Influence of fiber length, fiber content and alkali treatment on mechanical properties of natural fiber-reinforced epoxy composites

M. Bhuvaneshwaran[1), *)]**, P.S. Sampath**[2)]**, Suresh Sagadevan**[3)]

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Abstract: The composites of epoxy resin and *Coccinia indica* fibers (CIF) were obtained. The raw fibers were initially treated with 5 % NaOH. Compression molding technique was used for preparing the composites with four different fiber lengths (10, 20, 30, 40 mm) and various fiber loadings (25, 30, 35, 40 wt %). The mechanical properties of the produced composites were investigated and their structure was analyzed by using scanning electron microscope (SEM). It was found, that the composite matrix reinforced with 30 mm fiber length at 35 wt % fiber loading exhibited the best mechanical properties.

Keywords: natural fibers, epoxy composites, mechanical properties, SEM.

Wpływ długości i zawartości włókien oraz ich obróbki alkalicznej na właściwości mechaniczne kompozytów epoksydowych wzmacnianych włóknami naturalnymi

Streszczenie: Otrzymano kompozyty epoksydowe z udziałem włókien *Coccinia indica* (CIF). Surowe włókna poddawano obróbce alkalicznej przy użyciu 5 % NaOH. Metodą wytłaczania sporządzono kompozyty żywicy epoksydowej zawierające 25, 30, 35 lub 40 % mas. włókien CIF o różnej długości (10, 20, 30, 40 mm). Zbadano wytrzymałość mechaniczną otrzymanych kompozytów oraz przeprowadzono obserwacje ich struktury za pomocą skaningowego mikroskopu elektronowego (SEM). Stwierdzono, że najlepsze właściwości mechaniczne wykazywały kompozyty wzmocnione 35 % mas. włókien o długości 30 mm.

Słowa kluczowe: włókna naturalne, kompozyty epoksydowe, właściwości mechaniczne, SEM.

Day by day, the uses of natural fibers in composites are increasing due to their features such as being inexpensive, biodegradable, abundantly available and eco-friendly in nature [1], corrosion-resistant, lightweight and easily processed [2]. These features encourage the manufacturing of low weight natural fiber based polymer composites, which found uses in various fields such as furniture, packaging, automobile industry acoustics-vibration, aeronautics, impact energy absorption and naval applications [3–8]. However, natural fibers have some disadvantages, including inconsistency in the product performance because of natural variability in fiber characteristics [9]. The mechanical properties of natural fibers are totally dependent on the age, weather, method of extraction and quality of soil [2, 10]. Moreover, a hydrophilic nature of natural fibers affects negatively their adhesion to hydrophilic polymer matrices [11]. The advantage of using thermoset polymer matrix over thermoplastic ones in the preparation of natural fiber-reinforced composites is due to the fact that the fibers can be easily mixed with thermoset resin available in liquid form and the composites can be prepared at low pressure and temperature. Epoxy-based composites exhibit a range of advantageous properties when compared with thermoplastic counterparts, they also show better interfacial adhesion than those based on unsaturated polyester [12]. However, to overcome the disadvantage of hydrophilic nature of natural fibers, a suitable chemical modification of fiber surface to make it less hydrophilic is needed [13]. Using alkali treatment, it was found that the contents of hemicellulose, lignin and wax in the fibers were reduced [14]. The Borassus fruit fibers were treated with 5, 10 and 15 % NaOH; it was noted that 5 % NaOH treatment yielded a significant improvement in tensile properties of the fibers than the others [1]. Further increasing the alkali con-

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[1)] K.S.R. College of Engineering, Department of Mechanical Engineering, Tiruchengode, Tamil Nadu, India.

[2)] K.S. Rangasamy College of Technology, Department of Mechanical Engineering, Tiruchengode, Tamil Nadu, India.

[3)] AMET University, Centre for Nanotechnology, Chennai-603 112, India.

[*)] Author for correspondence:
e-mail: bhuvansparks2017@gmail.com

centration causes the reduction in the physical properties of the composite [15]. Fiber length, fiber load, and fiber orientation on the polypropylene composites were analyzed [16]. Using optimum fiber content ensured favorable fiber wetting by matrix polymer and resulted in a strong interfacial bond. The fiber length should have a certain minimum value known as critical fiber length [17]. The present work focused on the utilization of *Coccinia indica* fibers (CIF) as reinforcement in the preparation of epoxy composites. These fibers were extracted from the stem of *Coccinia indica* (CI). The composite samples were prepared with different fiber contents ranging from 25 % to 40 % by weight in the interval of 5 % and different fiber lengths ranging from 10 mm to 40 mm in the interval of 10 mm to obtain the optimum fiber length and volume from the viewpoint of the composite mechanical properties.

Fig. 1. Fiber extraction: a) full length plant, b) sliced CI, c) CI immersed in water after 3 weeks, d) hewed CI fibers

EXPERIMENTAL PART

Materials

Fiber preparation

The fiber was extracted from the stem of *Coccinia indica* plant, available in Chettipalayam village, Erode, Tamil Nadu, India. Figure 1 shows the actual process of extracting the fiber from the CI plant. The fiber was extracted from the stem of CI plant by immersing it in water for 3 weeks. The immersing of CI plant was done to allow for its microbial degradation so that it would become soft and both the inner and outer layer would be removed. The outer layer was removed and disposed, while the inner layer was retained for further separation of fibers by traditional combing process with the aid of long, fine metal teeth [18].

Fiber treatment

The available natural fibers are hydrophilic in nature, therefore a chemical treatment is required to render them hydrophobic. The most widely used surface treatment process is alkaline treatment, which eliminates hydrogen bonding in the network structure of the fiber. Fibers were soaked in 5 % NaOH solution at room temperature for an hour and then washed thoroughly with water to remove the excess of NaOH. Finally, they were washed with distilled water and dried in direct sunlight for 2–3 weeks [14].

$$\text{Fiber–OH} + \text{NaOH} \rightarrow \text{Fiber—O}^{-}\text{–Na}^{+} + \text{H}_2\text{O} \qquad (1)$$

Further, the fibers were cut in four different dimensions, namely 10, 20, 30, and 40 mm for preparing the laminates.

Epoxy resin and hardener

Epoxy resin with trade name LY556 and hardener with trade name HY951 were purchased from the Covai Seenu & Company, Coimbatore, Tamil Nadu 641012, India and used in the proportion of 10 : 1.

Preparation of CIF/epoxy composites

A mold made of rectangular steel plates with dimensions of 300 × 300 × 3 mm assembled a male die, spacer, and female die as shown in Fig. 2, was used for fabricating CIF/epoxy composite material. The molding was

Fig. 2. Compression molding machine: a) spacer in male die, b) spacer in female die, c) without spacer

performed at a pressure of 10.34 MPa and temperature of 80 °C for 3 hours. For easy removal of the composite from the mold, wax was applied on the mold before fabrication.

For easy identification and discussion the samples were named as N1025, N1030, up to N4040 depending upon the fiber length and fiber percentage in the composite. In these names the first two Arabic numerals denote the length of the fiber and the next two digits denote the weight percentage of the fiber.

Methods of testing

Tensile test

Computer-controlled Kalpak Universal Testing Machine was used to test the specimens according to ASTM D638-03 [19] with a head speed of 2 mm/min. Five specimens were tested for each set of samples and mean values were reported.

Compressive test

The test was carried out according to ASTM D 695-02a [20] using Kalpak Universal Testing Machine. The strain gauge readings and the load cell readings were noted when the axial cross head movement rate was 2 mm/min. Five specimens were tested for each set of samples and the mean values were reported.

Flexural test

Flexural tests were performed using the three point bending method of ASTM D 790-03 [21] using Kalpak Universal Testing Machine with cross head speed of 2 mm/min. Five specimens were tested for each set of samples and the average values were reported. With the specimen being freely supported by a beam, the flexural modules were calculated from the slope of the initial portion of the load curve when applying maximum load in the middle of the specimen.

Impact test

Izod impact tests were conducted according to ASTM D 256-05 [22]. Five specimens were tested to determine the impact resistance and average impact strength of composites were calculated. The notched-bar method of impact testing used lead to sudden fracture where a sharp stress raises at the notch and provided information under high velocity loading conditions. The entire test was carried out at room temperature.

SEM analysis

The failure surfaces of the alkali treated CIF/epoxy composites were analyzed by scanning electron microscopy (SEM), model VEGA3 TESCAN, to examine the interfacial properties like fiber matrix bonding, fiber breaking and fiber pull-out.

RESULTS AND DISCUSSION

Tensile strength

The effect of alkali treatment of *Coccinia indica* fiber on the tensile performance of CIF/epoxy composites with different fiber lengths and different fiber contents was investigated. Tensile load of the composites of all samples are shown in Table 1 and Fig. 3.

Among the examined samples, N3035 showed the highest tensile strength of 33.79 N/mm^2. When the fiber length and fiber content further increased, the strength began to reduce, due to insufficient resin for complete wetting of fibers. As reported in other study, tensile strength of the composites increased while using chemically treated sisal fibers [23]. The treated CIF/epoxy composites had 29 % higher tensile strength when compared with untreated sample. Comparable results were obtained by Maheshwari [24]. Figure 4 presents the SEM micrograph of tensile sample indicating that N3035 composite was noticed to have a good bonding interface between fiber and matrix. Moreover, the study on short coir fiber rein-

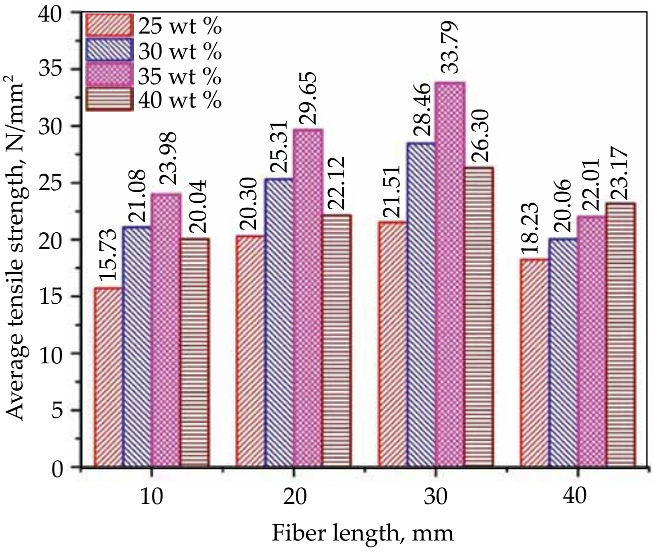

Fig. 3. Tensile test results of CIF/epoxy composites

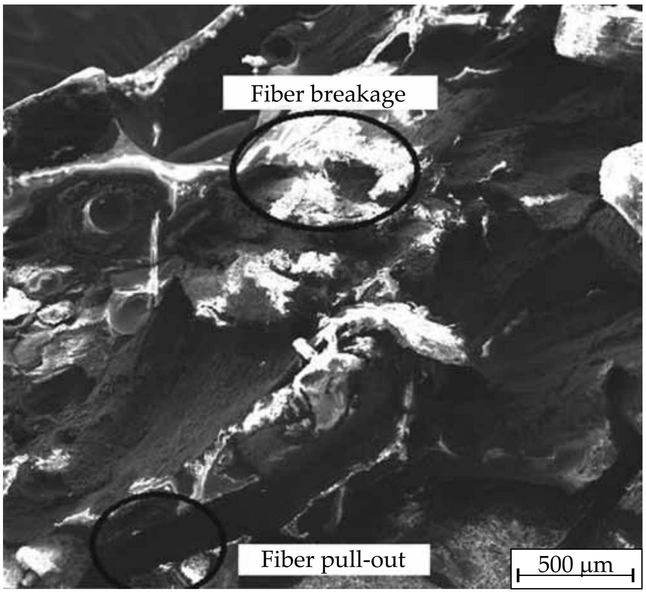

Fig. 4. SEM image of specimen N3035 after tensile test

Table 1. Mechanical properties of epoxy composites with 5 % NaOH treated fiber

Sample code	Average tensile strength, N/mm^2	Average compressive strength, N/mm^2	Average flexural strength, MPa	Average impact strength, J/m
N1025	15.73	28.33	36.47	256.80
N1030	21.08	33.82	45.04	284.09
N1035	23.98	41.03	66.86	406.89
N1040	20.04	39.22	56.52	286.70
N2025	20.30	29.82	41.72	324.09
N2030	25.31	34.69	53.60	355.17
N2035	29.65	41.05	73.09	423.53
N2040	22.12	38.53	53.15	391.89
N3025	21.51	31.94	54.64	466.82
N3030	28.46	37.31	72.49	518.53
N3035	33.79	45.48	79.86	580.09
N3040	26.30	37.98	62.16	293.45
N4025	18.23	29.08	44.88	247.80
N4030	20.06	36.06	66.82	371.84
N4035	22.01	40.71	69.25	438.61
N4040	23.17	37.43	56.23	325.94

forced natural rubber composites showed that the length of the fiber might have a major role in changing the tensile properties [25].

Compressive strength

The interfacial linkage between the matrix and the fiber played a vital role in controlling the compression properties of the composite material. The compressive strength of all the composites are illustrated in Fig. 5 and Table 1.

It is demonstrated that the compressive properties were improved due to the fact that chemical treatment reduced the hydrophilic nature of the cellulose fiber and improved the fiber matrix interfacial bonding [23]. N3035 sample showed an enhanced compressive strength of 45.48 N/mm^2 when compared to other materials. Average of 23 % compressive strength was achieved due to the improved transfer of stress from the matrix to the fiber. Alkali treatment resulted in rough surface and the interfacial bonding between fiber and matrix had been improved, which increased the strength behavior of the composites [11]. The discontinuities of fibers resulted in their breaking and pull out from the resin. Because of this, the voids were present in the matrices as shown in Fig. 6.

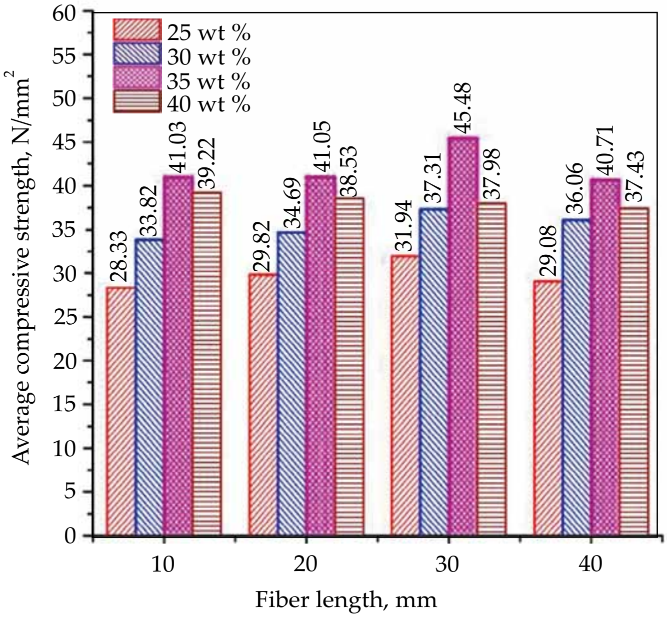

Fig. 5. Compressive strength of CIF/epoxy composites

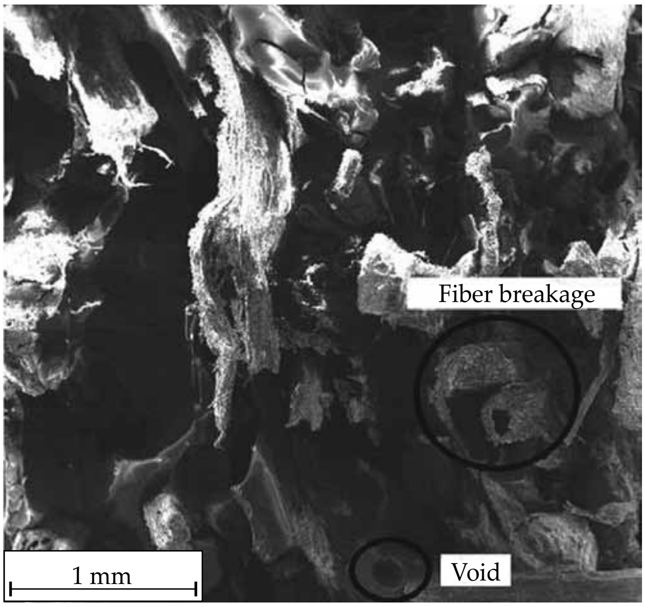

Fig. 6. SEM image of specimen N3035 after compression test

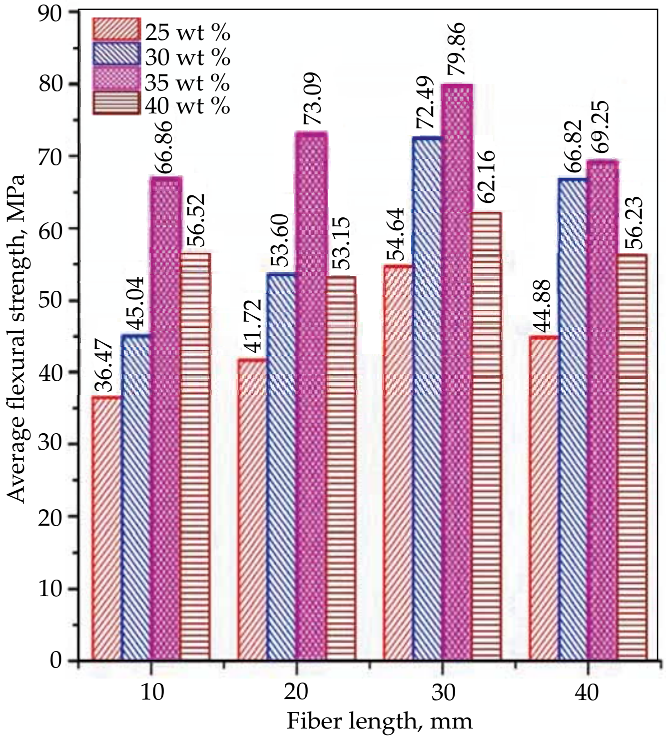

Fig. 7. Flexural test results of CIF/epoxy composites

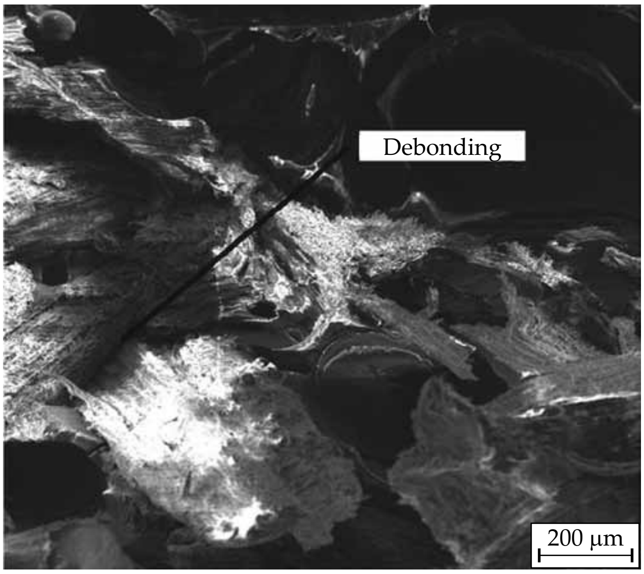

Fig. 8. SEM image of specimen N3035 after flexural test

Flexural strength

Table 1 and Fig. 7 illustrate the results of flexural testing of CIF/epoxy composites. The highest flexural strength of 79.86 MPa was determined for N3035 sample. An increase in the area of contact between the alkali treated fibers and the matrix improved the interfacial bonding [26]. When 30 mm fiber length and 35 wt % fiber content were used, the composite exhibited a better flexural properties. Further increase in the length and content of the fibers resulted in a decrease in the flexural strength. A date palm tree fiber reinforced epoxy composites showed similar trends [27]. Figure 8 represents the debonding and formation of crumbs in N3035 flexural test specimen, mainly due to epoxy resin was not able to penetrate into the fiber.

Impact strength

The impact resistance of a composite is measured by determining the total energy dissipated in the material prior to final failure. Figure 9 and Table 1 show the impact strength values of CIF/epoxy composites. An increase in the impact strength is observed when increasing fiber length up to 30 mm and fiber content up to 35 %, beyond that any increase in the fiber length and fiber content results in reducing the strength. The impact properties of composite can only be improved by decreasing the fiber length and by increasing the friction stress between the fiber and the matrix. The total energy dissipa-

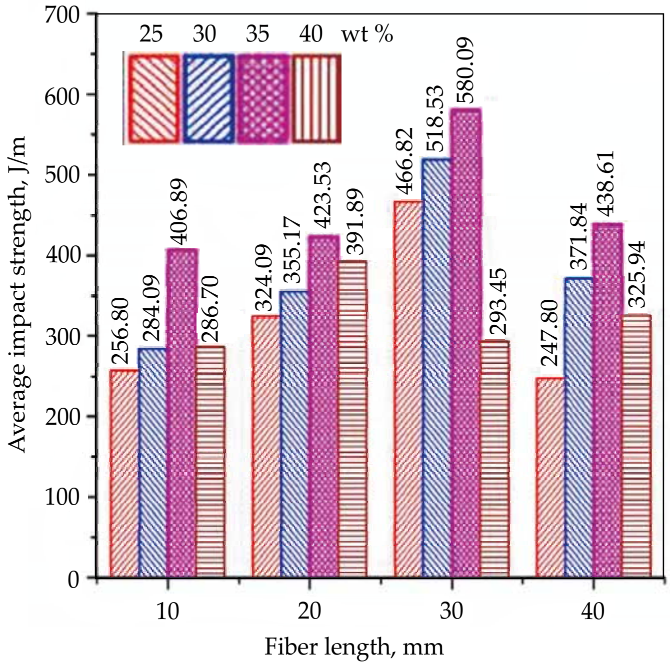

Fig. 9. Impact test results of CIF/epoxy composites

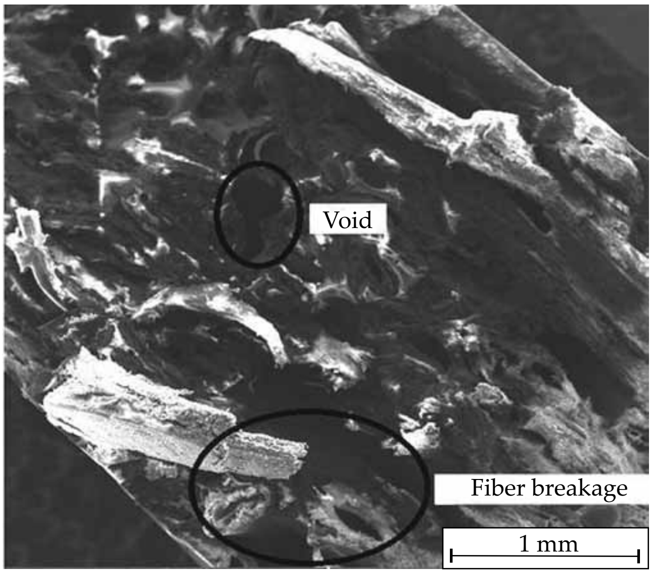

Fig. 10. SEM image of specimen N3035 after Izod test

ted in the composite before final failure occurs is a measure of its impact resistance [9]. N3035 epoxy composites gave maximum impact strength of 580.09 J/m and similar results were obtained in [15]. Corrales *et al.* observed that fiber/matrix interfacial properties, fiber aspect ratio, length distribution and orientation influenced the impact properties of short fiber reinforced composites [11]. The increased impact strength of N3035 composites with alkali treated fibers results from a better stress transfer between the epoxy matrix and fiber [28] as shown in Fig. 10 in SEM micrograph.

CONCLUSIONS

Effects of surface treatment of the fiber, volume fraction and dimension on the mechanical properties of CIF/epoxy composites were investigated. The conclusions are as follows:

– CIFs can replace the conventional materials due to their availability and enhanced mechanical properties; it has been confirmed by extensive experimental study.

– Epoxy composite contained alkali (5 % NaOH) treated CIFs exhibits higher strength when compared with untreated CIF/epoxy composites.

– The composite of 30 mm fiber length and 35 wt % fiber content gives overall better mechanical properties.

– The determined tensile, compressive, flexural and impact strengths of CIF composite (N3035) are 33.79 N/mm^2, 45.48 N/mm^2, 79.86 MPa and 580.09 J/m, respectively.

– SEM micrographs have revealed bonding between the epoxy matrix and the fiber, fiber pull-out, fiber breakage and voids in the composite.

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