Mechanical Properties and Water Absorption of Hemp Fibers–Reinforced Unsaturated Polyester Composites: Effect of Fiber Surface Treatment with a Heterofunctional Monomer

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Hemp fibers–reinforced unsaturated polyester (UP) composites were prepared by hand lay-out compression molding. Hemp fibers were treated with isocyanatoethyl methacrylate (IEM), using dibutyltin dilaurate as a catalyst. The results indicated that fiber treatment significantly increased tensile strength, flexural strength, flexural modulus, and water resistance of the resulting composites, and yet decreased the impact strength of the composites. The water absorption characteristics for composite samples immersed in water at room temperature followed Fickian behaviour, but for those evaluated at temperature 100 °C, there was a deviation from Fickian behaviour. Scanning electron microscope graphs of the tensile-fractured surface of hemp–UP composites revealed that fiber treatment with IEM greatly improved the interfacial adhesion between hemp fibers and UP resins. Fourier transform infrared analysis of the treated fibers showed that some IEM was covalently bonded onto hemp fibers.

Keywords: Hemp fibers; Unsaturated polyester; Composites; Fiber/matrix bond; Surface treatments; Mechanical properties; Water absorption

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INTRODUCTION

Natural plant fibers–reinforced polymer composites have received considerable attention for engineering applications in recent years. The advantages of natural plant fibers over inorganic fibers as reinforcers are high specific strength and modulus, economical viability, and good biodegradability. Several studies (Akil *et al*. 2009; He *et al*. 2012; Marais *et al*. 2005; Qiu *et al*. 2011; Ren *et al*. 2012) have been done on the replacement of inorganic fibers with bast fibers such as jute, flax, hemp, ramie, or kenaf for reinforcing polymer materials.

The main disadvantage of natural plant fibers in reinforcement for composites consists of the incompatibility between the hygroscopic natural fibers and the hydrophobic polymeric matrices. Physical and chemical methods can be used to improve the interfacial adhesion of such composites (Bledzki and Gassan 1999; Faruk *et al*. 2012). However, it was demonstrated that physical treatments could only modify a thin layer of fiber surfaces and did not change the hygroscopic characteristics of natural fibers. Many studies (Bessadok *et al*. 2008; Mehta *et al*. 2006; Sreekumar *et al*. 2009) have focused on chemical treatments for fibers and/or polymer matrices to improve the interfacial adhesion between fibers and polymer matrices. These treatments include silane treatment, alkaline treatment, acetylation, benzoylation, peroxide treatment for fibers, as well as using maleated coupling agents, and so on. It was found that there was an enhancement in the mechanical and thermal properties of hemp fibers–reinforced unsaturated polyester (UP) composites when the fibers were treated with alkali, silane, and acrylonitrile (Mehta *et al*. 2006). The modification of hemp fibers with alkaline solution, acetic anhydride, maleic anhydride, and silane was shown to improve the interfacial shear strength of hemp–reinforced polylactide, and hemp–reinforced UP composites (Sawpan *et al*. 2011). Alkalization, silane, and acetylation treatments on the hemp fibers were investigated for improving the mechanical properties of hemp–reinforced polyester composites (Kabir *et al*. 2012). The mechanical properties and water absorption of natural fibers–reinforced polymer composites also can be improved by using isocyanates as coupling agents. Isocyanates and blocked isocyanates were used as coupling agents to modify pine fibers, thus improving the interfacial adhesion between pine fibers and polypropylene composites (Gironès *et al*. 2007, 2008). By using *m*-isopropenyl-α,α-dimethylbenzyl-isocyanate as a novel compatibilizer, the mechanical properties of wood fibers–reinforced polypropylene composites were investigated (Karmarkar *et al*. 2007).

In previous studies, the treatment of hemp fibers with a combination of 1,6-diisocyanatohexane and 2-hydroxylethyl acrylate, or independently with *N*-methylol acrylamide and using sulfuric acid as the catalyst significantly improved the interfacial adhesion between hemp fibers and UP resins, and reduced the water-uptake of the resulting hemp–UP composites (Qiu *et al*. 2011, 2012). In this study, a novel isocyanate coupling agent, isocyanatoethyl methacrylate (IEM), was investigated for the treatment of hemp fibers. IEM is a heterofunctional monomer with a reactive isocyanate group and a vinyl polymerizable double bond (Thomas 1983). The two functions can be used to react independently with other vinyl monomers and an active hydroxyl of natural fibers. Most of the other known isocyanates are easy to decompose in water at room temperature. However, without specific catalysts, the IEM–H_2O reaction is negligible at or near room temperature. This was beneficial for easy handling when IEM was used for the treatment of hemp fibers.

EXPERIMENTAL

Materials

Hemp fibers with an average length of 3.86 cm and average fiber fineness of 7.69 dtex were obtained from Sanxing Hemp Industry (Anhui, China). UP resins (9231-1TP) were purchased from Shangwei Fine Chemical (Shanghai, China). Ethyl acetate, cobalt naphthenate, and methyl ethyl ketone peroxide (MEKP) were purchased from Sinopharm Chemical Reagent (Shanghai, China). IEM was obtained from Synasia (Suzhou) (Jiangsu, China). Dibutyltin dilaurate was obtained from Hongding International Chemical Industry (Jiangsu, China).

The Chemical Treatment of Hemp Fibers

Hemp-fiber mats with a width of 100 cm and a thickness of 0.5 cm were scutched from hemp fibers by using a cotton scutcher. The resulting fiber mats were cut into several mats with each mat being 22 cm long and 22 cm wide. Then the fiber mats were oven-dried at 103 °C for more than 24 h. The mixture of IEM (0.090 g) and dibutyltin dilaurate (0.018 g) dissolved in ethyl acetate (80 mL) was magnetically stirred at room temperature for 10 min and then immediately sprayed evenly onto both surfaces of each oven-dried hemp-fiber mat (90 g) with a spray bottle. The resulting IEM-coated fibers were then dried in an oven at 103 °C for 4 h and designated as IEM-1, meaning that the treated fibers contained 1 wt % of IEM based on the weight of the oven-dried hemp fibers and 20 wt % of dibutyltin dilaurate based on the weight of IEM. Using the same procedure, IEM solutions containing 3, 5, and 7 wt % of IEM based on the weight of oven-dried hemp fibers and 20 wt % of dibutyltin dilaurate based on the weight of IEM were used to treat hemp fibers to correspondingly generate IEM-3, IEM-5, and IEM-7.

The Preparation of Hemp–UP Composites

Untreated and IEM-treated hemp fibers were formed by hand into three 22 cm × 22 cm mats with uniform thickness. Each UP mixture was generated by mixing 90 g UP resins, 1 g styrene, 0.5 g cobalt naphthenate, and 7 g MEKP with a spatula for 1 min. The resulting mixture was immediately poured onto the surface of the hemp-fiber mats uniformly. Three hemp-fiber mats were stacked in the chamber of a stainless steel mold with the dimensions 22 cm × 22 cm × 0.3 cm. The mold was put onto the hot press with a pressure of 6 MPa for 5 min at room temperature, allowing the UP mixture to flow and wet the fiber mats well, and then moved to a press that was preheated to 110 °C. The platen temperature was maintained at 110 °C for 5 min and then increased from 110 to 140 °C while the pressure was maintained at 6 MPa. The hot-pressing duration at the final temperature was 30 min. After the hot-pressing was finished, the mold was removed from the press and cooled to room temperature while keeping the pressure at 6 MPa for 60 min on another press. Then the sample was removed from the mold for mechanical properties and water absorption tests.

Evaluation of Mechanical Properties of Hemp–UP Composites

Dumbbell specimens were prepared for the evaluation of tensile strength of the resulting composites. The composite panel was first cut off by 1 cm from each side, and then cut into rectangular specimens (150 mm × 20 mm × 3 mm) that were further cut to a dumbbell shape with a gripping length of 50 mm and a width of 10 mm in the center section. Rectangular specimens with the dimensions 80 mm × 10 mm × 3 mm were prepared for the evaluation of flexural and impact properties of composites.

Measurements of tensile strength and flexural properties of the hemp–UP composites were performed with a CMT6104 microcomputer control electronic universal testing machine (MTS System, Guangdong, China) in accordance with ISO 527-2010 and ISO 178-2010, respectively. The crosshead speed of the machine was 10 mm min^{-1}. The impact test was performed with a ZBC-25B Charpy Impact Tester (MTS System, Guangdong, China) in accordance with ISO 179-2010.

Water Absorption of Hemp–UP Composites

The water absorption of hemp–UP composites was measured by immersing the composite specimens in distilled water at different temperatures in accordance with ASTM D 5229M-04. The composite samples were cut into bars 7.62 cm long and 2.54 cm wide. All specimens were conditioned in an oven at 50 °C for 3 h, then put into a sealed plastic bag, and cooled at ambient temperature for 10 min. The specimens were weighed and immersed in a distilled-water bath at room temperature. At certain periods of soaking in the water, the specimens were removed from the water, wiped with tissue paper, weighed to measure the weight gain, and then put back in the water for continued soaking. Similarly, the specimens were immersed in a boiling water bath to determine water absorption at an elevated temperature. After a periodic time of immersion, the specimens were removed from the boiling water and cooled in distilled water for 15 min at room temperature before measuring the weight gain. The water uptake rate of the composite was calculated as the weight gain divided by the dry weight of the specimen.

Analysis of Interfacial Adhesion of Composites

Fractured surfaces of the specimens from the tensile tests were examined with a JEOL JSM-7500F SEM (JEOL Ltd., Tokyo, Japan) at an accelerating voltage of 5.0 kV. Specimens were coated with Au film (8 to 10 nm) before testing.

The untreated and IEM-treated hemp fibers were extracted with ethyl acetate and characterized with Fourier transform infrared analysis. The fiber samples (5 g) were first wrapped with filter paper and then extracted with ethyl acetate in a Soxhlet extractor for 6 h. The extracted fibers were dried at 103 °C for 24 h and then were powdered for characterization with FTIR. FTIR spectra were obtained on a Nicolet 5700 FT-IR spectrometer (Thermo Fisher Scientific, Florida, USA).

Statistical Analysis

Mechanical properties data were analyzed with one-way ANOVA (analysis of variance) using SPSS 11.5 (IBM Corp., USA). All comparisons were based on a 95% confidence interval.

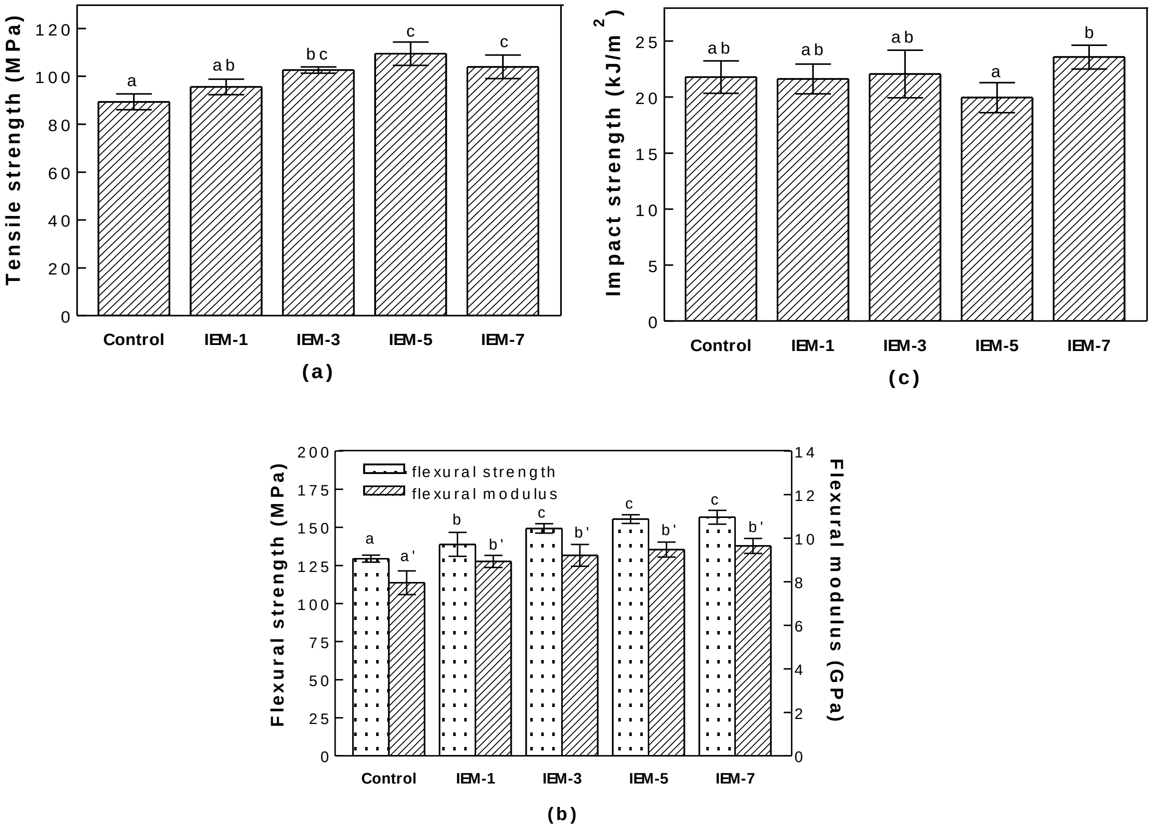
RESULTS

Mechanical Properties

The effect of fiber treatments with IEM on the tensile strength of hemp–UP composites is shown in Fig. 1a. IEM-1–resulting composites did not have a significant increase of tensile strength compared with the control (untreated-hemp–UP composites). When the IEM usage was raised to 3 wt % and 5 wt %, the tensile strength of composites significantly increased by 15 % and 23 % compared with that of the control, respectively. But the tensile strength of composites did not further increase when the IEM usage was raised from 5 to 7 wt %.

The IEM-treated-hemp–UP composites had higher flexural strengths than the control (Fig. 1b). The flexural strength of IEM-1–resulting composites was 7 % higher than that of the control. When the IEM usage was raised from 1 to 3 wt %, the flexural strength of composites further increased significantly. However, the flexural strength of composites did not significantly increase with the increasing of the IEM usage from 3 to 5 wt %, and from 5 to 7 wt %. Specifically, the flexural strengths of IEM-3, IEM-5, and IEM-7–resulting composites were comparable and were 15%, 20%, and 21% higher than that of the control.

Figure 1b indicates that the IEM-1–resulting composites had a much higher flexural modulus than the control. The flexural modulus of composites gradually increased when the usage of IEM was raised from 1 to 7 wt %. The flexural moduli of IEM-1, IEM-3, IEM-5, and IEM-7 were comparable and were 12%, 16%, 19%, and 21% higher than that of the control, respectively.



Note: IEM- means the usage of IEM based on the weight of the fibers. The error bar on the top of each column represents two standard deviations of the mean. There is a significant difference between any two groups when they have no common letter at the top of the bars; otherwise they did not differ significantly.

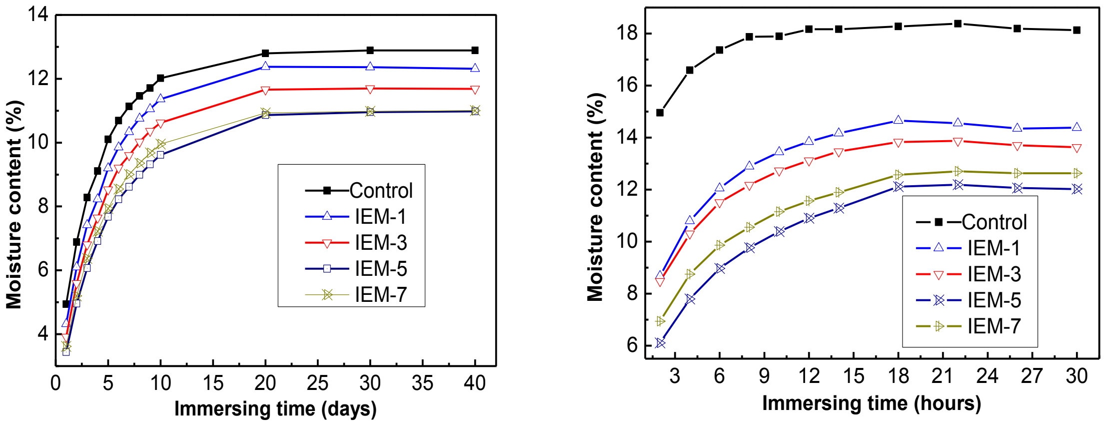
Fig. 1. Effect of surface treatment of hemp fibers on the (a) tensile strength, (b) flexural properties, and (c) impact strength of hemp–UP composites

The effect of fiber treatment with IEM on the impact strength of hemp–UP composites is shown in Fig. 1c. The impact strengths of IEM-1, IEM-3, and IEM-5–resulting composites were comparable and did not significantly differ from that of the control. But the impact strength of composites decreased slowly when the usage of IEM was raised from 3 to 5 wt %. When the usage of IEM was further raised from 5 to 7 wt %, the impact strength increased significantly, *i.e.*, IEM-5–resulting composites had the lowest impact strength among all the composites.

Therefore, in terms of tensile strength, flexural strength, and flexural modulus of the resulting composites, IEM-3 might be optimum for the treatment of hemp fibers. However, the impact strength of IEM-3–resulting composites exhibited little increase compared with that of the control.

Water Absorption

The water absorption curves of hemp–UP composites immersed in water are shown in Fig. 2. For hemp–UP composites immersed in water at room temperature (RT), the moisture content increased along with an increase in the immersing time when the immersing time was below 20 days, and then flattened out when the immersing time was longer than 20 days (Fig. 2a). The rate of water uptake for the composites immersed in water at an elevated temperature (100 °C) rapidly approached equilibrium compared with that at RT. The moisture saturation time of IEM-treated-hemp–UP composites soaked in boiling water was about 16 h (Fig. 2b). For those at RT, it was about 20 days, and much longer than that at 100 °C. Also, for the control or treated fibers–resulting composites with the same usage of IEM, the moisture content of composites at the equilibrium soaking at 100 °C was much higher than that at RT. In addition, at both temperatures the control had much higher moisture content than all IEM-treated-hemp–UP composites at each immersing time. There was a general trend that the moisture content decreased with the increasing of IEM usage from 1 to 5 wt % based on the fiber weight. And the moisture content of composites did not further decrease when the IEM usage was raised from 5 to 7 wt %, *i.e.* IEM-7–resulting composites had higher moisture content than IEM-5 at each soaking time. To summarize, the moisture content of different IEM usage–resulting composites had the following order: The control > IEM-1 > IEM-3 > IEM-7 > IEM-5.



(a) (b)

Note: IEM- means the usage of IEM based on the weight of the fibers.

Fig. 2. Effect of fiber-surface treatment of hemp fibers on the water uptake rate of hemp–UP composites immersed in water at (a) room temperature and (b) elevated temperature

Generally, the water absorption mechanism and kinetics of natural fibers–reinforced polymer composites can be analyzed from the following relationship (Osman *et al*. 2012; Sreekumar *et al.* 2009),

$$\log(M_t/M_\infty) = \log k + n\log t \qquad (1)$$

where M_t is the moisture content at time t; M_∞ is the moisture content at equilibrium, and k and n are constants. In Eq. 1, n and k give some information about the mechanism of diffusion taking place inside the composites. The value of coefficient n indicates the different diffusion behaviors. If the value of $n = 0.5$, the diffusion follows Fickian behavior. The diffusion is anomalous when the value of $n > 1$. For non-Fickian diffusion, the value of n is between 0.5 and 1. The diffusion coefficient can be calculated from the following formula (Sreekumar *et al*. 2009),

$$D = \pi(kh/4M_\infty)^2 \qquad (2)$$

where k is the slope of the linear part of water absorption curve and h is the initial specimen thickness. The diffusion coefficient represents the ability of the water molecules moving inside the composites.

The diffusion parameters of hemp–UP composites are given in Table 1. It is interesting that the values of k increased with increasing temperature. The values of n for hemp–UP composites immersed in water at RT were very close to 0.5, indicating that moisture absorption in the composites followed Fickian behavior. When the immersion temperature was 100 °C, the values of n decreased and were far from 0.5. It was demonstrated that moisture absorption in composites immersed in water at 100 °C could not be described appropriately by Fickian behavior. Furthermore, the diffusion coefficient of composites immersed in water at different temperatures had the same tendency: Thus, the control had a higher diffusion coefficient than that of IEM-treated-hemp–UP composites (Table 1). But the diffusion coefficient significantly increased when the temperature was raised from RT to 100 °C. In other words, the elevated temperature accelerated the ability of water molecules to move inside hemp–UP composites.

Table 1. Diffusion Parameters of Hemp–UP Composites

Condition	Samples	n	k(h^2)	Saturation Water Uptake M_∞ (%)	Diffusion Coefficient (D) ×10^{-13} (m^2/s)
Room temperature (RT)	Control	0.4320	0.0990	12.87	49.10
	IEM-1	0.4597	0.0827	12.35	32.33
	IEM-3	0.4762	0.0748	11.68	27.83
	IEM-5	0.4898	0.0673	10.93	27.39
	IEM-7	0.4810	0.0727	10.97	26.16
Elevated temperature (100 °C)	Control	0.1295	0.7544	18.21	2851.54
	IEM-1	0.2878	0.4945	14.48	1165.24
	IEM-3	0.2653	0.5151	13.37	1321.00
	IEM-5	0.3409	0.4004	12.09	769.56
	IEM-7	0.3055	0.4482	12.63	993.28

Analysis of FTIR Spectra

The FTIR spectra of untreated and IEM-treated hemp fibers are shown in Fig. 3.

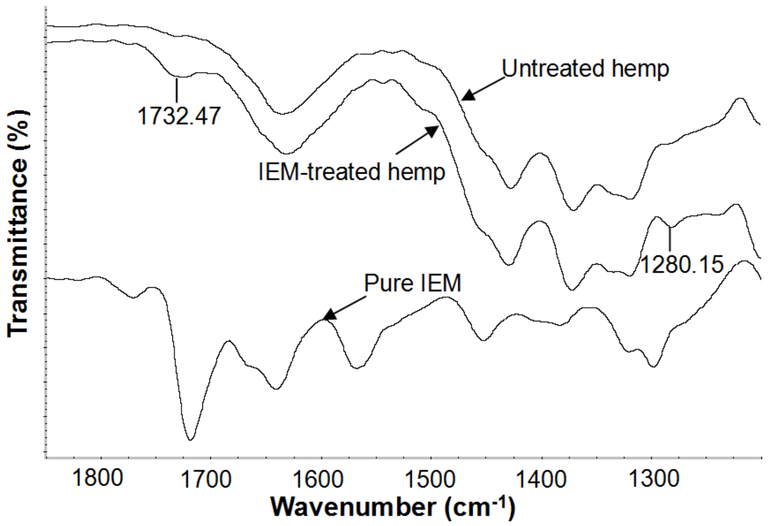
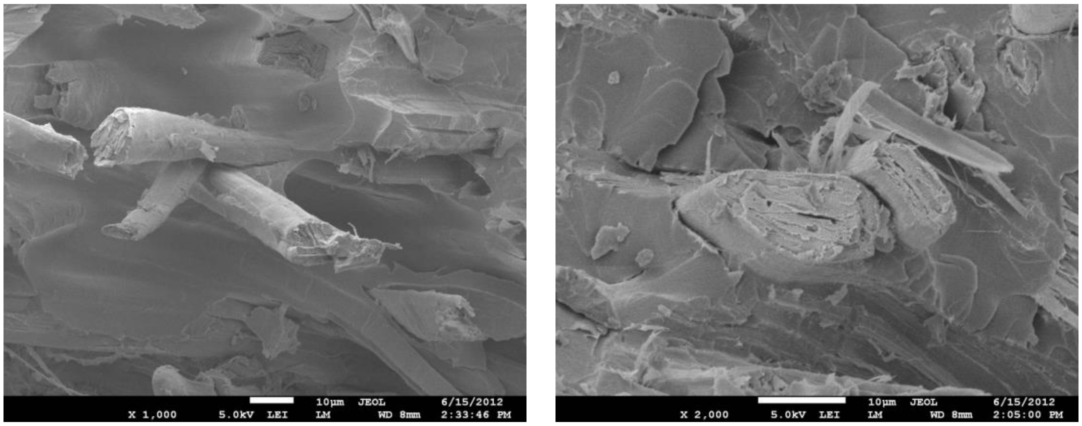


Fig. 3. FTIR spectra of untreated, IEM-treated hemp fibers, and pure IEM

IEM-treated fibers had a strong peak at 1732.47 cm^{-1} resulting from the stretch vibration of the carbonyl group, and a peak at 1280.15 cm^{-1} resulting from the stretch vibration of the C–N bond. Untreated fibers did not show peaks around 1732 cm^{-1} and 1280 cm^{-1}. The C=O groups might be attributed to bonds in IEM or in the carbamate resulting from the reaction products of the hydroxyl groups of hemp fibers and the isocyanate groups of IEM. But the C–N groups could only result from the structure of carbamate. This indicated that IEM was covalently bonded onto hemp fibers.

Interfacial Adhesion

The SEM graphs of the tensile-fractured surface of hemp–UP composites are shown in Fig. 4. Individual fibers and pull-out holes were observed on the fractured surface of the untreated-hemp–UP composites, which showed the poor interfacial adhesion between the untreated-fibers and UP resins (Fig. 4a). The smooth surface of fibers and deep holes indicated that the UP resins might not be able to wet the surface of fibers well to form good adhesion between fibers and resins. On the other hand, there were fractured fibers in the root and fewer pull-out holes could be seen from the SEM graphs of IEM-treated-hemp–UP composites, which indicated superior interfacial adhesion between the fibers and UP resins (Fig. 4b).



(a) (b)

Fig. 4. SEM graphs of the tensile-fractured surfaces of hemp–UP composites. (a) The control, *i.e.*, without IEM treatment for hemp fibers, (b) IEM-5–resulting composites

DISCUSSION

To achieve good mechanical properties, the stress must be transferred effectively to the fiber throughout the interface of fibers–reinforced composites, which requires a strong fiber/matrix bond (Montaño-Leyva *et al*. 2013). Hence, the hemp fibers were treated with IEM, using dibutyltin dilaurate as a catalyst, in order to improve the interfacial adhesion between hemp fibers and UP resins in this study. The possible reactions in the preparation of IEM-treated-hemp–UP composites are shown in Fig. 5. Hemp fibers would be coated by a solution of IEM and dibutyltin dilaurate after fiber treatment. During the progress of fiber drying, structure I in Fig. 5 was generated due to the reaction of the isocyanate groups of IEM and the hydroxyl groups of hemp fibers by using dibutyltin dilaurate as a catalyst. The C=C bonds on the IEM-treated fibers would have participated in the free-radical polymerization when the mixture of hemp fibers and UP resins were heated, where styrene was used as the crosslinking agent, and MEKP as the initiator. Structure II in Fig. 5 is only a representative copolymer among IEM-treated fibers, styrene, and UP backbones. The C=C bonds in structure I might be directly linked to UP backbones without the styrene unit in between.

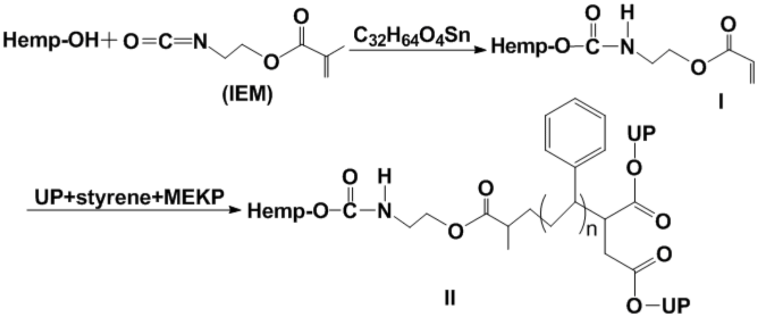


Fig. 5. Proposed reactions in the IEM-treated hemp–UP composites

FTIR analysis of the treated fibers indeed demonstrated that some IEM was covalently bonded onto hemp fibers. The SEM graphs of tensile-fractured hemp–UP composites also indicated that the treatments of hemp fibers with IEM improved the interfacial adhesion between the treated-hemp fibers and UP resins. These can be reasonably explained by the fact that IEM treatment for hemp fibers significantly increased the tensile strength, flexural strength, and flexural modulus of the resulting composites. However, the usage of IEM would be up to the saturation level when the addition of IEM was increased from 3 to 5 wt % based on the weight of the fibers. When the usage of IEM usage was above the saturation level, the excess IEM molecules that were not covalently bonded onto the hemp fibers could be loosely trapped at points on the surface of fibers and UP resins, thus forming weak interfacial layers between the hemp fibers and UP resins, and then slightly decreasing the tensile strength of the composites. The result was in agreement with the study of wood flour–reinforced polypropylene composites by using an isocyanate grafted polypropylene as a compatibilizer (Guo *et al*. 2012). On the other hand, when there is a strong bond at the interface of composites, the impact damage does not propagate into the surrounding area of the impacted point, and then local failure is created due to localized stress concentration. The strong interface leads to a brittle fracture mode with relatively low energy absorption, so the impact strength of composites is reduced (Dhakal *et al*. 2007b; Shahzad 2012). This effect can be responsible for the treatment of IEM-5 significantly decreasing the impact strength of the resulting composites.

The reactions between IEM and hydroxyl groups of hemp fibers will reduce the number of hydroxyl groups on the fiber surfaces, thus reducing the hydrophilicity of the fibers. The reduced hydrophilicity is part of the reason for the decreased water uptake and diffusion coefficient of the treated-hemp–UP composites. The free-radical polymerization between IEM-treated fibers and UP resins will form tight resin networks around the fibers, thus the water absorption of composites is hindered. The decrease of hydrophilicity and the improvement of interfacial adhesion may account for the fact that the IEM-treated fibers–resulting composites had superior water resistance than that of the untreated hemp fibers (Fang *et al*. 2013). Similarly, the residual IEM catalyzed with dibutyltin dilaurate will form weak layers between the fibers and the UP resins and react with water molecules. Those molecules also may form covalent linkages with UP resins during the curing process, but still be hydrophilic and tend to absorb water. Therefore, the water uptake rate of the composites increased when the usage of IEM was raised from 5 to 7 wt % based on the weight of the hemp fibers.

The water uptake content and diffusion coefficient of the composites immersed in boiling water were higher than that at RT. The higher and more rapid water absorption rate of samples immersed in boiling water may be attributed to the higher diffusivity of water molecules into the composites, leading to more interfacial cracks induced by moisture at an accelerated rate. In a high-temperature environment, with the developing of microcracks on the surface and inside of composites as well as fiber debonding in the interface region, water transfer becomes more active and more water molecules penetrate into materials at an accelerated velocity (Dhakal *et al*. 2007a).

CONCLUSIONS

1. The treatment of hemp fibers with IEM, using dibutyltin dilaurate as a catalyst significantly increased the tensile strength, flexural strength, and flexural modulus of the resulting hemp–UP composites, and yet decreased the impact strength of the composites.
2. Scanning electron microscope graphs of the tensile-fractured surface of hemp–UP composites revealed that fiber treatment with IEM greatly improved the interfacial adhesion between hemp fibers and UP resins. Fourier transform infrared analysis of the treated fibers validated that some IEM was covalently bonded onto hemp fibers which illustrated the improvement of mechanical properties and water resistance of IEM-treated hemp–UP composites.
3. The water-absorption characteristics for composites samples immersed in water at room temperature followed Fickian behavior, but for those at 100 °C, there was a deviation from Fickian behavior. The IEM-treated-hemp–UP composites had better water resistance compared with those of untreated hemp fibers.

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