Experimental investigation on dynamic mechanical and thermal characteristics of Coccinia Indica fiber reinforced polyester composites

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Abstract
Due to superior material properties of fiber reinforced composites, they are utilized in many structural fabrications. Even though many studies have been reported about various fiber reinforced composites, it is indeed to find more eco-friendly composites for modern applications. So, developing the new fiber reinforced composites and revealing its mechanical properties are vital. In this examination, the natural fiber reinforced polymer matrix composite was prepared by compression molding method. The natural fiber named as Coccinia Indica was used to fabricate the fiber reinforced composites. The impact of different fiber length on dynamic mechanical properties like loss modulus, storage modulus, and loss of weight in fiber reinforced composites was predicted using dynamic mechanical analysis and thermogravimetric analysis. The outcomes revealed that fiber length of 30 mm shows better values in storage modulus and nominal loss modulus owing to higher interfacial bonding among fiber and matrix. However, in other fiber lengths, the storage modulus depicts poor result and high loss modulus is due to inefficient stress transfer.

Keywords
Polymer matrix composites, Coccinia Indica fiber, compression molding, dynamic mechanical, polyester, thermogravimetric analysis

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Introduction
Fiber reinforced polymer (FRP) contains a polymer matrix with high-quality fibers.1 The added material is called reinforcement, and where it is added is called a matrix.2,3 The final developed material is a composite material. The polymer can typically be differentiated by two classes, namely, thermoplastics and thermosetting.4 In addition, fibers can also be distinguished as natural and synthetic fiber.5,6 The use of fibers such as natural or synthetic as an enhancing element in polymer matrix makes the polymer valid for many applications.7,8 The characteristics of fiber reinforced composite called as

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fibrous composite substances are especially dependent upon the properties of fiber and its microstructural parameters such as diameter and length of the fiber,9,10 fiber propagation, fiber orientation, and fiber volume division.11–17

In recent decades, researchers are more intrigued to find the use of biodegradable materials, including natural fibers, because they are more durable, recyclable, and cheap. The different mechanical characteristics of naturally fiber reinforced composites (NFRCs) are tested and obtained as a replacement for synthetic fiber reinforced composites (SFRCs). Comparisons of different mechanical characteristics of NFRC and SFRC18 show that although synthetic composites are superior to NFRC, the environmental risk of SFRC disposal remains unpleasant. Bodros and Baley19 researched the natural fiber of *Urtica dioica* and confirmed that it was possible to produce eco-friendly NFRC with natural fiber. The bio-composites may be made using bio-polymer matrix natural fibers. It has been stated by Boopathi and colleagues20 that one of the natural fibers known as Borassus Fruit fiber is more resistant and a less dense fiber which is important to manufacture lightweight materials. The composite composition produced with Jowar fiber shows better strength compared to sisal and bamboo fiber composites, and the mechanical characteristics of natural fibers such as Jowar, sisal, and bamboo with polyester matrix are compared. In addition, the researchers assimilated the Jowar fibers as low density,21 which can be used to create lightweight composites. Holbery and Houston22 state that epoxy resin is commonly used in the manufacture of natural fiber composite as a matrix component and is widely used in the automotive industry. In addition, the author stated the high efficiency of the epoxy resin.

In the study by Sapuan and Maleque,23 which deals with the telephone stand made of a natural composite of banana fiber and epoxy resin, the authors found the natural composite to be a good environment friendly option and to be used for making low-cost domestic appliances. *Schumannianthus dichotomus* (Murta) fiber supported by epoxy resin composite has excellent mechanical and thermal property and has also stated that optimum fiber loading length and weight percentages are important for achieving the desired characteristics.24 It has been stated by Sreenivasan et al.17 that the composite properties are very much dependent on the fiber length to produce maximum productive results and that the fiber length must be at least equal to the critical fiber length (lc). Recent literatures indicate that natural fiber composites play a key role in the current era, and lightweight composites are made by using low-density natural fibers. From the detailed literature analysis, it is found that no work has been reported earlier on the selected natural fiber composite. Hence, it is planned to fabricate the biodegradable FRCs by mixing the natural fiber (Coccinia Indica (CI)) and synthetic (polyester) resin. In addition, in this research work, the dynamic mechanical and thermal characteristics of CI fiber reinforced composites were assessed on the effect of different fiber length for successful composite manufacturing.

**Materials and preparation**

The CI plants were collected from Chinna chettipalayam village, Erode district, Tamil Nadu, India, which is a medicinal plant under the family of Cucurbitaceae.25,26 Figure 1 depicts the digital image of extracted CI fiber. Figure 2 shows the scanning electron microscope (SEM) photograph of CI fiber reinforced composites (CIFRCs). The technical properties of the CI fiber were determined as per the ASTM standards and are presented in Table 1. The unsaturated polyester of required amount was utilized as a suitable matrix material,27 which depicts better properties in both liquid and cured states. The curing catalyst used in this work is methyl ethyl ketone peroxide (MEKP). The function of this catalyst in the composite is to speed up the binding process between the fiber and resin. The accelerator utilized in this experiment is cobalt naphthenate which is used to alter the chemical bonds and to speed up the chemical process. The composites were fabricated using standard compression molding process.28 The steel mold of size 300 × 300 × 3 mm was utilized for composites manufacture. The internal surface of mold was coated with wax polish (a layer of release agent) to enable ease of removing the polymer composites from the mold. The CI fibers were hewed into various lengths such as 10, 20, 30, 40, and 50 mm. During curing process, the compressive
force of 5 bars was applied on the material for about 24 h. The polyester was used as a matrix material, and 2% MEKP and 0.5% cobalt naphthenate were employed as catalyst and accelerator, respectively.

**Experiments**

**Dynamic mechanical analysis**

It is a generally employed technique to determine the temperature dependence properties like storage modulus (\(G'\)), loss modulus (\(G''\)), and damping factor (\(\tan \delta\)).\(^{29}\) The instrument DMA 6100 was utilized to perform the experiments. The fabricated CIFRCs were sectioned into desired (ASTM D4440) size of \(53 \times 13 \times 3\) mm and they were used for the analysis. The tests were conducted by using a three-point bowing framework. The CIFRCs were dried in a fixed frequency of 1.0 Hz and oscillation amplitude of 0.3 mm. Experiments were conducted in the temperature range of 35°C–185°C with a plummeted rate of heating value as 0.08°C/s.

**Thermogravimetric analysis**

To determine the thermal stability and physiochemical properties of fabricated composite, thermogravimetric analysis (TGA) was performed on the CIFRC samples.\(^{30}\) The test was conducted in the air atmosphere between the temperature ranges of 20°C and 400°C. In TGA, a change in thermal stability was evaluated with respect to loss in weight percentage as a function of temperature. In the study, TGA estimates weight loss in developed CIFRCs under a controlled atmosphere with respect to temperature (or time) to decide the thermal strength as well as composition. As a rule, the thermal investigation of materials is very much necessary to assess the desired technical characteristics.

**Results and discussions**

In this research, the dynamic mechanical properties of CIFRCs like loss modulus, storage modulus, and damping factor have been determined by utilizing dynamic mechanical analysis (DMA) studies. The storage module (\(G'\)) indicates a viscoelastic material’s rigidity and is proportional to the loading cycle strength. The loss modulus (\(G''\)) indicates that a substance can dissipate energy as heat due to viscous motions within it. The ratio of loss modulus and storage modulus is generally expressed as damping factor (\(\tan \delta\)).

**Effect of heat on \(G'\) of pure polyester and composites with different fiber length** is represented in Figure 3. It is identified from Figure 3 that in all the investigated samples, the storage modulus reduces with an increase in temperature. The pure polyester shows the most minimal storage modulus in all temperature range compared with the tested CIFRCs. It is inferred from the figure that the CI reinforcement can build the storage modulus of CIFRCs because of the stiffening effect of fiber with the matrix. Faruk et al.\(^1\) clearly mentioned that a lower L/D can increase the strength of the fiber owing to the presence of fewer defects on the fiber. Furthermore, it is revealed from the figure that the highest \(G'\) up to around 120°C is for the CI fiber length of 30 mm, and the lowest for the length of 50 mm. Also for the CI fiber length of 10 mm, the \(G'\) is lower than for the lengths of 20 and 40 mm, which is due to the distribution and orientation of the reinforcing fibers.\(^{28}\)

Generally, the DMA result has two discrete regions called as a glassy plateau and a rubbery plateau, which are defined based on the temperature. The region below 110°C is named as the glassy plateau (mobilization) and the region above 110°C is named as the rubbery plateau (plasticized). It is revealed from Figure 3 that the pure polyester

![Figure 2. SEM photograph of CIFRCs.](image)

**Table 1.** Physio-chemical properties of the CI fiber.

| Cellulose (%) | Hemicellulose (%) | Lignin (%) | Wax (%) | Ash (%) | Moisture (%) | Density (kg/m³) | Elongation % | Tensile strength (MPa) |
|---------------|-------------------|------------|---------|---------|--------------|-----------------|--------------|----------------------|
| 72.56         | 15.18             | 21.21      | 0.24    | 2.39    | 8.95         | 1298            | 4.25         | 71.5                 |
G′ is lower than the CIFRCs in the glass transition region. This is because of the free molecular mobility of the polymer chains which can break the cross-linking between the molecular chains. Furthermore, it is identified from the figure that the reinforcement of CI into polyester increased the G′ drastically. Among the various fiber lengths, 40-mm fiber length reinforced composite has shown higher G′ in the glassy plateau due to the higher interfacial bonding between the fiber and matrix. However, it plummeted steeply with the increase in temperature. Furthermore, it is noticed from Figure 3 that all the curves of samples seem to be merged after the glassy plateau temperature which may happen based on the softening interfacial effect at elevated heat.28

Figure 4 explains loss modulus (G″) results of pure polyester and fiber reinforced composites. CIFRCs containing fiber length of 10 to 40 mm shows fluctuating values of G″ upto the temperature of 80°C and beyond that temperature sharp fall in G″ were observed. This is due to free sub-atomic versatility in the polymer chains. The fiber length of 30 mm was established to provide the upper most values around 40°C. The variation in the presence of bonds among the fiber and matrix may be the possible reason for the obtained results.30

Figure 5 shows the damping factor (tan δ) of all the examined samples. The tan δ of pure polyester is higher than all the other samples in the glassy plateau because of more degree of freedom in the atomic level. Only a small difference in the tan δ values is found among all the fiber lengths. However, the nominal damping factor was observed at reinforced composites with fiber length of 30 mm, that is, critical length (l_c). The length of fiber within or beyond the critical length demonstrates insignificant enhancement and tan δ reduction due to quicker dissipation of energy. The obtained results are well in conformity by means of the results obtained by Sreenivasan et al.17

The thermal stability of pure polyester and natural fiber reinforced CIFRCs under loading of different fiber length was estimated from thermogravimetry analysis with respect to temperature (or time) in controlled environment. Figure 6 demonstrates the result of TGA in which no considerable deviation in the humiliation pattern of raw polymer was observed. It is envisaged from the figure that upto 400°C, the degradation curves of samples are sharp, which is due to water loading content in the developed composites.30

Thermal behavior of the CI fibers was envisaged using thermogravimetry (TG) and derivative thermogravimetry (DTG) curves obtained from the thermogravimetry test as given in Figure 6. It is identified from the figure that the degradation of CI fibers happened in different stages. In
the first stage, temperature up to 70.16°C, evaporation of moisture content present in the CI fibers occurred with the mass change of 8.9%. In the second stage, temperature between 68.26°C and 203.36°C, the removal of remaining moisture and part of lignin content happened with the mass change of 18.36%. Similarly, in the third stage, temperature between 203.16°C and 373.3°C, the degradation of cellulose and remaining part of lignin was materialized with the mass change of 50.13%. In the final stage, the temperature ranges above 373.3°C, the degradation of part of cellulose and lignin occurs and the mass change was observed to be 8.12%.

Furthermore, it is observed that above 400°C, there is a major enhancement in thermal stability. This could be the reason for, at superior range of temperature, the interfacial bonding between the fiber and matrix, and the occurrence of cellulose and lignin content offers the withstanding capability of heat. Therefore, fiber length (l_c) of 40 mm results least loss in weight at the preceding degradation juncture as indicated in Figure 7.

**Conclusion**

DMA test revealed that the pure polyester storage modulus is much lower than the CIFRCs in the glass transition region. This is because of the free molecular mobility of the polymer chains which can break the cross-linking between the molecular chains. DMA result shows that 30-mm fiber length reinforced composite has shown higher storage modulus in the glassy plateau due to the higher interfacial bonding between the fiber and matrix. The loss modulus of pure polyester and higher fiber length is low owing to the lack of heat dissipation and the fiber length of 30 mm was found to have a high value among all fiber lengths. This is due to the variation in physical bond between the fiber and matrix. TGA result envisaged that up to 400°C, the degradation samples are more due to the level of water loading in the composite, and there is a significant improvement in the thermal stability beyond the temperature of 400°C. Among the various fiber lengths, the CIFRCs fabricated with the critical length of 30 mm fiber were found as an optimum value of modulus and damping factor. The SEM fractography results reveal that the major mechanisms involved for mechanical failure are fiber pull out, matrix, and fiber fractures.

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