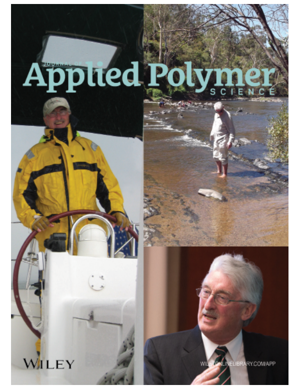
JOURNAL OF Applied Polymer SCIENCE

Special Issue: Sustainable Polymers and Polymer Science Dedicated to the Life and Work of Richard P. Wool

Guest Editors: Dr Joseph F. Stanzione III (Rowan University, U.S.A.) and Dr John J. La Scala (U.S. Army Research Laboratory, U.S.A.)



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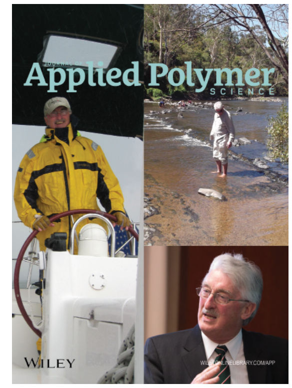

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Evaluation of the emulsion copolymerization of vinyl pivalate and methacrylated methyl oleate
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Mechanical properties of composites with chicken feather and glass fibers

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ABSTRACT: Chicken feather fibers (CFFs) have potential application in light weight composites. We investigated the physical properties of epoxy polymer composites reinforced with CFFs and glass fibers. CFFs or hybrid fiber (glass fiber and CFFs) composites reduced the density upto 30–40% when compared with glass fiber reinforced composites. The CFF composites has a storage modulus of about 3.5 GPa and a flexural strength of about 50–80 MPa. The hybrid fiber composite has better mechanical properties than CFF composites while having increased bio-based content. This study demonstrated a new way to utilize CFFs. © 2016 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2016**, *133*, 44013.

KEYWORDS: composites; fibers; mechanical properties

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INTRODUCTION

In the last decade, the development of sustainable bio-composite materials has become more and more attractive.[1–11] In particular, some bio-composites were made with chicken feather fibers (CFFs) and poly(lactic acid) or polypropylene recently.[12,13] CFFs are relatively new when compared with other natural fibers. The U.S. poultry industry produced 58.3 billion pounds of poultry in 2008.[14] The production of feathers is about 3.5 billion pounds per year in the U.S., given 6% of a bird's weight is feathers.[15] Most of the feathers are buried in landfills. The use of CFFs in composites offers an environmentally benign solution for feather disposal and also benefits the poultry industry by turning waste into a useful by-product. Past study showed that soy oil resins and CFF composites had potential applications in electronic devices, such as, printed circuit boards.[6,16]

Many different properties of CFFs have been investigated.[9,17,18] Feathers can be separated into two types of microcrystalline structure, barbs and rachis (see Figure 1) by some mechanical processes, such as grinding and air separation.[19] Only the barbs were used for composite in this study because the rachis is bulky and becomes particles after the grinding.[19] The CFFs obtained from the mechanical process have short length (<5 mm), i.e., a low aspect ratio (*l*/*d*, length divided by diameter). The low aspect ratio and the intrinsic structures of CFFs result in relatively weak mechanical strength. In general, it is not possible to use short CFFs to achieve high level of reinforcement attainable with long fibers. In practice, however, short CFFs are still used in composite materials because of their easy processability, low manufacturing cost, and sustainability. Shettar *et al.* studied CFFs reinforced polyester composites and found that bending strength and impact strength were increased with added CFFs. However, in their report, CFFs decreased the hardness and the tensile strength.[20]

In this study, we also utilized CFFs in composite application. Although CFFs showed relatively weak reinforcement as compared to glass fibers, it still provided some degree of thermal rigidization to the resin and did not negatively affect the mechanical properties of composite. More importantly, it increased the bio-content and decreased the density of composites. We fabricated the composites using the standard epoxy resin as the matrix material and CFFs as the reinforcement. The density, dynamic mechanical properties, and flexural properties of the composites were investigated. The properties of the composites made from the common E-glass fibers style 7628, were also studied. As a comparison, the properties of a composite made from bio-resin CB4-30 and CFFs were investigated as well.

EXPERIMENTAL

Materials

CFFs were obtained from Featherfiber Corp. (Nixa, MO). Woven E-glass fiber style 7628 was supplied by JPS Composite

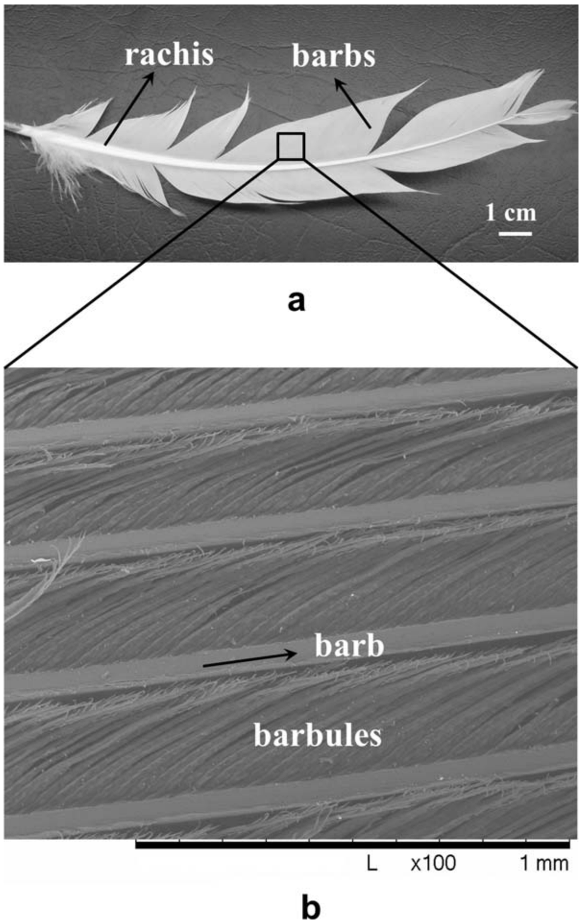

Figure 1. The branched structure of a chicken feather. (a) Digital photo of feather consisting of rachis and barbs and (b) low resolution SEM image of chicken feather barbs with barbules.

Materials (Statesville, NC) and was used as received. The diameter of single filament of E-glass is about 10 μm. EPON 862 epoxy resin (diglycidyl ether of bisphenol-F, epoxy equivalent is ~70 g/equiv) and curing agent Epikure W were obtained from Hexion Specialty Chemicals, Inc. (Houston, TX). CB4-30 bio-based resin was provided by Cara Plastics (Newark, DE). The CB4-30 resin is the mixture of 70 wt % acrylated triglyceride and 30 wt % styrene. *N-tert* butyl peroxybenzoate (Aldrich, Milwaukee, WI) was used as an initiator for the CB4-30 bio-resin.

Sample Preparation

TGA study at 50 °C showed that the weight of CFFs remained almost unchanged after 150 min.[21] To ensure the fibers were dry before use, CFFs were dried at 50 °C for 4 h to remove moisture and then were conditioned in a desiccator. For composites with EPON epoxy resin (weight ratio of EPON 862 to Epikure W was 100 to 26.4) and CFFs, various amounts (11–69%, all fiber contents in this report are volume basis.) of CFFs were mixed with the resin. The mixture was spread onto a mold which was then closed. To make composites with short glass fibers, glass fibers were cut to 10 mm long. Then the composites were made similarly with CFF composite. For composites with E-glass 7628, the fabric was cut to the size of the mold and eight layers of the fabric were used which made the final composites with a thickness of about 1.5–2.0 mm. For composites with hybrid fibers, one layer of E-glass 7628 was placed on each side of the fiber lay-up and around 29% CFFs were placed in between the glass fiber fabrics, making it a sandwich structure (see Figure 2).

All resins or resin/fiber mixtures were degassed to remove air bubbles before curing. The mold was then compressed using a hot press with various pressures in order to obtain composite panels with different fiber contents. Composites with CB4-30 resin were cured by free-radical polymerization with 1.5 wt % *N-tert* butyl peroxybenzoate as an initiator at 90 °C for 2 h, followed by a postcure at 120 °C for 2 h. Composites with EPON 862/Epikure W were cured at 120 °C for 4 h. The cured composites were then cut for various tests.

The nomenclature of the composites is listed in Table I.

Characterization

Apparent Bulk Density of CFFs and Composites. Due to the hollow structure of CFFs, we only measured the apparent bulk density, which was the density of the keratin protein including the air inside the voids. The apparent bulk density was measured according to ASTM C128-04a. A gravimetric method was used because of the tendency of fibers to float. An amount of 100–350 mg CFFs was placed in 100% ethanol in a pycnometer and air bubble was removed in vacuum. Then the pycnometer with CFFs was refilled with ethanol and subsequently weighed. All measurements were conducted at room temperature. Ten samples were measured and averaged.

The bulk density of the composites was measured according to ASTM C127. A minimum of three samples from each composite panel were measured. The matrix density was measured on samples of pure EPON 862/Epikure W which was cured simultaneously beside the composites.

The measured densities of matrix and CFFs were used to calculate the fiber volume fractions of the composites (assuming the void content is zero).

Dynamic Mechanical Analysis. Dynamic mechanical analysis (DMA) was performed using a Mettler-Toledo DMA/SDTA861e (Greifensee, Switzerland) according to ASTM D5023. A three-point bending fixture was used for the test. The dimensions of the samples for the DMA test were $70 \times 13 \times 2$ mm^3. The samples were cooled by liquid nitrogen and then heated from

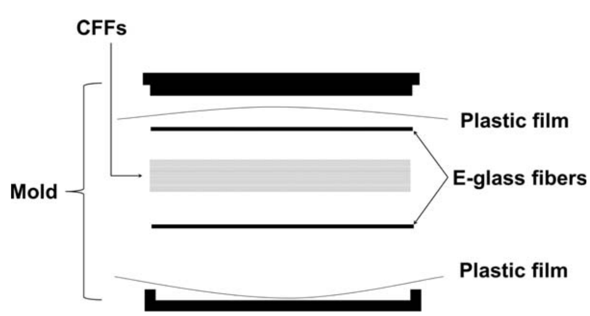

Figure 2. Mold setup for preparing EPON/Hybrid composites. The other composite panels were made using the same mold but with different fibers. The plastic films were for mold release.

Table I. Resins and Fibers Used in This Study and the Corresponding Fiber Volume Fraction

Composite designation	Resin	Fibers	Fiber content (vol %)
EPON/CFF	EPON 862	CFFs	Various (11–69%)
EPON/Short	EPON 862	Short glass fibers (10 mm)	29.0
EPON/7628	EPON 862	Glass fabric	48.7
EPON/Hybrid	EPON 862	Glass fabric and CFFs	glass 11.5 CFFs 28.9
CB4/CFF	CB4-30 bio-resin	CFFs	30.3

10 to 150 °C at a heating rate of 2 °C/min and a frequency of 1 Hz. Strain sweep tests were performed to determine the linear elastic region of the composites and matrix. A strain of 4×10^{-4} (displacement of 100 μm) was used according to the strain sweep tests, which fell into the linear elastic region.

Flexural Properties. The flexural tests were performed using an Instron 4201 (Norwood, MA) at room temperature with a three-point bending fixture (Wyoming Test Fixtures, Inc., Salt Lake City, UT) according to ASTM D790. The crosshead speed of the tester was set based on the support span length and sample thickness. The dimensions of the samples for the flexural tests were $70 \times 25 \times 2$ mm^3.

RESULTS AND DISCUSSION

Bulk Density of CFFs

CFFs contain a layer of fat on the surface.[22] To overcome the issue of feather wettability, ethanol with a known density was used instead of water to reduce the hydrophobic nature of feathers.[19,22] The measured density of CFFs was 1.01 ± 0.02 g/cm^3. Barone and Schmidt used 0.89 g/cm^3 to predict the densities of the composites.[23] They found that the measured densities of the composites were higher than the predicted values, which suggests that they underestimated the density of the CFFs. Kock also found that the density of feather fibers varied from 1.0 to 1.2 g/cm^3 depending on the sources.[24]

CFFs contain a significant amount of internal voids.[25,26] Wool (reported density as 1.28 g/cm^3 or 1.31 g/cm^3 [27,28]) is also a keratin material; however, it has virtually no internal voids. Assuming similar density of keratin from wool and CFFs without internal voids, the estimated volume fraction of voids inside CFFs was 22%.

Table II. Density of Different Fibers[29–31]

Fibers	Bulk density (g/cm^3)
CFFs	1.01 ± 0.02
Jute	1.3
Sisal	1.3
Flax	1.5
Hemp	1.48
Pineapple	1.56
E-glass fiber	2.5

The density of CFFs is the lowest when compared with the densities of other natural fibers (Table II). Jute, sisal, flax, hemp, and pineapple fibers are plant fibers and composed of cellulose, which forms rod-like crystalline microfibrils.[32] These microfibrils are highly packed; thus, the plant fibers have higher densities than CFFs. The density of CFFs is only 40% of that of the commercial glass fibers widely used in composite materials. Consequently, the CFF reinforced polymer composites can have lower overall densities than glass fiber reinforced polymer composites.

Density of Composites

The densities of natural fibers are lower than glass fibers. Thus, the natural fiber reinforced polymer composites have lower densities than glass fiber reinforced polymer composites. Figure 3 shows the densities of the CFFs and glass fiber reinforced polymer composites. The density of the neat resin was 1,198 kg/m^3, which is consistent with the reported value (1,200 kg/m^3).‡ With the addition of CFFs, the density of the composite is decreased. The theoretical density of EPON/CFF composite can be calculated by:

$$\rho = v_f \rho_f + \left(1 - v_f\right) \rho_m = \rho_m + \left(\rho_f - \rho_m\right) v_f \quad (1)$$

where ρ is the density, v is volume fraction, the subscript f denotes fiber and m denotes matrix. The equation indicates that the density of the glass fiber reinforced composites increases as the fiber volume fraction increases because $\rho_f > \rho_m$. In contrast, the density of EPON/CFF composite decreases as CFFs volume fraction increases because $\rho_f < \rho_m$. By applying linear regression, the density of EPON/CFF can be predicted using the following equation:

$$\rho = 1198 - 134.6 v_f \,(\mathrm{kg/m^3}) \quad (2)$$

The CB4 bio-resin has a lower density than the EPON epoxy resin; hence, the composites with the CB4 resin have even lower density. Although the density of EPON/7628 composites was higher than that of EPON/CFF composites at the same fiber loading, partially replacing glass fiber with CFFs (EPON/Hybrid composite) reduced the density significantly. The low density characteristic makes the CFFs composites have the great potential for light weight application.

‡Hexion, Product Bulletin SC:1183-02 EPIKOTE™ Resin 862/EPIKURE™ Curing Agent W. Hexion Resolution Performance Products LLC.

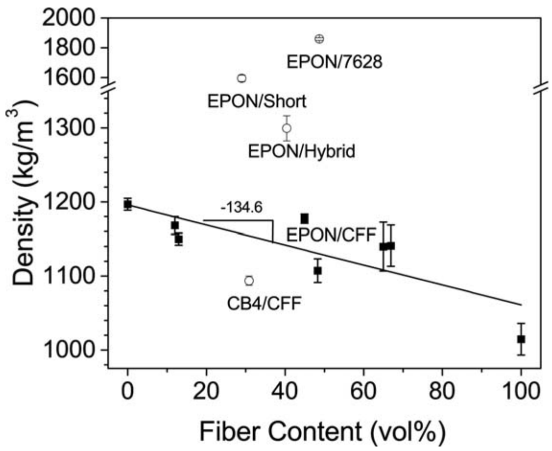

Figure 3. Densities of the composites. Filled squares: EPON/CFF composites; open symbols are indicated in the figure; solid line: linear fit of the EPON/CFF composites with a slope of −134.6. The error bars are the standard deviation of the measured samples.

Dynamic Mechanical Analysis

A good understanding of the storage modulus to temperature relation provides valuable insight into the material properties. DMA is highly sensitive to structure changes of the molecules, which in turn is adopted to study the molecular basis of the thermomechanical properties of materials. Figure 4 shows the storage moduli of the various composites. The modulus of the composite with 67% CFFs in the rubbery plateau was higher than that of the composite with 45% CFFs. In general, when fibers are incorporated into the matrix the stiffness of the materials increases due to higher stiffness of the fibers than matrix material; hence, a higher storage modulus is achieved. EPON/CFF composites also showed much higher storage moduli than CB4/CFF composites. Although CFFs are not as strong as glass fibers, it still provides thermal rigidization to the resin. This thermal rigidization can be seen in Figure 4 because the storage modulus of EPON/CFF increased with increased fiber content.

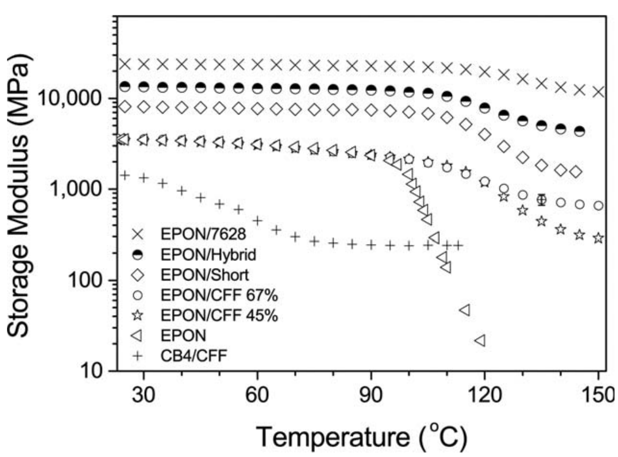

Figure 4. Storage modulus of the EPON/CFF and EPON/glass composites as a function of temperature. The error bar is a representative standard deviation for all samples.

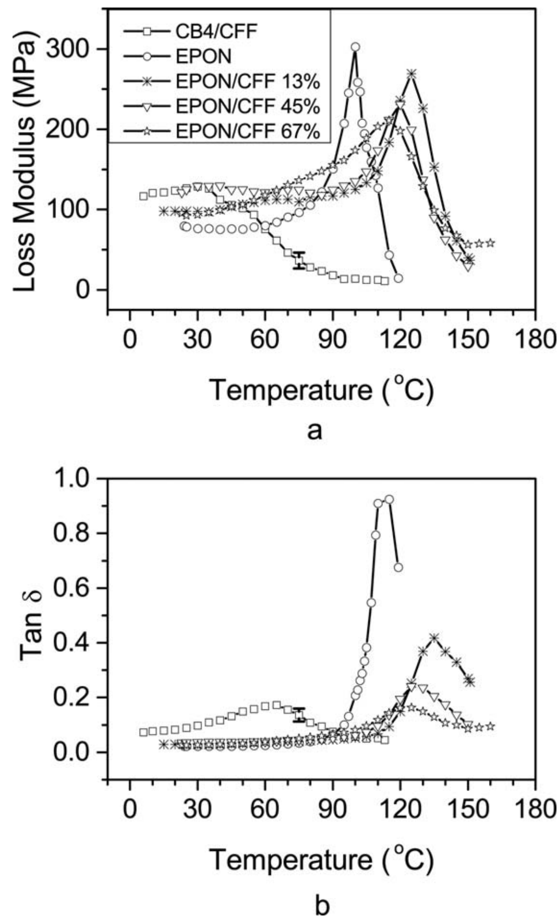

Figure 5. (a) Loss modulus and (b) tan δ of the EPON/CFF composites and CB4/CFF composite as a function of temperature. The error bars are representative standard deviations for all samples.

The reason that they still have much lower storage moduli than glass fiber reinforced composites are (a) CFFs have lower stiffness than glass fibers and (b) CFFs have lower aspect ratio than glass fibers. The average aspect ratio of CFFs is 200, while the aspect ratio of short glass fiber in this study is 1,000.[23]

Halpin and Tsai developed a model to predict the modulus of a composite consisting of randomly distributed short fibers with different aspect ratios[33]:

$$\frac{E}{E_m}=\frac{1+\xi\eta v_f}{1-\eta v_f} \tag{3}$$

$$\eta=\frac{(E_f/E_m)-1}{(E_f/E_m)+\xi} \tag{4}$$

where E is the modulus, v is volume fractions, the subscript f denotes fiber and m denotes matrix, respectively; ξ is a measure of reinforcement geometry (related to aspect ratio) which depends on loading conditions. When predicting longitude modulus of the composite, E_1, $\xi = 2l/d$; when predicting transverse modulus of the composite, E_2, $\xi = 2$.

Goettler and Lavengood[34] developed a rule-of-thumb expression for the modulus of a structure with random two-dimensional fiber orientation:

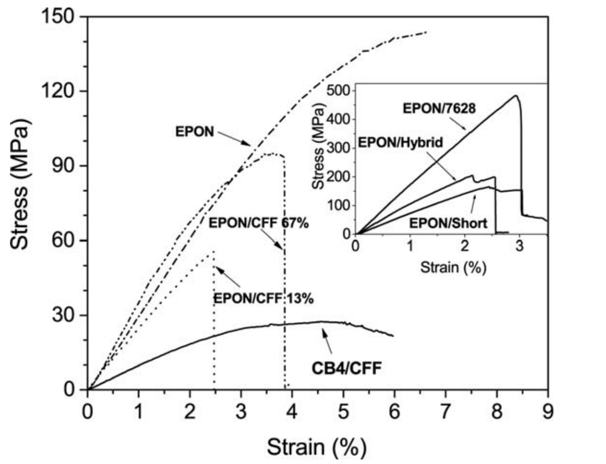

Figure 6. Flexural stress–strain plot of composites. The inset plot is the stress–strain of composites containing glass fibers due to different y-axis scale.

$$E=\frac{3}{8}E_1+\frac{5}{8}E_2 \tag{5}$$

where E_1 and E_2 are predicted by Halpin–Tsai equation.

The elastic modulus of CFFs is about 3.6 GPa, which was used for the calculation here.[18] With these numbers used in the Halpin–Tsai and Goettler equations, the calculated values of storage moduli at temperature range of 25–50 °C were within 8% error range of the experimental data. One of the reasons for this error is due to the heterogeneity of the CFFs, which in turn, affects the storage moduli of the composites.

If a higher modulus is desired, some of the CFFs can be replaced by glass fiber (EPON/Hybrid in Figure 4). The storage modulus of EPON/Hybrid composite (total fiber 40.4%) was 13.4 GPa at 25 °C, which was much higher than the value for EPON/CFF with a similar fiber fraction of 45% (3.4 GPa). The hybrid fiber composite has much better mechanical properties while having relatively significant amount of bio-based content. Given that most of poultry waste is disposed through burning and landfill, the use of CFFs in composites as filler could have cost advantage with increased biomass fraction.

Figure 5(a) shows the loss modulus of the composites. The peak of loss modulus decreased as the fiber loading increased. The peaks of the composites occur at a higher temperature than the pure EPON polymer. In addition, the peaks of the EPON composites were narrower than the CB4-30 bio-composite. This could be due to the lower molecular mobility of the EPON epoxy thermoset or because a wider distribution of monomers in the CB4-30 resin, which consists different triglyceride-derived molecules. Figure 5(b) shows the damping factor (tan δ) of the composites. Tan δ is related to the impact resistance of a material. The tan δ peak is associated with the dynamic percolating clusters existing near glass transition temperature T_g during the transition from the glassy state to the rubbery state.[35] The EPON polymer had the highest tan δ peak value, which indicates that it has a good molecular mobility; thus, better damping characteristics. The tan δ peak value decreases as the fiber volume fraction increases. The addition of CFFs hindered the mobility of the matrix molecules; thus, lowered it damping ability. The tan δ peak is also an indication of the glass transition temperature. The shift of the peak to higher temperatures showed that the composites had higher T_g than the neat EPON epoxy polymer.

Flexural Properties

Figure 6 shows the stress–strain curves of different composites. The EPON polymer and CB4/CFF composite was more ductile and did not break before the 5% strain limit. Both glass fiber and CFF reinforced EPON composites broke before 5% strain limit, indicating that the addition of fibers decreases the ductility of the materials.

Figure 7(a) shows the flexural strength of the EPON/CFF composites. The flexural strength was the highest with EPON thermoset. The CFFs do not show reinforcing effect for flexural strength of the composites. However, the extent of degradation was reduced with increasing amount of fiber addition once above a certain critical fiber fraction level, which is around 25%. The flexural strength subsequently increased with increasing fiber volume fraction. This common behavior was also found in many other composites, such as, cotton fiber reinforced polypropylene composites.[36] General composite theory regards composites as brittle-fibers and ductile–matrix system. If the fiber content is below its minimum volume faction, the stress on a composite may be high enough to break the fibers. These broken fibers can be treated as voids, which reduce the

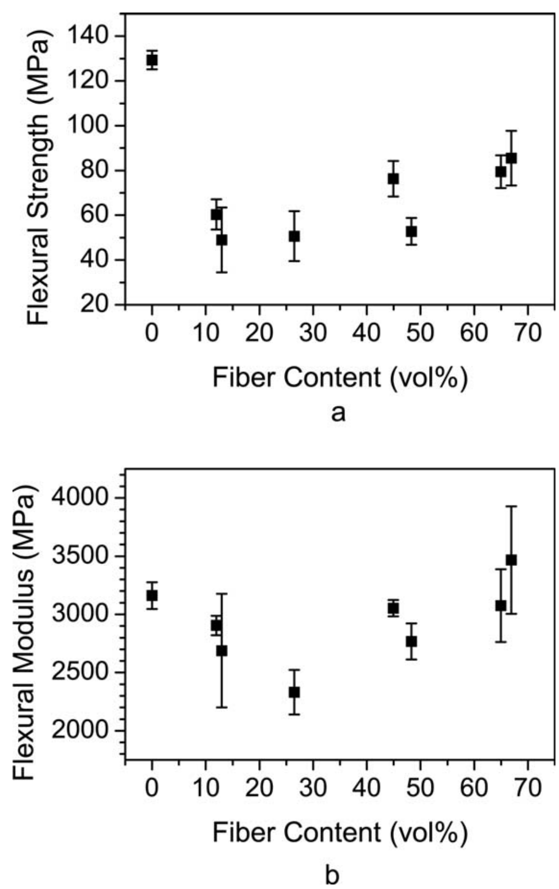

Figure 7. Flexural properties of EPON/CFF composite as a function of fiber volume fraction. (a) Flexural strength and (b) flexural modulus.

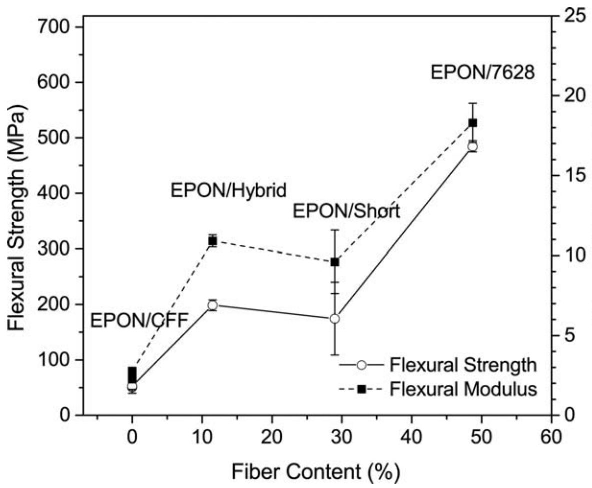

Figure 8. Comparison of the flexural strength and flexural module in different composites. Only glass fibers are treated as reinforcement in this figure. The CFFs are not included in the fiber fraction so that EPON/CFF has 0% fibers.

strength of the composites.[37,38] Similarly, the flexural modulus decreases initially but then increases with additional CFFs [Figure 7(b)], which was also reported for coir/glass fiber reinforced phenolic composite.[39]

The comparison of the flexural properties for different composites is shown in Figure 8. The composites consisting of glass fibers had higher values for both flexural strength and flexural modulus than the EPON/CFFs composites, due to the lower strength and low aspect ratio of CFFs. As discussed previously, the CFFs did not provide significant reinforcement. We therefore treated the CFFs as part of the resin here and only glass fibers were treated as fiber reinforcement in Figure 8. The EPON/Hybrid composite had a 300% increase in flexural strength and a 290% increase in flexural modulus than EPON/CFFs. It again shows that the hybrid fiber composite has better mechanical properties than EPON/CFF composites while having increased biomass content, which suggests a new way to utilize CFFs.

CONCLUSIONS

In this study, composite materials were developed from EPON epoxy, CFFs, glass fibers, and a mixture of CFFs and glass fibers (hybrid fibers). CFF or hybrid fibers composites reduced the density upto 30–40% when compared with glass fiber composites because CFF is composed of keratin protein with internal void. CFFs had a lower aspect ratio than glass fibers, which resulted in lower storage modulus of CFF composites. However, partially replacing CFFs with glass fiber (hybrid fiber composite) can increase the modulus and strength when compared to EPON/CFF composites. Overall, the light weight CFF and the hybrid fiber composites provides a new way to utilize CFFs and could have potential industrial applications, while having more bio-based content.

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