ARCHIVE OF MECHANICAL ENGINEERING

Volume 67 2020 Number 1

DOI: 10.24425/ame.2020.131683

Key words: areca nut husk fiber, dynamic mechanical analysis, storage modulus, loss modulus, elastic modulus, damping factor

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Dynamic mechanical characterization of epoxy composite reinforced with areca nut husk fiber

Natural fiber polymer composites are gaining focus as low cost and light weight composite material due to the availability and ecofriendly nature of the natural fiber. Fiber composites are widely used in civil engineering, marine and aerospace industries where dynamic loads and environmental loads persist. Dynamic analysis of these composites under different loading and environmental conditions is essential before their usage. The present study focuses on the dynamic behavior of areca nut husk reinforced epoxy composites under different loading frequencies (5 Hz, 10 Hz and 15 Hz) and different temperatures (ranging from 28°C to 120°C). The effect of loading and temperature on storage modulus, loss modulus and glass transition temperature was analyzed. Increase in storage modulus is observed with increase in loading frequency. The storage modulus decreases with increase in temperature. The glass transition temperature of the composite is determined to be 105°C. The elastic modulus calculated from the DMA data is compared with three point bending test.

1. Introduction

Mechanical properties of polymers strongly depend on loading rates and ambient temperature. Measuring the material behavior, using conventional material testing methods at various loading rates and at various ambient temperatures, is expensive and time consuming process which requires sophisticated material testing equipment. The viscoelastic nature of polymers can be studied by using

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Dynamic Mechanical Analyzer (DMA). A wide range of experimental conditions can be simulated in a single DMA test which saves time and money for the experimentation [1]. Loss modulus, storage modulus and loss factor are generally observed parameters in dynamic analysis of materials. These parameters obtained from the DMA cannot be used directly for engineering design. Elastic modulus of composites can be calculated using these parameters. Xianbo Xu et al. [2] studied the effect of carbon nanofibers on High Density Polyethylene (HDPE) composites using DMA. The obtained loss modulus and storage modulus data from DMA was used to calculate elastic modulus of the composite and they compared the results with conventional tensile testing results under similar loading conditions. The elastic modulus calculated by the authors using DMA data resulted in 13.4% error when compared to that of conventional tensile testing methods. Steven Eric Zeltmann et al. [1] performed dynamic mechanical analysis of High Density Polyethylene (HDPE) composites. These composites contain cenospheres of 20% and 40% by weight of the composite. Specimens were cast using injection molding process to avoid formation of air bubbles in the composite. Experiments were conducted at different temperatures and at different loading frequencies. Elastic modulus of the composite was estimated from the storage modulus and loss modulus data obtained from DMA testing and compared with the elastic modulus obtained from conventional tensile testing methods. Qiang Fu et al. [3] investigated the effect of asphalt content and water content on Dynamic Mechanical Properties of the cement mortar. Storage modulus, loss modulus and glass transition temperatures were observed by conducting the experiments under temperatures ranging from -40°C to 60° C. A decrease in storage modulus was observed with increase in asphalt content and water content in the cement motar.

Natural fibers and synthetic fibers are widely used by researchers as gap filling materials and reinforcing materials in cement concrete composites [4–6], bitumen composites [7] and polymer composites [8]. Suman Kumar Adhikary et al. [4] observed the improvement in bending strength and self-compacting property of cement concrete composites by reinforcing them with polyolefin fibers. Peltonen P.V. [7] studied the effect of cellulose fibers, polyester fibers and glass fibers on bitumen. He observed less strain in fiber bitumen composites when compared to that of the pure bitumen material. The polyester fibers were better reinforcing materials for bitumen material when compared to natural fibers and glass fibers. Mohammad Bellal Hoque et al. [8] observed improvement in tensile strength, tensile modulus, bending strength and bending modulus of polypropylene by adding pineapple fiber, jute fiber and cotton fiber to polypropylene. The strength of polypropylene composites decreased with increase in exposure time of composites to the soil. Shabbir Ahmed and Chad A. Ulven [9] conducted tensile tests on flax fibers and observed the brittle nature of Flax Fibers failure. Many researchers used natural fibers such as bagasse [10], jute [11], coir [12] and areca nut husk fiber [13, 14] for manufacturing of polymer composites. Researchers have concentrated more on static mechanical properties of areca nut husk fiber composites. Dynamic mechanical properties of areca nut husk fiber composites have not been studied extensively.

Subhakanta Nayak et al. [15] prepared composites with areca sheath fibers and polyvinyl chloride using injection moulding technique. They observed tensile, flexural, thermo gravimetric and dynamic mechanical properties of areca sheath fiber reinforced polyvinyl chloride composites. The thermo-gravimetric analysis revealed that areca sheath fiber reinforced polyvinyl chloride composites exhibit good biodegradable properties. The authors used the mechanical analysis of composites to note the suitability of areca sheath fiber reinforced polyvinyl chloride composites for automotive applications. Jawaid et al., [16] observed the tensile and dynamic mechanical characteristics of epoxy composites reinforced with different jute fiber loading. The composites were prepared with hand lay-up technique. The composites showed better tensile properties at higher jute fiber loading. The dynamic mechanical characteristics of epoxy composites containing oil palm increased with the addition of jute fiber to the composite. Geethamma et al. [17] conducted dynamic mechanical analysis of coir reinforced natural rubber composites. The damping factor decreased with increase in loading frequency whereas the storage modulus increased with increase in loading frequency. The authors observed the composites reinforced with surface treated coir fibers showed less energy dissipation during testing than that of the composites reinforced with untreated coir fiber due to good interfacial bonding between fiber and matrix material. Laly et al., [18] observed the effect of fiber loading, loading frequency and temperature on dynamic mechanical characteristics of banana fiber reinforced polyester composites. From the dynamic mechanical analysis, at 40% fiber loading, the authors observed that the reinforcing effect of banana fiber in polyester composites was greater at higher temperatures. Glass transition temperature increased with increase in fiber loading. The glass transition temperature increased with increase in loading frequency which revealed good fiber-matrix interaction that occurred at higher loading frequency.

In the present study loss modulus, storage modulus and glass transition temperature of the areca nut husk fiber composites were studied at 5 Hz, 10 Hz and 15 Hz loading frequencies. The observations of the present study can be used to calculate the elastic modulus of the areca nut husk fiber composites at different temperatures (from 28°C to 120°C) and at loading frequencies of 5 Hz, 10 Hz and 15 Hz. In the present study, elastic modulus is calculated from load-deflection data obtained from DMA and compared with the elastic modulus obtained from the static flexural tests.

2. Materials

The specimen used for the present study were made with epoxy resin (L12), room temperature hardener (camcure-2386) and areca nut husk fiber. Epoxy resins are polymer resins which have good adhesive capacity, resistance to corrosion and electrical resistance. The density of epoxy resin, provided by manufacturer, used in this study is 1.16 g/cc.

The areca nut husk fiber was extracted from areca nut shells by using water retting process [19]. Dried areca nut husk was collected from areca nut plantations. The dried areca nut husk was soaked in fresh water for 7 days, changing the water every day, to remove the soil particles attached to the fibers and for easy removal of fiber from areca nut husk. The extracted fibers were alkali treated with 6% NaOH solution [20] to improve mechanical bonding between areca nut husk fiber and epoxy [21, 22]. The density of areca nut husk fiber was measured using pycnometer method. The densities of areca nut husk fiber used for the present study is 0.947 g/cc. The threads of areca nut husk fiber were made using hand spinning yarn technique. The process of making areca threads is given in Fig. 1. The alkali treated areca nut husk fiber was de-husked properly as shown in Fig. 1a. First, leader areca nut husk fibers were twisted with hand and attached to the hand spin as shown in Fig. 1b. The fibers were joined slowly as shown in Fig. 1c while rotating the spinner. Fiber thread was separated from the spinner when the required length of thread was obtained. The prepared areca nut husk fiber threads are given in Fig. 1d.

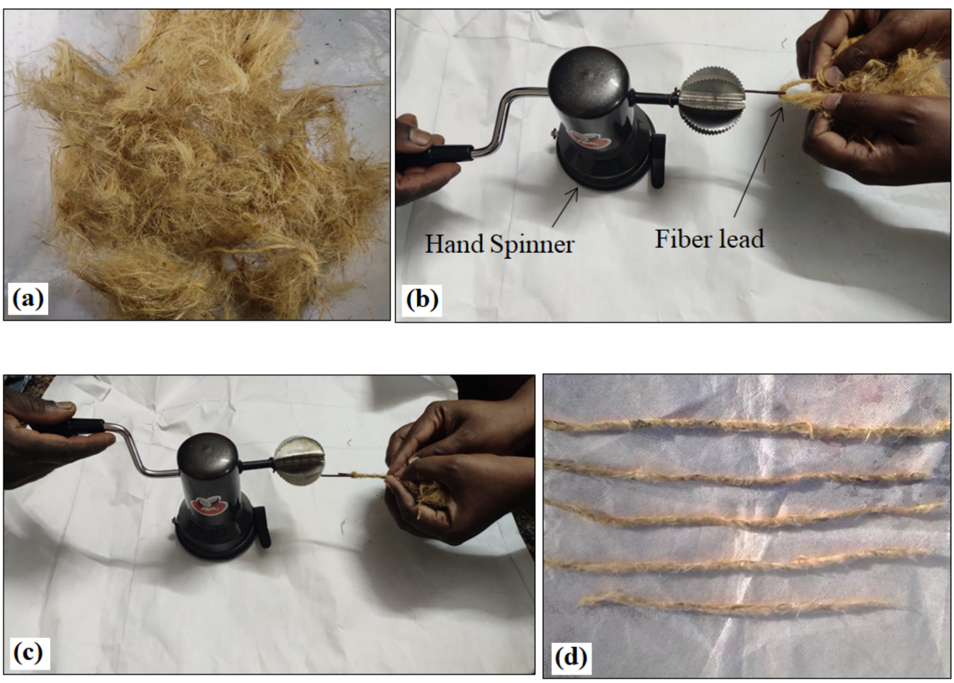

Fig. 1. The process of making areca threads: (a) alkali treated areca nut husk fiber, (b) attaching leader thread to hand spinner, (c) joining fibers to leader, (d) prepared threads using hand spinning yarn technique

3. Specimen preparation

The areca nut husk fiber reinforced epoxy composites were fabricated using hand lay-up technique. A mould having dimensions of 150 mm × 120 mm × 5 mm was used to cast the composite panel. The surface of cast mould and cover plate was coated with thin layer of wax (Fig. 2a). The prepared areca nut husk fiber threads were arranged in mould. Three layers of areca nut husk fiber threads were used to cast the composite panel, two layers in longitudinal direction and one layer in transverse direction as shown in Fig. 2b. The epoxy resin and hardener (10% of resin weight) were mixed thoroughly in a separate bowl. The epoxy resin mixer was poured on the top of arranged areca nut husk fiber threads arranged in the mould (Fig. 2c). The mould was closed with covering plate and weight of 150 kg was placed on mould cover plate to maintain uniform pressure on casting panel. The load applied on the casting mould helps in uniform distribution of epoxy resin, to minimize the void formation due to air and to get uniform panel thickness. The casting panel was cured for six hour period and removed from the casting mould. The composite panels were cast with areca nut husk fiber content of 15% by weight of the composite panel [23]. After removing from the

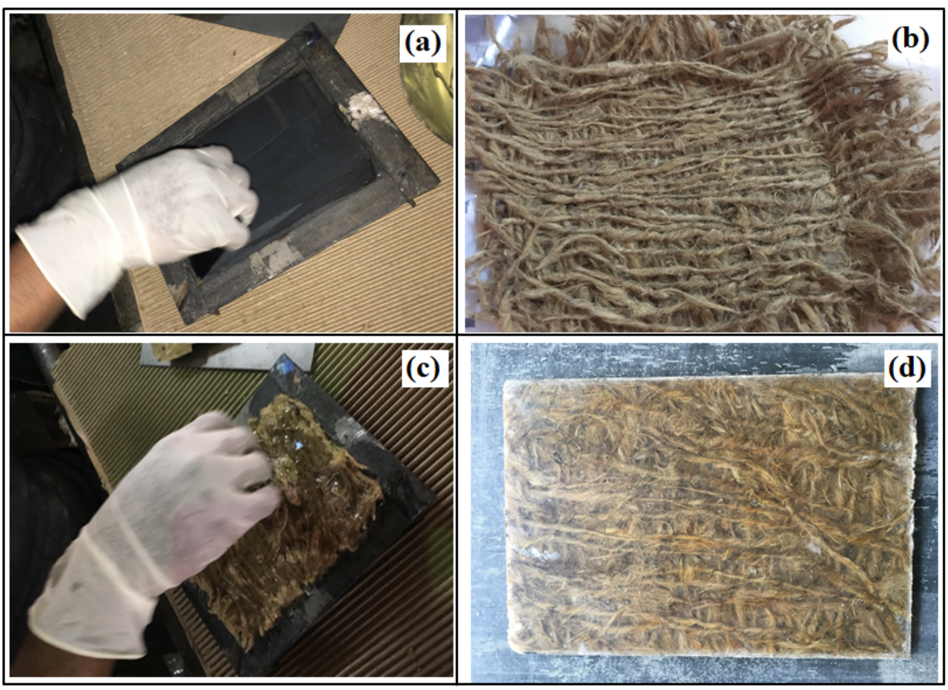

Fig. 2. Preparation of the specimen: (a) wax coating on mold surface, (b) areca nut husk fiber threads arrangement, (c) pouring epoxy resin arranged areca nut husk fiber threads, (d) cast areca nut husk fiber epoxy composite panel

mould the cast composite panel is shown in Fig. 2d. The specimen of dimensions 80 mm × 10 mm × 5 mm were cut, from the panel cast, for dynamic mechanical analysis.

4. Experimentation

4.1. DMA Experiments

Areca nut husk fiber composite specimens were subjected to dynamic loading at a temperature range of 28°C – 120°C, at a heating rate of 3°C/min. using DMA 50N equipment (Fig. 3a). The double cantilever mode (Fig. 3b) bending was used to test the composite samples at a loading frequency of 5 Hz, 10 Hz and 15 Hz. Generally, polymers have both elastic and viscous properties. The modulus obtained by testing polymers is a complex modulus which contains real component (elastic modulus) and imaginary component (viscous modulus). The elastic properties of the polymer materials can be obtained by means of dynamic mechanical analyzer (DMA). DMA applies force at required frequency on the specimen and observes the response of the materials with respect to time. Generally, DMA applies force on specimen in sinusoidal wave form of constant amplitude and collects the response in sinusoidal manner (Fig. 4). The polymers exhibit some phase lag (δ) between the force curve and the response curve. Pure elastic materials exhibit zero phase lag whereas the pure viscous materials exhibit phase change of 90°.

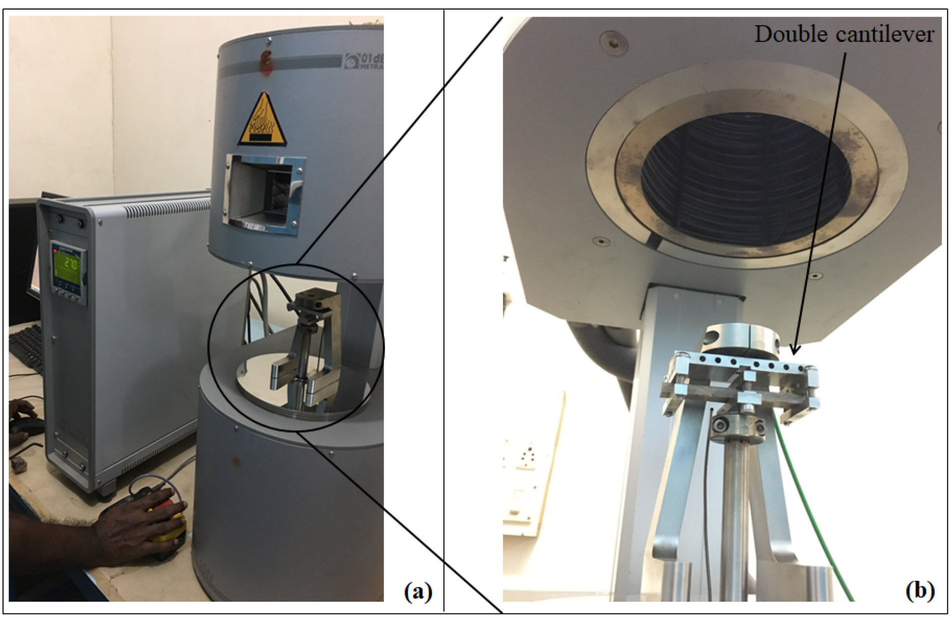

Fig. 3. DMA 50N equipment: (a) general view, (b) double cantilever beam arrangement

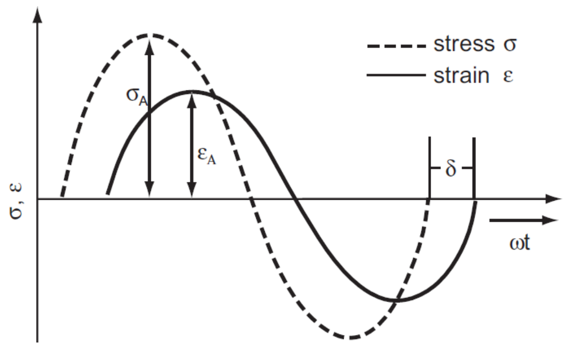

Fig. 4. Schematic picture of applied load and response curves from the DMA [24]

The schematic diagram of sinusoidal stress and sinusoidal strain curves are given in Fig. 4. The information of amplitudes of stress and strain curves and phase lag (δ) over a period of time are the base quantities required for the calculation of moduli of the material. The storage modulus (E'), loss modulus (E'') and $\tan(\delta)$ were calculated by using the equations (1), (2) and (3) respectively [24].

$$E' = \frac{\sigma_A}{\varepsilon_A}\cos(\delta), \tag{1}$$

$$E'' = \frac{\sigma_A}{\varepsilon_A}\sin(\delta), \tag{2}$$

$$\tan(\delta) = \frac{E''}{E'} . \tag{3}$$

4.2. Static Flexural Experiments

Experiments were conducted to find the flexural modulus of areca nut husk fiber reinforced epoxy composite at room temperature, according to relevant standards enlisted in ASTM-D790. Specimens were tested using three-point bending test at midpoint deflection rate of 0.5 mm/min. Specimen of dimensions 150 mm × 25 mm × 3.5 mm were used for conducting static flexural test. All the static flexural tests were conducted at support span length (l) of 100 mm. Three specimens of the composite were tested at same loading rate. Experimental setup used for three point bending test is given in Fig. 5. The elastic modulus of the composite was calculated from the equation (4) derived from Euler–Bernoulli bending theory.

$$E = \frac{l^3 m}{4bd^3} , \tag{4}$$

where: E – elastic modulus, m – slope of load-deflection curve, b – width of the beam (mm), d – depth of the beam (mm), l – support span length of the beam (mm).

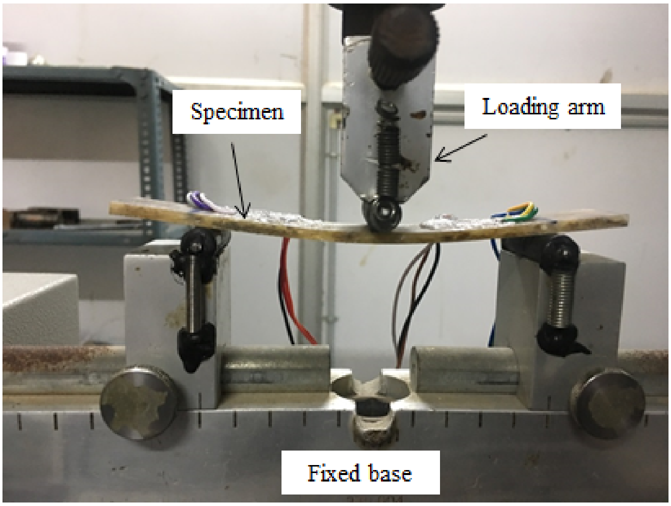

Fig. 5. Static three-point bending experimental setup used for the study

5. Results and discussion

5.1. Storage modulus (E')

The ability of material to store energy and release energy during loading and unloading process represents the storage modulus (E'). The storage modulus represents the elastic property of the material [24]. The storage modulus of the areca nut husk fiber composites increased (0.478 GPa, 0.573 GPa and 0.607 GPa) with the increase in loading frequency (5 Hz, 10 Hz and 15 Hz), as shown in Fig. 6.

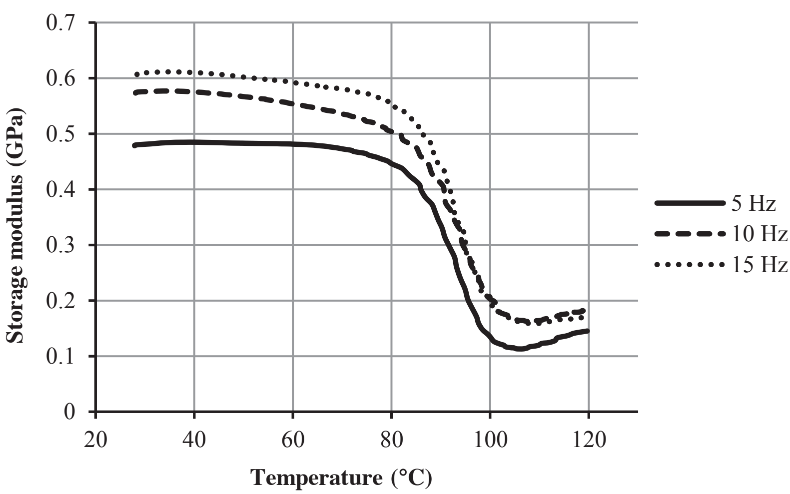

Fig. 6. The storage modulus variation at different loading frequencies

The material is able to retain its elastic property up to a temperature of 80°C and later it transforms to viscous material, which shows a decrease in storage modulus. From Fig. 6, consistency in storage modulus up to 80°C temperature represents the persistance of good bonding between fiber and epoxy [25, 26]. At higher loading frequencies, the composite does not have enough time to deform fully, which results in a higher elastic modulus [27]. Therefore the storage modus increases with increase in loading frequency (Fig. 6). The areca nut husk fiber composites exhibit better elastic properties at higher loading frequency. At lower temperature, composites exhibit higher elastic nature due to immobilization of polymer chains and strong interlocking of fiber and matrix [28]. As the temperature increases, the polymer chains move easily and the fiber-matrix interlocking diminishes which leads to decrease in storage modulus. At higher temperature, the specimens subjected to 10 Hz and 15 Hz frequency resulted in similar storage modulus.

5.2. Loss modulus (E'')

The ability of material to absorb energy (non-recoverable energy) represents the loss modulus (E''). The loss modulus gives the viscous property of the material and hence is referred to as viscous modulus. The viscous property of the material depends on phase lag between stress curve and strain curve. At 80°C, the loss modulus has increased suddenly which indicates the change in material behavior from elastic material to viscous material. Up to 70°C, loss modulus decreases with increase in temperature. Later, the sudden increase in loss modulus is due to dissipation of energy in intermolecular frictional forces [29]. The stored energy in the material dissipates suddenly in the glass transition region which results in rapid increase of loss modulus and rapid decrease of storage modulus. The peak of loss modulus curves (Fig. 7) increases with the increase in loading frequency.

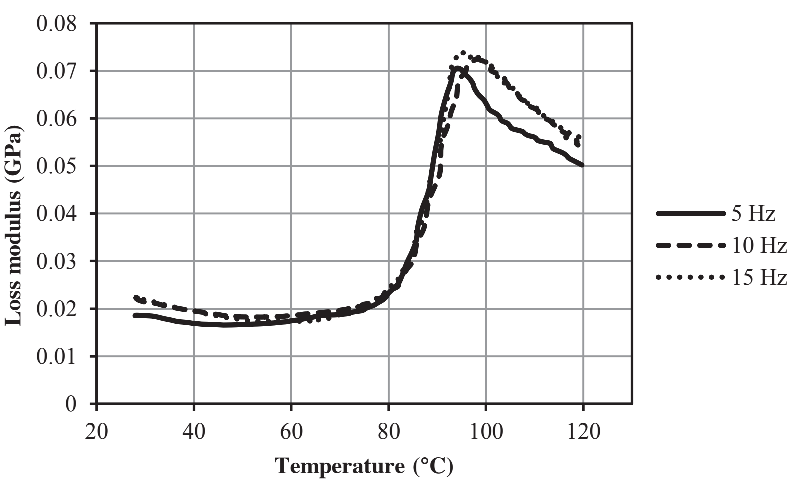

Fig. 7. The loss modulus variation at different loading frequencies

5.3. Damping factor (Tan-delta)

Tan-delta is the ratio of loss modulus to storage modulus. The storage modulus and loss modulus depend on geometry of the test specimens, but the tan-delta (δ) is a constant which does not depend on geometry of the test specimens [28]. The peak of the tan-delta (δ) curve represents the glass transition temperature of the material [29, 30]. From the experimental study, it is observed that the glass transition temperature of the areca nut husk fiber panels is not affected by the loading frequency and the glass transition temperature is observed as 105°C (Fig. 8). The peak of tan-delta (δ) curve characterizes the damping nature of a material [18]. The peak of tan-delta (δ) curve represents the internal molecular motion and dissipation of energy at fiber matrix interactions [18, 31]. In the present study, areca nut husk fiber epoxy composite has better damping characteristics at 5 Hz loading frequency (Fig. 8) when compared to 10 Hz and 15 Hz loading frequencies.

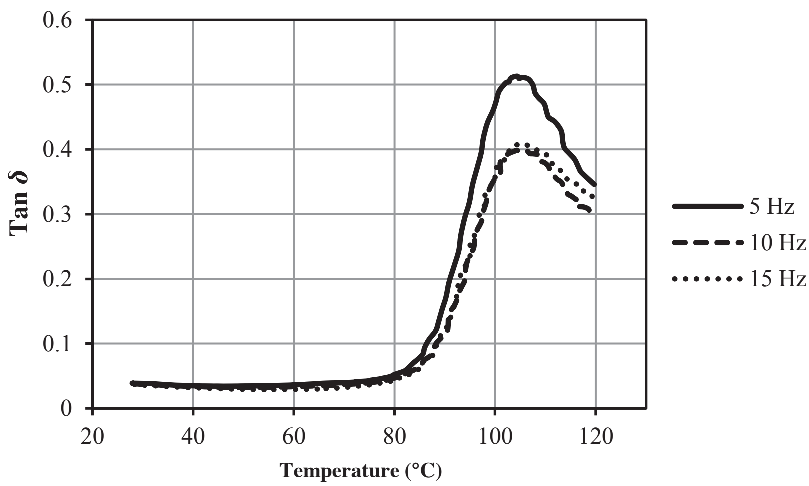

Fig. 8. The tan(δ) variation at different loading frequencies

Subhakanta Nayak et al. [15] observed storage modulus, loss modulus and glass transition temperature of both polyvinyl chloride matrix and areca sheath fiber reinforced polyvinyl chloride composites. As observed in the present study, a decrease in storage modulus, and an increase in loss modulus with increase in temperature was observed in both areca sheath fiber reinforced polyvinyl chloride composites and polyvinyl chloride matrix. At 20°C, the storage modulus of areca sheath fiber reinforced polyvinyl chloride composites and polyvinyl chloride matrix was observed as 1.58 GPa and 1.223 GPa respectively. Glass transition temperature of polyvinyl chloride matrix increased from 59.1°C to 66.09°C by reinforcing the polyvinyl chloride matrix with 18% by weight areca sheath fiber.

5.4. Elastic modulus calculation

Dynamic flexural experiments were conducted by applying maximum deflection of 0.1 mm at mid-span of double cantilever beam. The force required to give 0.1 mm deflection at an instant of time was acquired through DMA data acquisition system. The tests were conducted at ambient temperature ranging from 28°C to 120°C. The schematic representation of test specimen and the forces acting on the specimen during testing are shown in Fig. 9. The force required to give 0.1 mm deflection at various temperatures is given in Fig. 10. The elastic modulus of the composite material is calculated by equating the known central deflection (y = 0.1 mm) with the theoretical deflection (Eq. (5)) derived from Euler-Bernoulli bending theory [32]. The elastic modulus of the composite at different ambient temperatures is calculated and given in Fig. 11. The calculated elastic modulus decreases with the increase in temperature. At lower temperatures (from 28°C to 80°C), the elastic modulus is greater for higher loading frequency when compared to that of the lower loading frequency. Significant drop in elastic modulus at all loading frequencies is observed at 80°C temperature. At room temperature (28°C), the elastic modulus is observed as 3.4 GPa, 2.95 GPa and 2.7 GPa for loading frequencies of 15 Hz, 10 Hz and 5 Hz respectively.

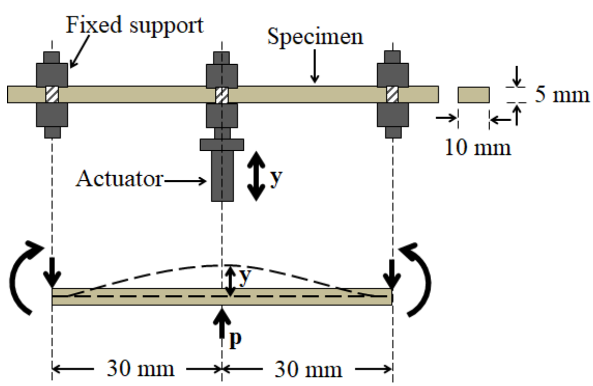

Fig. 9. Schematic picture of double cantilever beam and free body diagram of the beam

$$y = \frac{pl^3}{192EI}, \tag{5}$$

$$E = \frac{pl^3}{192yI}, \tag{6}$$

$$E = 108p, \tag{7}$$

where: y – applied vertical deflection at mid span of the double cantilever beam (0.1 mm), p – applied force at mid span of the double cantilever beam (N), l – length of the double cantilever beam (60 mm), I – moment of inertia of the section (104.17 mm^4), E – elastic modulus of the material (MPa).

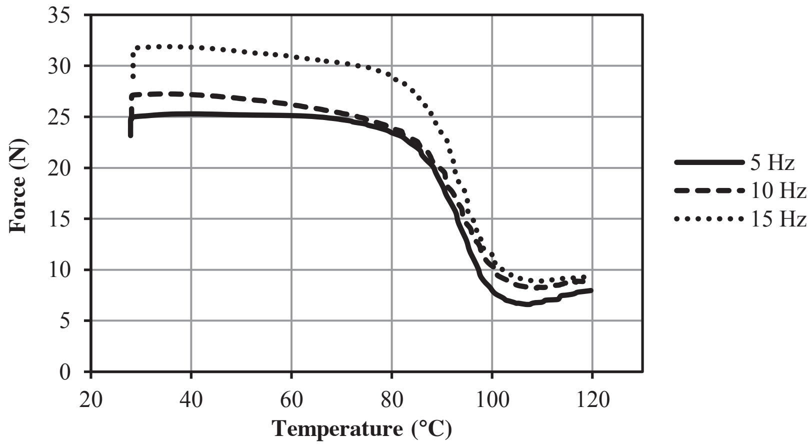

Fig. 10. Reaction force at mid span of the double cantilever beam at different temperatures

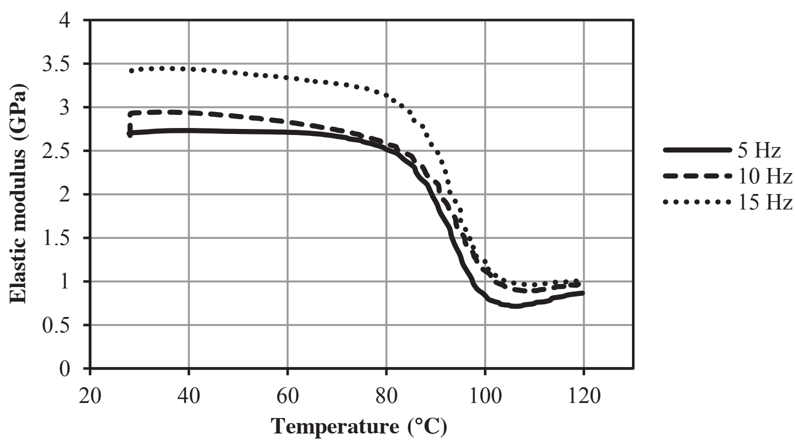

Fig. 11. Variation in elastic modulus with change in ambient temperature

5.5. Static three point bending results

The elastic modulus calculated from the DMA data is confirmed from the results of static flexural tests at room temperature. The load-deflection curves plotted from the experimental data are given in Fig. 12. The elastic modulus of the composite specimen, calculated from static flexural test data, is shown in Table 1. Subhakanta Nayak et al. [15] observed the elastic modulus of areca sheath fiber reinforced polyvinyl chloride composites as 2.38 GPa where the elastic modulus of areca nut husk fiber reinforced epoxy composites observed in the present study is varying from 3.13 GPa to 3.63 GPa.

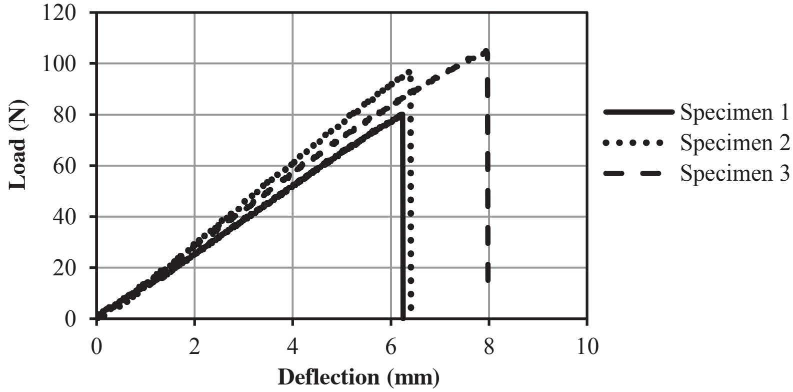

Fig. 12. Load-deflection curves of the static flexural experiments

Table 1.
Elastic modulus (GPa) of composite specimens obtained from static flexural experiments

	Elastic modulus (GPa)
Specimen 1	3.00
Specimen 2	3.63
Specimen 3	3.13

6. Conclusions

Dynamic characteristics of areca nut husk fiber composite were studied by using DMA at varying temperatures and different loading frequencies. The following conclusions are drawn from the study:

- The storage modulus increased with increase in loading frequency and variation of increment in storage modulus decreased with increase in frequency.
- At room temperature, the values of storage modulus are 0.478 GPa, 0.573 GPa and 0.607 GPa for loading frequencies of 5 Hz, 10 Hz and 15 Hz respectively.
- The areca nut composite can retain its storage modulus up to 80°C.
- The glass transition temperature of areca nut husk fiber composites is 105°C. Glass transition temperature of areca nut husk fiber composite is independent of loading frequency.
- The composite is having better damping properties at lower loading frequency [18].
- The elastic modulus, obtained from the dynamic analysis at room temperature, is ranging from 2.7 GPa to 3.4 GPa at loading frequencies of 5 Hz, 10 Hz and 15 Hz. Whereas the elastic modulus obtained from static flexural test at room temperature is in the range of 3 GPa to 3.63 GPa.

Manuscript received by Editorial Board, December 24, 2019;
final version, March 14, 2020.

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