

## Research Article

# Kenaf Bast Fibers—Part I: Hermetical Alkali Digestion

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The objective of this study was to develop a hermetical alkali digestion process to obtain single cellulosic fibers from kenaf bast. Kenaf bast were hermetically digested into single fiber using a 5% sodium hydroxide solution for one hour at four different temperatures (80°C, 110°C, 130°C, and 160°C). The hermetical digestion process used in this study produced fibers with high cellulose content (84.2–92.3%) due to the removal of lignin and hemicelluloses. The surface hardness and elastic modulus of the fibers digested at 130°C and 160°C were improved significantly compared with those digested at 80°C. The tensile modulus and tensile strength of the individual fibers reduced as the digestion temperature increased from 110°C to 160°C. Micropores were generated in fiber cell wall when the fibers were digested at 130°C and 160°C. The studies on the composites that were made from polypropylene reinforced with the digested fibers indicated that the compatibility between the digested fibers and polypropylene matrix was poor.

## 1. Introduction

Kenaf (*Hibiscus cannabinus*) an agricultural crop, is in the Malvaceae family, belonging to the division of *Magnoliophyta*. The history of kenaf cultivation can be traced back to ancient Africa [1]. Kenaf grows very quickly, rising to the heights of 12 to 14 feet in 4 to 5 months. In the United States, kenaf is mainly cultivated in Mississippi, Texas, and California, and so forth. Kenaf yields six to eight metric tons of bast and core per acre annually [1].

Kenaf bast fiber is a lignocellulosic fiber that has been used for pulp, paper, and textiles [2]. It is a good potential reinforcement material for polymer composites [3–8]. The lignocellulosic fiber mainly consists of cellulose, hemicelluloses, and lignin. Noncellulosic substances such as lignin, hemicelluloses, and pectins hold the cellulose fibrils together. The reinforcement effect for the lignocellulosic fiber is influenced by its cellulose content. Usually, high cellulose content can be obtained from the chemical retting [2, 9–14]. Retting is a term to describe a process to digest wood and agricultural stalks, especially bast crop stalks, into fibers. Alkali treatment is a standard procedure used in pulp and paper industries to remove lignin [15]. Through the alkali digestion process,

individual fibers can be obtained by separating the fiber bundles. A uniform fiber distribution in the polymer composites may be easily achieved by using the individual fibers as reinforcements. In addition, micropores in the fiber can be created [16], which provide spaces for downstream fiber treatment such as nanoparticle impregnation, and so forth.

In a hermetically alkali digestion process, the temperature is the most influential factor affecting fiber quality and determines the pressure level of the autogeneous vapor. High vapor pressure improves the penetration of digesting agents and accelerates the digestion process. The objective of this study was to investigate the effect of four digestion temperatures on the characteristics of kenaf bast fibers in hermetical alkali digestion process and to evaluate the reinforcement effects of the digested fibers in polymer composites.

## 2. Materials and Methods

**2.1. Materials.** Kenaf stalks were obtained from MSU North Farm. After the separation of the kenaf core and bast, the bast was cut into 50.8 mm lengths and dried to a moisture content of 7.4% at 103°C. Sodium hydroxide (NaOH) solution (5%, w/v) prepared with NaOH beads (Lab grade,

Thermo Fisher Scientific Inc.) and distilled water was used as digestion agent. Glacial acetic acid (17.4 N, Regent grade, Thermo Fisher Scientific Inc.) was used as a pH neutralizer. Polypropylene (PP) film (CO-EX Oriented Polypropylene), provided by Plastic Suppliers, Inc. Dallas, TX, was used to fabricate the laminated kenaf fiber-PP composites.

**2.2. Hermetical Alkali Digestion.** Kenaf bast fiber was digested with 5% NaOH solution (fiber: NaOH solution = 1:30 g/mL) in a hermetical reactor (Parr Instrument Co. 251 M) for one hour at four temperatures (80°C, 110°C, 130°C, and 160°C). The autogenous vapor pressures were 0.05 MPa at 80°C, 0.15 MPa at 110°C, 0.27 MPa at 130°C, and 0.60 MPa at 160°C. After digestion, the pH value of the fiber suspension was adjusted to 7.0 using acetic acid. The neutralized fibers were washed with tap water in order to remove the chemicals. Finally, the digested fibers were freeze dried for further analysis or oven dried for composites fabrication.

**2.3. Digested Fiber/PP Composites Fabrication.** The fibers digested at 80°C, 110°C, and 130°C were long fiber bundles. These fiber bundles were mechanically separated using a blender (Oster 6791) for five minutes. The disintegrated fibers could be dispersed in water and made into uniform fiber sheets. The fibers digested at 160°C had a better dispersion in water and could be made into uniform fiber sheets directly without further mechanical separation. For the fiber sheet forming, the fibers were first dispersed in water by vigorous stirring. The suspension was then passed through a screen (mesh 35), on which the fiber sheets were formed. The fiber sheets were dried at 80°C. The fiber sheets and polypropylene films were cut into 15.2 cm × 15.2 cm and layered in alternate fiber directions. The weight ratio of fiber to polypropylene was 50:50. The laminated mats were hot pressed at a temperature of 200°C and a pressure of 0.7 MPa for 2.5 minutes. The pressure was not released until the platen was cooled down to the room temperature. The laminated kenaf fiber/PP panels were removed from the press and stored in a desiccator for two days before the specimen preparation for mechanical testing. Three panels were fabricated for each digested fiber type.

**2.4. Chemical Components and Yields.** Chemical components, including holocellulose,  $\alpha$ -cellulose, Klason lignin, and ash content were determined for both the raw kenaf bast fibers and the digested fibers. The ash contents were measured following the procedure described in TAPPI standard T 211-om. 93 [17]. The determination of Klason lignin was based on the constituent insoluble in 72% sulfuric acid, which was estimated in accordance with the method #482 from the Institute of Paper Chemistry [18]. Holocellulose is the total polysaccharide fraction (cellulose and hemicelluloses) of the fibers and was estimated according to the method of Wise et al. [19]. Alpha cellulose is the part of cellulose which does not dissolve in 17.5% sodium hydroxide solution. The alpha cellulose was determined in accordance with the method from German Association of Cellulose Chemists and Engineers [20]. The yields of the

digested fibers were obtained based on the ratio of the oven-dry weights of the resultant fibers to the original weight of the raw kenaf bast fibers.

**2.5. Fiber Surface Morphology.** A Zeiss Supra™ 40-Gemini Scanning Electron Microscope (SEM) with an accelerating voltage of 15 kV was used for studying the morphology of the digested fibers. The fibers were treated with 15 nm gold sputter coating before being scanned in SEM. Seventy fibers from each digestion temperature were randomly selected, and the images were taken with the SEM. The dimensions of the fibers were measured by the image analysis. The distributions of their length, diameter, and aspect ratio were statistically analyzed.

**2.6. Fiber Surface Hardness and Elastic Modulus.** A Hysitron TriboIndenter with a Berkovich diamond tip was used to test the surface hardness and elastic modulus at a 400  $\mu$ N peak force and a 40  $\mu$ N/s loading rate using a 10-second segment time. The single fibers were mounted in a hardened epoxy matrix, and the load was perpendicular to the longitudinal direction of the fibers. According to the elastic punch theory and the method of Oliver and Pharr [21], the reduced elastic modulus was obtained based on the elastic contact stiffness.

**2.7. Tensile Properties of Single Fibers.** The single-fiber tests were conducted at the International Center for bamboo and Rattan (ICBR), Beijing, China, using a newly developed microtester (SF-I) [22]. Thirty individual kenaf fibers chemically digested at 110°C and 160°C were tested for tensile modulus and strength. The nominal gauge length was 0.7 mm, and the cross-head speed was 0.8 m/s. A multiple comparison with Fisher's least significance difference (LSD) method at  $\alpha = 0.05$  was carried out with SAS software (SAS Institute Inc. NC, USA).

**2.8. Mechanical Properties of the Composites.** Dog bone-shaped samples were cut from kenaf fiber/PP panels in accordance with ASTM 1037 [23] for the tension testing. The samples were stored in the desiccators with silica gel at the bottom for one week before they were used for mechanical tests. Tensile modulus and tensile strength of kenaf fiber/PP composites were tested using Instron 5869 (50 kN load cell) according to ASTM 1037. Nine replicates of each composite formulation were used. Multiple comparison of the results was conducted with Fisher's Least Square method at  $\alpha = 0.05$  using SAS 9.2 software (SAS Institute Inc. NC, USA). The fracture surfaces of the samples were observed using scanning electron microscopy (SEM, Zeiss Supra™ 40).

### 3. Results and Discussion

**3.1. Fiber Yields and Chemical Compositions.** Table 1 shows the yields and chemical composition of digested fibers and control fibers. It is shown that the fiber yield is gradually decreased as the digestion temperature increased. This result can be attributed to the removal of lignin and low molecular weight compounds as well as solubilization of hemicelluloses in hot alkali solution.

TABLE 1: Yields and chemical compositions of the digested fibers.

Digestion temperature (°C)	Yield (%)	Chemical composition (%)				
		Holocellulose	$\alpha$ -cellulose	Hemicellulose*	Lignin	Ash
(Control fiber)		75.8	46.0	29.8	19.1	5.1
80	56.8	84.1	81.4	2.7	10.5	3.0
110	57.3	85.8	84.0	1.8	10.0	3.2
130	53.2	87.8	86.6	1.2	8.1	2.8
160	44.6	94.2	92.3	0.9	0.24	2.7

\* Hemicellulose content is calculated from the difference between holocellulose and  $\alpha$ -cellulose content.



FIGURE 1: Images of the fibers digested at (a) 80°C, (b) 110°C, (c) 130°C, and (d) 160°C.

TABLE 2: Average and standard deviation of the fiber dimensions.

		Length ( $\mu\text{m}$ )	Diameter ( $\mu\text{m}$ )	Aspect ratio
Digested at 80°C, 110°C, and 130°C with mechanical disintegration	Ave.*	1578.8	20.3	89.0
	Stdev.*	524.8	9.4	41.7
Digested at 160°C	Ave.	468.0	18.2	16.0
	Stdev.	575.5	6.4	15.1

\* Ave.: average of 70 samples. \* Stdev.: standard deviation.

The total amount of holocelluloses is gradually increased as the increase of the digestion temperature. The holocellulose content increased from 75.8% for the control fibers to 94.2% for the digested fibers at 160°C. This increase in the total holocellulose content is probably related to the decrease of the lignin content. The total amount of pure cellulose in

the fibers expressed by  $\alpha$ -cellulose is remarkably increased as the increase of digestion temperature. The amount of hemicelluloses remained in the digested fibers were significantly decreased as the increase of digestion temperature. The above results indicate that the alkali digestion process used in the study solubilized most of hemicelluloses and removed most of lignin from the fibers by increasing the digestion temperature.

**3.2. Morphology.** From the appearance of the fibers, those digested at 80°C, 110°C, and 130°C were fiber bundles, which were bigger and longer compared with those digested at 160°C (Figure 1). The fiber bundles obtained at 80°C, 110°C, and 130°C were separated mechanically and made into fine fibers. Having been mechanically separated, these fibers have the same distribution in length, diameter, and aspect ratio, because they went through the same mechanical disintegration process. The fibers digested at 160°C were

TABLE 3: Surface hardness and elastic modulus of digested fibers.

	80°C	110°C	130°C	160°C
	Surface hardness (MPa)			
Ave.*	64.25	179.50	224.74	287.91
Stdev.*	45.23	124.07	31.88	113.75
LSD test	A	B	B	B
	Elastic modulus (GPa)			
Ave.	2.48	3.19	4.98	5.24
Stdev.	1.15	1.68	0.25	1.59
LSD test	A	A, B	B, C	C

\* Ave.: average of four samples. Stdev.: standard deviation. Means with the different letter are significantly different at  $\alpha = 0.05$ .

TABLE 4: Tensile properties of individual digested fibers.

Temperatures (°C)	Modulus (GPa)	LSD test	Strength (MPa)	LSD test	Elongation (%)	LSD test
110	23.6	A	1051	A	4.5	A
160	13.5	B	810	B	5.6	B

Average of 30 samples. Means with the different letter are significantly different at  $\alpha = 0.05$ .

fine individual fibers and were not subjected to mechanical separation. The distributions of the dimensions are shown in Figure 2. The average and its standard deviation of the dimensions are shown in Table 2.

After the mechanical separation, the lengths of the fibers digested at 80°C, 110°C, and 130°C ranged from 0.5 and 2.6 mm. The lengths of the fibers digested at 160°C were between 0.05 to 0.87 mm, which was much smaller compared to the other fibers. However, the fibers digested at 80°C, 110°C, and 130°C had aspect ratios ranging from 24 to 148, which were higher than those digested at 160°C.

Figure 3 shows the SEM images of the digested fibers. The micropores were seen in Figure 3 in cell wall structures of the fibers digested at 130°C and 160°C. The generated micropores are due to the removal of hemicelluloses and lignin. The size of the micropores was about 50 nm.

**3.3. Surface Hardness and Elastic Modulus.** Table 3 shows the results of surface hardness and elastic modulus of digested fibers tested by nanoindentation.

The higher the digestion temperature, the higher the hardness and elastic modulus were. The elastic modulus of the fibers digested at 160°C was two times higher than those digested at 80°C, while the hardness of the fibers digested at 160°C was four times higher than those digested at 80°C. This might be due to the increase of cellulose content and the decrease of lignin and hemicelluloses. Cellulose has more ordered crystalline structure than lignin and hemicelluloses, while lignin and hemicelluloses are in amorphous arrangement. The crystalline arrangement of the cellulose molecular chain may result in high surface hardness and elastic modulus [24]. However, cellulose also contains amorphous regions. Crystalline regions and amorphous regions alternately exist in a cellulose chain. Therefore, cellulose structure is not homogeneous. Moreover, the micropore

distribution in the digested fibers is not uniform, known from the SEM images. The nonuniform structure of cellulose and the micropore distribution in the fibers resulted in big variations of the indentation results.

**3.4. Tensile Properties of Individual Fibers.** The tensile properties of the individual fibers digested at 110°C and 160°C are shown in Table 4.

Significant differences were observed between the tensile modulus, tensile strength, and elongation of the fibers digested at 110°C and 160°C. The tensile modulus of the fibers digested at 110°C was 10.1 GPa higher than that of the fibers digested at 160°C. The fiber tensile strength reduced from 1,051 MPa to 810 MPa as the digestion temperature increased from 110°C to 160°C. However, the elongation of the fibers digested at 160°C was 1.1% higher than that digested at 110°C. The decrease in the tensile modulus and strength may be due to the removal of lignin and hemicelluloses. In the control kenaf fibers, lignin filled the spaces in the cell wall between cellulose and hemicelluloses. It is covalently linked to the hemicelluloses and thereby cross-linked the different polysaccharides. Thus, lignin conferred mechanical strength to the single fibers as a whole [25]. The removal of lignin and hemicelluloses resulted in a microporosity structure in the fiber cell wall and a loose connection between individual cellulose microfibrils in an individual fiber. Therefore, a lower tensile modulus and strength were obtained.

**3.5. Mechanical Properties of Composites.** The results of tensile modulus and tensile strength of the composites are shown in Table 5. Statistical analysis indicated that there was no significant difference in tensile modulus and tensile strength at the 95% significance level among the four types of composites.

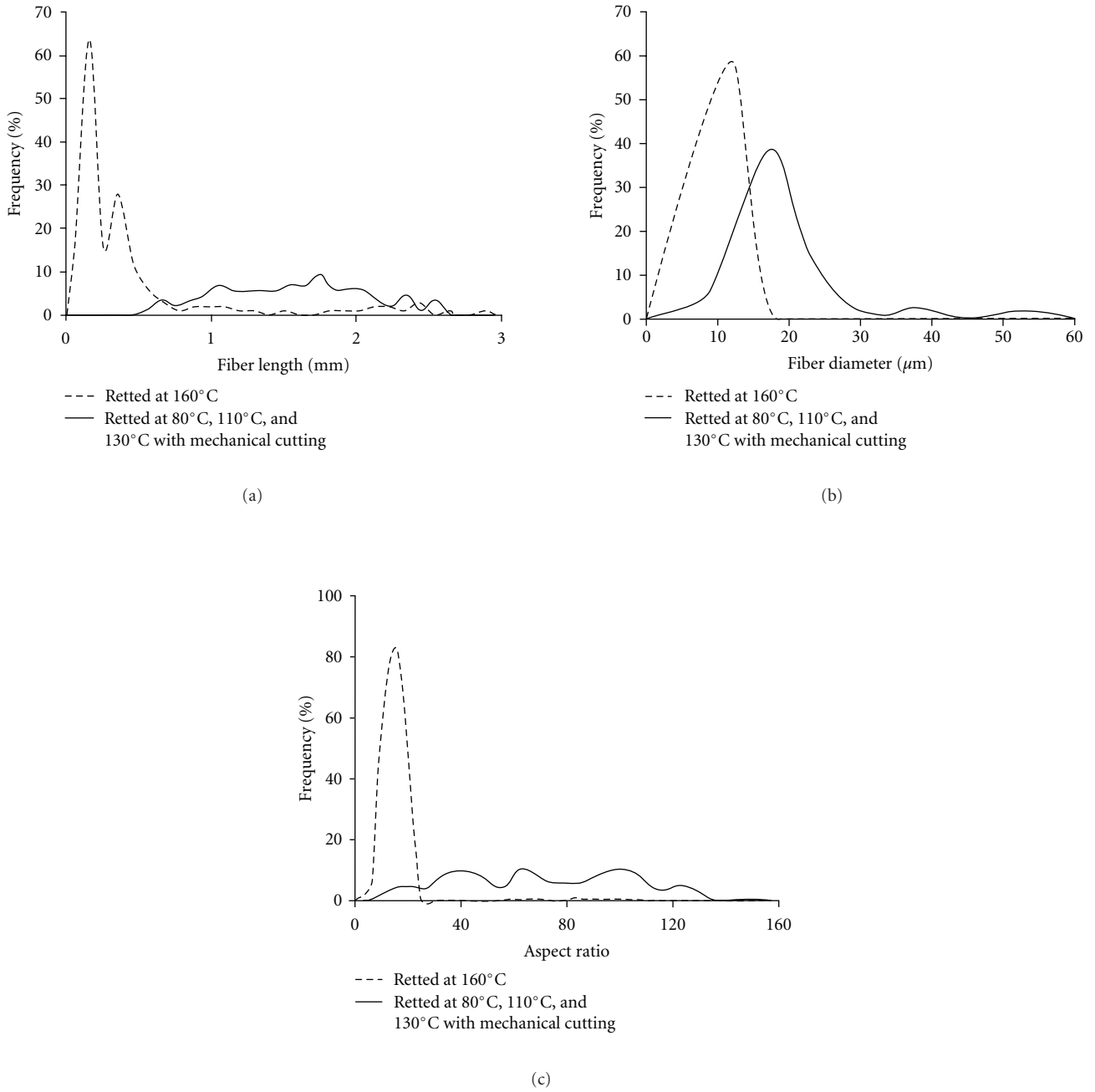


FIGURE 2: Dimensional distribution of the digested fibers. (a) length distribution, (b) diameter distribution, (c) aspect ratio distribution.

TABLE 5: Tensile properties of the kenaf fiber/PP composites.

Digestion temperatures (°C)	Tensile strength (MPa)			Tensile modulus (GPa)		
	Ave.*	Stdev.*	LSD Test	Ave.	Stdev.	LSD Test
80°C	42.44	7.12	A	1.57	0.11	A
110°C	47.35	2.20	A	1.53	0.09	A
130°C	45.20	3.57	A	1.67	0.12	A
160°C	46.77	3.43	A	1.70	0.20	A

\* Ave.: average of four samples. Stdev.: standard deviation. Means with the different letter are significantly different at  $\alpha = 0.05$ . Average of 27 samples. Means with the same letter are not significantly different at  $\alpha = 0.05$ .

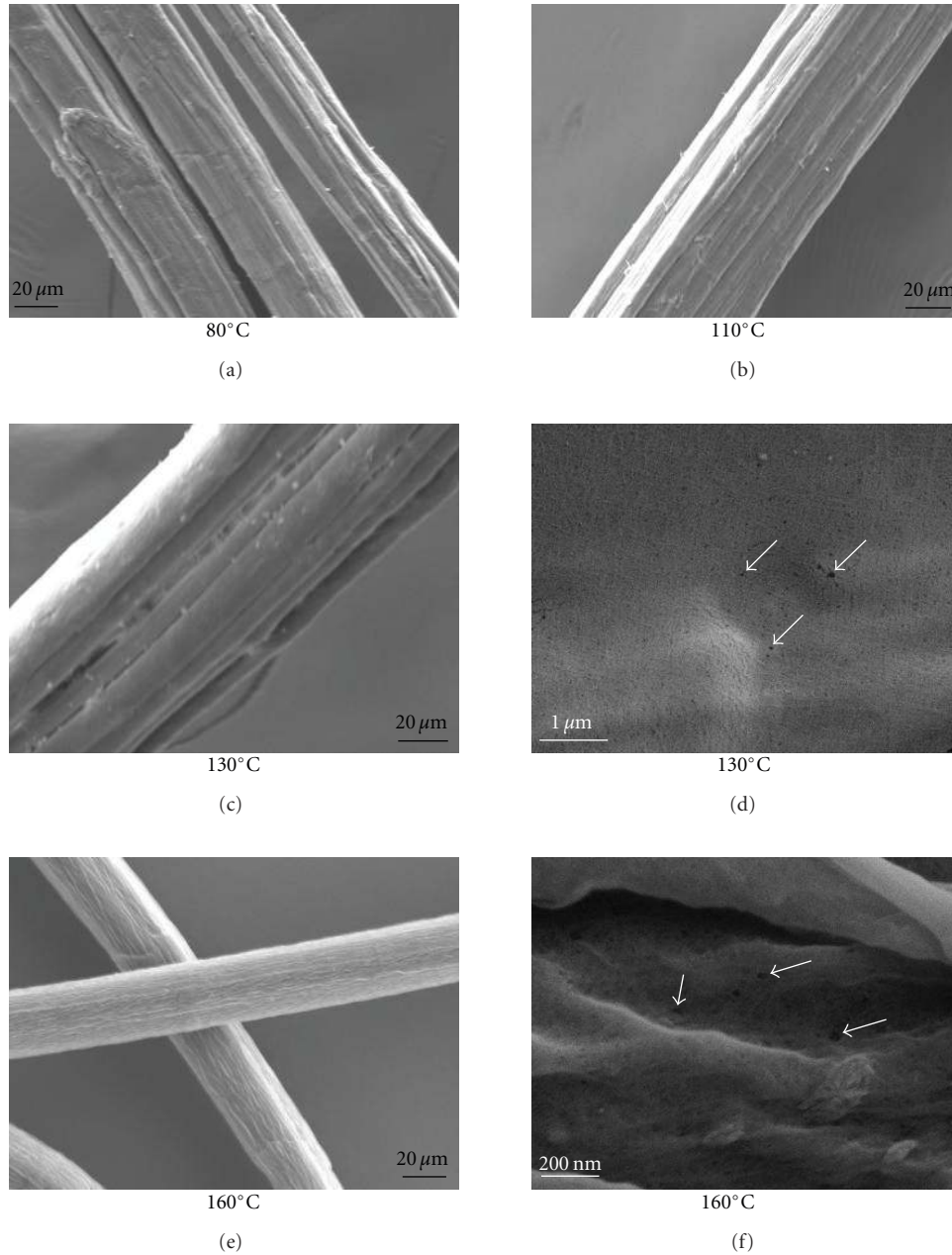


FIGURE 3: SEM images of the digested fibers. (The arrows designate the micropores).

Many factors may influence the reinforcement efficiency of the fibers including aspect ratio, chemical component, and surface characterization. Although the fibers digested at 160°C had a smaller aspect ratio than the other fibers, the resulting composites did not show a lower tensile modulus or tensile strength. This may be due to its higher cellulose content, which compensated the lower aspect ratio. The high cellulose content of the fibers resulted in a high fiber tensile modulus. More severe breakage of the fibers in the composite made from fibers digested at 160°C (Figure 4(a)) indicates a slightly better bondage between the fibers and the PP matrix but also probably resulted in their low strength at 160°C

(Table 4). Generally, the fracture surface images (Figure 4) illuminated that the compatibility between the digested fibers and polypropylene was poor as evidenced by the complete separation between the fibers and polypropylene matrix. Therefore, surface modification for the digested fibers is necessary in order to improve the compatibility between the fibers and the PP matrix

#### 4. Conclusions

Hermetical alkali digestion process effectively removed the lignin and hemicelluloses from kenaf bast fibers at 160°C.

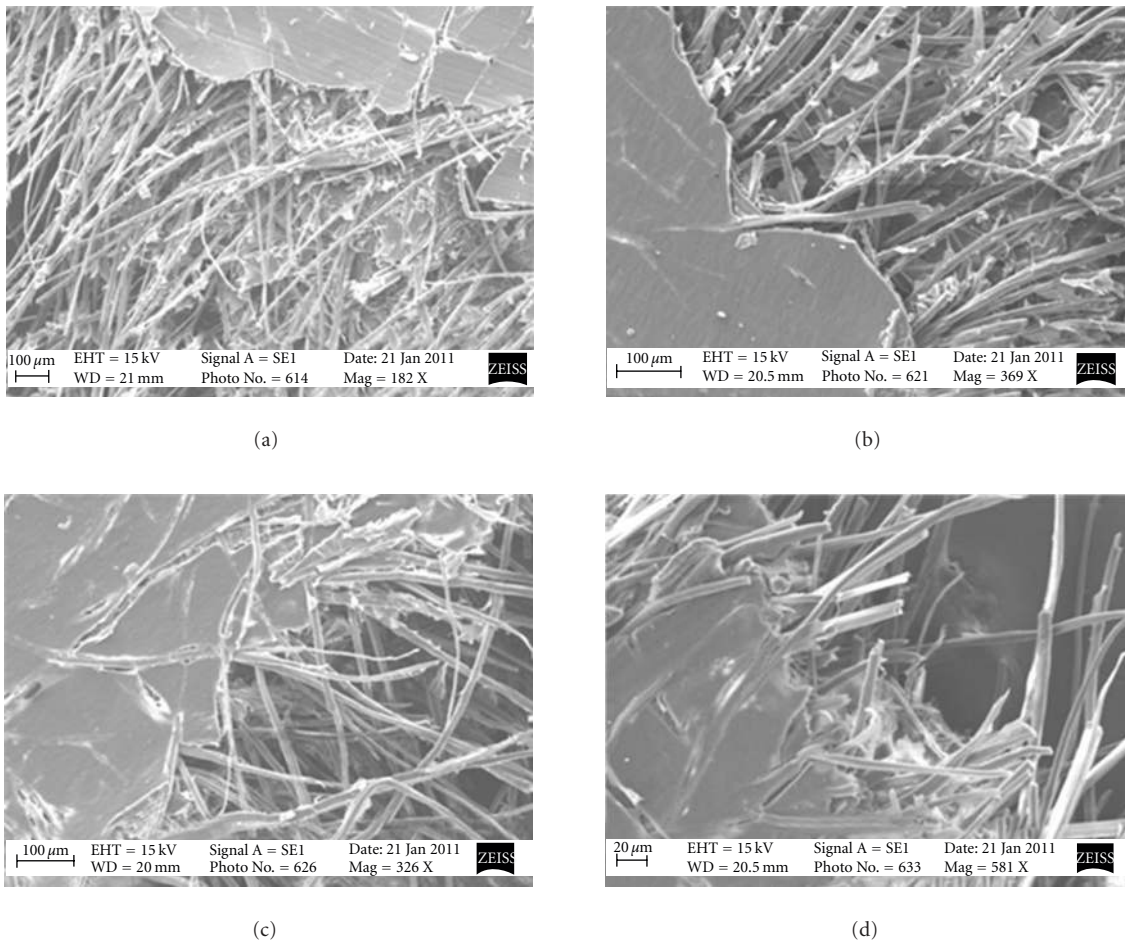


FIGURE 4: SEM images of the fracture surface of the PP composites reinforced with kenaf fibers digested at (a) 80°C, (b) 110°C, (c) 130°C, and (d) 160°C.

The  $\alpha$ -cellulose content of the fibers was 92%. The average surface hardness and elastic modulus of the fiber digested at 160°C yielded improvement of 348.1% and 111.3%, respectively, compared with those digested at 80°C. The increase of cellulose content of the digested fibers resulted in an improved fiber surface hardness and elastic modulus. The digestion temperature had a significant effect on tensile modulus and tensile strength properties of the fiber. When the digestion temperature increased from 110°C to 160°C, the tensile modulus and tensile strength of individual fibers were reduced by 42.8% and 22.9%, respectively, while the elongation increased by 1.1%. The SEM images showed that the micropores were generated in cell wall structures for the fibers digested at 130°C and 160°C, providing the possibility to anchor nanoparticles into the cell wall. The digested fibers without surface modification had a poor interfacial compatibility with the polypropylene matrix.

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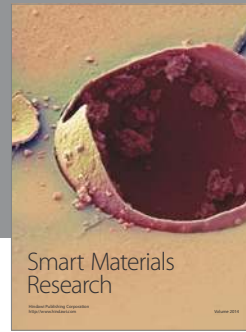
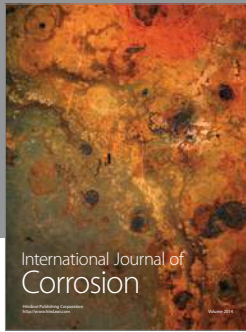
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