

BEHAVIOR OF KENAF FIBERS AFTER IMMERSION IN SEVERAL WATER CONDITIONS

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Behaviors of kenaf fibers after long-term immersion under several environmental water conditions were studied. Water absorption tests were carried out by immersing kenaf fibers in distilled water, sea water (pH 8.4), and acidic solution (pH 3) at room temperature for 140 days. The test results indicated significant differences among the kenaf fibers immersed in different environmental conditions, with kenaf fiber immersed in sea water exhibiting the highest level of water absorption, whereas kenaf fiber immersed in acidic solution showed the lowest water absorption values. The tensile strength of the immersed kenaf fibers decreased with increasing immersion time, implying degradation of the fiber. Investigation on the microstructure of immersed kenaf fibers using SEM reveal the degradation of the kenaf fiber with the development of micro-cracks and increased surface roughness of the fiber.

Keywords: Kenaf fiber; Water absorption; Fiber bundle test

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INTRODUCTION

Nowadays, natural fibers are becoming very widely known and extremely valuable due to their characters. Numerous research studies have been carried out concerning natural fibers based on plant fibers (Herrera-Franco et al. 2004; Rao 2007; de Andrade Silva et al. 2008; Liu et al. 2010). Natural fiber (NF) has good specific strengths and modulus, economical viability, and low density. It poses no risk to human health when the particle is inhaled, and it is biodegradable (Krishan Kumar Chawla 1998; Joseph et al. 2003; Mohd Edeerozey et al 2007). There are varieties of natural plant fibers such as hemp, sisal, banana, coconut, Vakka, bamboo, jute, and including kenaf.

As a result of intensive research, kenaf plantations in Malaysia can be harvested three times a year instead of once, as reported for many locations other than Malaysia. Kenaf is the common name given to fiber from the bast of stems of plants of the genus *Hibiscus*, family *Malvaceae*, especially the species *H.cannabinus L.* Normally, these kenaf plants are grown from seeds, but some are grown as perennials in the tropics and subtropics. Kenaf can grow to 2.5-4 m in height at optimum temperatures between 22 and 30 °C with a minimum moisture requirement of 150 mm in a soil pH between 6.0 and 6.8, as shown in Table 1 (Roger 2007). Kenaf is an annual plant with rapid growth that is considered as the renewable source with good fiber quality (Andrew 1992) and low cost (Natsuno Nishimura et al. 2002). Abdul Khalil et al. (2010) have studied the characteristics and physical properties of kenaf fibers cultivated in the region of Malaysia.

Table 1. Climatic Requirement for Growing Kenaf (Krishan Kumar Chawla 1998)

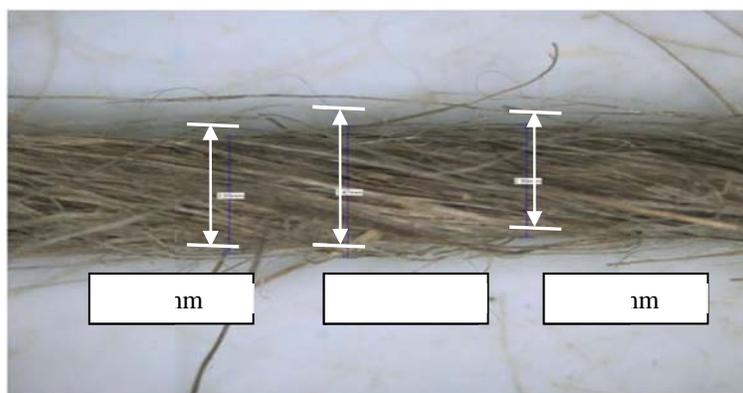
Plant	Optimum temperature (°C)	Minimum moisture (mm)	Optimum Soil (pH)	Growing Cycle (days)	Fiber Yield (kg/ha)
Kenaf	22-30	120	6.0-6.8	150-180	1700

Much of the interest on the part of researchers and engineers has shifted from attempting to use natural fibers in product applications with monolithic materials to fiber-reinforced polymeric material composites (Wambua et al. 2003; Joshi et al. 2004; Reis 2006; Alves 2009). The research is not just focused on mechanical loading but also the degradation behavior after the material is exposed to severe environmental conditions. Most researchers have reported the water absorption behavior of fiber in composite form rather than the fibre alone. Lee and Eiteman (2001) studied the filtration characterization of kenaf core for use in paper production. Steef et al. (2009) studied the water absorption characteristics of kenaf core to use it as animal bedding material. Thus, the present work provides results on the effect of several water conditions on the tensile properties of kenaf fiber. Our aim was to establish changes into fiber prior to immersion, which later will be used as effective reinforcement fibers for making composites. Various conditions of exposure were considered to provide differences in terms of the nature of the real environments. SEM was used to provide microstructural information pertaining to the physical effects of the environments on the kenaf fibers.

EXPERIMENTAL

Material

Kenaf fibers were obtained from herbaceous annual plant extracted from stem provided by Malaysian Tobacco Board (LTN) Malaysia in twisted yarn form. The size of the fiber bundle ranged from 3 to 3.5 mm. Figure 1 shows the picture of the kenaf fiber bundle used in this study.

**Fig. 1.** Kenaf fiber supplied by LTN

Water Absorption Test

A bundle of kenaf fibers was immersed into distilled water (pH 7), sea water (pH 8.4), or acidic solution (pH 3). The fibers were kept in a covered bottle for 140 days at room temperature.

The measurements of weight were done by removing the kenaf fibers from the water, wiping them dry, and then weighing them using an electron balance (accurate to 10^{-4}). The moisture content was calculated using Equation 1 by weighing a fixed amount of dry fiber before immersion (w_o) and after immersion (w_t).

$$Mt (\%) = \frac{w_t - w_o}{w_o} \times 100 \quad (1)$$

Chemical Analysis of Kenaf Fibers

The chemical properties of the kenaf fiber bundles before and after immersion in distilled water, seawater, and acidic solution were evaluated by the Forest Research Institute Malaysia (FRIM) for both immersed and standard (without immersed) kenaf fibers for comparison purposes. Five measures of the chemical composition were evaluated, which were the holocellulose and alpha-cellulose content, lignin content, pentosans, and moisture content.

FTIR Spectroscopy

Fourier transform infra-red (FTIR) analysis was performed on both immersed and standard kenaf fiber bundles in the range of 4000 to 400 cm^{-1} within 64 scans using an FTIR machine Model Perkin-Elmer system 2000. Kenaf fibers (5 mg) together with potassium bromide, KBr (95 mg) were compacted into thin pellets with a hydraulic (8 tons) force and pressure (400 psi) for about 2 minutes. Then the spectra scanned on the pellets were recorded using OMNIC software.

Tensile Fiber Test

Uniaxial tension properties of kenaf fiber were evaluated according to ASTM Standard C 1557. Fiber bundle tensile tests were performed using an Instron machine 3366 with a crosshead speed of 5 mm/minute. Generally, the kenaf fiber samples were glued between two rectangular cardboard pieces with a dimension of (30 x 70) mm. The cardboard contained a circular hole right at the centre with a diameter of 20 mm. A detailed illustration of the fiber bundle tensile test configuration is shown in Fig. 2. At least 15 samples of each kenaf fiber bundle were tested for each condition.

Microstructural Analysis

The microstructure of kenaf fiber immersed in distilled water, seawater, and acidic solution were then compared and analyzed. Differences in microstructure can be expected to provide one of the key explanations to the differences in terms of water absorption behavior.

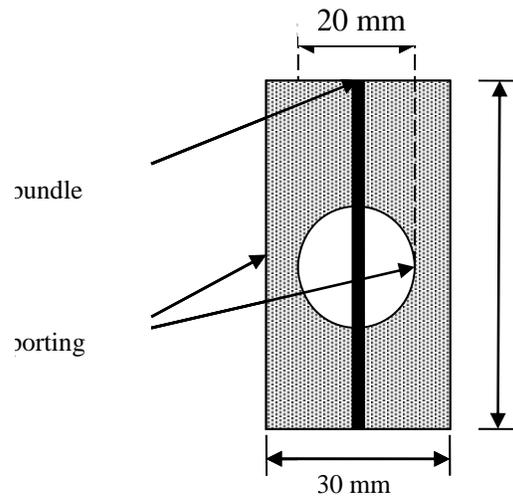


Fig. 2. Illustration of cardboard which was used for fiber bundle tensile tests

RESULTS AND DISCUSSION

Sorption Behavior of Kenaf Fiber

The experimental results for moisture content as a function of the square root of time for each immersed specimen in three different solutions for a period of 140 days are shown in Fig. 3. The relationship of the moisture content with the square root of time appeared to obey non-Fickian's behavior. The water absorption trend for all specimens was linear in the beginning, and then slowly decreased after prolonged time.

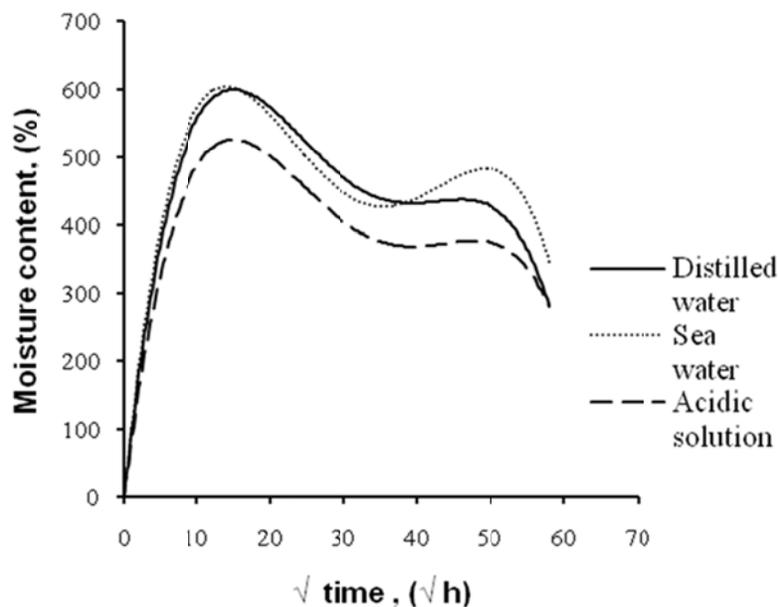


Fig. 3. Water absorption curves in three conditions for kenaf fiber bundles

So far, not many papers published previously have dealt with non-Fickian's behavior. As reported earlier, Daly et al. (2007) observed the same water absorption trend as non-Fickian. This phenomenon can be explained by considering the water absorption characteristics of kenaf fiber. The kenaf fiber swells when it is exposed to moisture. Pursuant to fiber swelling, micro-cracking occurred and wrecked the fiber. The hydrophilic characteristic of the kenaf fiber further contributed to more water penetrating into the micro cracks, creating swelling stresses, and leading to failure.

To obtain a better understanding, a comparison of the pH for each immersed specimen can be considered. It can be concluded that kenaf fibers showed greater absorption at higher pH: seawater (pH 8.4) > distilled water (pH 7) > acidic solution (pH 3). This could be explained in terms of a change in chemical compositions of the fiber after the immersion period between the three kinds of water solutions studied. The chemical composition of kenaf fibers (before and after immersion) was analyzed after an immersion period of 140 days. Table 2 presents the description of chemical compositions of the fiber before and after immersion in various solutions. There are several explanations that could be deduced from these differences. The differences were found to be important, especially with respect to cellulose and lignin content. Theoretically, the fibers with higher lignin content would be expected to exhibit the lowest values of water absorption, due to the fact that lignin is hydrophobic, thus providing resistance against the hydro-degradation of the kenaf fibers. Therefore, standard kenaf fiber had the highest content of lignin in comparison to fiber that being immersed in acidic solution, distilled water, and seawater according to regulation, which are listed from lowest to highest moisture absorption. If we compare the immersion, the kenaf fiber immersed in seawater had been treated, and it eliminated the lignin content. After the lignin was eliminated, the water molecules were absorbed in through the cellulose fiber. Therefore, it is widely accepted that lignin is hydrophobic in nature and cellulose is hydrophilic upon immersion.

Table 2. Properties of Kenaf Bundles

Description (%)	Test Method	F	FDW	FSW	FAS
Moisture content	T 264 om-88 Preparation of wood for chemical analysis	8.92	15.4	16.50	10.60
Holocellulose content	In-house method	87.40	89.90	80.50	89.70
Alpha-cellulose	T 203 os-74 Alpha-, Beta-, and Gamma-Cellulose in Pulp.	68.13	74.90	61.70	80.80
Lignin content	T 222 om-02 Acid-Insoluble Lignin in wood and pulp	52.77	6.71	6.47	7.33
Pentosan	In-house method	28.90	7.21	7.53	7.61

(F= kenaf fiber without immersed; FDW=kenaf fiber immersed in distilled water; FSW=kenaf fiber immersed in seawater; FAS=kenaf fiber immersed in acidic solution).

As the water penetrates into kenaf fiber, degradation of the cellulose becomes active. This degradation is caused by the hydrolysis mechanism along the fiber. The hydrolysis of cellulose is expected to be more pronounced in seawater (alkaline) and

acidic solution as compared to distilled water. The proposed hydrolysis mechanisms of cellulose such as kenaf fiber in seawater (alkaline) and acidic solution are depicted in Figs. 4 and 5, respectively. In seawater (pH 8.4), the electrostatic interaction was more electronegative and the moisture penetrates completely, swelling the kenaf fibers with high concentration positive charge. The increase in the degree of swelling in seawater could be attributed to the interaction between COO^- of the kenaf fiber cellulose chain and OH^- groups. A large concentration of OH^- ions absorbed into the kenaf fibers causes the formation of swelling stresses and leads to fiber failure. However, the decreasing of water absorbed in the acidic solution indicates the high content of lignin of the kenaf fiber and the hydrogen bonds in affecting the degradability of the kenaf fibers. The proposed mechanism of the acidic solution in Fig. 5 shows the H^+ in the reaction was acting as the catalyst and minimally affects the medium. Therefore, in this study the swelling of the kenaf fibers was greater in the seawater rather than in the acidic solution.

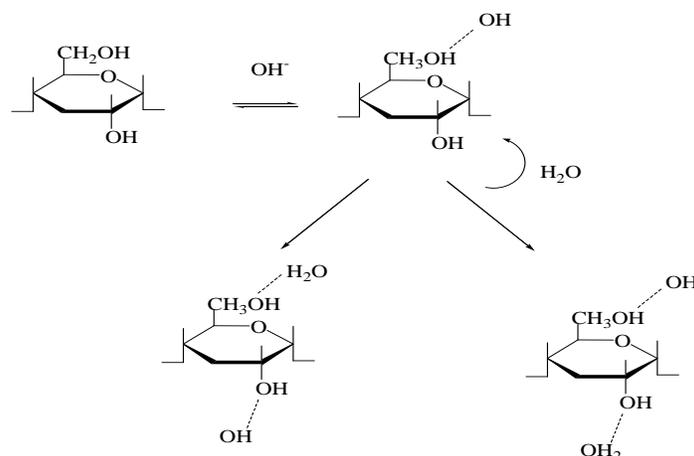


Fig. 4. Hydrolysis of the cellulose linkage in alkaline medium

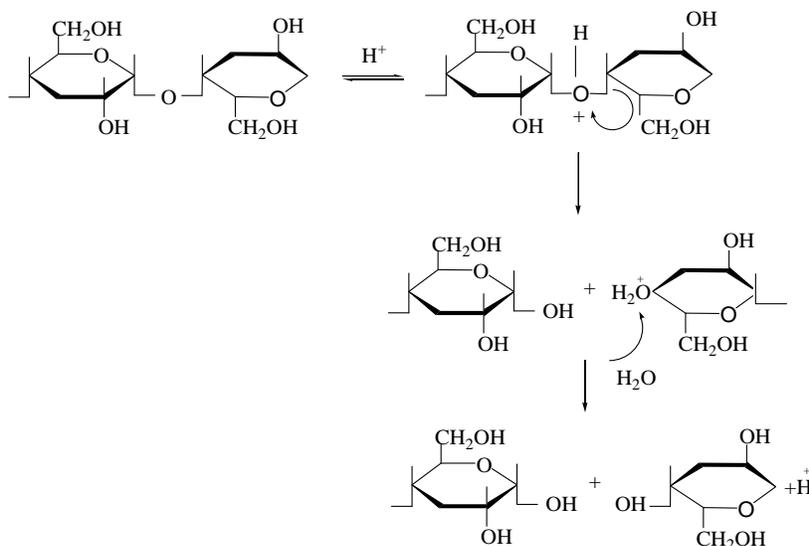


Fig. 5. Hydrolysis of the cellulose linkage in acidic medium

FT-IR Spectra

FTIR was used to study the chemical changes of kenaf fiber subjected to various immersion solutions. FTIR spectra of kenaf fiber immersed in various solutions after 140 days period of immersion are summarized in Fig. 6. It is observed that the treatments affected the compositions of kenaf fiber. The trend shows the changes, which were almost the same for kenaf fibers before and after immersion. The difference was only in terms of the value of absorbance; the more water absorbed into kenaf fiber, the more higher will be the value of transmittance. These results were recorded in a range of 4000 to 400 cm^{-1} . Noticeable difference was observed particularly within the range 3200 to 3400 cm^{-1} , which corresponds to hydrogen bonding. The maximum absorption band $-\text{OH}$ group stretching of fiber SW is more than other immersions kenaf fiber samples for fiber DW and fiber AS. A broad absorption band at 3394 cm^{-1} confirms the stretching of the $-\text{OH}$ group, and the small band at 2990-2850 cm^{-1} corresponds to the C-H stretching frequency. The band peak at 1648 cm^{-1} in the region 1648-1632 cm^{-1} is the mode of the absorbed water. The small peak at 1434-1421 cm^{-1} in all samples relates to CH_2 symmetric bending, while a peak in region of 1375-1368 cm^{-1} shows the O-H bending. The absorbance at 1260 cm^{-1} showed the $-\text{C}-\text{O}-\text{C}-$ stretching, and 1249-1229 cm^{-1} is due to OH in a plane bending cellulose. The band at 1055 cm^{-1} in the range at 1065-1015 cm^{-1} is due to the stretching of $-\text{CH}_2-\text{O}-\text{H}$.

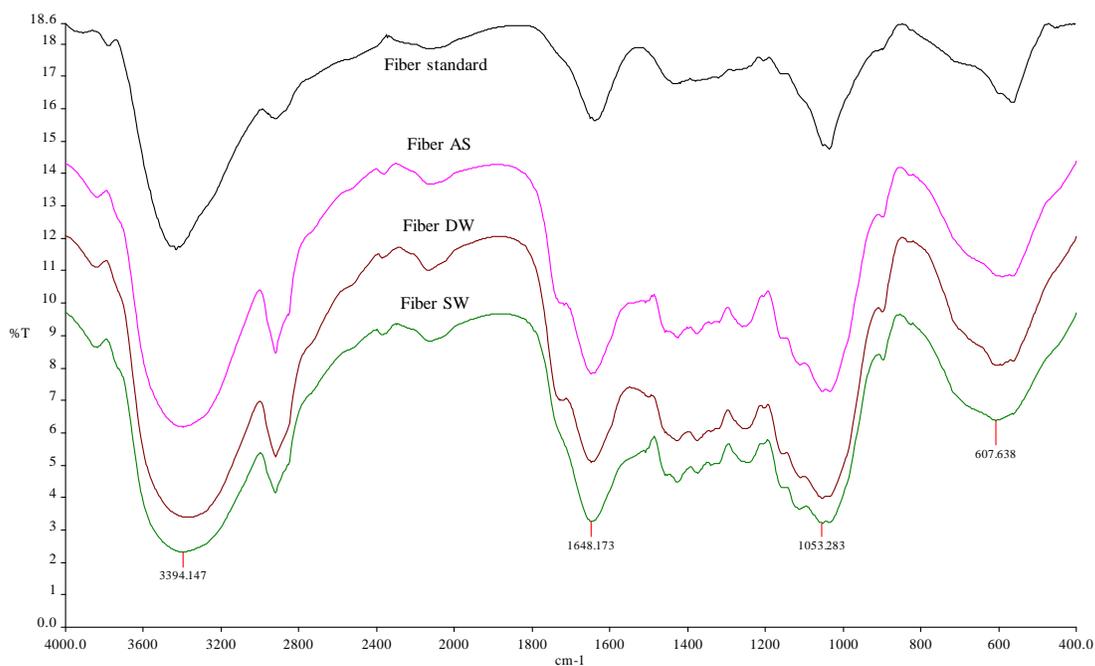


Fig. 6. FT-IR spectra of different conditions of kenaf fibers cellulose (Fiber standard= kenaf fiber without immersed; Fiber DW=kenaf fiber immersed in distilled water; Fiber SW=kenaf fiber immersed in seawater; Fiber AS=kenaf fiber immersed in acidic solution).

Tensile Test of Kenaf Bundles

A detailed investigation was carried out to determine the effects of water treatment on the mechanical characteristics of kenaf fiber. Ideally, single fibers should be considered. Significant changes in strength were observed for each of the kenaf fibers

after water treatment, as shown in Fig. 7. The figure shows typical uniaxial tensile stress-strain curves of specimens for standard and immersed in distilled water, sea water, and acidic solution at room temperature. Fibers immersed in acidic solution displayed brittle failure within the maximum applied stress, but other samples that had been immersed in distilled water or sea water were able to resist breakage and continued elongation after the maximum stress. The tensile strength, failure strain, and elastic modulus of kenaf fiber bundles are summarized in Table 3. The accelerated growth of cracking, involving the formation of voids and porosity, would allow moisture to penetrate into fibers, which can accelerate the failure of the structure. However, after immersion in the acidic solution the strength and modulus values were less than those immersed in distilled water and sea water. This may be caused by some unexpected residual stresses, which induced earlier failure at the method when grip the kenaf fiber before test and including the variation or inconsistencies in the length and diameter of kenaf fibers. Moreover, the cations and anions in acidic solution would penetrate along with the water molecules into the fibers and causing rotting to take place more easily. This finding is in agreement with previously research work by Huang (2009).

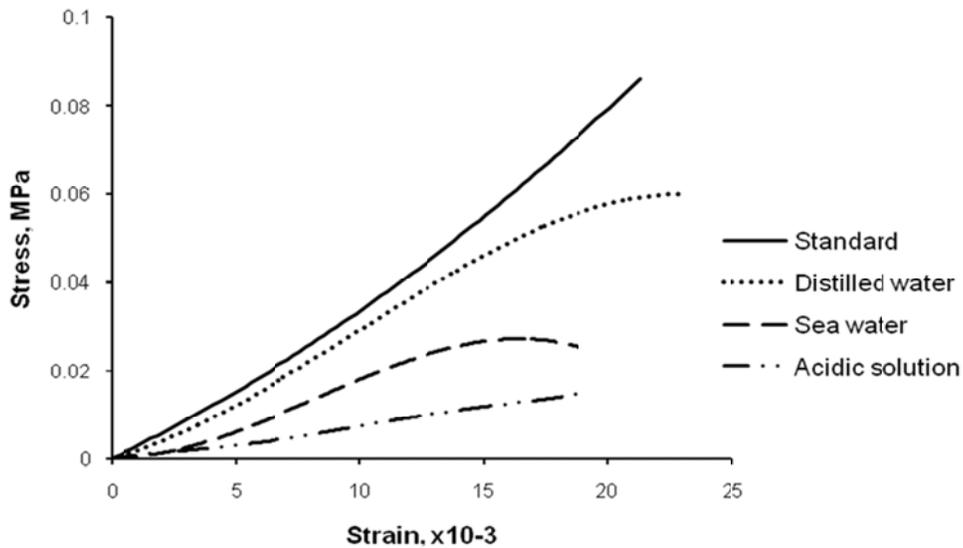


Fig. 7. Stress-strain curves of kenaf fiber bundles under different conditions

Table 3. Statistical Tensile Properties of Kenaf Fiber Bundles Before and After Immersion under Different Environment; Distilled Water, Sea Water, and Acidic Solutions for 140 Days

Kenaf fibers bundles	Tensile strength (MPa)		Failure strain (%)		Young's modulus (MPa)	
	Mean	Std. Dev.	Mean	Std. Dev.	Mean	Std. Dev.
Standard	118.30	25.54	8.31	1.22	2416.50	460.14
Distilled water	5.87	2.42	2.45	1.50	372.43	223.51
Seawater	3.41	1.35	1.68	0.37	291.76	105.55
Acidic solution	1.82	0.38	2.40	0.52	145.96	48.98

(Standard= kenaf fiber not immersed; Distilled water=the fiber immersed in distilled water; Seawater=the fiber immersed in seawater; Acidic solution=the fiber immersed in acidic solution).

Microstructural Analysis

The SEM-micrographs display four surfaces of kenaf fibers in different conditions to illustrate the possible failure modes. All micrographs in this work were taken with 1.00k times (1.00k x) magnification. In Fig. 8a the fiber without immersion (standard) shows a neatly smooth surface. After 140 days of immersion in distilled water, seawater, or acidic solution, the kenaf fiber's surface had been seriously roughened and damaged, as shown in Fig. 8. One might see a gauzy covering on the fiber surface after immersion in seawater or acidic solution. The roughness surface of all immersed samples is attributable to the swelling and permeation, due to the hydroxyl groups (-OH) forming hydrogen bonds with cellulose fibers. The findings of Aziz (2004) and Sombastsompop (2004) support this statement.

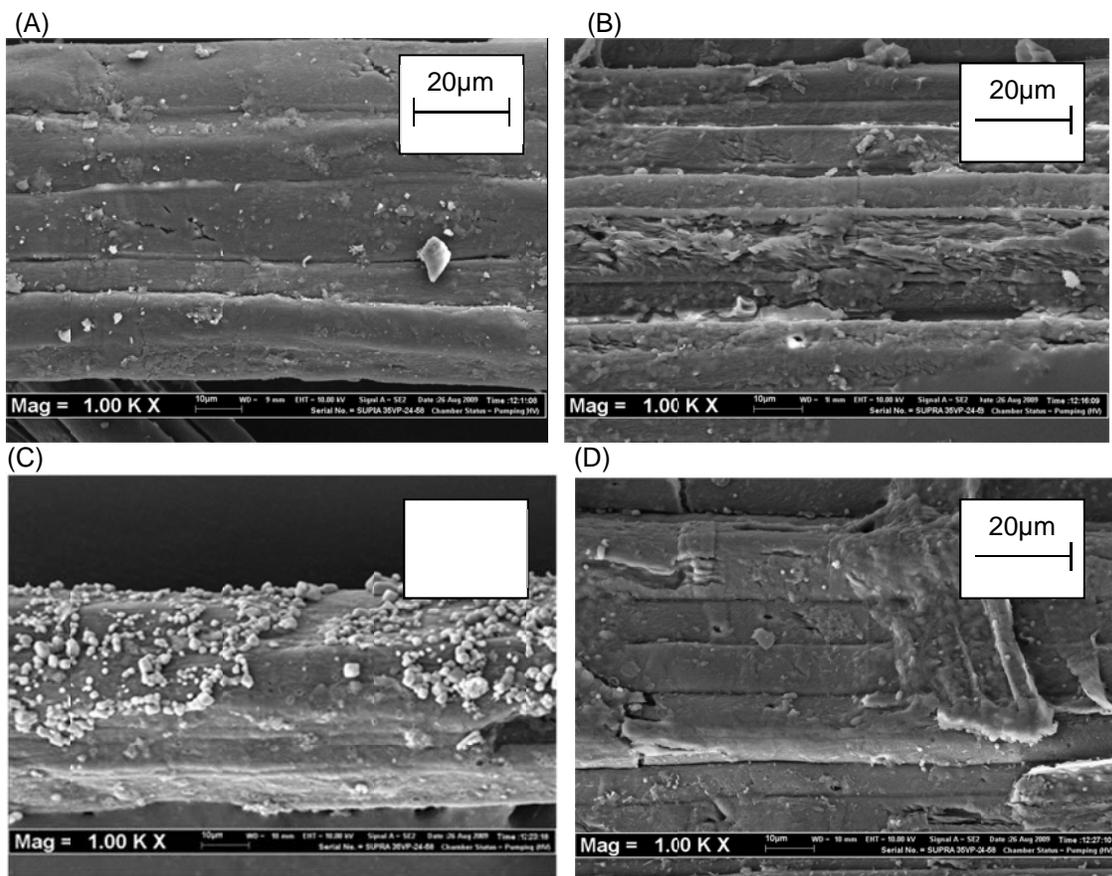


Fig. 8. SEM-micrographs of kenaf fiber bundles surface: (A) standard; (B) immersed in distilled water; (C) immersed in sea water; and (D) immersed in acidic solution

CONCLUSIONS

The behavior of kenaf fibers after exposure to several environmental conditions has been studied following immersion in distilled water, seawater, or acidic solution at room temperature. Results showed that the water absorption pattern of the kenaf fibers is

non-Fickian's, where the moisture uptake behavior is radically altered due to moisture-induced degradation. The kenaf fibers immersed in seawater showed the highest absorption characteristics in comparison to distilled water and acidic solution, respectively. A significant correlation could be found between the chemical composition and the water retention. These indicate that the kenaf fiber absorbed more water when the lignin content was lower. The authors also were concerned about the strength of the kenaf fibers, which might be weakened after being immersed. Among the three conditions under which the kenaf fibers immersed, the strength and modulus of the kenaf immersed in acidic solution were the lowest. The strength was low due to the form of damage in kenaf fibers caused by the ions and chemical degradation. Scanning electron microscopy (SEM) confirmed that the kenaf fiber immersed under those three different environment conditions suffered mechanical damage.

ACKNOWLEDGEMENTS

The authors wish to thank Malaysian Tobacco Board (LTN) and Universiti Sains Malaysia (USM) (grant no. 1001/PBAHAN/8032018) for their raw material supply, technical, and financial assistance that has resulted in this article.

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Article submitted: July 23, 2010; Peer review completed: August 21, 2010; Revised version received: February 7, 2011; Accepted: February 12, 2011; Published: February 15, 2011.