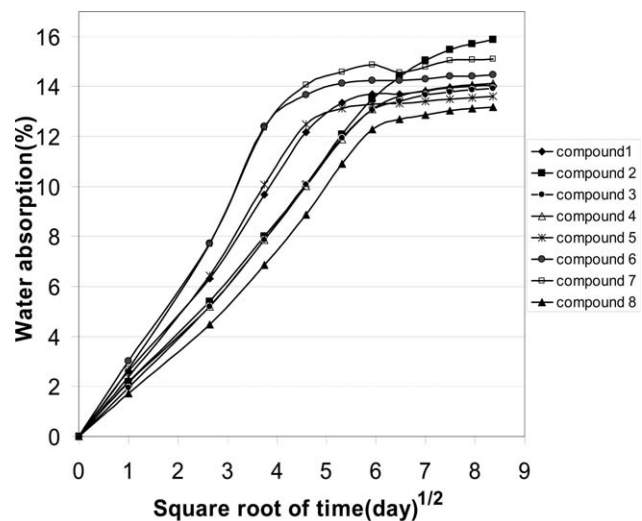


Figure 1 Water absorption (%) versus the square root of time [(day)^{1/2}] for the eight compounds.

where D_A is the apparent diffusion constant, $\%M_{\text{sat}}$ is the percentage weight gain at saturation, $\%M_1$ is the percentage weight gain at time t_1 , $\%M_2$ is the percentage weight gain at time t_2 , and h is the thickness.

The apparent diffusion constant given by eq. (3) is one-dimensional. Therefore, it does not account for the diffusion taking place through the coupon edge. This can be calculated with a geometric edge correlation factor (ECF), which was given by Rao and coworkers:^{7–9}

$$\text{ECF} = \left(1 + \frac{h}{l} + \frac{h}{w} \right) \quad (4)$$

$$D = \frac{D_A}{\text{ECF}} \quad (5)$$

where l and w are the length and width of the sample, respectively, and D is the corrected diffusion constant.

EXPERIMENTAL

High-density polyethylene (HDPE) was compounded with spruce sawdust. In all of the formulations, the level of sawdust was 50% of the total weight of each compound. The weight percentage of magnesium stearate lubricant was constant (5 wt %) in all of the formulations.

A heat stabilizer (Irgafos 168, 0.5 wt %) was used in all of the formulations. Table I shows the materials used in this research to make compounds and their specifications.

Sawdust was dried in an oven at 100°C. All components were weighed according to design (Table II) and physically mixed.

The weighed and mixed formulations were compounded in an extruder. A single-screw extruder

TABLE I
Materials and Specifications

Material	Appearance	Specifications	Supplier
HDPE	Granule	Grade: 44005 Melting point: 134°C MFI: 0.4–0.7 g/10 min	Arak Petrochemical Co., Arak, Iran
Sawdust spruce	Fine powder	Mesh: 30–80 Density: 0.4–0.7 g/cm ³	Alvar Plast Sepahan co, Isfahan, Iran
Talc	Powder	Mesh: 600–700	Eksir Shimi, Iran
Magnesium stearate	Powder	Mesh: 325	Sabatsaz (Iran)
MAPE	White powder	MFI: 0.8–1.8 g/10 min Graft: 0.8–1.3%	Kimia Javid Sepahan (Isfahan-Iran)
Irgafos 168	Powder	Molecular weight: 647	Ciba, Iran
Zinc borate	White, crystalline powder	Mesh: 325	Alpha Products, Karlsruhe, west Germany

MFI = melt flow index.

with three heat zones and a length-to-diameter ratio of 30 was used for compounding. The temperatures of the extruder zones were 137–140, 146–150, and 155–165°C in zones 1, 2, and 3, respectively. The screw speed was the same for all compounding that was carried out. Furthermore, the extrudate was milled in a granulator.

Standard samples for water absorption and tensile and bending testing were prepared by compression molding.

Design of the experiment

Four factors in two levels were chosen, and the effects of each factor on the water absorption and mechanical properties were investigated.

Table III shows the variable factors (additives) and their levels.

L8 orthogonal array which has 8 combinations of levels. Each column in this array correspond to a factor. An experiment with two levels each can be run using L8 (2⁷).¹⁰

In the one-step mixing procedure, all of the materials in the formulation were mixed together and compounded at once.

In the two-step mixing procedure, 45 wt % HDPE with all of the additives was mixed and compounded

in an extruder. Then, the extrudate was compounded again with the remaining 55 wt % HDPE.

Water absorption test

Sheets of wood–plastic with dimensions of 152 × 152 × 4.2 mm³ were prepared by the compression-molding method with a hot press. From each compound, three sheets were molded. Each sheet was dried in an oven at 100°C until a constant weight was reached. Then, all of the sheets were immersed in distilled water. Periodically, the weights of sheets were measured until they reached a constant weight. These experiments took 70 days for the eight compounds to reach equilibrium. The curves of the relative weight versus the square root of time were similar to absorption curves, which obeyed Fick's law. Figure 1 shows the percentage water absorption of the eight compounds versus the square root of time. In all curves, the first part of each curve was linear. The diffusion constant was calculated from eqs. (3)–(5).

Mechanical properties testing under dry conditions

Tensile- and bending-strength tests were performed according to ASTM D 638 and ASTM D 790, respectively. For each test, three samples were used.

TABLE II
L8 Array of the Taguchi Method, Design of the Eight Compounds,
and Variable Factors

Compound no.	Factor A: Talc (wt %)	Factor B: Zinc borate (wt %)	Factor C: MAPE (wt %)	Factor D: Method of mixing
1	5	0	4	1
2	5	0	6	2
3	5	1	4	2
4	5	1	6	1
5	15	0	4	2
6	15	0	6	1
7	15	1	4	1
8	15	1	6	2

TABLE III
Variable Factors and Their Levels

Material/ method	Variable factor	Level 1	Level 2
Talc	A	5 wt %	15 wt %
Zinc borate	B	0 wt %	1 wt %
MAPE	C	4 wt %	6 wt %
Mixing	D	One-step mixing	Two-step mixing

Mechanical properties testing under wet conditions

The prepared tensile- and bending-strength samples were immersed in distilled water at room temperature. Every 24 h, the surface of specimens were dried with a cloth and weighed, the results were recorded, and again, the specimens were immersed in water until of the sample weights reached a constant value. Each of the eight compounds had a different saturation time.

Tensile- and bending-strength tests (under wet conditions) were performed according ASTM D 638 and ASTM D 790, respectively. For each test, three samples were used.

RESULTS AND DISCUSSION

Water absorption and diffusion constant

Figure 1 shows the mean percentage water absorption versus the square root of time for each of eight compounds. Three samples for each compound were tested, three curves and three slopes of the approximately linear part of the curves were obtained, and three diffusion constants were obtained. Figure 1 shows the means of the results for each compound.

The coefficient of determination (R^2) of the linear parts of the curves were calculated to be greater than 0.98.

The mean of the slope of the linear part of each curve was calculated, and the values are presented in Table IV. Consequently, the mean and standard deviation (SD) values are reported. The diffusion constants of the eight compounds were calculated

from eqs. (3)–(5). The calculated means of the diffusion constants are listed in Table IV. The values of SD for the diffusion coefficients were taken for three sample of each compound.

The direct comparison of the diffusion constants obtained from this research with previous studies was difficult, as the matrix polymer, compounding method, and testing conditions were not the same. The magnitude of the diffusion constant obtained in this research was 0.782×10^{-12} to 1.83×10^{-12} m²/s. Wang et al.¹¹ reported a diffusion constant of 4.63×10^{-13} m²/s for hot-pressed 50 wt % rice hull–HDPE composites coupled with MAPE. Espert et al.¹² reported a diffusion constant of 1.09×10^{-12} m²/s for PP composites containing 30 wt % coir fiber and a diffusion coefficient of 1.83×10^{-12} m²/s for composites containing 30 wt % luffa fiber. Tajvidi et al.¹ prepared injection-molded 50 wt % WF–PP composites with 2 wt % MAPE and reported a diffusion constant of 1.33×10^{-11} m²/s. Adhikary et al.⁵ reported a magnitude of the diffusion constant of 2.76×10^{-12} to 9.45×10^{-12} m²/s for wood–HDPE composites and wood–PP composites.

In the next step, the effects of the four factors at two levels on the diffusion constant were determined by the Taguchi method. Figure 2 shows the effect of four factors at two levels on the diffusion constant. Statistical analysis by 90% confidence showed that factors A, B, C, and D had significant effects on the diffusion constant. As shown in Figure 2, a higher talc level increased the diffusion constant.

Increasing the zinc borate level decreased the diffusion constant. This was due to the formation of a cellulose–boron complex. One boron atom could change to a metal organic complex by a hydroxyl group. This complex could improve the interface strength between the wood and HDPE.¹³

Increasing the MAPE level in the composite compound reduced the diffusion constant.

The diffusion constant in the two-step mixing method was lower compared to that in one-step mixing. This was due to the formation of a layer of polymer on the wood particles that prevented the exposure of wood particles to water and water diffusion.

TABLE IV
Water Absorption (%) Versus the Square Root of Time

Compound no.	Mean slope of the linear part of the curve (day ^{-1/2})	SD of the slope (day ^{-1/2})	Mean diffusion constant (m ² /s)	SD of the diffusion constant (m ² /s)
1	2.61261	0.0640	1.37×10^{-12}	0.1008×10^{-12}
2	2.24774	0.2130	0.818×10^{-12}	0.051437×10^{-12}
3	2.17856	0.1463	0.884×10^{-12}	0.079622×10^{-12}
4	2.15258	0.3035	0.885×10^{-12}	0.106715×10^{-12}
5	2.73445	0.1014	1.54×10^{-12}	0.200073×10^{-12}
6	3.24618	0.2044	1.83×10^{-12}	0.119606×10^{-12}
7	3.26370	0.2725	1.75×10^{-12}	0.099284×10^{-12}
8	1.91020	0.1370	0.782×10^{-12}	0.006261×10^{-12}

The data were obtained from Figure 1.

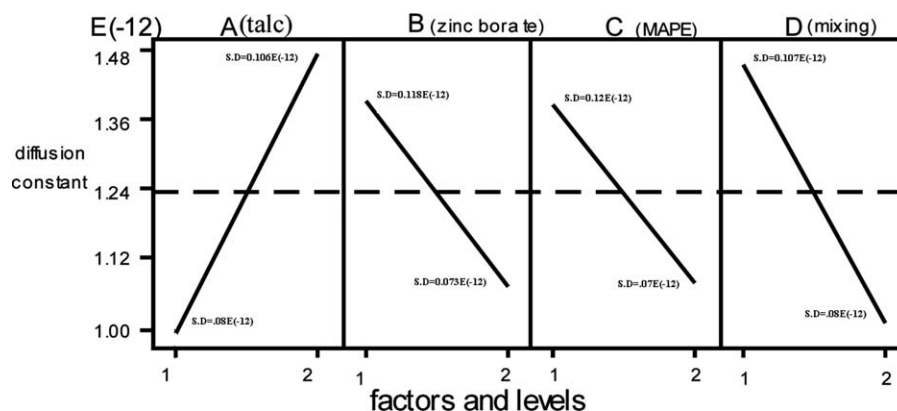


Figure 2 Effect of four factors at two levels on the diffusion constant.

A minimum diffusion constant was desired. Therefore, the diffusion constant was minimized when factor A (talc) was at level 1 (A_1), factor B (zinc borate) was at level 2 (B_2), factor C (coupling agent) was at level 2 (C_2), and factor D (method of mixing) was at level 2 (D_2). Under these conditions, the optimum diffusion constant (D_{opt}) was calculated from eq. (6):¹⁰

$$D_{opt} = \text{Mean} + (A_1 - \text{Mean}) + (B_2 - \text{Mean}) + (C_2 - \text{Mean}) + (D_2 - \text{Mean}) \quad (6)$$

$$D_{opt} = 0.445 \times 10^{-12} \text{ m}^2/\text{s}$$

Therefore, the diffusion constant was at a minimum when the talc concentration was 5 wt %, the zinc borate concentration was 1 wt %, the MAPE concentration was 6 wt %, and the two-step mixing method was used.

Mechanical properties under wet conditions

Tensile strength

For each compound, six specimens were prepared according to ASTM D 638. Tensile testing of three samples were performed under dry conditions, and three other specimens were immersed in distilled water

for wet conditions testing. Tensile testing was performed after the saturation of the samples in a water bath. In all of the compounds, the tensile strengths of the samples under wet conditions decreased.

Table V shows the tensile strength under dry and wet conditions and the percentage decrease in the tensile strength.

Stark⁴ reported a tensile-strength decrease of 14% for a 40% WF-PP composite that was water-soaked.

The time to reach to equilibrium for each compound was different. So the immersion time was not the same for each of the eight compounds.

With the Taguchi method, the effect of each factor on the tensile strength was determined. The results were statistically analyzed with 90% confidence by the analysis of variance of means. The statistical analysis showed that factors A, C, and D had significant effects on lowering the tensile strength because of saturation in distilled water. Figure 3 shows effect of each factor at two levels on the tensile-strength reduction percentage. Factor B had no significant effect on the tensile-strength reduction percentage.

As shown in Figure 3, the change in the level of talc from 5 to 15 wt % (factor A) increased the tensile-strength reduction percentage. A higher content of talc meant lower level polymer in the compound. Therefore, water diffusion accelerated with higher contents of talc and affected the wood-HDPE interface. Zinc

TABLE V
Tensile Strength Under Dry and Wet Conditions and Tensile-Strength Reduction (%)

Compound no.	Mean tensile strength: Dry conditions (MPa)	SD of the tensile strength: Dry conditions (MPa)	Mean tensile strength: Wet conditions (MPa)	SD of the tensile strength: Wet conditions (MPa)	Tensile-strength reduction (%)
1	11.24	0.91	5.9	0.05	47.5
2	13.78	0.12	9.7	0.79	29.8
3	12.95	1.09	8.6	0.4	33.6
4	13.31	1.08	8.1	0.37	39.5
5	12.91	1.36	6.0	0.06	53.4
6	11.13	1.54	4.7	0.17	57.8
7	12.89	1.40	4.2	0.55	67.4
8	15.14	1.56	8.6	0.51	43.4

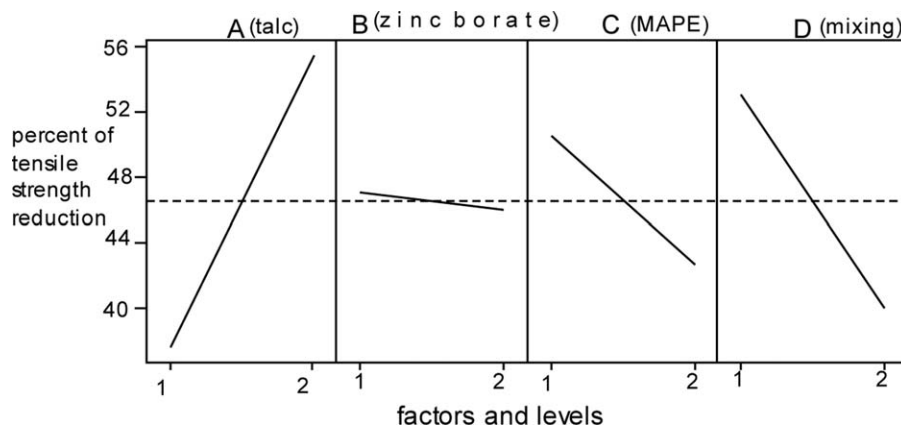


Figure 3 Effect of four factors at two levels on the tensile-strength reduction percentage (wet condition).

borate (factor B) had no significant effect on the tensile-strength reduction percentage.

An increase in the coupling agent (factor C) concentration from 4 to 6 wt % caused a decrease in the tensile-strength reduction percentage. The coupling agent enhanced the wood–HDPE interface adhesion and increased the bending strength under wet conditions. The change of mixing method (factor D) from one-step mixing to two-steps mixing decreased the tensile-strength reduction percentage.

The minimum tensile-strength reduction occurred when the talc concentration was 5 wt % (A_1), the coupling agent concentration was 6 wt % (C_2), and the two-step mixing method (D_2) was used. Under these conditions, the optimum tensile-strength reduction percentage (Y_{opt}) was calculated from eq. (7):¹⁰

$$Y_{opt} = \text{Mean} + (A_1 - \text{Mean}) + (C_2 - \text{Mean}) + (D_2 - \text{Mean}) \quad (7)$$

where $Y_{opt} = 24.17\%$. So under these conditions, a minimum tensile-strength reduction percentage occurred.

Bending strength

For each compound, six specimens were prepared according to ASTM D 790. The bending strengths of

three specimens were measured under dry conditions, and the other three specimens were immersed in distilled water. The bending-strength test was carried out after saturation of the samples in the water bath. In all of the specimens, a bending-strength reduction was seen. Table VI shows the bending strength under dry and wet conditions, and the bending-strength reduction percentage. The bending-strength reduction percentage was calculated from eq. (8):

$$\begin{aligned} &\text{Bending strength reduction percentage} \\ &= (\text{Mean bending strength under dry conditions} \\ &\quad - \text{Mean bending strength under wet conditions}) \\ &/ (\text{Mean bending strength under dry conditions}) \times 100 \end{aligned} \quad (8)$$

The statistical analysis showed that factors A, B, C, and D had a significant effect on the bending-strength reduction percentage. Figure 4 shows that the method of mixing had the greatest effect on the bending-strength reduction percentage.

By the Taguchi method, the effect of each factor on the bending-strength reduction was determined.

The results were statistically analyzed with 90% confidence by the analysis of the variance of means. As the level of the talc, zinc borate, and coupling agent increased, the bending-strength decreased.

TABLE VI
Bending Strength Under Dry and Wet Conditions and Bending-Strength Reduction (%)

Compound no.	Mean bending strength: Dry conditions (MPa)	SD of the bending strength: Dry conditions (MPa)	Mean bending strength: Wet conditions (MPa)	SD of the bending strength: Wet conditions (MPa)	Bending-strength reduction (%)
1	18.3	1.55	9.5	0.38	48.8
2	21.4	1.56	11.8	0.23	44.7
3	19.8	0.46	11.1	0.12	43.9
4	20.6	0.83	8.8	0.58	57.5
5	19.07	1.58	10.9	0.15	43
6	17.97	0.45	8.1	1.14	54
7	16.8	1.27	6.7	1.64	60
8	21.6	0.89	10	0.67	53.6

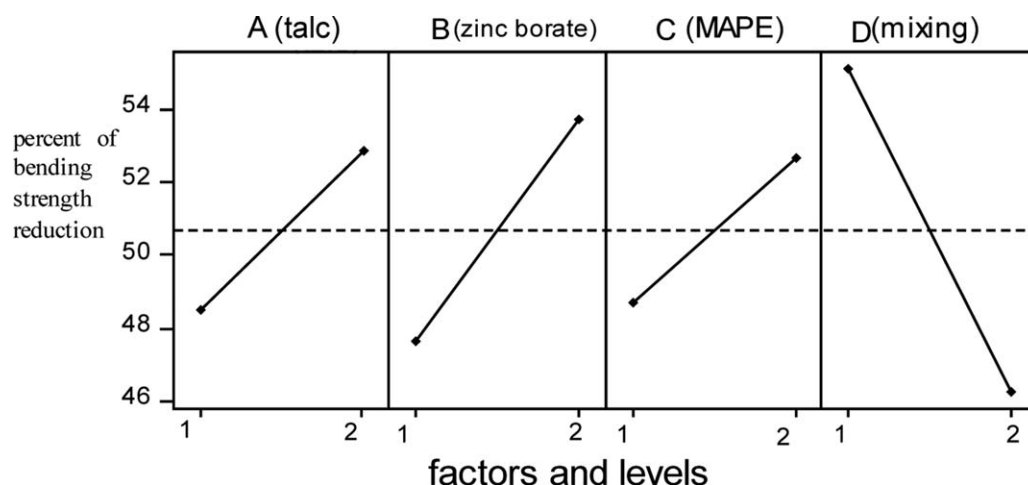


Figure 4 Effect of four factors at two levels on the bending-strength reduction percentage (wet condition).

The minimum reduction of bending strength occurred when the two-step mixing method was chosen (D_2), the zinc borate level was 0 wt % (B_1), the talc concentration was 5 wt % (A_1), and the coupling agent concentration was 4 wt % (C_1).

The optimum bending-strength reduction percentage (B_{opt}) was estimated from eq. (9):¹⁰

$$B_{opt} = \text{Mean} + (A_1 - \text{Mean}) + (B_1 - \text{Mean}) + (C_1 - \text{Mean}) + (D_2 - \text{Mean}) \quad (9)$$

where $B_{opt} = 39.1\%$.

Correlation between the diffusion constant and the tensile-strength reduction

As shown in Figure 5, there was a linear correlation between the diffusion constant and the tensile-strength reduction percentage because of saturation in water. This means that a higher diffusion constant resulted in

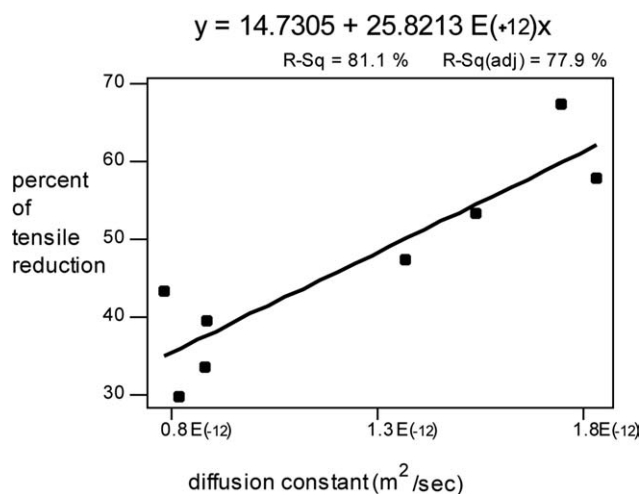


Figure 5 Tensile-strength reduction percentage (wet condition) versus the diffusion constant.

a lower tensile strength. This was reasonable because water diffusion inversely affects the strength of the wood-polymer interface. So, the tensile strength of the wood-HDPE composites decreased as water diffusion took place. Factors A, C, and D affected both the diffusion constant and the tensile-strength reduction percentage because of saturation in water.

Scanning electron microscopy (SEM) analysis

Figures 6 and 7 show the SEM images of the surfaces of compound 8 (two-step mixing method) and compound 6 (single-step mixing method), respectively. These images were prepared by a Philips XL 30 scanning electron microscope. They represent better dispersion and an improved wood-HDPE interface in compound 8 compared to compound 6.

CONCLUSIONS

The immersion of wood-HDPE composites in water adversely affected the mechanical properties. The

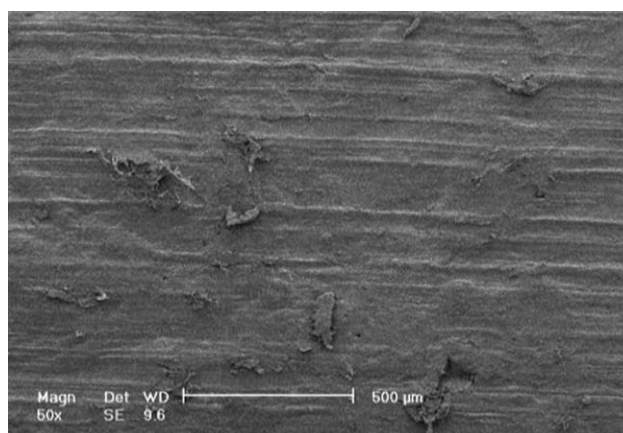


Figure 6 SEM image of the surface of compound 8 (two-step mixing).

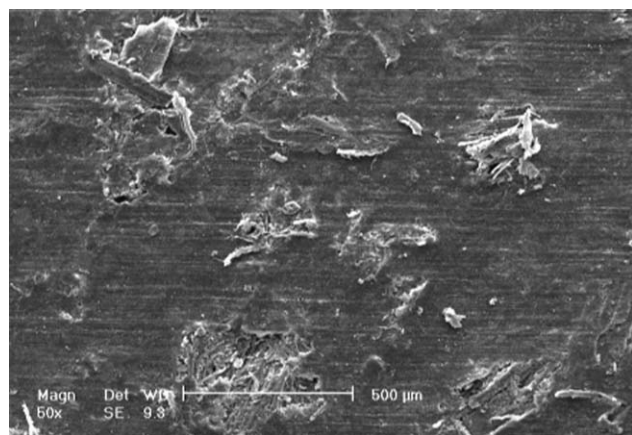


Figure 7 SEM image of the surface of compound 6 (one-step mixing).

absorption of water by the wood component of the composite caused the wood to swell. This could have led to a degradation of the interface quality and resulted in a decrease in the composite strength. Both the tensile and bending strengths of the samples immersed in the water bath decreased because of the weakening wood–HDPE interface adhesion.

The advantages of the two-step mixing method were as follows:

1. Lower water diffusion constant.
2. Lower tensile-strength reduction percentage due to saturation in distilled water.
3. Lower bending-strength reduction percentage due to saturation in distilled water.

These effects occurred because of the better dispersion of wood particles and the HDPE matrix and the formation of a layer of polymer on the wood particles, so it prevented the exposure of wood particles, water diffusion, and wood swelling.

A higher percentage of talc (15 wt %) caused a higher diffusion constant and lower mechanical

properties. The presence of zinc borate decreased the diffusion constant but increased the bending-strength reduction.

A higher level of coupling agent (6 wt % cf. 4 wt %) led to a lower diffusion constant and a reduction in the tensile strength. However, it caused a higher bending-strength reduction.

Two-step mixing resulted in a better dispersion of ingredients in the formulation and better mechanical properties under both dry and wet conditions. SEM images confirmed better mixing in the two-step mixing method.

There was a linear correlation between the diffusion constant and the tensile-strength reduction percentage because of saturation in water. A higher diffusion constant resulted in lower tensile and bending strengths.

References

1. Tajvidi, M.; Kazemi Najafi, S.; Moteei, N. *J Appl Polym Sci* 2006, 99, 2199.
2. Springer, G. S.; Shen, C. H. *Environ Effects Compos Mater* 1988, 3, 15.
3. Rangaraj, S. Master's Thesis, Washington State University, 1999.
4. Stark, N. J. *Thermoplast Compos Mater* 2001, 14, 421.
5. Adhikary, K. B.; Pang, S.; Staiger, M. P. *Chem Eng J* 2007, 142, 190.
6. Springer, G. S. *Environ Effects Compos Mater* 1988, 3, 1.
7. Rao, R. M.; Balasubramanian, N.; Chanda, M. *Environ Effects Compos Mater* 1988, 3, 75.
8. Gopalan, R.; Rao, R. M.; Murthy, M. V.; Dattaguru, B. *Environ Effects Compos Mater* 1988, 3, 96.
9. Rao, R. M.; Balasubramanian, N.; Chanda, M. *Environ Effects Compos Mater* 1988, 3, 89.
10. Mori, T. *The New Experimental Design: Taguchi's Approach to Quality Engineering*; ASI: Dearborn, MI, 1991.
11. Wang, W.; Sain, M.; Cooper, P. A. *Compos Sci Technol* 2006, 66, 379.
12. Espert, A.; Vilaplana, F.; Karlsson, S. *Compos A* 2004, 35, 1267.
13. Duan, X.; Lu, J. Z.; Wu, Q.; Lian, K. *Proc Pacific Rim Bio-Based Compos Symp* 2004, 7.