

Modifications of Kenaf Fibers with *N*-Methylol Acrylamide for Production of Kenaf-Unsaturated Polyester Composites

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ABSTRACT: Kenaf fiber mats were modified with *N*-methylol acrylamide (NMA) solution with either sulfuric acid or *p*-toluenesulfonic acid (TsOH) as a catalyst. The (unmodified-kenaf)-UPE composites and (NMA-modified-kenaf)-UPE composites were obtained by a compression molding process. Orthogonal experiments were used for determination of optimum modification variables for maximizing the flexural strength of the resulting (NMA-modified-kenaf)-UPE composites. Three variables with three levels (NMA usage: 1, 3, and 5 wt %; usage of acid catalyst: 0.1, 0.2, and 0.3 wt %; and oven-dry temperature for NMA-treated kenaf fiber mats: 80, 103, and 125°C) for each variable were used in the orthogonal experiments. Modifications of kenaf fibers with TsOH as a catalyst significantly increased

the flexural strength. Modifications of kenaf fibers with either sulfuric acid or TsOH as a catalyst significantly enhanced the flexural modulus and significantly reduced the water-uptake rate of the resulting kenaf-UPE composites. The improved interfacial adhesion between kenaf fibers and UPE resin was confirmed by characterization of NMA-modified fibers with Fourier transform infrared spectroscopy, as well as scanning electron microscopy images. The mechanisms for the improved strengths and water-resistance by the NMA modifications of the kenaf fibers were discussed. © 2012 Wiley Periodicals, Inc. *J Appl Polym Sci* 125: 2846–2853, 2012

Key words: kenaf fiber; surface modification; unsaturated polyester; interfacial adhesion; *N*-methylol acrylamide

INTRODUCTION

Glass fibers are one of the major reinforcing materials used in composites industry. They are heavy and are derived from nonrenewable materials. Use of renewable plant fibers for replacement of glass fibers has drawn much interest in recent years.^{1–3}

Kenaf is one of the most important plant fibers that can potentially be used for replacement of glass fibers. Kenaf fibers are abundant, renewable, inexpensive, and readily available.^{4,5} Unsaturated polyester (UPE) is one of the most commonly used thermosetting resin in the production of fiber-reinforced polymer composites. It has many advantages over other thermosetting resin in terms of mechanical properties, room temperature curing capability, and transparency.⁶ Kenaf-UPE composites have been investigated for use as interior automobile parts such as interior door trim and seating back.^{7–9}

Hydrophilic kenaf fibers are incompatible with hydrophobic UPE, resulting in low adhesion

between kenaf fibers and UPE, and high water-uptake of the kenaf-UPE composites.¹⁰ The water-uptake typically leads to strength loss and swelling, that is, dimensional instability of the composites, thus being an important property for many applications, especially exterior applications, of the composites. Surface modifications of kenaf fibers are one of the most effective ways of improving the interfacial adhesion between kenaf fibers and UPE and reducing the water-uptake of the kenaf-UPE composites. The modification of kenaf fibers with alkali and methacryloxypropyltrimethoxy silane has been studied.^{6,11} The alkaline modification of kenaf fibers enhanced the flexural strength and flexural modulus of the kenaf-UPE composites to 123 MPa and 13 GPa, respectively.⁶ However, those modifications did not improve the water-resistance of the composites. Surface coating and edge sealing improved the water resistance of the composites, but did not improve the mechanical properties, with the flexural strength and flexural modulus being below 90 MPa and 9 GPa, respectively.¹²

More study on surface modifications of the kenaf fibers is needed so that the kenaf-UPE composites can have superior strengths and low water-uptake rate. In this study, a new coupling agent, *N*-methylol acrylamide (NMA), was investigated for the

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modifications of kenaf fibers. NMA has been incorporated into adhesives and coatings and served as a crosslinking point.^{13,14} However, little has been published on whether NMA can serve as an effective coupling agent for kenaf-UPE composites. The objective of this study is to investigate modifications of kenaf fibers with NMA and effects of the modifications on strength and water resistance of the resulting (NMA-modified-kenaf)-UPE composites.

EXPERIMENTAL

Materials and equipment

Kenaf fibers were purchased from Wilhelm G. Clasen services (Dhaka, Bangladesh). Aropol 7030 (a mixture of about 60% unsaturated polyester and 40% styrene) and LP-4016 (poly(vinyl acetate)) were obtained from Ashland Chemical (Columbus, OH). Styrene, NMA as a 48 wt % aqueous solution, and *tert*-butyl peroxybenzoate (TBPB) were obtained from Sigma-Aldrich (St. Louis, MO). Zinc stearate and *p*-Toluenesulfonic acid (TsOH) monohydrate were obtained from Acros Organics (Morris Plains, NJ). Chloroform was purchased from J. T. Baker (Phillipsburg, NJ). Differential scanning calorimetric (DSC) graphs were obtained from a TA DSC 2092 (TA Instruments, New Castle, DE). The hot-pressing was performed on an automatic Benchtop Carver press (Carver, Wabash, IN). Mechanical properties were obtained from a Sintech machine (MTS Systems, Enumclaw, WA). Scanning electronic microscope (SEM) graphs were obtained by a FEI Quanta 600 SEM (Hillsboro, OR). The FTIR spectra were obtained with a Nexus 470 spectrometer (Thermo Nicolet, Madison, WI), using a KBr pellet method.

Preparation of kenaf fiber mats

Wet kenaf fibers (100 g, 2 in. length) were fed into a LOUET drum carder for tearing apart fiber bundles and forming oriented kenaf fiber mats through a carding, layering, and needle-punching process. This carding process oriented the fiber length along the length of the fiber mat. The resulting fiber mats were cut by a paper cutter into 5 mats, with each mat having the dimensions of 200 × 200 × 10 mm³. The fiber mats were stacked horizontally in an aluminum tray and oven-dried at 103°C for at least 20 h before use.

Preparation of UPE resin

Aropol 7030 resin (62.2 parts) and LP-4016 (28.6 parts) were mixed together to form a solution. Styrene (4.8 parts) and zinc stearate (4.4 parts) were added to the solution and the resulting mixture was

mechanically stirred at room temperature for 2 h to form a UPE resin.

DSC characterization of the UPE resin

The UPE resin (40 g) and TBPB (0.56 g) were mixed well by hand. The mixture (12 mg) was put in a hermetic aluminum pan with a lid, and an empty aluminum pan with a lid was used as a reference. Argon was used as a purge gas, and the flow rate was 40 mL/min. The furnace temperature was raised from 25 to 250°C at 10°C/min.

Experimental design for optimal processing parameters

Orthogonal experiments of three variables with three levels (NMA usage: 1, 3, and 5 wt %; usage of acid catalyst: 0.1, 0.2, and 0.3 wt %; and oven-dry temperature for NMA-treated kenaf fiber mats: 80, 103, and 125°C) (all the weight are based on the 78 g oven-dried kenaf fiber mats for each composite) for each variable were conducted to determine the optimum modification parameters for kenaf-UPE composites. The flexural strength of composites was used as the criterion for the process optimization. Variance analysis was conducted to determine the effects of different variables on the flexural strength of the composites.

Treatment of kenaf fiber mats with NMA

The 48% NMA aqueous solution (1.63 g) and 10% sulfuric acid solution (0.78 g) were dissolved in deionized water (80 mL). The mixture was magnetically stirred at room temperature for 10 min and was then sprayed onto five oven-dried kenaf fiber mats (78 g). More specifically, the resulting solution (about 16 mL) was evenly sprayed onto both surfaces of each oven-dried kenaf fiber mat (about 15.6 g) by a spray bottle. The resulting five NMA-modified fiber mats were stacked horizontally in an aluminum tray and oven-dried at 80°C for 24 h. The resulting NMA-modified fiber mats contained 1 wt % NMA and 0.1 wt % sulfuric acid based on the weight of kenaf fiber mats.

This procedure was used for preparation of NMA-treated kenaf fibers that contained 1, 3, or 5 wt % of NMA with 0.1, 0.2, or 0.3 wt % sulfuric acid or TsOH as a catalyst, and were oven-dried at 80, 103, and 125°C, respectively.

After the oven-drying, the NMA-treated fiber mats were immediately used for making kenaf-UPE composite boards.

Hot-press procedure for kenaf-UPE composites

UPE resin (78 g) and TBPB (1.2 g) were mixed well and the resulting mixture (15.8 g) of the mixture was uniformly poured onto an oven-dried untreated or NMA-treated kenaf fiber mat that had been placed into a stainless steel mold with the dimensions of $200 \times 200 \times 3 \text{ mm}^3$. The second mat was stacked above the first mat in a way that the kenaf fibers were oriented in the same direction, and the resulting UPE-TBPB mixture (15.8 g) was then uniformly poured onto the second mat. This process was repeated until a stack of five mats was made. The mold filled with the stack and UPE resin was placed onto the lower platen of an automatic benchtop Carver press, and pressed at 3.24 MPa for 10 min at room temperature allowing for thorough penetration of UPE resin into fibers. The curing was carried out at 160°C with a pressure of 4.24 MPa for 20 min, followed by cooling for 100 min under pressure. The resulting composites had a fiber loading of 50 wt %. The (unmodified-kenaf)-UPE composite with 50 wt % oven-dried kenaf fiber loading was used as the control.

Determination of mechanical properties of the kenaf-UPE Composites

For the flexural test, each specimen had a rectangular dimension of $70 \times 14 \times 3 \text{ mm}$. The longitudinal direction of the test specimen was parallel to the longitudinal direction of the fibers. The three-point flexural test was performed on a Sintech testing machine in accordance with ASTM D 790-03, with a 50-mm span, crosshead moving downward at a speed of 5 mm/min. The flexural strength and flexural modulus were obtained from the test.

Water-uptake rate of the kenaf-UPE composites

Method 1: Soaking the composite specimens in water at room temperature

Each specimen had a dimension of $80 \times 25 \times 3 \text{ mm}$. All specimens were weighed and then soaked in water at room temperature in accordance with ASTM D 5229 M-04. At a predetermined time, the specimens were removed from water, wiped with tissue paper, weighed, and then put back to water. The water-uptake rate was obtained from the weight gain divided by dry weight of the specimen.

Method 2: Soaking the composite specimens in boiling water

Each specimen was cut into a dimension of $80 \times 25 \times 3 \text{ mm}$. All the specimens were weighed and then soaked in boiling water for 4 h. The specimens were

then removed from water, wiped with tissue paper, and weighed. The water-uptake rate was obtained from the weight gain divided by dry weight of the specimen.

Characterization of fractured kenaf-UPE composites with SEM

The SEM images were used for characterization of the fractured surface of the composites. Specimens after the flexural tests were cut to small pieces for SEM imaging, the cross area of fractured surface having dimensions of $5 \times 3 \text{ mm}$. All the specimens were coated with an Au-Pd film in the coater for 50 s before testing to prevent the electrical charging effect. The SEM images were obtained at an accelerating voltage of 10.0 kV.

Characterization of untreated and NMA-treated kenaf fibers with FTIR

The FTIR was used for detection of the newly formed covalent bond between NMA and kenaf fibers. The 48% NMA aqueous solution (1.04 g) and 10% sulfuric acid (0.1 g) were dissolved in deionized water (10 mL). The mixture was magnetically stirred at room temperature for 10 min. Oven-dried kenaf fibers (10 g) were immersed into the resulting mixture, and oven-dried again at 80°C for 24 h for forming NMA-treated kenaf fibers with sulfuric acid as a catalyst. The same procedure was used for the preparation of NMA-treated kenaf fibers with TsOH as a catalyst.

Untreated kenaf fibers and NMA-treated kenaf fibers (3 g) with sulfuric acid or TsOH as a catalyst were wrapped with filter paper and then extracted with chloroform in a Soxhlet extractor for 48 h, respectively. The extracted fibers were oven-dried at 80°C for 24 h and then cut into small pieces for FTIR characterization. FTIR spectra were recorded using a KBr pellet method and the number of scans for all three samples was 64.

Statistical analysis

Data from flexural tests were analyzed with two sample t-test method using R statistical software (Free Software Foundation, Boston, MA). All *P*-values from tests were based on a 95% confidence interval.

RESULTS AND DISCUSSION

A DSC graph of the UPE resin is shown in Figure 1. The exothermic graph indicated that the UPE had a peak around 135°C. The peak started at around 115°C, and came to an end at around 156°C. These

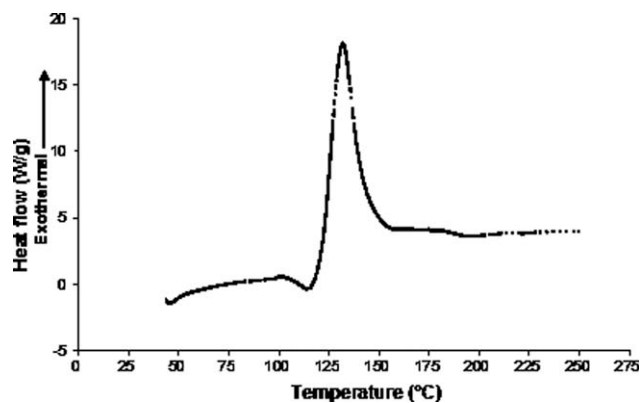


Figure 1 The DSC curve of UPE resin.

results implied that the resin could be cured at 156°C. To make sure the UPE resin could cure completely, 160°C was used as the hot-press temperature in this study.

Three NMA usages (1, 3, and 5 wt %), three catalyst usages (0.1, 0.2, and 0.3 wt %), and three oven-dry temperatures (80, 103, and 125°C) were selected as variables in orthogonal experiments of making (NMA-treated kenaf)-UPE composites. The experimental design and results of orthogonal experiments are shown in Tables I and II. With sulfuric acid as the catalyst, maximum flexural strength was obtained under the following variables: A3, B2, and C2, that is, 5 wt % NMA usage, 0.2 wt % acid usage, and 103°C oven-dry temperature of kenaf fiber mats (Table I). An independent experiment further confirmed that the kenaf-UPE composite made under these optimum variables had the flexural strength of 130.24 MPa that was higher than all other nine combinations of process variables shown in Table I.

With TsOH as the catalyst, the highest flexural strength was obtained under the variables of A3, B1, and C3, that is, 5 wt % NMA usage, 0.1 wt % acid

usage, and 80°C oven-dry temperature (Table II). Independent experiment confirmed that the kenaf-UPE composite made at these variables had the flexural strength of 143.65 MPa that was the highest among all combinations of variables investigated, and was also significantly higher than the published values.^{6,12}

Results from the variance analysis (Tables I and II) for the orthogonal experiments indicated that the oven-dry temperature for kenaf mats had the highest maximum difference among the three variables, 17.61 (Table I) and 13.96 (Table II), respectively. Thus the oven-dry temperature was the most influential variable on the flexural strength of the kenaf-UPE composites for both sulfuric acid and TsOH as a catalyst. The optimum temperature was 103°C for sulfuric acid as the catalyst and 80°C for TsOH as the catalyst, indicating the reactions between NMA and fibers required a higher temperature for sulfuric acid than for TsOH.

Under optimum process variables, the flexural strength of (NMA-treated kenaf)-UPE composites with sulfuric acid as the catalyst was not significantly higher than that of (untreated-kenaf)-UPE composites (the control) ($P = 0.174$) (Fig. 2). However, the flexural strength of (NMA-treated kenaf)-UPE composites with TsOH as the catalyst was 20.8% higher than that of the control and 11.5% higher than that of (NMA-treated kenaf)-UPE composites with sulfuric acid as the catalyst. The enhancement in the flexural properties of composites after surface treatment can be attributed to the improved interfacial adhesion between the fibers and UPE resin. The flexural modulus with sulfuric acid as the catalyst was 14.0% higher than that of the control ($P = 0.023$) and was 10.1% lower than that with TsOH as the catalyst. Under optimum process variables, both flexural strength ($P = 0.013$) and

TABLE I
Orthogonal Experiments for Optimal Processing Parameters of (NMA-Modified Kenaf)-UPE Composites with Sulfuric Acid as the Catalyst

Experiment No.	Factor A, NMA loading (wt %)	Factor B, acid catalyst loading (wt %)	Factor C, oven-dry temperature for kenaf mats (°C)	Flexural strength (MPa)
1	A1(1)	B1(0.1)	C1(125)	105.54
2	A1	B2(0.2)	C2(103)	127.22
3	A1	B3(0.3)	C3(80)	119.34
4	A2(3)	B1	C2	124.72
5	A2	B2	C3	129.14
6	A2	B3	C1	107.16
7	A3(5)	B1	C3	120.90
8	A3	B2	C1	115.82
9	A3	B3	C2	129.42
Mean 1	117.37	117.05	109.51	
Mean 2	120.34	124.06	127.12	
Mean 3	122.05	118.64	123.13	
Maximum difference	4.68	7.01	17.61	

TABLE II
Orthogonal Experiments for Optimal Processing Parameters of (NMA-Modified Kenaf)-UPE Composites with TsOH as the Catalyst

Experiment no.	Factor A, NMA loading (wt %)	Factor B, acid catalyst loading (wt %)	Factor C, oven-dry temperature for kenaf mats (°C)	Flexural strength (MPa)
1	A1(1)	B1(0.1)	C1(125)	123.28
2	A1	B2(0.2)	C2(103)	121.05
3	A1	B3(0.3)	C3(80)	131.28
4	A2(3)	B1	C2	133.95
5	A2	B2	C3	131.10
6	A2	B3	C1	121.80
7	A3(5)	B1	C3	143.65
8	A3	B2	C1	119.05
9	A3	B3	C2	130.65
Mean 1	125.20	133.63	121.38	
Mean 2	128.95	123.73	128.55	
Mean 3	131.12	127.91	135.34	
Maximum difference	5.92	9.90	13.96	

flexural modulus ($P = 0.001$) with TsOH as the catalyst were significantly higher than those with sulfuric acid as the catalyst, respectively, which implied that TsOH was a better catalyst than sulfuric acid in terms of increasing flexural properties for the production of (NMA-treated-kenaf)-UPE composites. It appeared that TsOH was more effective in catalysis of grafting NMA onto kenaf fibers than sulfuric acid.

The water-uptake rate of the composites versus time during a long-term water immersion at room temperature is shown in Figure 3. The (NMA-treated-kenaf)-UPE composites, either with sulfuric acid or TsOH as a catalyst, had a significantly lower water-uptake rate than the control at each predeter-

mined soaking time. The water-uptake rates almost approached equilibrium after 40 days. The greater water-uptake rate for the control was attributed to the hydrophilicity of the kenaf fibers. NMA-modification of kenaf fibers could make the fibers more hydrophobic and improve interfacial adhesion between the fibers and UPE resin, thus reducing the water-uptake rate. (NMA-treated kenaf)-UPE composites with TsOH as the catalyst had a higher water-uptake rate than those with sulfuric acid as the catalyst. Results indicated that sulfuric acid was a preferred catalyst to TsOH in terms of reducing the

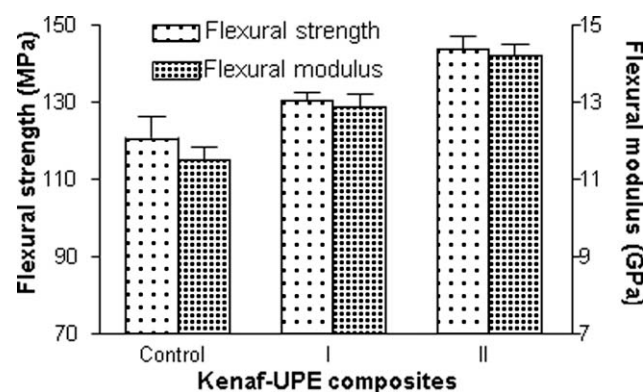


Figure 2 Flexural properties of (NMA-treated kenaf)-UPE composites made under optimum modification variables. (Control, (unmodified kenaf)-UPE composites; I, (NMA-modified kenaf)-UPE composites with sulfuric acid as the catalyst (5 wt % NMA, 0.2 wt % sulfuric acid, 103°C oven-dry temperature of kenaf fiber mats); II, (NMA-modified kenaf)-UPE composites with TsOH as the catalyst (5 wt % NMA, 0.1 wt % TsOH usage, 80°C oven-dry temperature of kenaf fiber mats). Data are the mean of at least five replicates and the error bars represent a standard error of the mean).

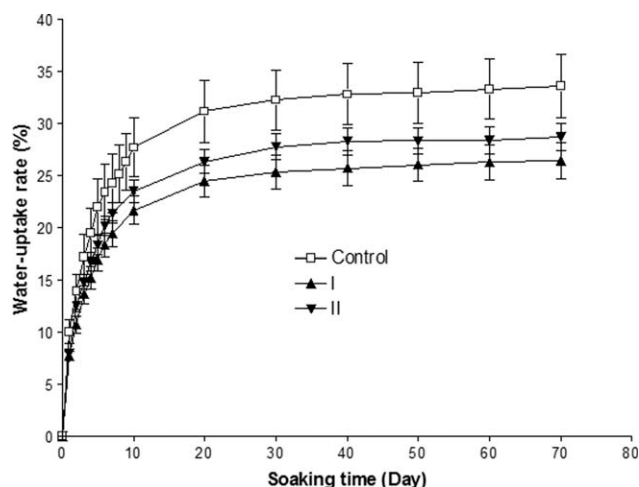


Figure 3 The water-uptake rate of the composites made under optimum modification variables in water at room temperature. (Control, (unmodified kenaf)-UPE composites; I, (NMA-modified kenaf)-UPE composites with sulfuric acid as the catalyst; II, (NMA-modified kenaf)-UPE composites with TsOH as the catalyst. Refer to Figure 2 legend for usage of NMA and acids, and oven-dry temperature of kenaf fiber mats. Data are the mean of at least four replicates and the error bars represent a standard error of the mean).

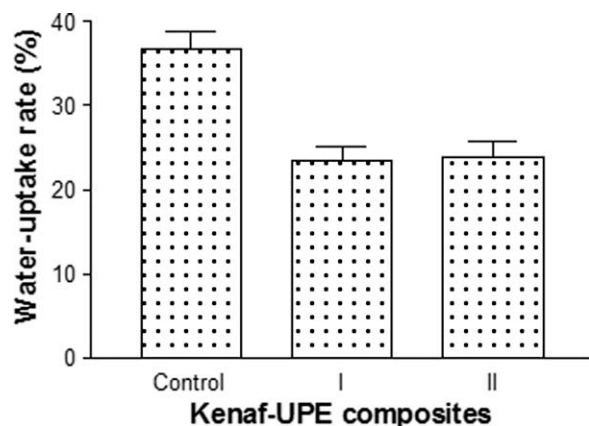


Figure 4 The water-uptake rate of the composites made under optimum modification variables in boiling water. (Control, (unmodified kenaf)-UPE composites; I, (NMA-modified kenaf)-UPE composites with sulfuric acid as the catalyst; II, (NMA-modified kenaf)-UPE composites with TsOH as the catalyst. Refer to Figure 2 legend for usage of NMA and acids, and oven-dry temperature of kenaf fiber mats. Data are the mean of at least four replicates and the error bars represent a standard error of the mean).

water-uptake rate of the resulting (NMA-treated kenaf)-UPE composites at room temperature. These results can be used for predicting the relative water-uptake rate and the maximum amount of water-uptake of the composites in a wet environment at room temperature.

The boiling water test is an accelerated aging test for the composites. This test can be used for predicting the water-uptake rate of the composites when they are used in a hot and wet environment such as an exterior wet environment under direct sunlight. (NMA-treated kenaf)-UPE composites with either TsOH or sulfuric acid as a catalyst had a lower water-uptake rate than the control in boiling water (Fig. 4). The water-uptake rate of (NMA-treated kenaf)-UPE composites with TsOH as the catalyst was comparable to that with sulfuric acid as the catalyst in boiling water ($P = 0.8483$) (Fig. 4).

The possible reactions during the fiber treatment with NMA and the compression molding process are shown in Figure 5. The *N*-hydroxymethyl group in NMA can readily form covalent linkages with hydroxyl groups of kenaf fiber components such as cellulose and hemicelluloses under an acid catalysis (Fig. 5). A separate experiment has demonstrated that NMA solution was completely soluble in chloroform. The NMA-treated kenaf fibers were extensively extracted with chloroform, which removed residual NMA that were not covalently bonded onto fibers. The FTIR spectra of NMA-treated kenaf fibers with either sulfuric acid or TsOH as a catalyst had a strong peak at 1670 cm^{-1} corresponding to stretching of ester carbonyl ($\text{C}=\text{O}$) functional groups as well as a strong peak at 1550 cm^{-1} corresponding

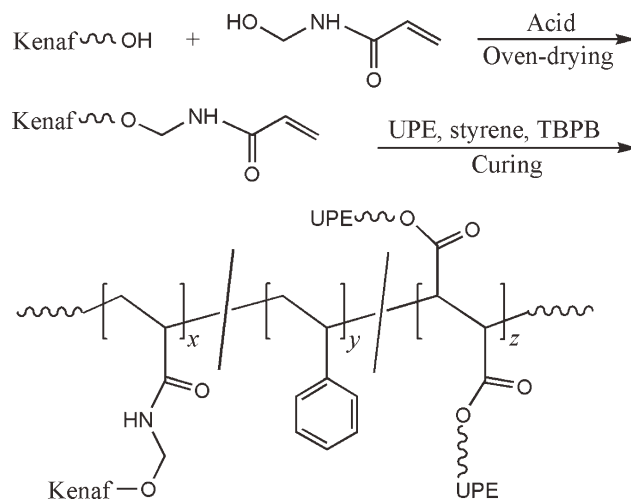


Figure 5 Proposed reactions in the formation of the (NMA-treated kenaf)-UPE composites.

N-H bending, whereas the FTIR spectrum of untreated kenaf fibers did not have these peaks, which demonstrated that NMA was covalently bonded onto kenaf fibers (Fig. 6). The peak intensity at 1670 cm^{-1} with TsOH as the catalyst was stronger than that with sulfuric acid as the catalyst, which indicated that more NMA was grafted onto the fibers with TsOH as the catalyst than that with sulfuric acid as the catalyst. This was consistent with the result that (NMA-treated-kenaf)-UPE composite with TsOH acid as the catalyst had higher flexural strength and flexural modulus than that with sulfuric acid as catalyst. The NMA-treated fibers had pendant vinyl groups that could readily react with the $\text{C}=\text{C}$ bonds in UPE through a free radical polymerization process during the compression molding process (Fig. 5).

The SEM image of unmodified kenaf fibers showed the kenaf fibers have relatively smooth

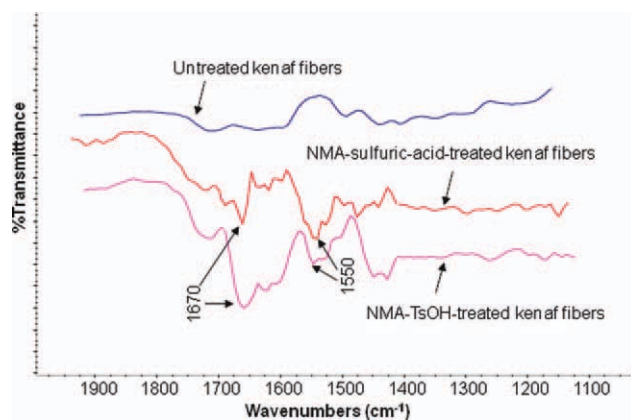


Figure 6 FTIR spectra of chloroform-extracted kenaf fibers with or without NMA treatments. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

surfaces (Fig. 7). The UPE resins might not be able to penetrate well the surfaces for formation of good mechanical interlocks between the UPE resins and the kenaf fibers. This is why a surface modification of the kenaf fibers with a coupling agent is typically required for improving the interfacial adhesion between the UPE resins and the kenaf fibers. The SEM image of the fractured surface of the unmodified kenaf-UPE composites further confirmed the poor interfacial adhesion, evidenced by the clean fiber surfaces and fiber pullouts [Fig. 8(a)]. For the NMA-treated-kenaf-UPE composite, there were extensive fiber breaks and no obvious gap between the kenaf fibers and the UPE resins could be seen, which indicated superior interfacial adhesion between the fibers and the UPE matrix [Fig. 8(b)]. The enhanced interfacial adhesion could also reduce the water penetration to the kenaf fibers, thus increasing the water-resistance of the resulting kenaf-UPE composites.

The covalent linkages between kenaf fibers and the UPE accounted for the improved adhesion between kenaf fibers and the UPE matrix. The improved adhesion enhanced effective stress transfer from the UPE matrix to the fibers, thus increasing the flexural strength and flexural modulus of the (NMA-treated kenaf fiber)-UPE composites. The enhanced adhesion also minimized the gap between kenaf fiber and UPE matrix, resulting in improved water resistance.

The treatment of kenaf fibers with NMA also reduced the amount of hydroxyl groups on fiber surfaces. The covalent linkages between NMA-treated fibers and UPE resin also limit water from penetrating kenaf fibers. The reduced amount of

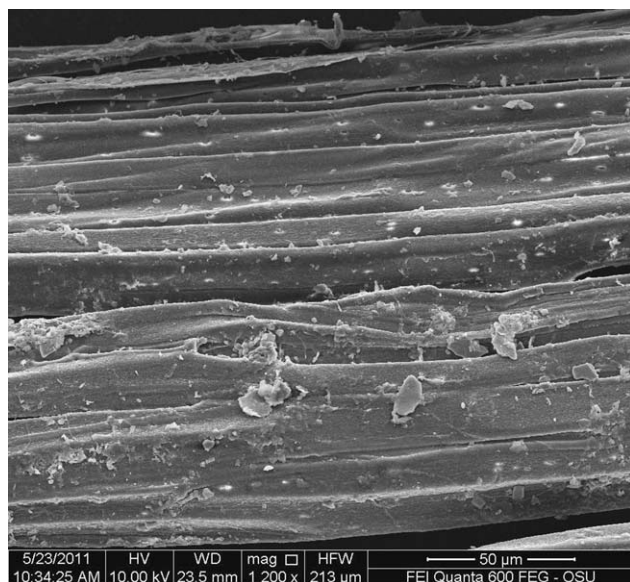


Figure 7 A SEM image of unmodified kenaf fiber surfaces.

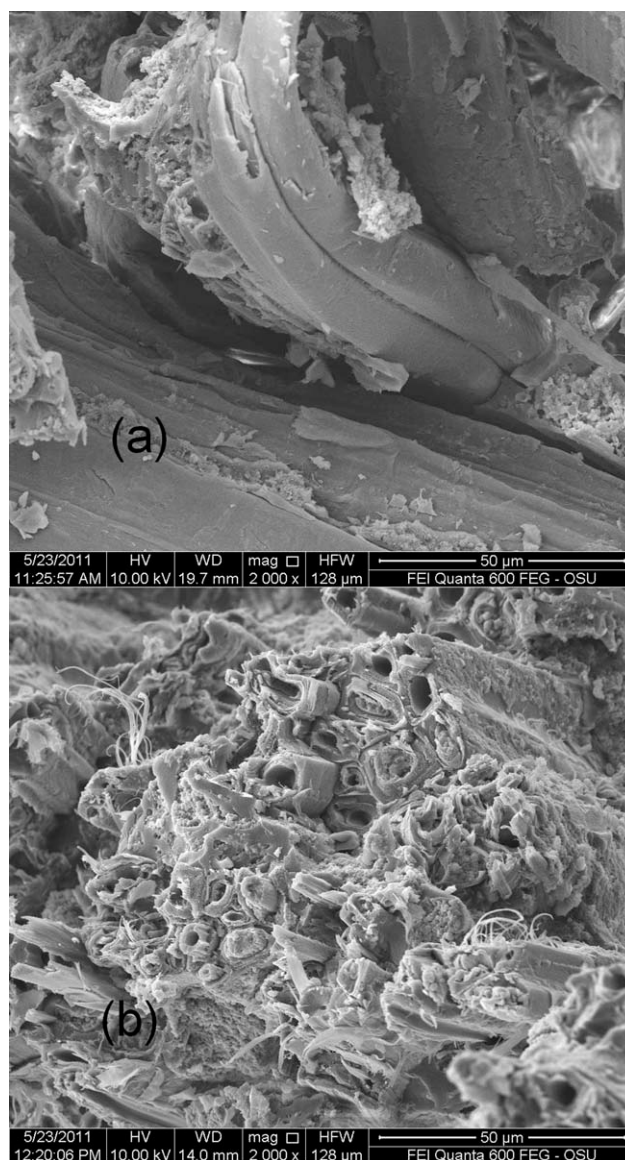


Figure 8 SEM images of flexural-fractured surfaces of kenaf-UPE composites. (a) (Unmodified kenaf)-UPE composites and (b) (NMA-modified kenaf)-UPE composites.

hydroxyl groups and the covalent linkages, as well as the reduced gaps, contributed to the reduced water-uptake rate of the (NMA-treated fiber)-UPE composites.

It is still poorly understood that the composites with TsOH as a catalyst had higher strengths, but also a higher water-uptake rate in water at room temperature than those with sulfuric acid as a catalyst.

CONCLUSION

NMA improved the compatibility and interfacial adhesion between kenaf fibers and UPE resin. Modifications of kenaf fibers with NMA with TsOH as the catalyst significantly increased flexural strength and flexural modulus of the resulting kenaf-UPE

composites, and significantly reduced the water-uptake rate of the composites as well. Modifications of kenaf fibers with NMA with sulfuric acid as a catalyst did not significantly increase flexural strength, but improved flexural modulus and reduced the water-uptake rate of the composites. Under corresponding optimum variables, TsOH as the catalyst resulted in higher flexural strength, flexural strength, as well as higher water-uptake rates of the composites at room temperature than sulfuric acid as the catalyst.

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