

Resistance of paper mill sludge/wood fiber/high-density polyethylene composites to water immersion and thermotreatment

Xiaohui Yang,¹ Weihong Wang,¹ Haibing Huang²

¹Key Laboratory of Biobased Material Science & Technology (Education Ministry), Northeast Forestry University, Harbin 150040, China

²Heilongjiang Wood Science Research Institute, 134 Haping Road, Harbin 150081, China

Correspondence to: W. Wang (E-mail: weihongwang2001@aliyun.com)

ABSTRACT: The disposal of paper mill sludge (PMS) is a difficult environmental problem. Thus, PMS has been used as a substitute for wood fiber (WF) to reinforce high-density polyethylene (HDPE). In this study, we compared PMS–WF–HDPE composites with composites without PMS after water immersion and thermal treatment. Water immersion and thermal treatment were conducted at 25 and 70°C, respectively. The results show that the composites with PMS absorbed less water but lost more of their original flexural properties after immersion; thereby, their strength was compromised. These reduced mechanical properties could be partially restored after redrying. After the thermotreatment, the composites with added PMS lost their weight and flexural properties, whereas the composites without PMS gained flexural strength. The results show that the thermotreatment improved the impact strength of the composites when no more than one-third of WF was replaced with PMS. Fourier transform infrared spectroscopy and energy-dispersive X-ray energy-dispersive spectroscopy showed that the wood index of the PMS composite decreased more than the index of the non-PMS composite, whereas the carbonyl index increased more. However, the PMS composite showed a lower increase in the total oxygen/carbon weight ratio. This study suggested that limited amounts of WF could be substituted with PMS to reinforce HDPE. However, WF–PMS–HDPE composites should not be used in hot, humid environments for long periods. © 2014 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2015**, *132*, 41655.

KEYWORDS: aging; cellulose and other wood products; properties and characterization; thermoplastics

Received 6 October 2013; accepted 20 October 2014

DOI: 10.1002/app.41655

INTRODUCTION

Paper mill sludge (PMS) is a byproduct of pulping or paper recycling. Generally, the production of 1 ton of paper generates 6.5 m³ of paper sludge, which contains 90% water.¹ In 2012, more than 102 million tons of paper and paperboard were produced in China.² The disposal of such a large quantity of PMS poses complex environmental problems because most sludge requires disposal into landfills.³ Furthermore, the disposal cost for paper sludge is approximately half the cost of wastewater treatment. Hence, the paper manufacturing industry is highly interested in the reduction of disposal costs for paper sludge through recycling and reuse.^{4,5}

PMS generally contains fine, fibrous, and inorganic materials, including lime, clay, calcium carbonate, and trace metals.⁶ It offers several benefits as a substitute for typical inorganic reinforcement fillers that are used in the manufacturing of thermoplastic polymer composites.³ Girones *et al.*⁷ added recycled PMS to polypropylene and found that an increase in the sludge con-

tent in the composites resulted in materials with higher Young's moduli but lower tensile strengths and deformations at break. Hamzeh *et al.*⁸ reported that composites made with ink-eliminated sludge (from paper recycling), wood fiber (WF), and high-density polyethylene (HDPE) exhibited superior flexural properties. Huang *et al.*⁹ found that PMS could be used to substitute part of the WF to reinforce HDPE.

Thermoplastic composites reinforced with WF are most often used in exteriors for decks, rails, and land building materials.^{10,11} They are, therefore, exposed to rain and air oxidation. Several studies have indicated that the moisture content is the key factor in the deformation of biofiber-reinforced HDPE composites and that the effectiveness of the filler content is significant.^{12–15} On the other hand, researchers have also shown that temperature affects the performance of WF-reinforced plastic composites.^{16–19} For example, plastic deformation occurs in biofiber-filled polypropylene composites and results in a decrease in tensile strength and a decrease in the modulus of the composites at higher test temperatures.²⁰

Table I. Formulations of the PMS–WF–HDPE Composites

Formula	PMS (%)	WF (%)	HDPE (%)	MAPE (%)
0	0	60	36	4
1	10	50	36	4
2	20	40	36	4
3	30	30	36	4

Both wax and PE wax were added at 1% on the basis of the total amount of PMS, WF, HDPE, and MAPE.

However, studies on the properties of PMS-reinforced composites are still limited, and the important issues of water immersion and thermotreatment have not been explored. The purpose of this study was to examine the behavior of PMS–WF–HDPE composites subjected to water immersion and thermotreatment. The results may be used to develop efficient approaches for the use of large quantities of PMS generated from paper manufacturing.

EXPERIMENTAL

Main Raw Materials

HDPE (grade: 5000s, melting flow index = 0.8–1.1 g/10 min at 190°C, density = 0.949–0.953 g/cm³) was purchased from Petrochina Daqing Petrochemical Co. Poplar WF (particle size = 40–80 mesh, length = 1–2.8 mm, aspect ratio = 9–12) was purchased from a local market. PMS with a composition of 39% fine fibers and 61% inorganic materials was provided by a waste-paper recycling company. It was dried, ground, and sieved through 20–100 mesh to obtain particles with a size of 1–2.2 mm and an aspect ratio of 1–3. Polyethylene grafted with maleic anhydride (MAPE; grafting percentage: 0.9%) was obtained from Shanghai Sunny New Technology Development Co., Ltd. It was used as a coupling agent to improve the compatibility between the biofiber and HDPE because anhydride moieties in MAPE undergo esterification with the surface hydroxyl groups of wood flour.²¹ Wax and polyethylene (PE) wax were obtained from Shanghai Hualing Health and Machinery Firm and Shangdong QiLu Petrochemical Co., Ltd., respectively. Both waxes were used as lubricants.

Preparation of the PMS–WF–HDPE Composites

WF and PMS particles were oven-dried separately (at 105°C) to reduce their moisture content to less than 3%. As shown in Table I, the components were mixed at 86°C in a high-speed mixer for 10 min. The mixture was then fed into a twin-screw extruder. The rotation speed of the twin screw was 100 rpm, and the feeding speed was 12 rpm. The barrel temperatures of the twin-screw extruder are listed in Table II. In this stage, HDPE and WF (with or without PMS) were compounded at 150–175°C. The blends were broken into small particles by a pulverizer. Finally, the pellets were fed into a single-screw

Table II. Barrel Temperatures of the Twin-Screw Extruder

Zone	1	2	3	4	5	6	7
Temperature (°C)	155	165	170	175	175	165	150

Table III. Barrel Temperatures of the Single-Screw Extruder

Zone	Die							
	1	2	3	4	5	6	7	8
Temperature (°C)	165	165	170	170	165	165	165	165

extruder and then extruded into plates with cross-sectional areas of 40 × 4 mm². The rotation frequency of the single screw was 14.00 Hz. The barrel temperatures of the single-screw extruder are listed in Table III.

Material Treatment

Water Immersion. The specimens were submerged in water at 25°C for a predetermined duration and then removed from the water for weighing. The dimensions of specimens were determined with a specific testing apparatus. Before weighing, the water on the surface of the specimens was wiped off. The specimens were tested after they were immersed in water for 120, 360, and 600 h.

Thermotreatment. The specimens were placed in a test chamber for thermal oxidative weathering (LR225, Chongqing, China) set at 70°C. After 120, 360, and 600 h of treatment, the specimens were removed for analysis. Before testing, the specimens were cooled to room temperature.

Mechanical Property Tests

Mechanical property measurement was conducted on the specimens at room temperature (relative humidity ≈ 50%) before and after immersion. Samples that were previously submerged in water for 600 h were tested in the dry, wet, and redried states.

Flexural tests were carried out in a universal testing machine (RGT-20A, Shenzhen Reger Instrument Co., Ltd.), in accordance with the procedure “Fiber-Reinforced Plastic Composites—Determination of Flexural Properties” (GB/T 1449–2005). The specimens had dimensions of 80 × 13 × 4 mm³ and a span length of 64 mm. A loading speed of 2 mm/min was used for testing. Five specimens in each group were tested to obtain values for the flexural modulus and flexural strength.

The unnotched impact strength was examined with an impact testing machine (XJ-50Z, Chengde Precision Testing Machine Co., Ltd.) on the basis of GB/T 1043.1–2008 (“Plastics, Determination of Charpy Impact Properties, Part 1: Noninstrumented Impact Test”). The specimens had dimensions of 80 × 10 × 4 mm³ and a span length of 60 mm. The striking velocity of the tests was 2.9 m/s, and the pendulum energy was 2 J. Six specimens of each sample were tested to determine the impact strength.

Scanning Electron Microscopy (SEM)

To characterize the structural changes, samples were frozen in liquid nitrogen and then broken. Broken surfaces were sputter-coated with gold and then analyzed under a scanning electron microscope (FEI Quanta 200).

The surface of the extruded composite before and after thermotreatment was examined by energy-dispersive X-ray spectrometry without nitrogen freezing to characterize changes in the oxygen and carbon content and to determine the atomic weight ratio of

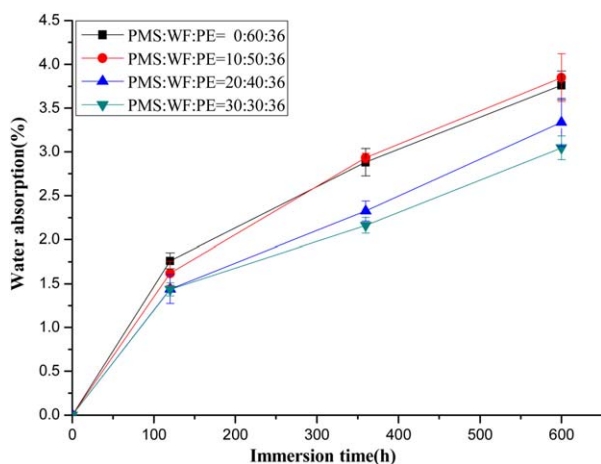


Figure 1. Water absorption of the composites after immersion in water. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Oxygen to Carbon (O/C weight ratio). The O/C weight ratio was calculated according to eq. (1):

$$O/C = \frac{O_{wt\%}}{C_{wt\%}} \times 100\% \quad (1)$$

where wt % denotes the weight percentage of the respective element.

Fourier Transform Infrared (FTIR) Spectroscopy

FTIR spectroscopy was conducted on a Magna-IR 560 spectrometer (Nicolet) to detect the functional groups present on the surface of the extruded composites. Scans were recorded in absorbance units from 4000 to 400 cm^{-1} . The carbonyl index and wood index were calculated from eqs. (2) and (3), respectively:

$$\text{Carbonyl index} = \frac{I_{1738}}{I_{2913}} \times 100\% \quad (2)$$

$$\text{Wood index} = \frac{I_{1030}}{I_{2913}} \times 100\% \quad (3)$$

where I represents peak intensity at various wavelengths.²²

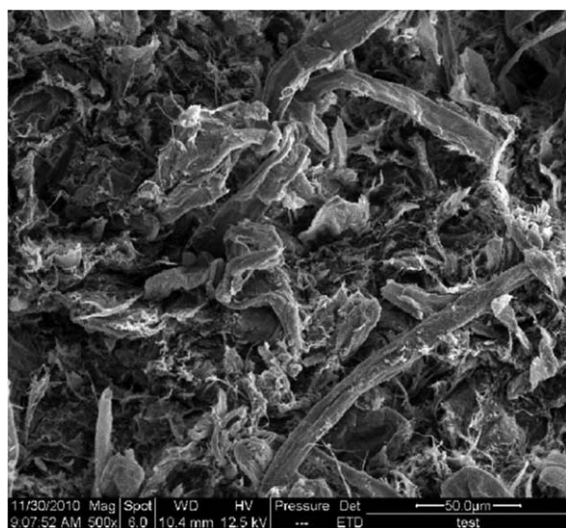


Figure 2. SEM image of PMS.

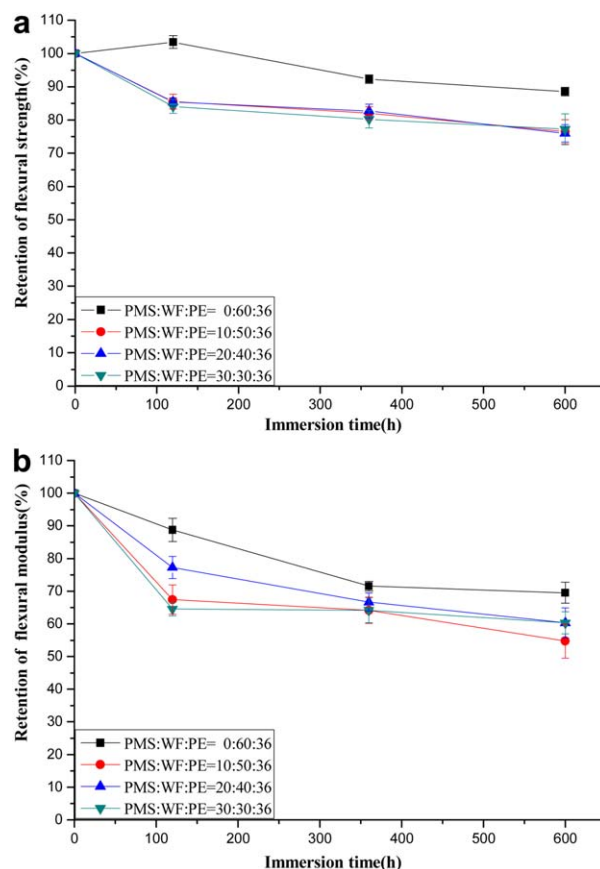


Figure 3. Retention of the (a) flexural strength and (b) modulus of the composites after water immersion (without drying). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Statistical Analysis

Significant differences between the untreated and treated values ($\alpha = 0.05$) were determined by analysis of variance.

RESULTS AND DISCUSSION

Effect of Water Immersion on the Composite Properties

Water Absorption by the Composite. Water absorption by all specimens increased during immersion (Figure 1). After immersion for 600 h, the composites containing 20 and 30% PMS absorbed markedly less moisture than did the composites filled with 0 or 10% PMS. As the hydrophilicity of the natural biofibers enabled the composites to absorb water,^{23,24} a higher WF content led to a larger amount of absorbed water. However, PMS contained both cellulose fibers and other inorganic fillers (Figure 2). The amount of hygroscopic materials, such as cellulose and hemicelluloses, in the PMS–WF–HDPE composites decreased because of the partial replacement of WF with PMS. Therefore, the addition of PMS to the WF–HDPE composite decreased the moisture absorption.

Mechanical Properties of the Composites After Immersion.

Flexural tests were performed on the specimens before and after water immersion. Both the flexural strength and flexural modulus significantly decreased (Figure 3) after the immersion of the composites in water. Once the moisture penetrated the composite materials, WF tended to swell. We presumed that this

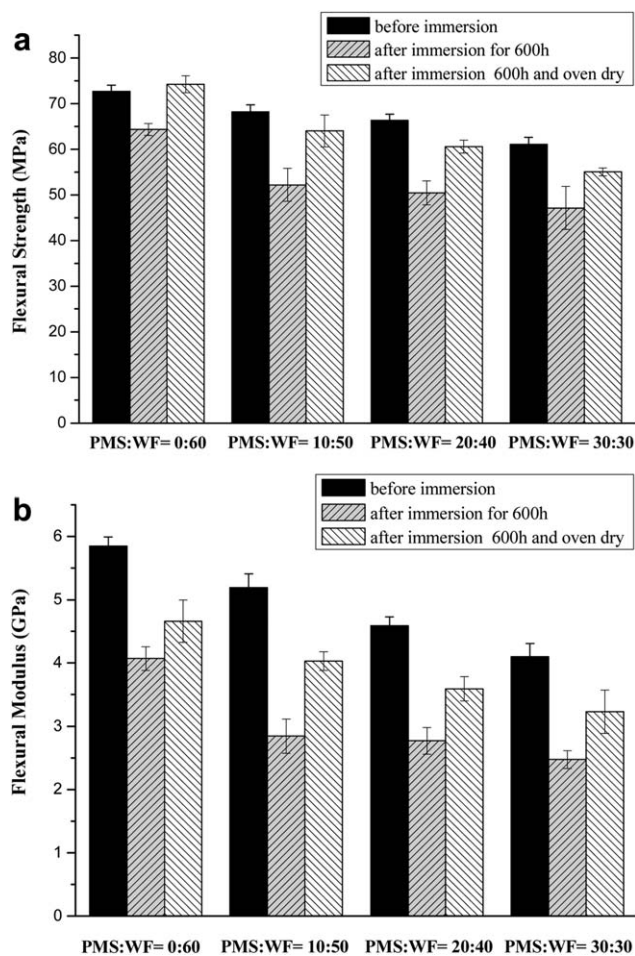


Figure 4. (a) Flexural strength and (b) modulus of the composites before immersion and after immersion for 600 h without drying and with redrying.

swelling caused the debonding or weakening of the interface between the WF and the thermoplastic polymer. Meanwhile, WF became wet and less rigid than dry WF when the composites absorbed water. Thus, the flexural modulus decreased more than the flexural strength.

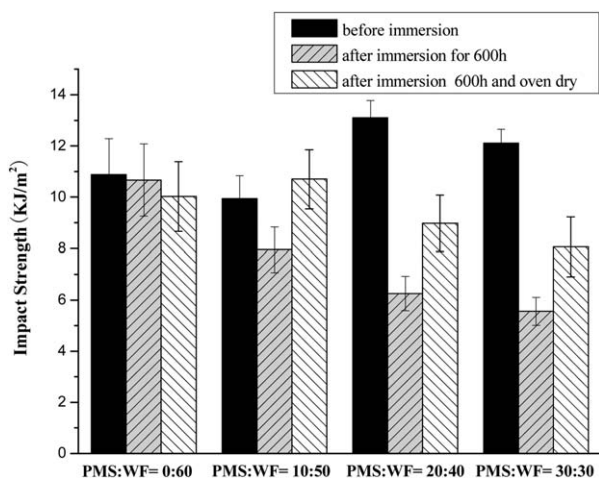


Figure 5. Change in the impact strength of the materials before immersion and after immersion for 600 h without drying and with redrying.

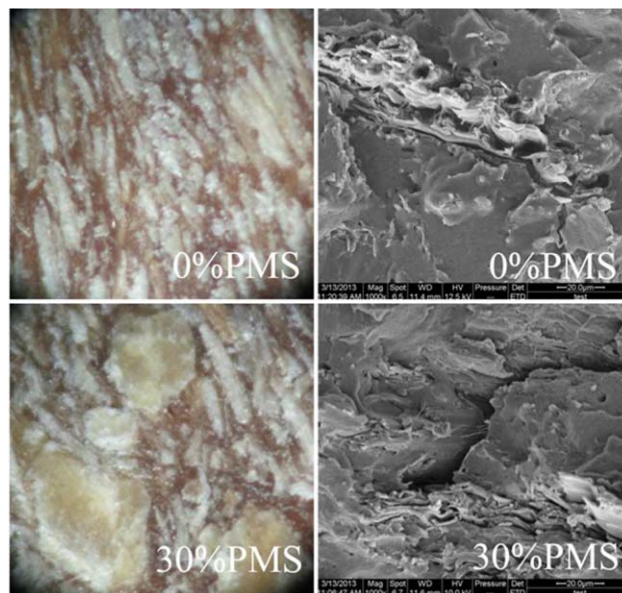


Figure 6. Photographs and microstructures of the cross sections of the composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

In PMS, the inorganic material was the matrix, within which fine WF was distributed (Figure 2). Inorganic materials enclosed the small fibers, which did not make complete contact with the HDPE. The reinforcing function of the small fibers partly depended on the bond between the inorganic material and HDPE. However, the inorganic particles were very hard and were impenetrable to molten HDPE; this caused inadequate mixing of the materials. During immersion, water penetrated the gaps between the PMS and HDPE and further damaged their combination. Thus, composites containing PMS lost more flexural strength and flexural modulus than did the control composite. For example, the retention rate of the flexural strength for the specimens with PMS was about 78%, whereas that for specimens without PMS was about 90% after immersion for 600 h (Figure 3). Similarly, the composite containing PMS showed a greater reduction in the flexural modulus (retention rate = 55%–60%) than the composite without PMS (retention rate = 70%).

When the samples were dried again, the flexural strength and flexural modulus were partly recovered (Figure 4). The drying process evaporated moisture in the composite. Dry WF was stronger than the wet fiber, and the PMS became harder. Thus, the redried specimens showed significant improvements in flexural strength and flexural modulus compared with the undried specimens. However, part of the interfacial bond between HDPE and WF had already been damaged by the swelling and shrinking of WF. Thus, the flexural properties could not be restored to presubmersion levels.

The addition of PMS decreased the flexural strength and flexural modulus (Figure 4) and increased the impact strength of the composite (Figure 5). This phenomenon was explained in our previous work. Round PMS particles behaved like spherical glass beads in the composite structure (Figure 6) and improved

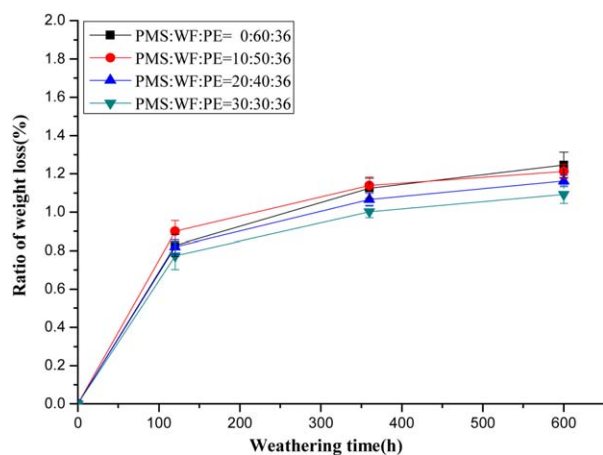


Figure 7. Weight loss of the composites after thermotreatment. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

its flow behavior.^{25,26} Spherical particles became uniformly distributed and embedded in the HDPE matrix. The spherical shape was an advantage because it prevented the accumulation of stress at sharp corners in the fibers. Thus, the impact resistance increased with the addition of PMS. Another possible reason for the improvement was the presence of gaps between the PMS particles and the HDPE matrix (Figure 6) due to incompatibility. These gaps helped absorb energy during shock.

After immersion for 600 h in water, the impact strength of the composites decreased by 5, 27.7, 52.3, and 54.1% upon the addition of 0, 10, 20, and 30% PMS, respectively (Figure 5). Water, which also softened the wood component, contributed to the absorption of the impact energy. Thus, the impact strength decreased slightly in the wet state of the composites with 0–10% PMS. However, the addition of more PMS resulted in a greater loss of impact strength because less WF and more PMS (which contributed stiff particles) remained. These changes decreased the ability of the composites to absorb energy upon impact. On the other hand, water penetration might have adversely affected the interbonding between the PMS and HDPE matrix. This may explain why redried specimens containing PMS recovered more impact strength than did those without PMS (Figure 5). That is, water that evaporated and left voids provided better structural resistance to impact. Similar to the flexural properties, the impact strength could not be restored to the level before immersion because of the damaged bonding between HDPE and PMS.

Effect of Thermotreatment on the Composite Properties

Weight Loss of the Composites After Thermotreatment. All specimens lost part of their weight during the thermal treatment (Figure 7), especially in the first 360 h. The composites containing 20 and 30% PMS lost less than the composites filled with 0 and 10% PMS. The smaller weight loss of the WF–PMS–HDPE composite may have been linked to the lower amounts of volatile components in the PMS. WF used in the HDPE-based composites was pulverized from wood chips. The main components of WF were cellulose, hemicelluloses, lignin, and small amounts of extractives and water. The evaporation of

water, extractives, and other small molecules may have contributed to weight loss in the composites. Thus, a higher WF content led to a greater weight loss. However, we obtained the PMS in this study from recycled paper, which had lost almost all of its lignin and extractives. Small WF in PMS contains mainly cellulose and a small amount of hemicelluloses.

Mechanical Properties of the Composites After Thermotreatment. Specimens without PMS showed a slight increase in the flexural strength throughout the thermotreatment period (Figure 8). Wang *et al.*²⁷ also found that the flexural properties improved after the heat treatment of rice-husk powder/HDPE composites for 128 h. A possible explanation for the flexural strength improvement was that the HDPE molecules rearranged at high temperature, and this resulted in improved compatibility with the WF and a gradual release of its internal stress. On the other hand, the rearrangement of the polymer led to shrinkage of the composite and an increase in its density.

The flexural strength of the specimens containing PMS decreased in the first testing stage (120 h). The addition of greater amounts of PMS caused a greater decrease in the flexural strength. The rearrangement of HDPE molecules at high temperatures might have led to shrinkage,¹⁸ and this resulted in the separation of HDPE from the stable PMS. This separation provided more channels for oxygen penetration and created

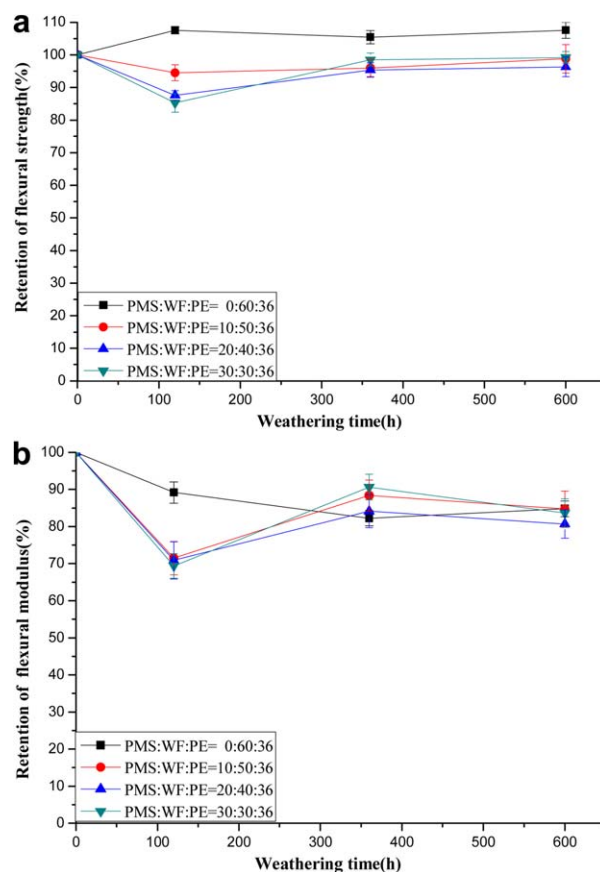


Figure 8. Retention of the (a) flexural strength and (b) modulus of the composites after thermotreatment. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

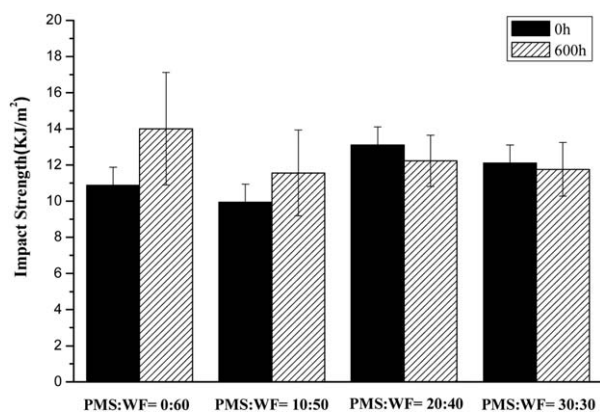


Figure 9. Impact strength of the composites before and after thermotreatment.

larger cracks, which caused the specimens filled with PMS to significantly lose flexural strength.

With continuing thermotreatment, HDPE rearranged and WF shrinkage stopped when oxygen penetrated the deeper layers. In addition, the further crosslinking of HDPE occurred.²⁸ This effect was dominant compared with the degradation effect. Thus, specimens containing PMS showed a recovery trend through the second testing period (120–360 h). Although thermooxidative degradation occurred at the early stage, degradation occurred in the induction period and was not dominant. During thermotreatment, there was a balance between molecular rearrangement and degradation. Therefore, the flexural strength of the specimens showed nearly no change after 360 h when weathering was complete.

The results also show that the flexural moduli of all of the reinforced HDPE composite samples decreased significantly after thermotreatment. Similar to the flexural strength, the flexural modulus of the specimens filled with PMS decreased in the first 120 h and then recovered to a level similar to that of unfilled specimens (Figure 8). Thus, the addition of WF to HDPE might have increased stiffness of the composite. However, thermal oxidation for extended periods damaged the WF and resulted in a greater reduction in the flexural modulus compared to that of the flexural strength.

After thermotreatment, the composite without PMS or the composite with a small amount of PMS exhibited an increased impact strength (Figure 9). This was due to the improved compatibility between WF and HDPE. Upon substitution of more than one-third of WF with PMS, the composites decreased their impact strength. This loss may have been due to the increased stiffness that resulted from the hard PMS particles that remained after thermotreatment. However, this change was not significant. Thermotreatment had little adverse effects on the impact strength of the WF–PMS–HDPE composites.

Table IV. Carbonyl Indices, Wood Indices, and O/C Ratios on the Surface of Composites Before and After 600 h of Thermotreatment

Sample formula	Carbonyl index (%)		Wood index (%)		O/C ratio (%)	
	0 h	600 h	0 h	600 h	0 h	600 h
PMS/WF/PE = 0:60:36	3.0	4.0	27.1	23.8	8.4	12.9
PMS/WF/PE = 30:30:36	2.9	4.1	25.7	19.7	11.9	16.3

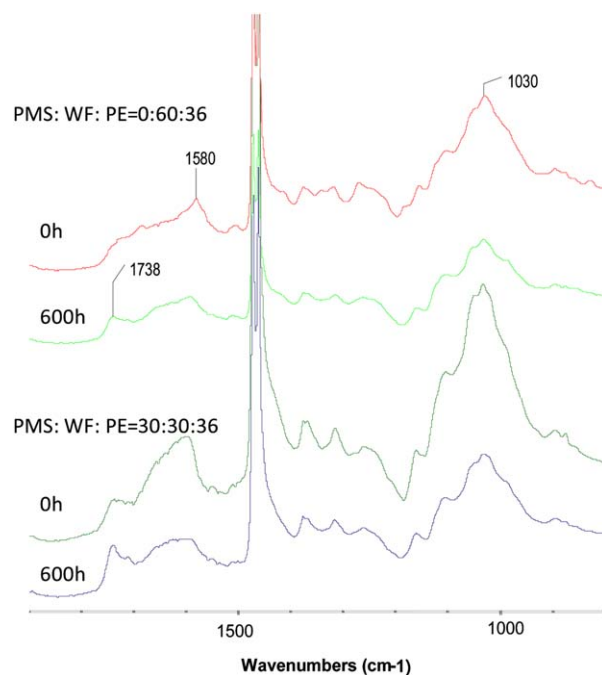


Figure 10. FTIR spectra of the surface of the composites before and after 600 h of thermotreatment. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Elemental Analysis of the Composite Surface After Thermotreatment. The O/C weight ratio was taken as an indicator of surface oxidation. Results show that the total O/C weight ratio of all of the specimens increased after 600 h of thermal weathering (Table IV). This increase indicated that the sample surface was oxidized. PMS contains C, O, Al, Si, and Ca, some of which are mainly present in kaolin ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$) and CaCO_3 . These components were stable during the thermotreatment and led to less change in the total O/C weight ratio of the sample containing PMS compared with that of the sample without PMS (Table IV).

Figure 10 shows the FTIR spectra of the surface of the extruded composites. The peak at about 1030 cm^{-1} was a characteristic peak and was associated with C–O stretching in cellulose, hemicellulose, and lignin of WF. The peak at about 1738 cm^{-1} represented C=O stretching in polysaccharose, which is involved in degradation via polymer oxidation. The decrease in the wood index and the increase in carbonyl index may have indicated degradation of the wood component and polymer oxidation, respectively.²² The results of the calculations are listed in Table IV.

Before thermotreatment, the wood index and carbonyl index of the sample containing 30% PMS were slightly lower than those

of the samples without PMS because the former contained less wood component. However, after thermotreatment for 600 h, the carbonyl index of the sample filled with 30% PMS increased and the wood index decreased more significantly than did the corresponding indices of the control sample. Although the inorganic materials in PMS were stable under the thermotreatment conditions, the results of the FTIR analysis show that PMS accelerated the degradation of the WF–HDPE composites during thermal oxidation and generated more C=O.

Yang *et al.*²⁹ and Valadez-González *et al.*³⁰ also demonstrated that HDPE-based composites filled with kaolin or CaCO₃ were easily oxidized. On the other hand, PMS–WF–HDPE composites contained more gaps than did the control samples because of incompatibility between PMS and HDPE and the dimensional change in HDPE after thermotreatment. Oxygen penetrated the gaps, and this resulted in the oxidation of more WFs.

CONCLUSIONS

In this study, we showed that partial substitution of WF with PMS to produce WF–HDPE composites could enhance the utility of PMS in dry environments. The addition of PMS to WF–HDPE composites could decrease their capacity for water absorption; however, water penetration decreased the flexural properties and impact strength of the composites. Fortunately, this reduction in performance could be partially restored after the composites were redried. After thermotreatment, the composites containing PMS showed a deterioration in their flexural properties, whereas the impact strength remained nearly unchanged. Therefore, WF–PMS–HDPE composites should not be used in hot, humid environments for extended periods. Furthermore, the use of thermotreatment to improve the mechanical properties of the WF–PMS–HDPE composites was problematic.

ACKNOWLEDGMENTS

This research was supported by Forestry industry research special funds for public welfare projects (201204802) and the Fundamental research funds for central universities (DL12EB06-01).

REFERENCES

1. Li, Q. L. *Heilongjiang Pulp Pap.* **2011**, *2*, 59.
2. China Papermaking Industry Report, 2012. http://www.fjppi.com/www/news_show.asp?varid=1799 (accessed September 20, 2013).
3. Son, J.; Kim, H. J.; Lee, P. W. *J. Appl. Polym. Sci.* **2001**, *82*, 2709.
4. Springer, A. M. *Industrial Environmental Control: Pulp and Paper Industry*; Wiley: New York, **1986**.
5. Taramian, A.; Doosthoseini, K.; Mirshokraii, S. A.; Faezipour, M. *Waste Manage.* **2007**, *27*, 1739.
6. Xiao, C. Q.; Ma, L. Q.; Sarigumba, T. *J. Environ. Qual.* **1999**, *28*, 321.
7. Girones, J.; Pardini, G.; Vilaseca, F.; Pelach, M.; Mutje, P. *J. Polym. Environ.* **2010**, *18*, 407.
8. Hamzeh, Y.; Ashori, A.; Mirzaei, B. *J. Polym. Environ.* **2011**, *19*, 120.
9. Huang, H. B.; Du, H. H.; Wang, W. H.; Shi, J. Y. *Polym. Compos.* **2012**, *33*, 1628.
10. Beg, M. D. H.; Pickering, K. L. *Polym. Degrad. Stab.* **2008**, *93*, 1939.
11. Butylina, S.; Hyvärinen, M.; Kärki, T. *Polym. Degrad. Stab.* **2012**, *97*, 337.
12. Adhikary, K. B.; Pang, S. S.; Staiger, M. P. *Compos. B* **2008**, *39*, 807.
13. Najafi, S. K.; Tajvidi, M.; Chaharmahli, M. *J. Appl. Polym. Sci.* **2006**, *102*, 3907.
14. Simonsen, J.; Jacobsen, R.; Rowell, R. *Forest Prod. J.* **1998**, *48*, 89.
15. Wang, W.; Sain, M.; Cooper, P. A. *Compos. Sci. Technol.* **2006**, *66*, 379.
16. Ishak, Z. A. M.; Yow, B. N.; Ng, B. L.; Khalill, H. P. S. A.; Rozman, H. D. *J. Appl. Polym. Sci.* **2001**, *81*, 742.
17. Wang, W.; Sain, M.; Cooper, P. A. *Polym. Degrad. Stab.* **2005**, *90*, 540.
18. Klyosov, A. A. *Wood Plastic Composites*; Wiley: Hoboken, NJ, **2007**.
19. Hrabalova, M.; Gregorova, A.; Wimmer, R.; Sedlarik, V.; Machovsky, M.; Mundigler, N. *J. Appl. Polym. Sci.* **2010**, *118*, 1534.
20. Yang, H. S.; Kim, H. J.; Son, J.; Park, H. J.; Lee, B. J.; Hwang, T. S. *Compos. Struct.* **2004**, *63*, 305.
21. Matuana, L. M.; Balatinecz, J. J.; Sodhi, R. N. S.; Park, C. B. *Wood Sci. Technol.* **2001**, *35*, 191.
22. Stark, N. M.; Matuana, L. M. *Polym. Degrad. Stab.* **2004**, *86*, 1.
23. Bledzki, A. K.; Gassan, J.; Theis, S. *Mech. Compos. Mater.* **1998**, *34*, 563.
24. Espert, A.; Vilaplana, F.; Karlsson, S. *Compos. A* **2004**, *35*, 1267.
25. Hamzeh, Y.; Ashori, A.; Mirzaei, B. *J. Polym. Environ.* **2011**, *19*, 120.
26. Geng, X. L.; Zhang, S. Y.; Deng, J. *Soc. Wood Sci. Technol.* **2007**, *26*, 345.
27. Wang, W. H.; Yang, X. H.; Bu, F. H.; Sui, S. J. *Polym. Compos.* **2014**, *35*, 2180.
28. Yang, R.; Liu, Y.; Yu, J.; Zhang, D. Q. *Polym. Eng. Sci.* **2008**, *48*, 2270.
29. Yang, R.; Yu, J.; Liu, Y.; Wang, K. H. *Polym. Degrad. Stab.* **2005**, *88*, 333.
30. Valadez-González, A.; Veleza, L. *Polym. Degrad. Stab.* **2004**, *83*, 139.