Research Article

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Effect of natural *Indocalamus* leaf addition on the mechanical properties of epoxy and epoxy-carbon fiber composites

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Abstract: In this study, *Indocalamus* micro/nanofibers (IMFs) were extracted from natural *Indocalamus* leaves by physical processing and alkaline treatment. IMFs reinforced epoxy resin (EP) and their carbon-fiber composites (IMFs/CFRP) were fabricated. The effects of IMF on the mechanical properties of the EP and CFRP composites were studied. Infrared spectroscopy and scanning electron microscopy (SEM) were used to characterize the functional groups and microstructure of IMF, EP, and CFRP. The experimental results showed that the strength of the EP increased as the IMF content increased from 0% to 20%, but on further increase in IMF content of 25%, the strength of the EP reduced. In addition, the mechanical properties of the IMF/CFRP were slightly higher as compared with the control CFRP. The SEM observations on IMFs/EP and IMFs/CFRP composites reveal that the alkali-treated IMFs facilitate the interfacial interlocking structure and improve the interfacial adhesion of the composites.

Keywords: epoxy resin, carbon fiber, composites, natural *Indocalamus* leaf, mechanical properties

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1 Introduction

Carbon fiber-reinforced epoxy resin composites (CFRP) have a wide range of applications in the fields of automotive, transportation, construction, and aerospace due to their excellent performances and good design abilities (1,2). Epoxy resin (EP) is a typical thermosetting polymer material and transfers external loads in the CFRP during the loading. Therefore, the mechanical properties of EP play an important role in the mechanical properties of the CFRP.

Natural plant fillers are considered desired substitute for traditional synthetic micro/nanofillers to improve the mechanical properties of the EP and other polymers because they have the advantages of renewable resource, abundant sources, low cost, high strength and stiffness, easy manufacturing and processing, good biodegradability, etc. (3,4). And, therefore, they have been used in automobile bumpers, seat back panels, and doors (4). The researchers have investigated the EP composites mixing with extracting fiber fillers from many natural plants, such as corn stalks, bamboo, jute, pineapple leaves, and coconut shells (5–7), and confirmed that natural plant fibers have enhancing effects on the mechanical and thermal properties of the EP and its composites.

Among natural plant fibers, bamboo fiber (BF) is an important component of the bamboo biostructure. Bamboo is a natural lingo-cellulose composite containing fibers (bast fibers in vascular bundles) and matrix (8,9), which has many amazing characteristics, including a short growth cycle, abundant resources, high toughness, and low density (3). These characteristics of bamboo have attracted extensive interest from researchers using bamboo as a reinforcement for polymer composites in the form of micro/nanofiber filler, woven, and fabric. The BF has a high specific strength and specific modulus (10,11), which can be obtained from raw bamboo by various extraction methods, including retting, steam explosion, alkali treatment, degumming, micro-grinding, and cryo-crushing (12,13). Many reports demonstrated that BF has great potential to improve the mechanical properties of EP. Yang et al. (5) added BFs into EP and found that the tensile strength and modulus increased significantly after incorporating BFs into EP, which indicates that the reinforcing effect of BFs on the EP is obvious. Daniel et al. (6) developed hybrid composites using various stacking-order BFs and jute fibers as reinforcements. The hybrid composites had superior mechanical properties when the stacking order was composed of BFs as the outer layer and jute fibers as the inner layer. Chin et al. (14) investigated the mechanical properties of BF-reinforced composites with fiber content ranging from 0 to 40 vol% in three thermoset resins (epoxy, polyester, and vinyl ester). The tensile and flexural properties of all the composites were directly proportional to the fiber volume fraction. The epoxy matrix composites with 40 vol% fiber exhibited the highest tensile and flexural strength than polyester and vinyl ester matrix composites. Phong et al. (15) extracted micro/nano-BFs from raw bamboo and investigated the effect of BF on the mechanical properties of CFRP composites. They found that fracture toughness, tensile modulus, and fatigue life were increased. These studies indicate that the addition of BF is in favor of the mechanical properties of EP and its carbon fiber composites.

The desired mechanical properties and physical properties of BF-reinforced epoxy composites could be achieved by modification techniques (16,17). The main constituents of BFs are cellulose, hemicellulose, and lignin, which contain many polar functional groups, leading to poor interfacial compatibility between fiber and EP. These factors reduce the transfer effect of stress transfer at the interface (18,19) and limit the mechanical properties of epoxy composites. To solve the problem, some modification methods have been used, including physical modification (steam explosion, heat treatment, etc.) and chemical modification (alkali treatment, silane coupling agent treatment, graft copolymerization, acetylation treatment, etc.) (16–20). Silane coupling agents and sodium hydroxide are usually applied to increase the tensile strength, elastic modulus, flexural strength, and bending modulus of bamboo/epoxy composites by improving the interface (21–25). Shih (21) investigated the morphology, mechanical, and thermal properties of BF-reinforced composites treated with silane coupling agents. The silane coupling agent-treated fibers had better compatibility with polymers compared to untreated fibers. Zhang et al. (25) treated BFs with different mass concentrations of NaOH solutions and prepared BF-reinforced epoxy composites. The interfacial shear strength between the fibers and the epoxy matrix was significantly improved due to the removal of impurities of exposed hydroxyl groups on the fiber surface by the alkali treatment.

The BF extracted from raw bamboo varies greatly due to the different species, growing regions, and parts of the raw bamboo, all of which affect the properties of BF (26). Awalludin et al. (27) compared five different bamboo species, whose tensile strengths range from 144.93 to 233.98 $N\cdot mm^{-2}$. Owing to the wide range of sources of natural bamboo, more BFs from different species of raw bamboo need to be explored. In this study, the *Indocalamus* leaf is used to produce the *Indocalamus* micro/nanofiber (IMF) by a series of processing treatments, and then these IMFs are mixed with EP to produce the modified EP and its carbon fiber composites. *Indocalamus* leaf is a good packaging material, which could package food, e.g., traditional food and tea, and it is also used as a bucket hat and boat canopy liner. It is noted that the information on IMF extracted from *Indocalamus* leaf as a reinforcement in EP is limited. Therefore, in this study, the effect of the IMF content on the mechanical properties of EP was studied at first, and a better composition was obtained. Afterward, the effects of IMF on the mechanical properties of carbon fiber-reinforced EP composites (CFRP) were studied, and the fracture morphology of CFRP was observed by scanning electron microscopy (SEM) to explore the mechanical strengthening mechanism of IMF on composites. Through this study, it is anticipated that an available IMF filler could be developed for the preparation of EP composites. It is expected that the EP composites would provide a potential option for automotive applications, such as inner door panels, luggage compartments, and inner lining panels.

2 Experimental procedure and preparation method

2.1 Raw materials

The EP used in this experiment is a bisphenol-A E-51 epoxy resin (Yehao Co., Ltd. WX, China), which is widely used as a matrix for the CFRP by its high adhesive strength and good processability (28), and the carbon fabric is selected as T300 grade. The *Indocalamus* leaf is plucked as the source of the IMF (HNC, China). Broad-leaved *Indocalamus* leaf is a genus of *Indocalamus* bamboo of the family Gramineae, and it often grows in the forest understory or mountain slopes, as shown in Figure 1.

2.2 Preparation of fiber

The preparation process of IMF is shown in Figure 2. The fresh *Indocalamus* leaves were gone through a series of

Figure 1: Image of *Indocalamus* leaves.

wash-treatment processes to make their surface clean. Then, the leaves were dried after baking, as shown in Figure 3a. The dried *Indocalamus* leaves were repeatedly broken in a crusher, and the fine chips were selected through a sieve. Subsequently, the planetary ball mill was used for 5 h to ground the leaves into powders.

The *Indocalamus* powder was repeatedly soaked for 2 h in 5 wt% NaOH solution to dissolve lignin and hemicellulose. After alkali soaking, the powder was repeatedly washed with water, and then, the powder was put into the oven to dry. Finally, they were picked up after the sieving process, as shown in Figure 3b.

2.3 Preparation of EP composites

Figure 4 shows the preparation process of IMF-reinforced EP composites (IMF/EP). First, a proper amount of IMFs was ultrasonically dispersed into 50 mL of acetone solution, and then they were mixed with a little EP liquid. Afterward, the mixture was put into the vacuum-drying oven to remove the air bubbles. Then, the well-dispersed IMF/acetone solution was added to the EP liquid to obtain the IMF content from 0 to 25 wt% under ultrasonic stirring for 1 h at a power of 40 kHz. The mixture was put into the oven to remove the acetone before the curing agent was added proportionally. And then, the mixture was poured into the pre-prepared mold. Finally, the EP samples were cured and cooled to room temperature.

2.4 Preparation of CFRP

The vacuum molding process was used to fabricate the CFRP. The carbon fiber (CF) unidirectional fabric was selected as the reinforcement, and EP was fluidly immersed into the fabric. And then, the CF prepreg was prepared through a series of processes. Afterward, the CFRP laminate was prepared by a unidirectional lay-up process, and the multilayer unidirectional prepreg was cured under 0.5–2 MPa pressure using vacuum compressive equipment, and finally, the CFRP laminates were obtained after they were cooled down to room

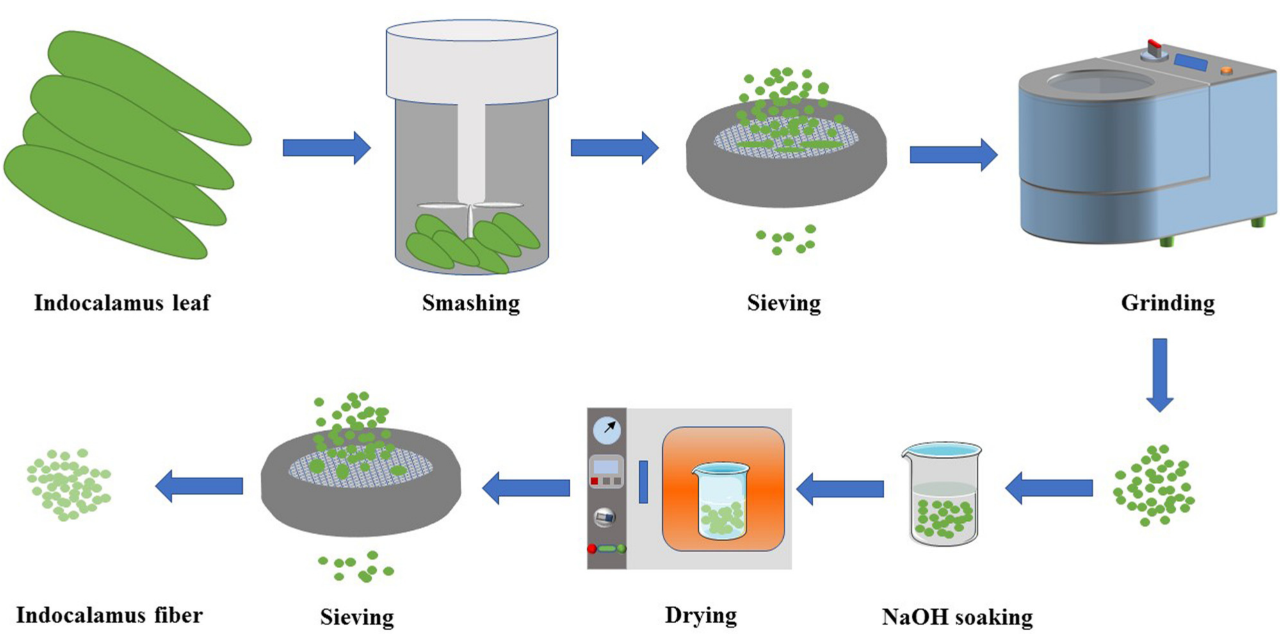

Figure 2: Preparation process of the IMF.

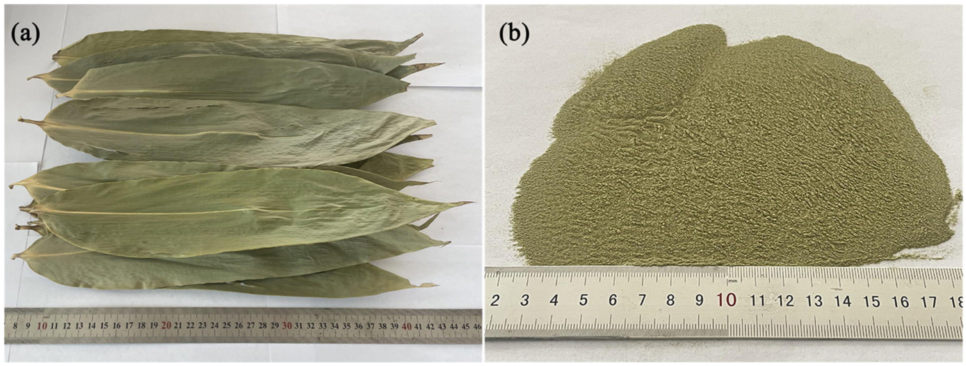

Figure 3: Macroscopic morphologies of (a) *Indocalamus* leaves after drying and (b) IMFs.

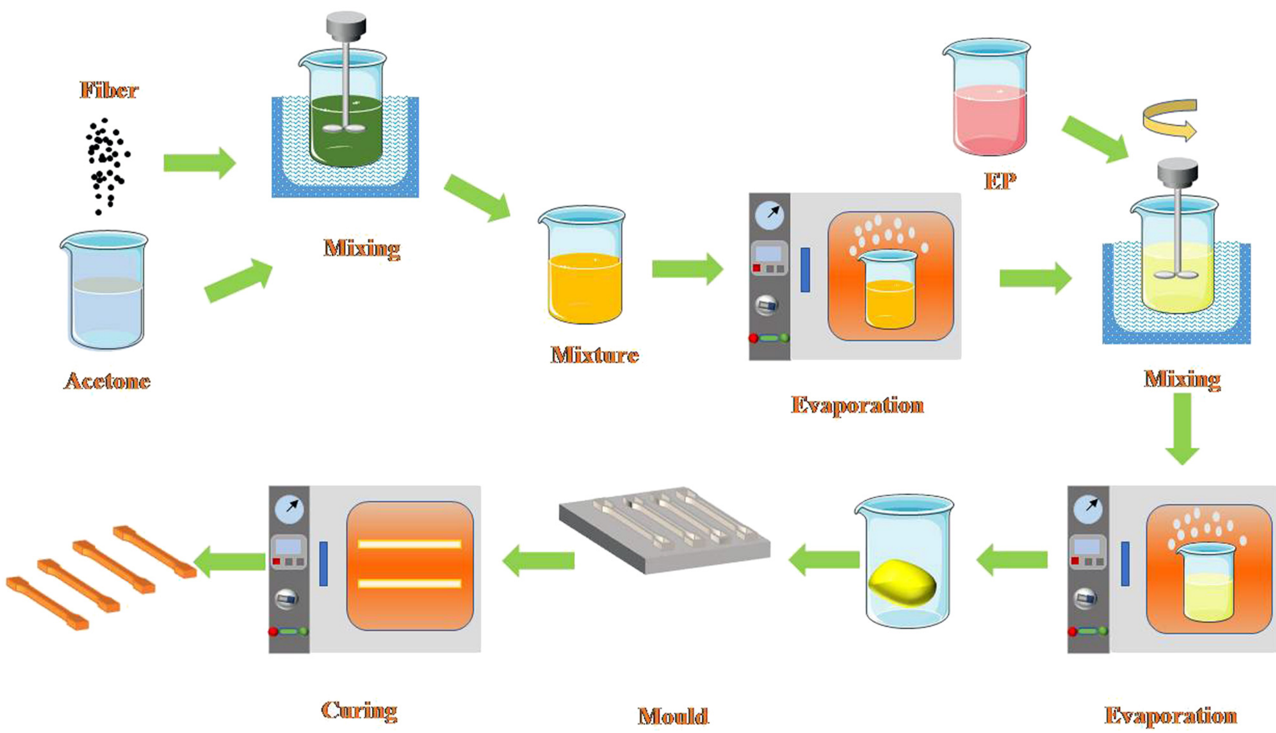

Figure 4: Preparation process of IMF/EP composites.

temperature. Figure 5 shows the local image of the CFRP laminates.

2.5 Characterization and mechanical test

Fourier transform infrared spectroscopy (FTIR, Thermo Scientific, MA) was used to test the infrared spectra of the samples. The scanning range was between 500 and 3,700 cm^{-1} with a resolution of 4 cm^{-1}, and the infrared spectra were plotted after the measurements were completed.

The tensile test of the EP sample was performed on a universal testing machine (MTS810, USA) at a speed of 2 $mm{\cdot}min^{-1}$, according to GB/T2567-2008. The typical tensile specimens are shown in Figure 6. The tensile test of the CFRP was referred to ASTMD 3039. The three-point bending test was conducted on a universal testing machine with a bending test speed of 2 $mm{\cdot}min^{-1}$ according to ASTM D7164. To ensure the accuracy of the data, five specimens were tested and the average value was calculated.

SEM (VEGA3, TESCAN) with an energy spectrometer (EDS, Link-ISIS) was used to observe the microstructure and the morphology of IMF under an acceleration voltage

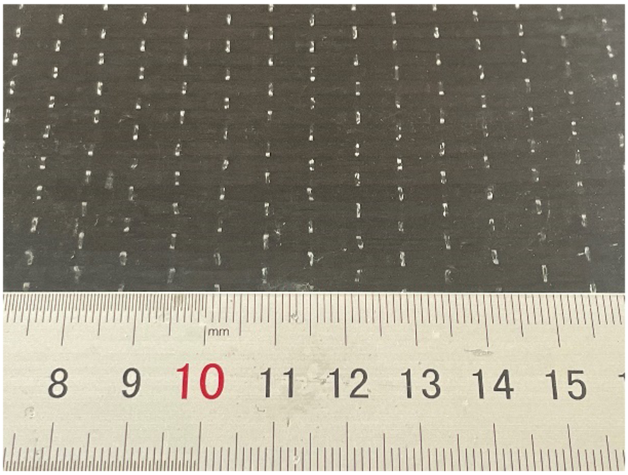

Figure 5: Macrograph of the CFRP.

of 10–15 kV. The fracture morphologies of epoxy and CFRP after the tensile test were observed using SEM. Before the SEM observation, the samples were sprayed with the conductive metal layer.

3 Results and discussion

3.1 Microstructure of the IMF

After the extraction treatment, *Indocalamus* leaf has variable forms, such as micro flakes, fine granules, and micro/nanofibers. The IMF has wide variable diameters ranging from a few hundred nanometers to several micrometers. Figure 7a shows the SEM images of a typical IMF with a diameter of about 15 µm. Figure 7b exhibits that the IMF are rough surface with many nanosized bumps. This is similar to the observations on alkali-treated natural fiber reported by some previous researches (29–31). The rough surface morphology and higher surface area allow for better interfacial interaction and mechanical interlocking between the natural fibers and the polymer matrix, resulting in superior mechanical properties of the composites (29–31). Therefore, it is inferred that the rough surface of the IMF would promote good interfacial properties of the EP composites in this study.

3.2 Tensile properties of EP composites

Figure 8 shows the typical tensile curves of EP composites with different contents of IMFs. It can be seen that the strength and modulus of the EP composites increased as the IMF content increased from 0% to 20%, but on further increase in the IMF content of 25%, there was a decrease in the strength and modulus of the EP composites. In other words, the tensile strength and modulus reached the maximum value at 20 wt%. The mechanical properties of the EP composites do not continuously increase with the increase of filler content. Similar experimental results were found on EP/cornstarch composites (32). The strength and elastic modulus increased first and then decreased when the starch content increased from 0 to 10 wt%, and they reached maximum values at a starch content of 2.5 wt%, which were 9% and 33% higher than those of neat EP, respectively. The enhancement effect might be due to the mechanical properties of starch, dispersibility, and interfacial interaction (32).

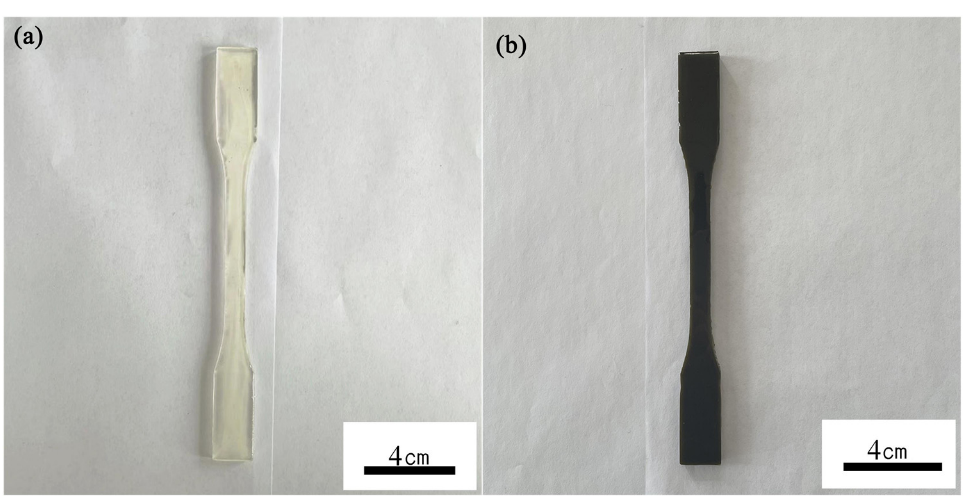

Figure 6: Macrograph of (a) neat EP and (b) IMF/EP composites.

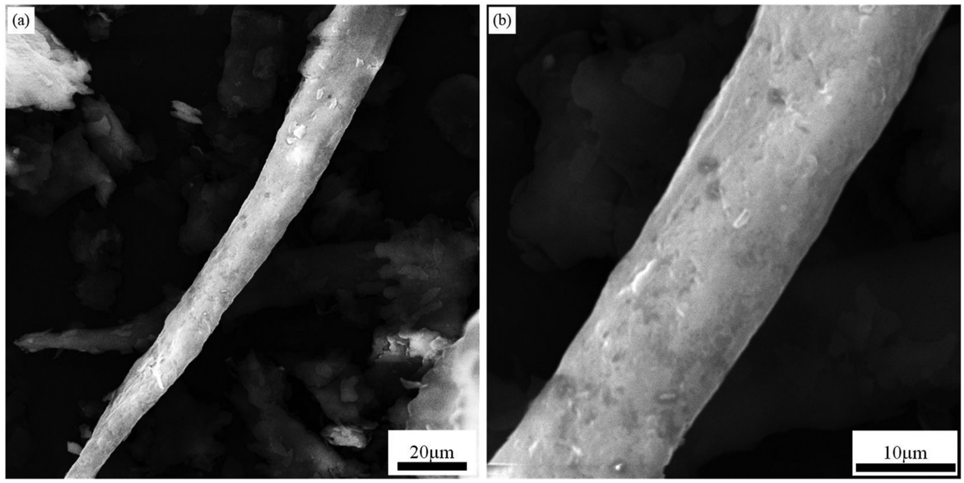

Figure 7: SEM images of IMFs: (a) low and (b) high magnification.

The tensile strength of resin-natural fiber composites has changed irregularly, depending on the fiber type, fiber content, and resin/fiber interfacial properties, e.g., agave is approximately tripled the tensile strength at 15 wt%. Wheat straw, wood charcoal powder, bamboo, and rattan bamboo slightly improved the tensile strength of polyester composites (33). In this study, compared with the neat EP, the tensile strength and tensile modulus of the EP composites are increased by 16.6% and 12.2%, respectively. It is generally believed that alkali-treated BFs could enhance the mechanical properties of plastics, which are mainly attributed to two aspects (34–36): first, alkali treatment can remove impurities from the surface of BFs and rearrange the fiber filaments along the tensile direction to increase their tensile strength. Second, alkali treatment could separate the fibers into proto-fibers and facilitate the dissolution of hemicellulose as well as lignin and provide good compatibility between the BF and the substrate. Therefore, the incorporation of high-performance BFs into the polymer matrix improves its energy absorption for dissipating applied loads.

Figure 9 shows the tensile fracture morphology of the neat EP. The fracture surface is relatively flat. There are several crack striations and a few short crack branches are growing along the initial matrix crack. These results indicate that the neat EP is a highly brittle fracture and has low resistance against the crack extension (37). The flat and smooth fracture morphology of the neat EP was also reported in the previous study (32), and it was found that the fractures were neatly arranged on the fracture plane, which shows a typical brittle fracture characteristic for neat EP.

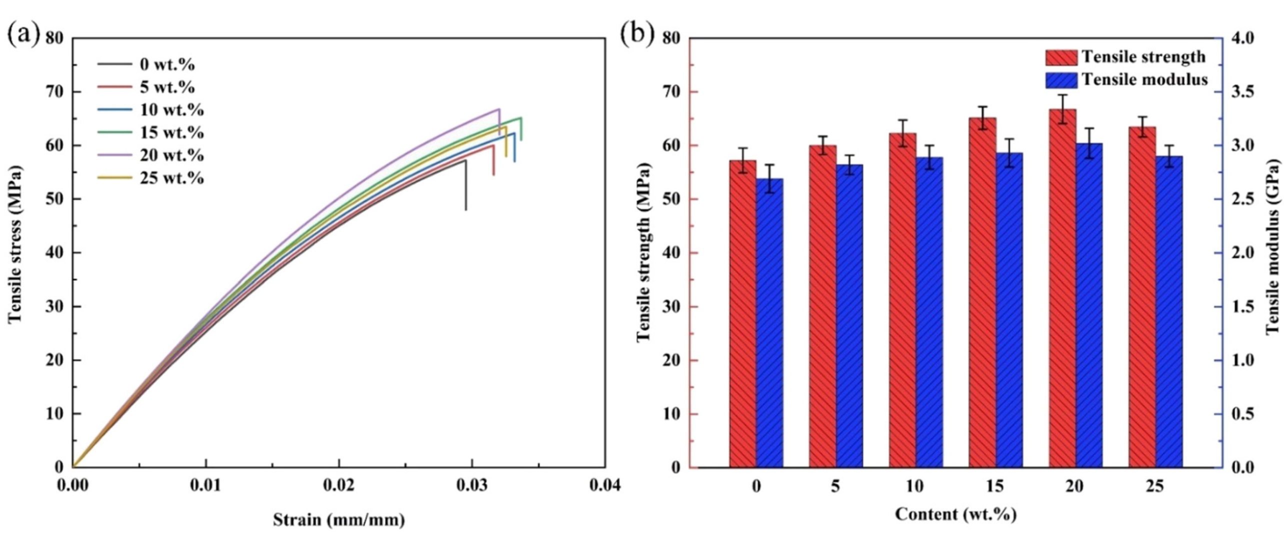

Figure 8: Effect of IMF content on tensile properties: (a) tensile stress–strain curve and (b) tensile property.

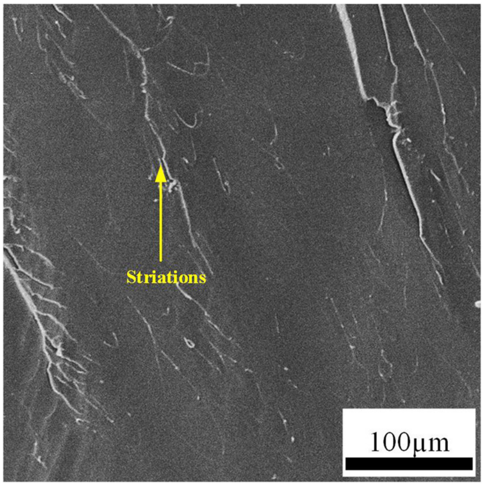

Figure 9: Tensile fracture morphology of neat EP.

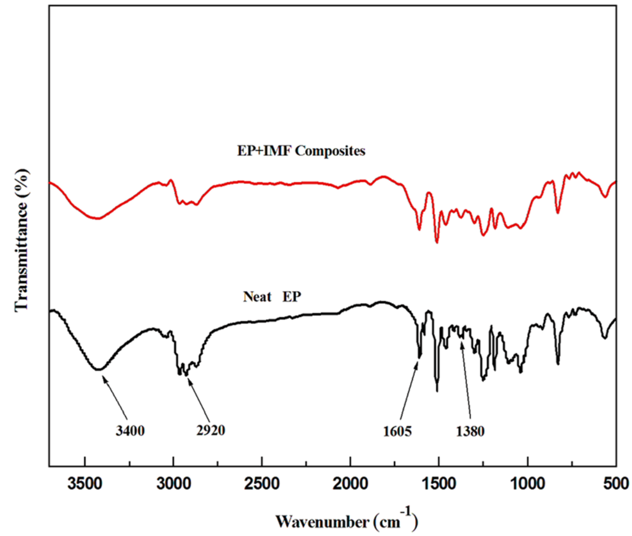

Figure 11: Infrared spectroscopy of neat EP and IMF/EP composites.

Figure 10 shows the tensile fracture morphology of the EP composites. It can be seen from Figure 10a that the fracture surface is rough and uneven. There are many crack branches and some tortuous crack paths are expanding. As shown in Figure 10b, the IMFs with a diameter of a few hundred nanometers are pulled out from the EP matrix. The fracture morphology reflects the role of the filler on the mechanical properties of the composites. A typical case is that pineapple leaf fiber-reinforced composites exhibit lower tensile strength because of the presence of voids in the composites (34), but the microscopic fracture of the EP/cornstarch particle composite exhibits that the particles induce microcracks initiation in the EP matrix, and many localized shear-type stepped failures in the EP matrix mean more energy would be dissipated during the tensile process (32). In this study, the microscopic observation results on the fracture morphology indicate that the IMF impedes crack expansion in the forms of crack deflection, crossover, and twisting, reducing the stress concentration.

3.3 FTIR result of the EP composites

Figure 11 shows the infrared spectra of the neat EP and IMF/EP composites. It can be seen that the broad peak around 3,400 cm^{-1} belongs to the stretching vibration peak of the –OH group, and the peak at 2,920 cm^{-1} is the C–H bond; the peak of 1,605 cm^{-1} denotes the C=C bond, and the peak at 1,380 cm^{-1} is related to the –OH bending vibration of the carboxyl –COOH group (38–40). Although some

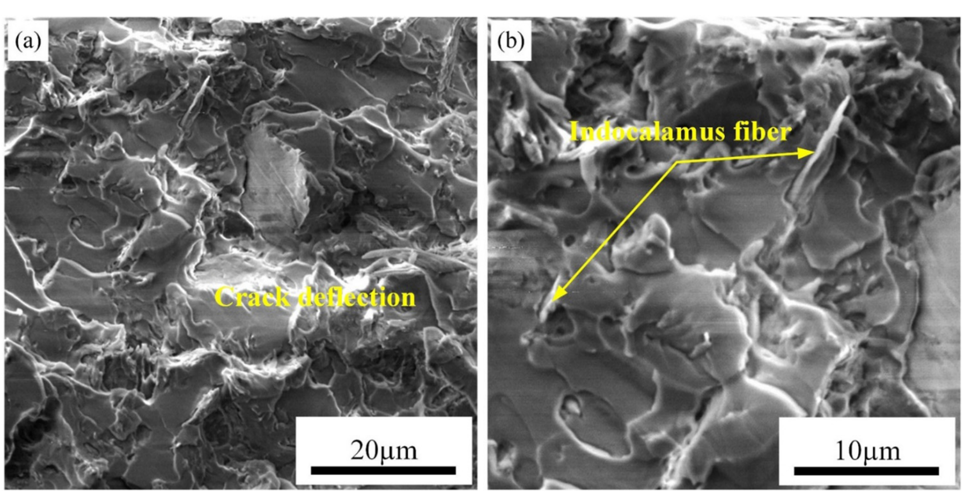

Figure 10: Tensile fracture morphology of IMF/EP composites: (a) crack deflection and (b) pull-out of the IMF.

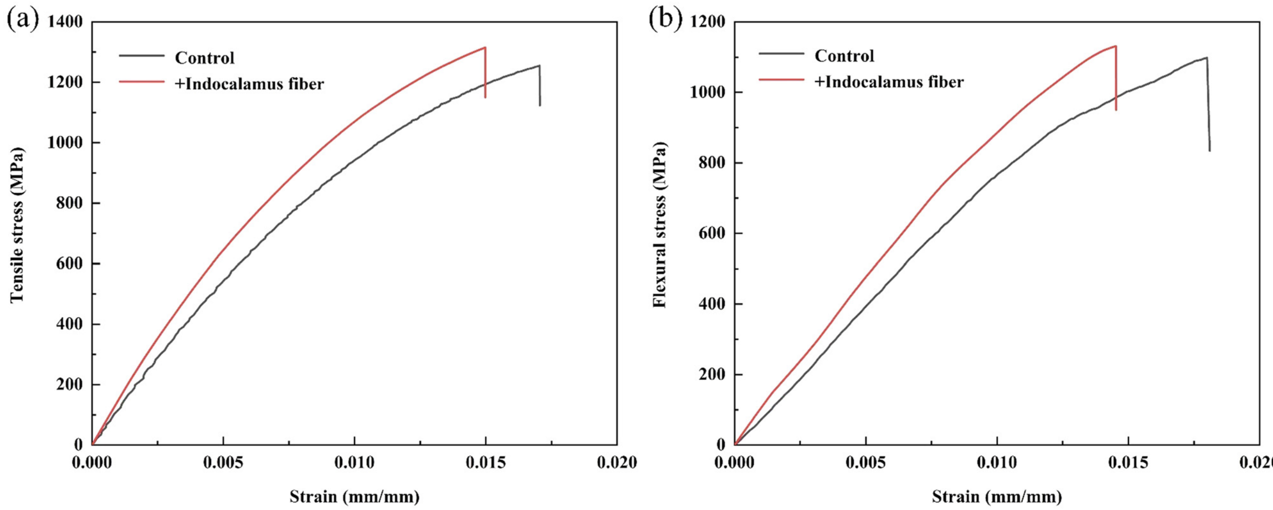

Figure 12: Mechanical curve of the CFRP: (a) tensile stress–strain curve and (b) bending stress–strain curve.

peaks have different intensities, the spectrum of IMF/EP composites is almost identical to that of neat EP. This indicates that the addition of IMF does not produce new functional groups in the EP matrix.

3.4 Mechanical properties of CFRP

Figure 12 shows the mechanical curves of the CFRPs. It can be concluded from Figure 12a that compared with the control CFRP, the IMF/CFRP are 4.78% and 1.65% higher in tensile strength and tensile modulus, respectively. Previous studies have shown the complex effect of micro/nanofiller on the tensile properties of long fiber-reinforced EP composites. It was found that, although there is an increased tendency in tensile modulus of CF/EP composites filled with micro/nano-bamboo fibrils (15) or microfibrillated cellulose (41), the increased value in tensile strength is not significant (15,41). For kenaf fiber-EP composites, the addition of 2 vol% cellulose filler enhances the tensile strength by 45% because the filler improves the bonding between fiber and resin (42). It also can be seen that the bending strength and bending modulus of IMF/CFRP are increased by 3.01% and 0.95%, respectively, as compared with those of control CFRP. Kaliappan et al. found that when 40 vol% kenaf fibers were added into neat EP, the flexural strength increased by 30%. And further addition of 2 vol% cellulose filler enhances the flexural strength by 44% for the kenaf/EP composites (42).

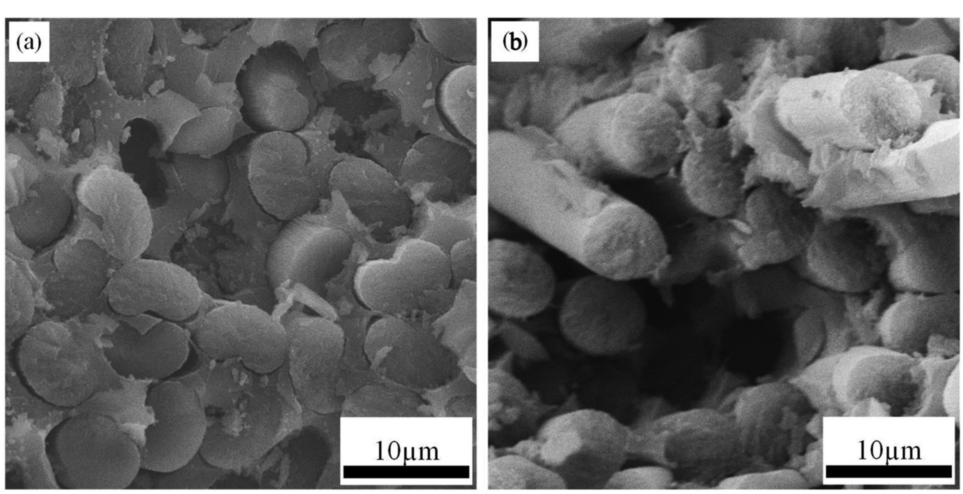

Figure 13: SEM fracture morphology of: (a) control CFRP and (b) IMF/CFRP.

It is indicated from Figure 12 that the addition of IMF slightly enhanced the mechanical properties of the composites. This is mainly attributed to the high interlocking density facilitating load transfers from EP matrix to fiber (43). It is also reported that fillers aid in obtaining higher mechanical and thermal properties in the fiber-reinforced composites because fillers increase the adhesion between fiber and matrix (44,45).

Figure 13a shows the fracture morphology of the control CFRP. It can be seen that the fracture surface is relatively smooth. The failure characteristics observed on the tensile fracture surface contain the fracture of EP, interfacial debonding, and a few pull-out of carbon fiber, displaying typical brittle fractures. Figure 13b shows the fracture morphology of IMF/CFRP. It can be seen that the fracture surface is rougher. Various fracture modes are also observed, including the fracture of EP, interfacial debonding, and pull-out of carbon fiber. On the fracture surface of IMF/CFRP, EP is obviously adhered to the fiber surface, indicating that the interfacial adhesion of the composites has been improved (46). Besides, the previous study confirmed that the micro/nanofibril would fill up the gaps between fiber and polymer matrix, resulting in a strong interfacial adhesion (46). Under the action of high interfacial adhesion, stress can be effectively transferred from the polymer matrix to the fibers, which provides a benefit to mechanical properties.

From this study, the IMF filler is beneficial for the mechanical properties of EP and its CF composites. The IMF is considered to be a green and renewable resource with promising applications because of its abundant sources and low energy consumption. IMF is environmentally friendly, easy to obtain, low cost, low density, and high specific strength, and if it is used as filler in resin-based composites, it would not only save energy and protect the environment but also improve the performance and promote the automobile application of the composites.

4 Conclusion

The EP and CFRP composites were modified by adding IMFs from natural *Indocalamus* leaf treated with micro-grinding and alkaline soaking. The addition of IMF significantly improved the tensile strength and modulus of the EP. As the content of IMF is increased from 0 to 25 wt%, the mechanical properties of the EP first rise and then decrease. And the mechanical properties of EP composites reach the optimized values when the MIF content was 20%. The addition of IMF slightly improves the mechanical properties of the CFRP. Compared with those of control CFRP, the tensile strength and tensile modulus of IMF/CFRP are increased by 4.78% and 1.65%, respectively; and the flexural strength and flexural modulus are increased by 3.02% and 0.95%, respectively. This is attributed to an improving interfacial bonding between the EP and IMF.

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