

# Supercritical Carbon Dioxide Treated Kenaf Bast Pulp Fiber Reinforcement in Epoxy Composite

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**ABSTRACT:** Due to environmental concerns, green composites have become a highly researched material. In the present study, kenaf fiber was used as reinforcement in epoxy-based composite with weight fraction ranges from 0, 5, 10, and 15% (w/w of resin). The ratio of epoxy to hardener was 65:32.5. Prior to incorporation, kenaf bast fiber underwent Soda-AQ pulping followed by total chlorine-free bleaching (OAZP sequence). The obtained pulp was then subjected to supercritical carbon dioxide extraction (SCE) treatment. It was observed that epoxy composite with 10% of fiber loading demonstrated the highest mechanical properties with a tensile strength of 64 MPa, tensile modulus of 1.64 GPa, flexural strength of 83 MPa, and flexural modulus of elasticity of 2.94 GPa. The observed improvement was due to the SCE process which enhanced the wetting process of fiber. However, at 15% mechanical properties decreased due to void formation since fiber could not be dispersed properly in epoxy matrix at high concentration.

**KEYWORDS:** Kenaf fiber, supercritical carbon dioxide extraction, epoxy composite, water absorption, mechanical properties

## 1 INTRODUCTION

Environmental concerns recently have led to the increasing demand for green composites in order to achieve sustainable industrial development. Therefore, it is necessary for researchers to explore green materials and technology to replace their conventional counterparts. Kenaf is an industrial crop which is highly suitable for green composite because of its high yield, low density, and mechanical strength comparable to synthetic fiber [1]. Owing to its properties, kenaf bast is commonly utilized as a reinforcing fiber in composites to enhance their mechanical characteristics. One of the most frequently used methods for extraction of fiber from kenaf is soda pulping. The soda-anthraquinone (AQ) process requires less bleaching chemical for non-wood fiber such as kenaf bast fiber than wood. Furthermore, recovery of chemicals required in soda-AQ pulping also minimizes its harmful effect on the

environment. For removal of lignin from pulp, totally chlorine-free (TCF) bleaching needs fewer chemicals than soxhlet extraction [2]. The conventional lignin extraction procedure involves solvent that has highly volatile organic compounds and such chemicals will emit harmful effluent into the environment. However, TCF bleaching involves a sequence of bleaching known as OAZP. Where O stands for oxygen bleaching followed by acidic treatment that is annotated with the letter A and ozone bleaching annotated with Z; and the final stage is hydrogen peroxide bleaching denoted with the letter P. By controlling the bleaching conditions, cellulose degradation can be avoided and this gives a higher yield.

Supercritical fluid extraction (SFE) process is another alternative to replace the conventional method in order to minimize the use of harmful chemicals. Besides the extraction of lignin, SFE is also used for polymer synthesis, extraction of metal, and phase separation of polymer blend [3]. By optimizing the conditions of SFE researchers can predict and control reactions which are difficult to achieve conventionally, thus SFE can be used for the

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production and separation of nanofiber from lignocellulose [4]. Supercritical carbon dioxide is an environmentally friendly solvent that has the potential to be used in nanofiber production. The solubility of many extracted compounds in CO<sub>2</sub> varies with pressure and temperature, thus permitting selective extractions [4]. Hence, it is of interest to study the effect of supercritical CO<sub>2</sub> on pulp fiber properties, including morphology, chemical structure, and thermal properties. Fiber-reinforced composite materials are rapidly grabbing attention for various industrial applications due to their low cost and biodegradability. Thermoset resins are widely used as a matrix for fiber-reinforced composites because of their resistance against chemical corrosion and high thermal stability. Pure epoxy resin is brittle and sensitive to temperature and moisture [5]; however, epoxy-based reinforced composites have excellent mechanical properties [6]. Composite materials have a few drawbacks such as sensitivity against humidity, aging, and poor interfacial adhesion [7]. Furthermore, noncompatibility between nonpolar matrix, such as epoxy resin, and highly polar kenaf fiber restrict the resin from wetting the fiber surface, thus resulting in poor interfacial adhesion [8]. Concentration and temperature of supercritical CO<sub>2</sub> may change the morphology and particle size of fiber, thus altering the interfacial adhesion between fiber and matrix [9].

To the best of our knowledge, no studies have been done on extraction of kenaf fiber using supercritical CO<sub>2</sub> and subsequent reinforcement of extracted fiber in epoxy resin to enhance composite characteristics. The main objective of the present study is to investigate the effect of supercritical CO<sub>2</sub> extraction and bleaching on fiber properties. Furthermore, the effect of pulp fiber reinforcement in epoxy composite on the physical and morphological properties was also investigated.

## 2 EXPERIMENTAL

### 2.1 Material

Kenaf bast fiber was collected from Nibong Tebal Paper Mills, Seberang Perai, Pulau Pinang, Malaysia. Bast fiber was separated from its inner core by using a Sprout Bauer refiner (USA). Supercritical CO<sub>2</sub> was obtained from ZARM Scientific and Supplies (Malaysia). Epoxy used in this study was bisphenol A diglycidyl ether (BADGE) with an epoxide equivalent weight of 182 to 192 units. The curing agent was clear epoxy hardener 8161 (isophorone diamine [IPD]) with an amine value of 260 to 284 (mg KOH gm<sup>-1</sup>). Epoxy resin and commercial curing agent were obtained from ZARM Scientific and Supplies (Malaysia).

### 2.2 Pulping

First, 3600 g of oven dried kenaf fiber was pretreated in 25% weight equivalent NaOH and 0.2% (w/v) anthraquinone (AQ) solution having fiber-to-liquid ratio of 10:1 for 1 hour. The pretreated fiber was then cooked in the digester at 165 °C for 120 minutes. Cooked pulp was remixed in a hydra-pulper for 10 min and then the pulp slurry was washed with water. Later pulp was added to the disintegrator for 30000 revolutions at a pulp consistency of 2% before screening with 0.15 mm slits on a flat-plate Sommerville screener.

### 2.3 Bleaching

Oxygen bleaching was done in a 650 mL stainless steel autoclave equipped with oxygen gas inlet and a stirrer (Parr Instrument Company, USA). First, 60 g of dried pulp was mixed with 1% MgSO<sub>4</sub>·7H<sub>2</sub>O and 1.5% NaOH; then distilled water was added until 10% consistency was reached. The obtained mixture was closed in an autoclave and the oxygen inlet was opened. Pressure and temperature inside the autoclave were kept constant at 0.55 MPa and 95 °C, respectively, for 30 min. The obtained pulp was washed and spin-dried. Later, acidic pretreatment of pulp by sulphuric acid was carried out at pH 2 in a plastic bag at 30 °C for 120 min. For ozone bleaching, a MarkV high intensity mixer (Quantum Technologies Inc., USA) was used. A 0.5% concentration of ozone was added to 20 g of dried pulp and the reaction temperature was maintained at 20 °C for 2.5 min. Later, the consistency of pulp slurry was adjusted to 15% by using 0.05N aq. NaOH. The pulp mixture was then inserted into a heat resistant plastic bag and a corresponding amount of 3% H<sub>2</sub>O<sub>2</sub> and 0.3% NaSiO<sub>3</sub> was added and temperature was set at 70 °C for 60 min. Finally, bleached pulp underwent SCE process by using Aiston pump p300 LGPS 0 Separex supercritical fluid technology (France). Temperature was set at 80 °C, pressure was 500 bars at inlet, and extraction time was 2 hours.

### 2.4 Fiber Analysis

Fiber analysis was carried out using a Sherwood Instruments fiber analyzer with Bauer McNett classifier according to Tappi T-233 standard. One gram of pulp was immersed in water for 24 hours and then dispersed in distilled water by using a disintegrator to ensure that pulp were softened and well dispersed before the fiber test. Average fiber length, length weighted average and fine content percentage analysis were determined.

## 2.5 Composite Preparation and Analysis

Composites were prepared using epoxy matrix and varying fiber weight fraction of 0%, 5%, 10%, and 15% (w/w of resin). Fiber was mixed with 65 g of BADGE at 50 rpm for 60 min; later, the mixture was vacuumed for 60 min for removal of bubbles. Then, 32.5 g of hardener was added to the sample and slowly mixed for 12 min to avoid premature gelatinization, followed by vacuumization for 30 min. The obtained solution was poured evenly into a mold having a dimension of 148 mm × 118 mm × 3 mm and placed in an oven at 85 °C for 120 min. Later, the oven temperature was raised to 125 °C for 3 h for curing of composites. All samples were cut using a Hitachi CB75F electrical band saw. Composite of dimension 20 mm × 20 mm × 3 mm was cut for density and water absorption testing. Density of composites was measured according to ASTM D792 standard. Each sample was analyzed five times using Equation 1:

$$\text{Density (g/cm}^3\text{)} = \frac{\text{Mass of Composite (g)}}{\text{Volume of the Composites (cm}^3\text{)}} \quad (1)$$

Water absorption was calculated as per ASTM D5229 standard. Percentage of water absorption equilibrium was calculated using Equation 2:

$$\text{Water Absorption (\%)} = \frac{w_n - w_d}{w_d} \times 100\% \quad (2)$$

where  $W_n$  is the weight of composite sample after immersion and  $W_d$  is the weight of composite sample before immersion.

Mechanical strength of composites was investigated by using a GoTech universal testing machine (model 1114). Tensile testing of composites having a dimension of 120 mm × 15 mm × 3 mm was carried out according to ASTM D3039 at crosshead speed of 5 mm/min and gauge length of 6 cm. Flexural testing of samples with dimension of 120 mm × 20 mm × 3 mm was carried out at crosshead speed of 10 mm/min and gauge length of 9 cm according to ASTM D790. Izod impact testing of sample having a dimension of 65 mm × 20 mm × 3 mm was performed using a 2-Joule hammer on a Universal Fractoscope (model CEAST 6546) according to ASTM D256-93.

## 2.6 Scanning Electron Microscopy

The morphology of SCE treated fibers and fractured surface of all composites after flexural testing was examined by using an SEM model S 360 (Leica Cambridge Ltd.) at an accelerating voltage of 5 kV. Prior to analysis, composite was kept in an oven for one day at 60 °C for removal of moisture. Sample was sputtered with gold prior to observation.

## 3 RESULTS AND DISCUSSION

### 3.1 Fiber Length Analysis

Weighted fiber length average uses fiber mass instead of fiber count as the numerical quantifier. Fiber mass is correlated with fiber strength or viscosity and, hence, provides a clear relationship between fiber length and sheet strength. Average fiber length was  $0.92 \pm 0.04$  mm, length weighted average was  $1.59 \pm 0.08$  mm, and fine content was  $4.7 \pm 0.3\%$  of the SCE treated fiber (Table 1). Longer fiber length suggested that its incorporation into composites will result in improved mechanical properties. It was observed from Table 1 that the SCE process resulted in a decrease in fiber length and increase in fine content. The SEM images of fibers suggested that the SCE process induced compression force which acted on fibers and thus resulted in splitting of individual fibers, as shown in Figure 1a. In voids formed during bleaching, however, fine content of fibers covered the pores (Figure 1a) [10]. High pressure during SCE acted in one direction toward the fiber and this broke the crosslink between the microfibrils [11]. This phenomenon further resulted in increased specific surface area per unit volume. Compression force during SCE also flattened fiber (Figure 1b). Flattened fibers have high surface area, thus contributing to the wettability of composites which resulted in enhanced mechanical properties.

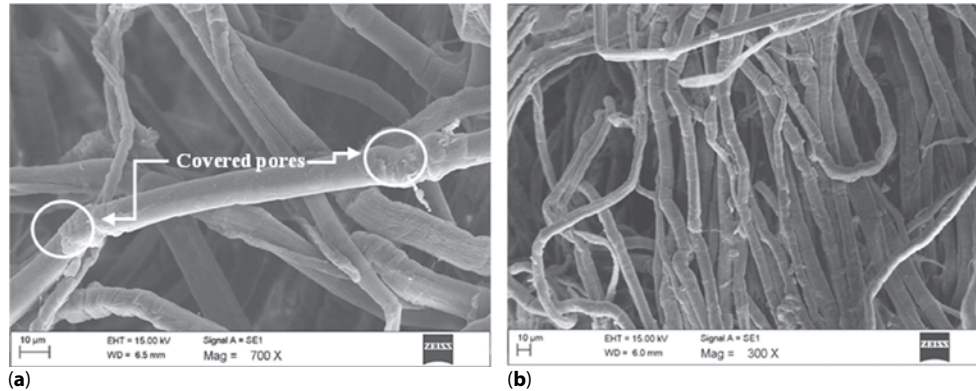
### 3.2 Density and Water Absorption of Composites

Water absorption capacity and density of composites are given in Figure 2. Density of composite increased with the addition of fiber from  $1.1095 \pm 0.01$  kg/m<sup>3</sup> to  $1.1820 \pm 0.02$  kg/m<sup>3</sup> for 0% and 15% epoxy composite,

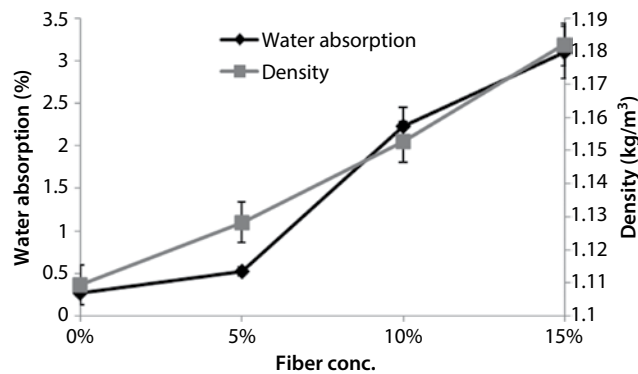
**Table 1** Average fiber length, length weighted average and fine content of fiber.

Sample	Average fiber length (mm)	Length weighted average (mm)	Fine content (%)
Raw fiber	$1.03 \pm 0.05^a$	$1.96 \pm 0.06^a$	$0.3 \pm 0.08^b$
SCE treated fiber	$0.92 \pm 0.04^b$	$1.59 \pm 0.08^b$	$4.7 \pm 0.3^a$

Any two means in the same column followed by the same letter are not significantly ( $p > 0.05$ ) different.



**Figure 1** SEM micrographs of supercritical carbon dioxide extracted fibers at (a) 700x and (b) 300x.



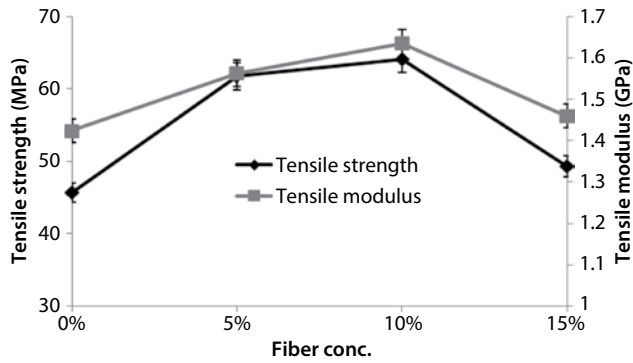
**Figure 2** Density and water absorption of the epoxy composite.

respectively. There was only a 6.53% increase in density when 15% of fiber was incorporated as compared to non-reinforced epoxy composites. The observed small increase in density was due to the low density of kenaf fiber [12]. Water absorption depends on types, orientation, and amount of fiber added, presence of void, porosity of composite, surrounding temperature, etc. [13, 14]. The blank epoxy sample showed lowest water absorption of  $0.27 \pm 0.02\%$  since the epoxy matrix is hydrophobic. The water absorption capacity increased with the addition of pulp fiber since the pulp fiber is hydrophilic in nature, thus resulting in the composite's easy absorption of moisture from surroundings. At 15% of fiber addition, composite demonstrated water absorption of  $3.1 \pm 0.3\%$ . Moreover, bubbles formed in composite reinforced with 15% of fiber because of poor dispersion of fiber in the epoxy matrix, which resulted in further absorption of moisture in voids created from bubbles after curing [15]. Furthermore, voids also promote the formation of cracks within the composite that allow moisture to diffuse into the sample [16]. Both phenomena as discussed above promoted water absorption in composites, as observed in the present study. At high water absorption percentage,

hydrophilic fiber absorbs a large amount of diffused water molecules and this phenomenon reduces the interfacial adhesion between fiber and epoxy matrix. The disruption of interfacial adhesion resulted in low mechanical strength of composites.

### 3.3 Tensile Properties of Composites

From Figure 3 it was observed that fiber concentration had a significant effect on tensile strength of the composite. The tensile strength of the epoxy composites increased due to the incorporation of the BSFE fiber up to 10% with a further decrease in tensile strength at 15% of fiber concentration. With the addition of 10% of pulp fiber, tensile strength increased to  $64.12 \pm 4.2$  MPa from  $45.68 \pm 3.5$  MPa at 0% of fiber content. Fiber had higher specific surface area per unit volume due to the SCE process, thus enhancing the wetting process of fiber which resulted in improved tensile strength [4]. However, at 15% of fiber content, tensile strength decreased to a value of  $49.32 \pm 3.8$  MPa [17]. This might be due to void formation since fiber could not be dispersed properly in epoxy matrix at high concentration. Poor dispersion of pulp fiber also inhibited its wetting by epoxy, thus matrix might not get completely cured by hardener, which further resulted in poor tensile strength [18]. A similar trend was observed for the tensile modulus of composites. Tensile modulus is described as stiffness of composite [19]. Tensile modulus with a value of  $1.64 \pm 0.03$  GPa was highest at 10% of fiber loading and for pure epoxy it was  $1.42 \pm 0.01$  GPa. However, a further increase in fiber concentration resulted in tensile modulus of  $1.46 \pm 0.02$  GPa at 15%. The observed reduction at higher fiber content was due to improper dispersion of hydrophilic filler in hydrophobic matrix [18]. Elongation at break decreased in fiber concentration in a dependent manner. It decreased from  $9.5 \pm 0.4\%$  for pure epoxy to  $6.2 \pm 0.3\%$  at 15% of fiber in epoxy composites (Figure 5).



**Figure 3** Tensile strength and tensile modulus of composite with various fiber concentrations.

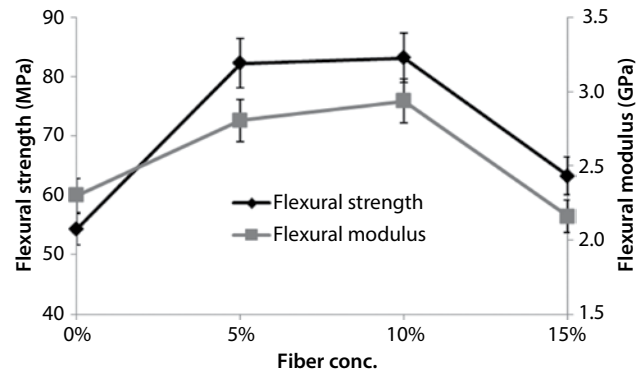
Kenaf fiber is highly rigid as compared to epoxy, thus in the present study fiber incorporation resulted in a decrease in percent elongation at break.

### 3.4 Flexural Properties of Composites

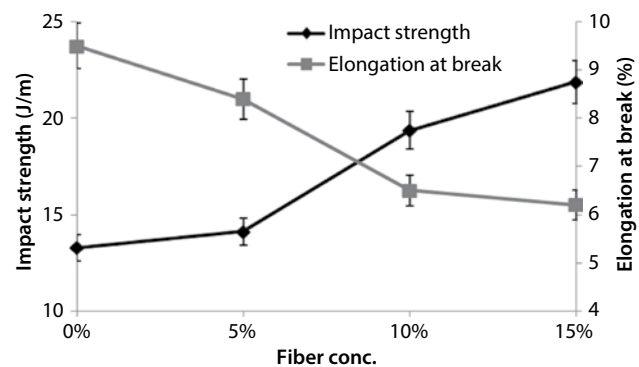
Incorporation of pulp fiber also improved the flexural modulus and flexural strength of composite samples (Figure 4). At 0% of fiber, composite demonstrated a flexural modulus of 2.3 ± 0.15 GPa; however, the highest modulus was observed at 10% with a value of 2.94 ± 0.18 GPa. A further increase in fiber concentration to 15% resulted in a reduction in flexural modulus to a value of 2.16 ± 0.17 GPa. Incorporation of pulp fiber up to 10% increased the ability of composite to withstand a higher load. Beyond that, poor dispersion of fiber led to a reduction in interfacial adhesion and thus resulted in lower flexural modulus [20]. A similar trend was followed by the flexural strength of composites, as shown in Figure 4. With an increase in fiber loading, flexural strength increases to a maximum value of 83.14 ± 6.3 MPa at 10% and for pure epoxy it was 54.3 ± 3.7 MPa. However, at 15% of fiber concentration the flexural strength of composites was reduced to a value of 63.24 ± 4.2 MPa. The observed reduction was due to agglomeration of hydrophilic fiber at higher concentration in hydrophobic epoxy matrix; as a result the compatibility between fiber and matrix was reduced, which led to ineffective stress transfer in composite [11].

### 3.5 Impact (IZOD Test)

Surprisingly, the addition of pulped fiber improved the impact strength of composites at all concentrations, therefore brittleness of blank epoxy composite was overcome in the present study by addition of fiber (Figure 5). This demonstrated increase in impact strength might be due to the high flexibility of fiber, which enables the composite to resist



**Figure 4** Flexural strength and flexural modulus of epoxy composites.

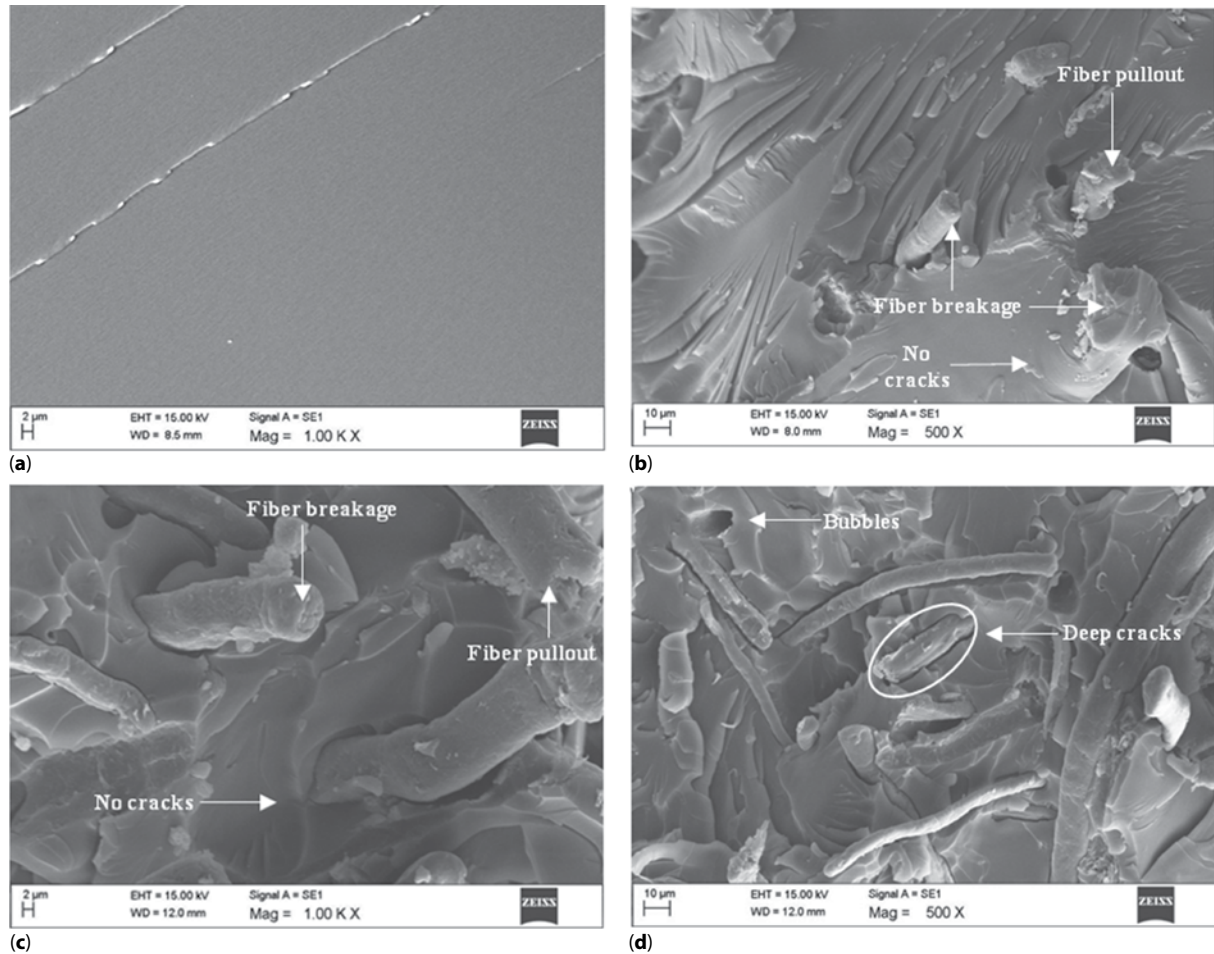


**Figure 5** Impact strength and percent elongation at break of epoxy composite with various fiber concentrations.

sudden load as compared to blank epoxy sample [21, 22]. The highest impact strength was 21.88 ± 1.5 J/m for composites having 15% of fiber and for 10% it was 19.38 ± 1.1 J/m and for pure epoxy it was 13.28 ± 0.9 J/m. Based on the mechanical analysis of composite it can be concluded that the composite with 10% of pulp fiber had the highest mechanical strength as compared to all other samples in the present investigation.

### 3.6 Scanning Electron Microscopy of Fractured Surface of Composite

Figure 6 shows the morphology of the fractured surface of epoxy-based composite incorporated with different pulp fiber concentrations. It was observed that the fractured surface of composite without pulp fiber was smooth, as shown in Figure 6a. However, all fiber incorporated composites had uneven fractured surface (Figure 6b–d). Fibers were pulled out from the fractured surface of all fiber loaded composites.



**Figure 6** SEM images of fractured surfaces of epoxy composite with fiber concentration at (a) 0%, (b) 5%, (c) 10% and (d) 15%.

Fracture toughness is not affected by fiber diameter [23] but fiber length affects the energy absorption in composites [24]. Unlike blank epoxy sample, stress was evenly distributed on every single fiber of composites and this led to improved toughness of composite material and finally resulted in fiber pull-out during fracture [25]. However, some fibers were broken instead of being pulled out and this shows that the stress act on epoxy matrix was transferred to fibers during mechanical testing, which resulted in their breakage [26]. This further demonstrated that pulped fibers were brittle. Zhandarov *et al.* [25] stated that the broken fibers contributed little to the overall toughness. Wang and Chung [11] stated that fiber breakage is less in composite with higher modulus and a similar observation was made in the present study. Composite with 10% fiber loading showed the highest modulus and least fiber breakage as compared to 5% and 15% fiber incorporated composites. Agglomeration of fiber occurred at 15%, which resulted in its lower compatibility with epoxy as observed by deep cracks at the

interface (Figure 6d). However, at lower concentration no such cracks were observed at the interface between matrix and fiber. Thus, the decrease in mechanical properties of composite at 15% is explained by SEM image analysis. There was no bubble formation at the interface between fiber and epoxy resin at 5% and 10% of composite, which indicated the good dispersion of filler in matrix (Figure 6b,c). However, bubble formation was spotted for 15% fiber-containing composite (Figure 6d). This indicated that the fiber resisted its mixing at higher concentration with epoxy and resulted in bubble formation, which further led to a reduction of mechanical properties of the composite [27]. Furthermore, all filler incorporated composites had flattened fiber caused by supercritical CO<sub>2</sub> process. Flattened fiber was found to break close to the fractured surface without any pull-out. Flattened surface also indicated enhanced specific surface area per unit volume of fiber, which aided in its wetting action and therefore improved the compatibility between fiber and matrix [27].

## 4 CONCLUSION

In the present study, green composites based on SCE treated kenaf fiber reinforced in epoxy matrices were prepared and various mechanical properties were analyzed. Composites reinforced with 10% of fiber demonstrated the highest mechanical properties among all studied samples. Tensile strength and tensile modulus of 10% fiber-reinforced composite increased to 40.37% and 14.83%, respectively, as compared to 0% fiber-reinforced composite. A similar trend was observed for flexural strength and flexural modulus with a respective increase of 53.11% and 27.6% as compared to non-reinforced composite. Up to 10%, flattened fibers enhanced the wettability of composites due to their high surface area and thus improved the mechanical characteristics of composites. A higher percentage of fiber agglomeration led to a decrease in composite properties. However, impact strength increased with an increase in fiber percentage which was due to the natural rigidity of fiber. SEM image analysis suggested that there was fiber pull-out and fiber breakage in epoxy-based composites after flexural testing. This showed that the load applied on the composite was effectively transferred to the fiber itself, thus further strengthening the fact that fiber incorporation resulted in better mechanical properties.

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## REFERENCES

1. S. Karimi, P.M. Tahir, A. Karimi, A. Dufresne, and A. Abdulkhani, Kenaf bast cellulosic fibers hierarchy: A comprehensive approach from micro to nano. *Carbohydr. Polym.* **101**, 878–885 (2014).
2. M. Kleen, J. Sjöberg, O. Dahlman, S. Johansson, K. Koljonen, and P. Stenius, The effect of ECF and TCF bleaching on the chemical composition of soda-anthraquinone and kraft pulp surfaces. *Nord. Pulp. Pap. Res. J.* **17**(3), 357–363 (2002).
3. M. McHugh and V. Krukoni, *Supercritical Fluid Extraction: Principles and Practice*, H. Brenner (Ed.), Elsevier, Massachusetts, USA (2013).
4. M.F. Kemmere and T. Meyer (Eds.), *Supercritical Carbon Dioxide in Polymer Reaction Engineering*, John Wiley & Sons, Weinheim, Germany (2005).
5. A.B. Maureen, C.J. Martin, and D.N. John, Epoxy resins, in *ASM Handbook*, vol. 21, pp. 78–89, Hexcel Corporation, ASM International, Materials Park, Ohio, USA (2001).
6. C. May (Ed.), *Epoxy Resins: Chemistry and Technology*, 2nd ed., CRC Press, New York, USA (1987).
7. S. Matei, M. Stoicanescu, and A. Crisan, Composites with short fibers reinforced epoxy resin matrix. *Procedia Technology* **22**, 174–181 (2016).
8. J.D. Lincoln, J.C. Earthman, O.A. Ogunseitan, J.D. Saphores, and A.A. Shapiro, Renewable-resource printed wiring board design using natural fibers and a bio-based thermosetting matrix, in *Proceedings of the 2007 IEEE International Symposium on Electronics and the Environment*, pp. 190–195, Institute of Electrical and Electronics Engineers (2007).
9. M. Mukhopadhyay, *Natural Extracts Using Supercritical Carbon Dioxide*, CRC Press, New York, USA (2000).
10. T. Hiraoka, K. Asakawa, and Y. Hotta, Method of manufacturing a material having a fine structure, US Patent 6565764, assigned to K.K. Toshiba (2003).
11. X. Wang and D. Chung, Fiber breakage in polymer-matrix composite during static and fatigue loading, observed by electrical resistance measurement. *J. Mater. Res.* **14**(11), 4224–4229 (1999).
12. M.V. Madurwar, R.V. Ralegaonkar, and S.A. Mandavgane, Application of agro-waste for sustainable construction materials: A review. *Constr. Build. Mater.* **38**, 872–878 (2013).
13. N. Hameed, P.A. Sreekumar, B. Francis, W. Yang, and S. Thomas, Morphology, dynamic mechanical and thermal studies on poly(styrene-co-acrylonitrile) modified epoxy resin/glass fibre composites. *Compos. Part A-Appl. S.* **38**(12), 2422–2432 (2007).
14. P.A. Sreekumar, K. Joseph, G. Unnikrishnan, and S. Thomas, A comparative study on mechanical properties of sisal-leaf fibre-reinforced polyester composites prepared by resin transfer and compression moulding techniques. *Compos. Sci. Technol.* **67**(3), 453–461 (2007).
15. A.K. Bledzki, V.E. Sperber, and O. Faruk, *Natural and Wood Fibre Reinforcement in Polymers*, Rapra Technology Ltd., Shropshire, UK (2002).
16. L.W. Davies, R.J. Day, D. Bond, A. Nesbitt, J. Ellis, and E. Gardon, Effect of cure cycle heat transfer rates on the physical and mechanical properties of an epoxy matrix composite. *Compos. Sci. Technol.* **67**(9), 1892–1899 (2007).
17. F.G. Garcia, B.G. Soares, V.J.R.R. Pita, R. Sánchez, and J. Rieumont, Mechanical properties of epoxy networks based on DGEBA and aliphatic amines. *J. Appl. Polym. Sci.* **106**(3), 2047–2055 (2007).
18. H. Anuar and A. Zuraida, Improvement in mechanical properties of reinforced thermoplastic elastomer composite with kenaf bast fibre. *Compos. Part B-Eng.* **42**(3), 462–465 (2011).
19. M. Jawaid, H.A. Khalil, A. Hassan, R. Dungani, and A. Hadiyane, Effect of jute fibre loading on tensile and dynamic mechanical properties of oil palm epoxy composites. *Compos. Part B-Eng.* **45**(1), 619–624 (2013).
20. G. Murray, C.V. White, and W. Weise, *Introduction to Engineering Materials*, CRC Press, New York, USA (2007).

21. X. Cao, H. Dong, and C.M. Li, New nanocomposite materials reinforced with flax cellulose nanocrystals in waterborne polyurethane. *Biomacromolecules* **8**(3), 899–904 (2007).
22. A. Jabbar, J. Militký, B.K. Madhukar, S. Rwawiire, Y. Nawab, and V. Baheti, Modeling and analysis of the creep behavior of jute/green epoxy composites incorporated with chemically treated pulverized nano/micro jute fibers. *Ind. Crops. Prod.* **84**, 230–240 (2016).
23. J. Wells and P. Beaumont, Debonding and pull-out processes in fibrous composites. *J. Mater. Sci.* **20**(4), 1275–1284 (1985).
24. J.A. Nairn, C.H. Liu, D.A. Mendels, and S. Zhandarov, Fracture mechanics analysis of the single-fiber pull-out test and the microbond test including the effects of friction and thermal stresses, in *Proceedings 16th Annual Technical Conference of the American Society Composites*, VPI, Blacksburg VA (2001).
25. S. Zhandarov, E. Pisanova, E. Mäder, and J. Nairn, Investigation of load transfer between the fiber and the matrix in pull-out tests with fibers having different diameters. *J. Adhes. Sci. Technol.* **15**(2), 205–222 (2001).
26. K.K. Chawla, Fiber fracture: An overview, in *Fiber Fracture*, M. Elices and J. Llorca (Eds.), pp. 5–25, Elsevier Science Ltd., Oxford, UK (2002).
27. Y. El-Shekeil, S. Sapuan, K. Abdan, and E. Zainudin, Influence of fiber content on the mechanical and thermal properties of kenaf fiber reinforced thermoplastic polyurethane composites. *Mater. Des.* **40**, 299–303 (2012).