



An investigation of fatigue, creep, and dynamic mechanical behavior of bio-fiber-reinforced PLA and their hybrid biocomposites

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Abstract

In the present research work, three combinations of composite samples, jute and nettle fibers reinforced with polylactic acid (PLA) composites, and their hybrids (jute/PLA, Nettle/PLA, and jute/nettle/PLA) were fabricated using compression molding process. Present work aimed at the experimental investigation of fatigue, creep, dynamic mechanical behavior (DMA), and X-ray diffraction (XRD) of all fabricated composite specimens. Hybridization of jute and nettle fiber enhanced the fatigue cycles and creep strain rate of the developed jute/nettle/PLA hybrid composite as compared to neat PLA and other composite specimens. Experimental results of DMA focused that it is observed by the damping factor ($\tan \delta$); all composite specimens obtained higher value of glass transition temperature as compared to neat PLA. Jute/PLA and nettle/PLA composites achieved the maximum glass transition temperature of 64 °C at a damping factor of 1. Jute/nettle/PLA hybrid composites obtained the uppermost value (4775.9 MPa) of storage modulus which is more than four times of neat PLA at room temperature. Jute/nettle/PLA hybrid composite obtained the highest loss modulus 550.36 MPa. In XRD, jute and nettle fibers showed small peaks at diffraction angles of 17 to 30° which indicated the presence of various constituents such as cellulose, lignin, and pectin present in these natural fibers.

Keywords Bio-fibers · Polylactic acid · Fatigue cycle · Creep strain · Dynamic mechanical analysis · Surface roughness · X-ray diffraction

1 Introduction

Plastic wastage influences the environment and human health very dreadfully. Different synthetic plastic is used by many industries and humans, and after usage, they throw this plastic into the landfill. This synthetic plastic spreads non-toxic gases in the environment, which is a very serious concern for our present and future generations [1]. To overcome this problem, strong laws against synthetic plastic

usage to stop hazardous gases and chemical pollution play a significant role in controlling the huge consumption of synthetic plastics. Natural resources develop the non-toxic and biodegradable composite materials which increases sustainability of the materials. Material scientists and researchers are exploring the potential inclusion of natural products to make composite materials [2–4]. Natural resources such as plant fibers extracted from the plant leaves and plant stems are used as reinforcement in composite materials. Similarly, various animal fibers like sheep wool and horse hairs and various minerals like asbestos have potential applications as a reinforcement in composite materials. Polymers are used as a matrix material to provide the shape and structure that surrounds the whole fiber phase and develop the polymer composite materials [4–7]. The incorporation of natural fiber with biodegradable polymers develops biodegradable polymer composites. This biodegradable composite serves as a non-toxic, lightweight, and sustainable material [1]. Lightweight, biodegradability, and high specific strength of these composite materials stand them in every field of engineering for structural and nonstructural components. The

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high glass transition temperature of fibers as well as matrix provides them good thermal stability. In various applications such as automobile and aerospace components, natural fiber reinforced polymer composites achieved good strength with good thermal and structural stability. These biodegradable fiber-reinforced polymer composites are replacing the frequently used ceramics, metals, and several synthetic plastics in numerous applications [8–10].

These composites are an obvious devotion in automotive, aerospace, power plants, distinct machines, etc. During the application, these composite materials experienced static and dynamic loadings. These loading conditions raise the importance of fatigue, creep, and dynamic mechanical analysis of developed composite material before their fitment in real applications. In previous studies, researchers have focused on fatigue, creep, and dynamic mechanical analysis of polymeric materials. Durante et al. [11] performed the creep and dynamic mechanical analysis of woven hemp/polylactic-acid composite. The authors concluded that creep strength of the developed composite achieved a substantial increment as compared to neat PLA (polylactic acid). They also stated that glass transition temperature is enhanced after the reinforcement of woven mat with PLA. Liber-Knec et al. [12] studied the fatigue behavior of flax/PLA composites. Authors stated that the incorporation of flax fiber with PLA achieved better fatigue strength as compared to flax/thermoplastic starch composite. They also found that flax reinforced with PLA and thermoplastic starch showed higher value of fatigue strength as compared to neat PLA specimen. Therefore, it is very valuable to perform their dynamic mechanical analysis. Dynamic mechanical analysis helps to predict the glass transition temperature of composite materials with their damping factor and storage and loss modulus. Every composite has a specific property that is utilized according to actual needs in the required application [13, 14]. One of the most important properties of composite materials is their viscoelastic nature. Viscoelastic nature represents the thermal ability of composites under the application of sinusoidal force. These properties were examined by conducting dynamic mechanical analysis (DMA) at numerous temperatures and frequencies [15, 16]. The dynamic mechanical analysis parameters like glass transition temperature, storage modulus, and loss modulus of biocomposites are mostly dependent on various parameters like different types of reinforcement architecture such as loose fibers (unidirectional and bidirectional mat), alignment of fibers (normal, parallel, and anti-parallel), treatment of fibers, dry and wet contact conditions, and weight percent of fibers [4–6]. Hence, it is very important to investigate the dynamic mechanical behavior of biodegradable fiber-based polymer composites. Figure 1 represents the different types of synthetic fibers, natural fibers, and polymer matrix to develop the fiber-reinforced polymeric composite materials.

In this paper, jute and nettle as fiber reinforcement and polymer as a biodegradable matrix are used. These reinforced biodegradable composites are made of jute, nettle, and polymer matrix. Nettle-based composites have good surface roughness and glass transition temperature compared to jute-based composites. Also, their hybrid has good damping capability with improved glass transition temperature. The interface bonding between biodegradable fiber and matrix was examined by SEM.

Based on the literature and the authors' best knowledge, no research was available on the hybridization of jute and nettle fibers which performs fatigue, creep, and dynamic mechanical behavior of biocomposites. The present study focused on the effect of the hybridization of jute and nettle fiber and their effect on fatigue, creep, glass transition temperature, storage modulus, and loss modulus of developed composite specimens. The significance of the present work stands for the application of developed biocomposites in the areas where static and dynamic loads act with different temperature ranges. The abbreviations used in this study are shown in Table 1.

2 Materials and methods

2.1 Fibers and matrix used to develop the composite specimens

Jute and nettle fiber mats were supplied by Compact Buying Services, Faridabad (India). Bi-directional fiber mats of jute and nettle fibers were reinforced with epoxy to develop the biocomposites. Each fiber mat has a 0.75 mm thickness, and each developed composite has a 4 mm thickness. The GSM of the jute fiber mat was 250 GSM, and the nettle fiber mat was 230 GSM. Before the fabrication of the composite specimen, fiber mats were placed in the oven to remove moisture content in it. Figure 2 represents the jute and nettle fibers with its SEM images.

The polylactic acid used as a polymer matrix in the present study was in a pellet form having a density of 1.24 g/cm³. The glass transition temperature and melting temperature of polylactic acid are 60 ± 2 °C and 180 °C.

Table 1 Abbreviations used in the paper

Abbreviations	Full form
PLA	Polylactic acid
DMA	Dynamic mechanical analysis
XRD	X-ray diffraction

Fig. 1 Types of fibers and polymer matrix for polymeric composite materials

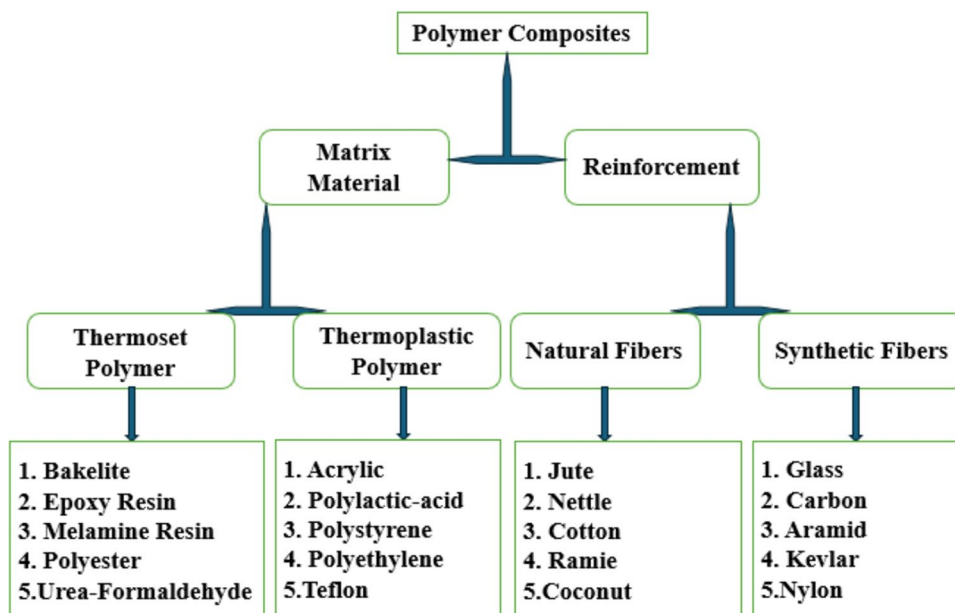
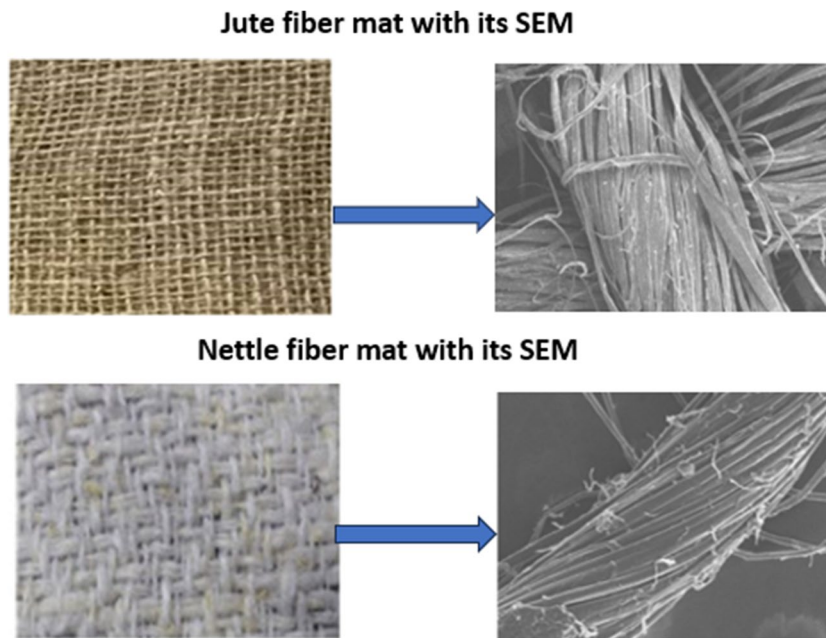


Fig. 2 Jute and nettle fiber mat with SEM micrograph of single fiber [4]



2.2 Processing of composite specimens

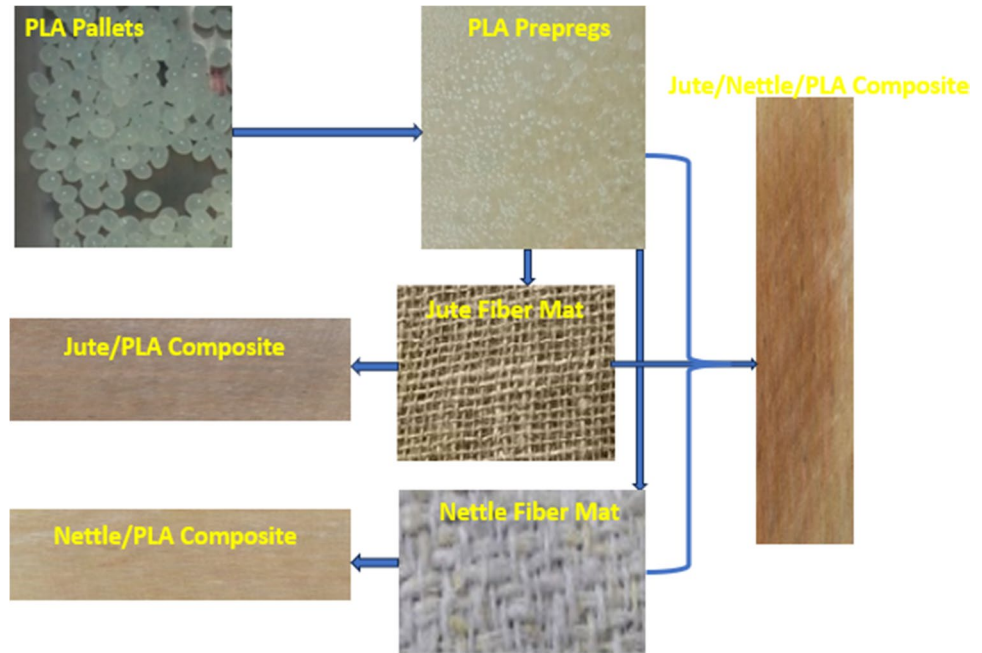
In this study, the compressive molding technique was used to manufacture all the composite material. The weight percent of fiber reinforcement kept 30% by weight and the matrix was 70% by weight. In the case of hybridization both jute and nettle fibers were 15% and 15% respectively. In compressive molding, prepregs of PLA were created between the upper and lower part of the steel mold and then fiber laminate was pressed by the proper arrangement of heat and pressure applied by the compression molding machine. After some time reduce the pressure and temperature and open

the upper and lower part of the mold to pull out the sheet of composite material. This process was repeated for each type of composite material. Reduce the excessive edge and cut to the standard form of samples. PLA pallets and PLA prepregs with Jute/PLA, Nettle/PLA, and hybrid composite laminates (Jute/Nettle/PLA) are shown in Fig. 3.

2.3 Fatigue testing

The fatigue testing specimens were prepared according to ASTM D3479 standards. Based on ASTM D3479, the dimensions of the composite specimen were 200 mm ×

Fig. 3 PLA pellets, prepregs and fabricated samples of jute/PLA, nettle/PLA, and hybrid jute/nettle/PLA composites



25 mm × 3 mm (length × breadth × thickness) [17]. Fatigue tests were performed to characterize the fatigue response of the developed composites under fluctuating loading. Fatigue tests were performed on 5900 horizontal fatigue testing machines supplied by P.S.I. Sales Pvt. Ltd. Fatigue testing results were used to plot the load and deflection over a number of fatigue cycles.

2.4 Creep testing

The creep testing specimens were prepared according to ASTM D7337 standards. During the creep test, the operating load was selected by 40% of the ultimate tensile strength of the developed composite. Maximum creep timing was 15,000 s and the operating temperature was 45 °C.

2.5 Dynamic mechanical analysis (DMA)

DMA was accomplished on Anton Paar Rheometer (MCR-102). During DMA, a twisting load is applied at various temperature ranges from 30 to 100 °C, and a cyclic frequency of 1 Hz. The specimen of DMA was prepared according to ASTM D7028. Dynamic mechanical analysis, storage modulus, loss modulus, and damping capability are the outcomes that analyze the viscoelastic behavior of the material.

2.6 High-resolution X-ray diffraction (XRD)

High-resolution X-ray diffraction was accomplished on D8 Discover HR-XRD. All the developed composite materials were crushed into small particles and filled in the pallet

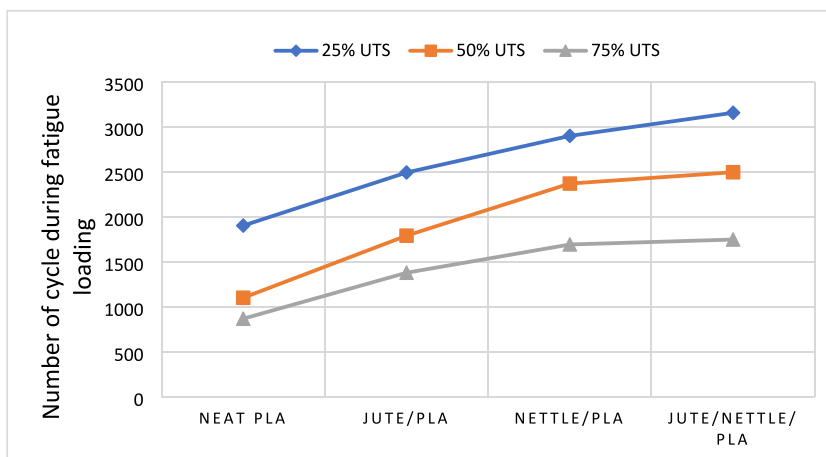
after mixing with KBr. Experimental investigation of diffraction intensity of all the developed composite specimens was recorded at a diffraction angle (2θ) of 5 to 35° at a scan rate of 40 min.

3 Result and discussions

3.1 Analysis of fatigue testing

The fatigue behavior of all developed composite specimens is represented in Fig. 4. The number of cycles was calculated by applying the different percentages of ultimate tensile strength (UTS) of all developed composites. As the percentage of ultimate tensile strength increased from 25 to 75%, then the number of cycles reduced for all developed composites as shown in Fig. 3. For every percentage of ultimate tensile strength, neat PLA achieved lower value of cycles as compared to other composite specimens before fracture. Neat PLA achieved 1900, 1098, and 865 number of cycles at 25, 50, and 75% of UTS. The lower value of number of cycles showed the brittle nature of neat PLA matrix as compared to fiber-reinforced PLA matrix composites. Hybridization of jute and nettle with PLA matrix achieved maximum value of number of cycles during fatigue loading [17, 18]. Jute/nettle/PLA showed 3154, 3493, and 1745 at 25, 50, and 75% of UTM. Hybridization of jute and nettle provides different surface properties as jute fiber has rough surface property and nettle fiber has smooth surface properties which offers the better fluctuating properties during the cyclic loading [17, 18]. Jute/PLA and nettle/PLA showed the

Fig. 4 Analysis of fatigue behavior of composite samples at different stress ration



2490, 1788, and 1376 and 2897, 2367, and 1690 number of cycles at 25, 50, and 75% of UTS. Smooth surface of nettle fiber offers better flexibility to the nettle/PLA composite during fluctuating load as compared to jute/PLA composites.

3.2 Analysis of creep testing

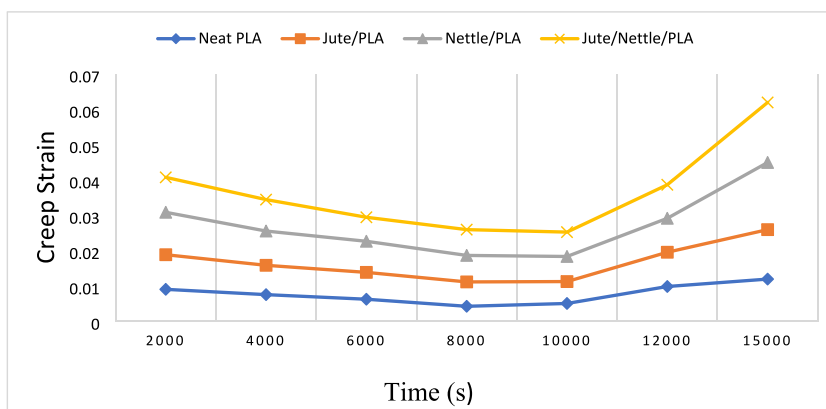
Creep testing was performed to analyze the creep strain obtained with respect to different times as shown in Fig. 5. Neat PLA achieved a minimum value of creep strain as compared to all other developed composites. The incorporation of fiber reinforcement increases the creep strain as compared to neat PLA which upheld the elastic behavior of developed fiber-reinforced composites. Neat PLA achieved 0.0091, 0.0076, 0.0063, 0.0043, 0.0051, 0.0099, and 0.012 creep strain in 2000, 4000, 6000, 8000, 10,000, 12,000, and 15,000 s. Creep strain is more for all developed composites and neat PLA in the initial second and decreases continuously from 2000 to 8000 s. After 8000 s, the creep rate is rapidly increasing up to a measured creep strain. Hybridization of jute and nettle fiber with PLA offers adequate elastic performance which enhanced the creep behavior of developed composite [19, 20]. Jute/nettle/PLA hybrid composite

achieved highest creep strain as compared to all developed composites. Jute/nettle/PLA hybrid composite achieved 0.0099, 0.0089, 0.0068, 0.0073, 0.0069, 0.0095, and 0.017 creep strain in 2000, 4000, 6000, 8000, 10,000, 12,000, and 15,000 s. Nettle/PLA achieved the second-highest value of creep strain. The rough surface of jute fibers is not competent to impart the elastic properties after incorporation of jute with brittle behavior of PLA, while the elastic behavior of nettle fiber with a smooth surface easily integrates the proper elasticity with ductility which results in terms of high creep strain during the creep test [19, 20].

3.3 Damping characteristics of developed biocomposites versus temperature at fixed frequency 1 Hz

The outcome of various temperatures on the damping characteristic of neat PLA, jute/PLA, nettle/PLA, and nettle/jute/PLA composites was investigated at a constant frequency of 1 Hz as shown in Figs. 6, 7, 8 and 9. The damping factor is the ratio of loss modulus and storage modulus. The incorporation of natural fibers with the PLA matrix influences the damping capability. Figures 6, 7, 8 and 9, where different

Fig. 5 Analysis of creep strain at different times for developed composite samples



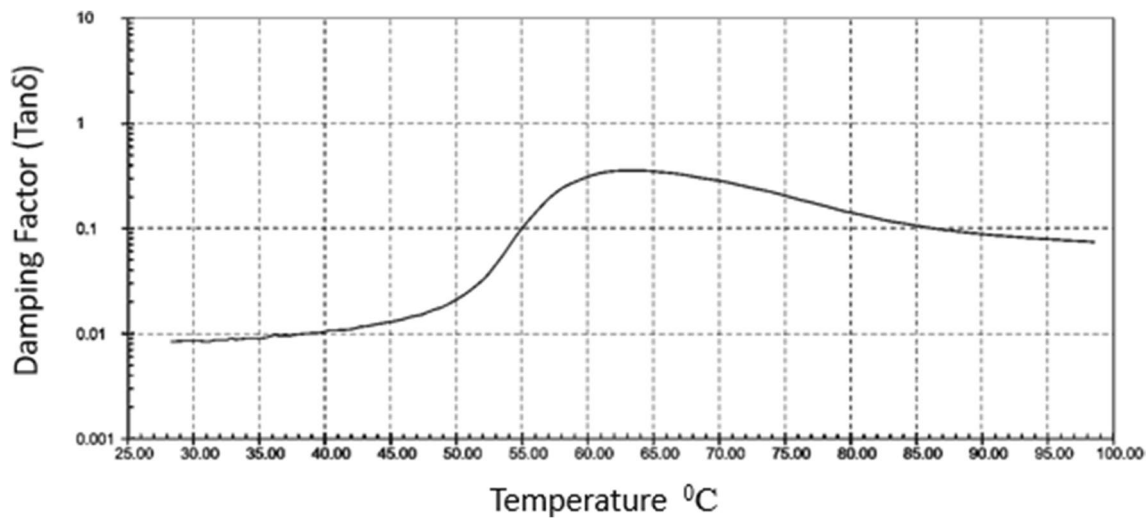


Fig. 6 Damping factor of neat poly(lactic acid) (PLA)

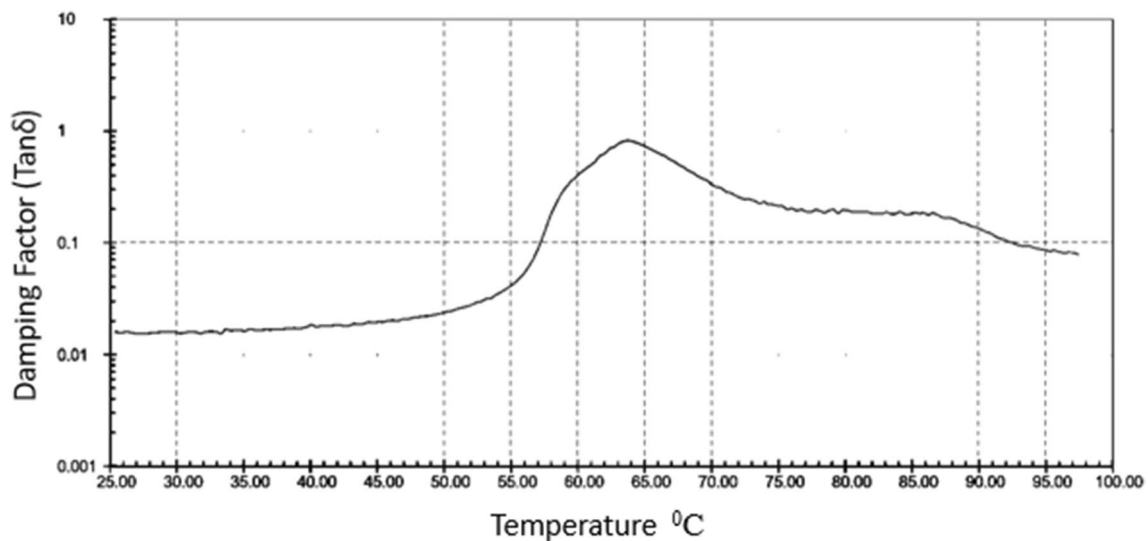


Fig. 7 Damping factor of jute/PLA composites

tan delta peaks are obtained for different composites. Every composite has the capability to achieve a glass transition temperature (T_g). The highest temperature peak value in graph between damping factor and temperature presents the glass transition temperature of the composite sample. After achieving the glass transition temperature, the polymer composites specimen start transforming from a glassy state to a rubbery state. It is observed by the damping factor ($\tan \delta$), all composite specimens obtained a higher value of glass transition temperature as compared to neat PLA. Neat PLA obtained 0.8 $\tan \delta$ at the glass transition temperature of 62°C. Damping capability represents the composite characteristics such as elastic and non-elastic nature [11–14]. Damping capability speaks for the energy absorbance and

dissipation during the temperature and sinusoidal force. Addition of fibers in composite providing the sharp peak of damping factor to the composite specimen which indicated the more elastic nature and higher glass transition temperature at highest peak of all developed composite represents the high thermal stability during energy absorbance and dissipation [11–14]. Jute/PLA and Nettle/PLA composites achieved the maximum glass transition temperature of 64°C at damping factor of 1. Hybrid jute/nettle/PLA composite achieved glass transition temperature of 62°C at damping factor of 0.9. Various authors carried out their study on damping factor and glass transition temperature of polymeric composite materials. Manral et al. [21] fabricate kenaf fiber/poly(lactic acid) (PLA) based composites with different

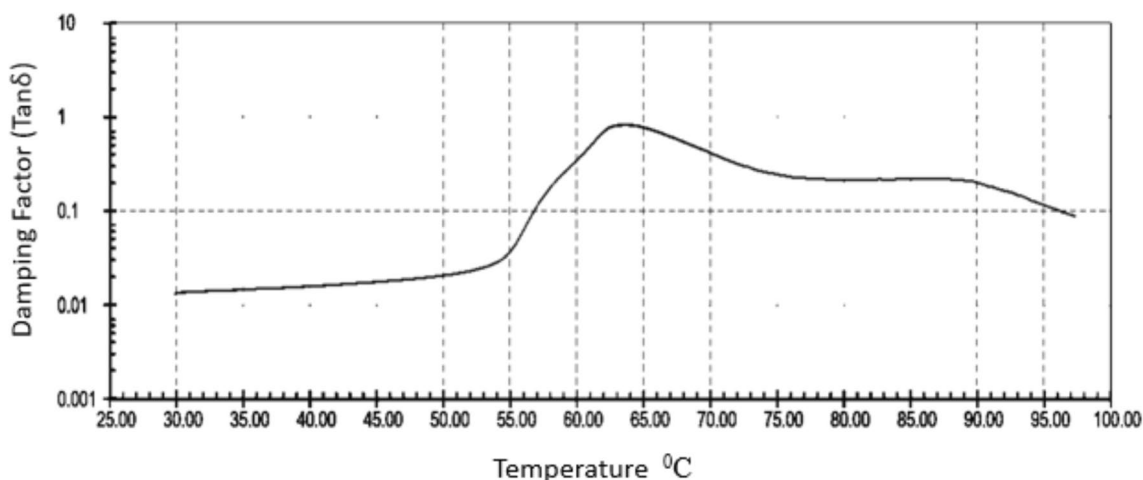


Fig. 8 Damping factor of nettle/PLA composites

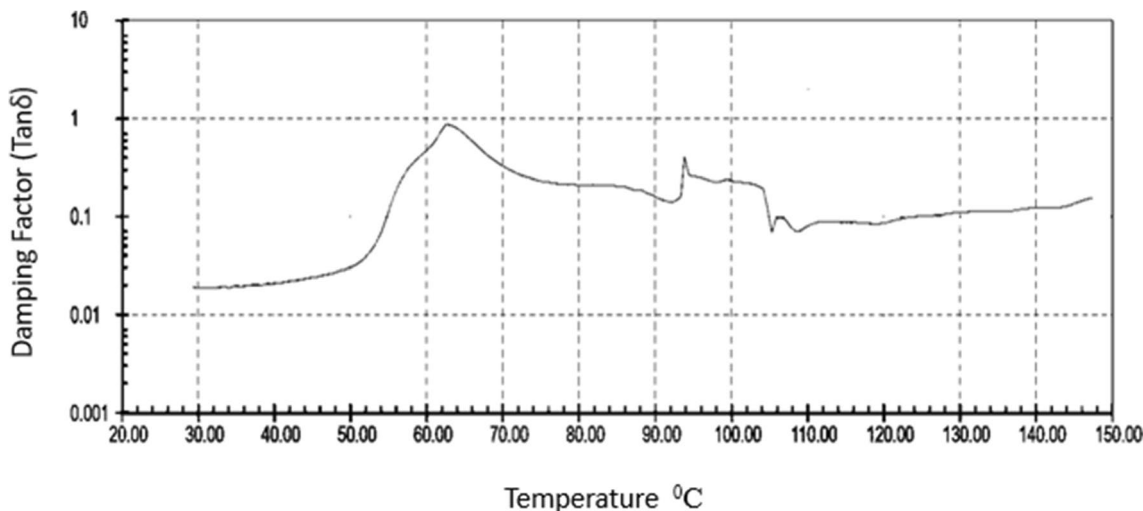


Fig. 9 Damping factors of jute/nettle/PLA hybrid composites

orientations of reinforcement such as (bidirectional, unidirectional, and randomly oriented). Authors concluded that incorporation of fiber reinforcement reduced the damping factor and enhanced the glass transition temperature of all developed composite as compared to neat PLA specimen. They also concluded that damping factor of randomly oriented fiber reinforced PLA composites obtained the higher value under the dynamic loading condition as compared to another developed composite specimens.

3.4 Storage modulus (G')

Storage modulus is the most vital property of materials which represents the qualifications in the provisions of energy stored by the composites under the applied sinusoidal load and temperature. Higher values of energy stored

offer a higher value of storage modulus while lesser values of damping factors. The storage modulus of composite graphs is shown in Figs. 10, 11, 12, and 13. Storage modulus and damping factors are reciprocal to each other. Storage modulus represents the stiffness behavior of the material [11–14]. Incorporation of fibers with polymer enhanced the storage modulus or stiffness of fabricated composites. Nettle/jute/PLA hybrid composites obtained the uppermost value (4775.9 MPa) of storage modulus which is more than four times of neat PLA at room temperature. The second highest value of nettle/PLA composites is 2011.9 MPa. The values of jute/PLA and neat PLA are 1286.3 MPa and 1174.2 MPa, respectively. A higher value of storage modulus represents the higher stiff nature of nettle/jute/PLA composites compared to other fabricated composites. Nagendra et al. [22] fabricated nano

Fig. 10 Storage modulus and loss modulus of neat PLA

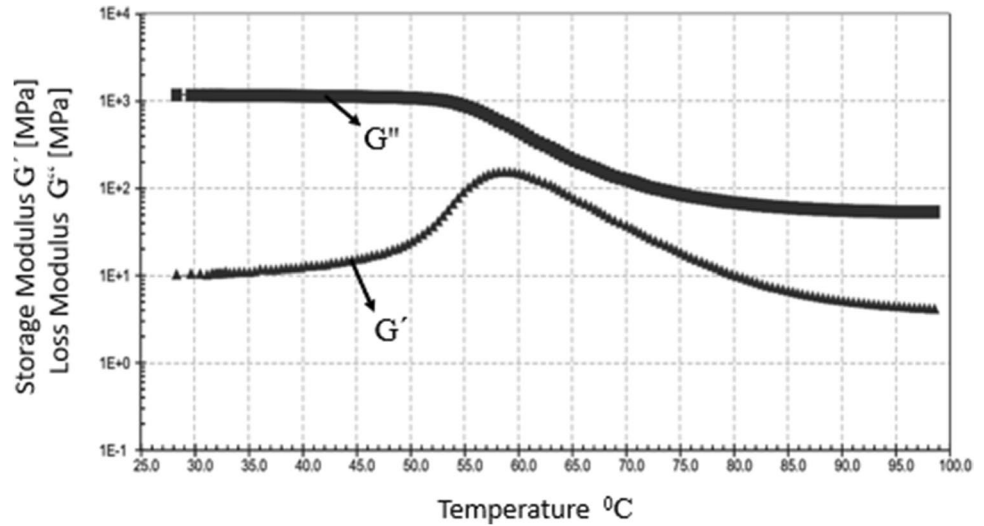


Fig. 11 Storage Modulus and loss modulus of jute/PLA composite

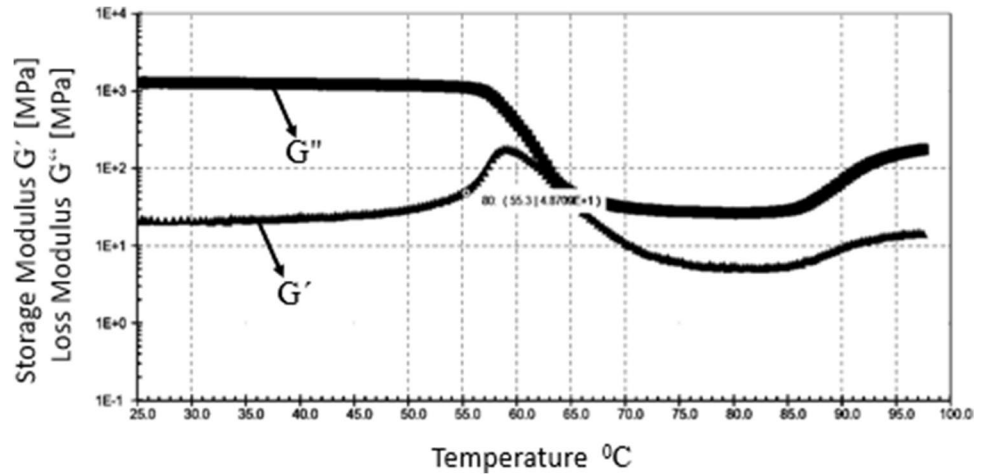
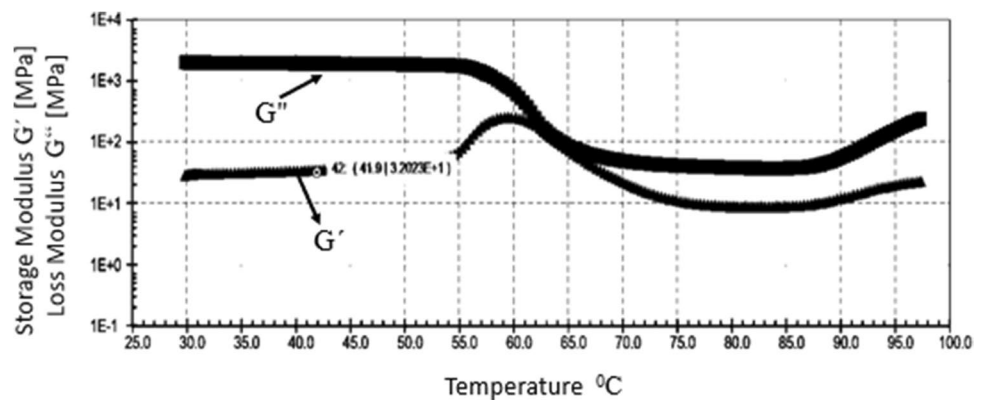


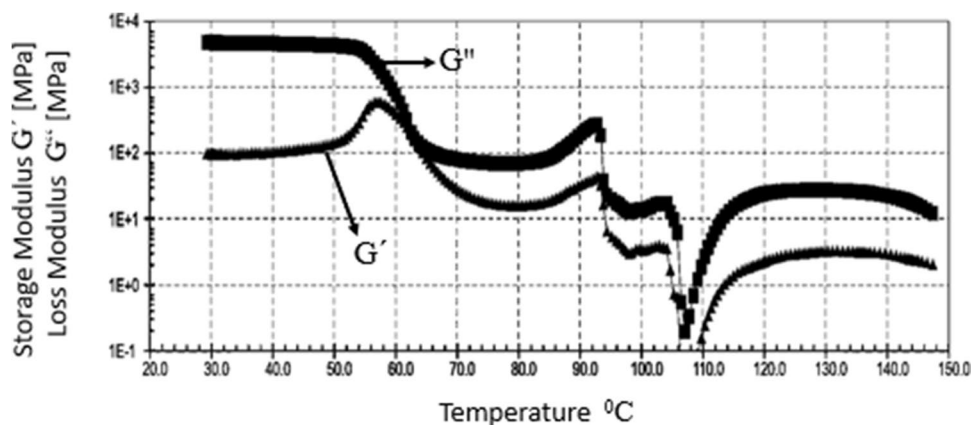
Fig. 12 Storage Modulus and loss modulus of nettle/PLA composite



banana particles/glass fiber/polyester-reinforced composites. DMA probes the behavior of composites with respect to temperature and frequency. The author concluded that the storage and loss modulus increase with increasing the fiber weight percent. The storage modulus was higher for

the neat polyester composite during lower temperatures. Idicula et al. [23] manufactured sisal/banana/polyester-based-reinforced composites and their hybrids. Static and dynamic mechanical parameters are analyzed. The author concluded that maximum storage modulus of fiber volume

Fig. 13 Storage Modulus and loss modulus of nettle/jute/PLA hybrid composite



fraction was obtained at above the glass transition temperature (T_g) of the matrix material.

3.5 Loss modulus (G'')

Loss modulus indicates the viscous behavior of composites. Loss modulus achieved the highest value in the transition temperature. The loss modulus of composite graphs is shown in Figs. 10, 11, 12, and 13. At higher temperatures, polymer molecules start movements and represent the viscous behavior. Materials have some energy that is generated due to intermolecular force. After the glass transition temperature, the value of loss modulus decreased very sharply. Reinforcement of the fibers enhanced the stiffness of neat PLA and increased the loss modulus value of composites. Jute/nettle/PLA hybrid composite obtained the highest loss modulus 550.36 MPa. The rest of the composites achieved higher values compared to neat PLA. Nettle/PLA, jute/PLA, and neat PLA loss modulus values are 233.04 MPa, 169.03 MPa, and 147.18 MPa, respectively. The same results were found in previous studies. Nagendra et al. [22] performed the dynamic analysis of nano banana particles/glass fiber/polyester-reinforced composites. The authors concluded that loss modulus increases after the incorporation of nanofillers and fiber reinforcement with polymer matrix.

3.6 Analysis of high-resolution X-ray diffraction (XRD)

Figure 14 represents the X-ray diffractograms of neat PLA, jute, and nettle fiber. Diffraction intensities were recorded at different diffraction angle (2θ). Diffractograms of all the samples achieved the two major crystalline peaks. The primary longest peak occurs at a diffraction angle of 17° and a second peak occurs at 22° for neat PLA, jute, and nettle fiber. Neat PLA showed a clear peak at a diffraction angle of 17° which indicates the crystalline behavior of neat PLA, while jute and nettle fibers showed the small peaks at diffraction angle of

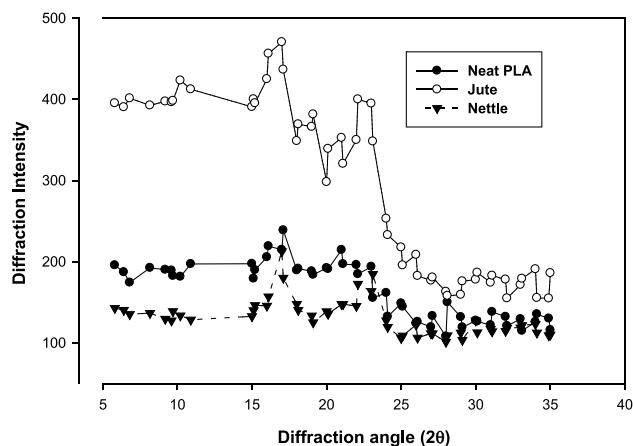


Fig. 14 X-ray diffraction spectra of neat PLA, jute, and nettle fibers

17 to 30° which indicated the presence of various constituents such as cellulose, lignin, and pectin present in these natural fibers. Various authors carried out their research work on the X-ray diffraction of different fibers and composites to indicate the different chemical constituents present in developed composites. Sawpan et al. [24] conducted the X-ray diffraction analysis for hemp fiber. They concluded that sharp peak of cellulose shows diffraction angle (2θ) of 220° . A similar study was performed by Sathishkumara et al. [25] for *Sansevieria* fiber. They concluded that two diffraction peaks occur at a diffraction angle (2θ) of 26.3° and 14.26° . The peak at a diffraction angle (2θ) of 26.3° represents the α -cellulose and 14.26° characterizes nanocellulose material such as hemicellulose and lignin in a natural fiber.

4 Conclusions

In the present study, various types of biodegradable composites and their respective hybrid composites were fabricated with jute and nettle fiber reinforcement combined

with biodegradable PLA matrix. Composites are fabricated with the help of compression molding machine. The following conclusions were made by the findings of dynamic mechanical analysis and XRD of biodegradable composites specimens.

- Incorporation of natural fiber enhancing the fatigue cycle as compared to neat PLA.
- Creep strain of all developed composite obtained higher as compared to neat PLA which shows the elastic behavior of all developed composite.
- Incorporation of natural fibers (jute and nettle) with PLA polymer enhanced the glass transition temperature of all the developed composites.
- Jute/nettle/PLA hybrid composite obtained maximum storage modulus at room temperature.
- Jute/nettle/PLA hybrid achieved the highest loss modulus compared to neat PLA.
- Neat PLA achieved maximum damping factors that means neat PLA has brittle behaviors.
- In X-ray diffractograms, neat PLA showed a clear peak at a diffraction angle of 17° which indicates the crystalline behavior of neat PLA.
- Jute and nettle fibers showing the small peaks at diffraction angle of 170 to 300° which indicated the presence of various constituents such as cellulose, lignin, and pectin present in these natural fibers.

5 Future scope

Future scope has enough space to investigate the current field of study. The present research can be expanded to investigate the additional aspects of composites by increasing the percentage of reinforcement, chemical treatment of fibers, and evaluation of their parameters such as acoustic behavior, thermal analysis, and analysis of tribological properties.

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Author contribution Vijay Chaudhary: writing—original manuscript; Shashi Prakash Dwivedi and Pallav Gupta: ideas, conceptualization, supervision, Bhasha Sharma and Partha Pratim Das: writing—reviewing and editing

Data availability Not applicable

Declarations

Ethical approval Not applicable

Competing interests The authors declare no competing interests.

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