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# Fibrous biocomposites from nettle (*Girardinia diversifolia*) and poly(lactic acid) fibers for automotive dashboard panel application

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ABSTRACT

This work deals with fibrous biocomposites prepared by using nettle and poly(lactic acid) fibers and employing carding and compression-molding processes. The role of carding process in determining the tensile strength of the biocomposites was analyzed. The tensile, bending and impact properties of the biocomposites were found to increase initially with the increase of nettle fiber content till 50 wt% and decrease afterwards. The thermogravimetric analysis inferred that the thermal stability of the biocomposites increased with the increase of nettle fiber content. The dynamic mechanical analysis showed that the biocomposites exhibited high storage modulus, low loss modulus, and low damping factor. Further, the biocomposites exhibited excellent biodegradability and their biodegradability increased with the increase of nettle fiber content. Overall, the biocomposite prepared with equal weight proportion of nettle and poly(lactic acid) showed high potential for automotive dashboard panel application.


## 1. Introduction

Fibrous biocomposites, consisting of biofibers reinforced with biodegradable matrix, is a bioproduct that is derived from renewable sources, stable in its desired lifetime, and fully biodegradable after disposal in composting environment. They are non-toxic, easily processable, and fully biodegradable, they offer high strength-to-weight ratio, and they may be recycled to reduce the carbon footprint of materials. The main area of increasing usage of such composites is predominantly lying with the automotive industry [1,2] as they result in significant weight savings of automotives and corresponding cost savings on fuel consumption. The end-of-life vehicle (ELV) directive by European Union, stating that by 2015, vehicles must be constructed of 95% recyclable materials, with 85% recoverable through reuse or mechanical recycling and 10% through energy recovery or thermal recycling, led to a tremendous usage of biocomposites in automotives [1,3]. It is reported that besides automotives, the fibrous biocomposites have the potential to be used in construction, infrastructure, consumer, industrial, and aerospace applications [1,4–6]. According to a report on Global Natural Fiber Composites Market 2014–2019: Trends, Forecast and Opportunity Analysis, the market for biocomposites is likely to be worth of 531.2 million USD by 2016 with an expected annual growth rate of 11% during 2014–2019 [7].

Of the biofibers used in biocomposites, the naturally occurring bast fibers such as jute, flax, hemp, ramie, and kenaf are mostly used. Nevertheless, there is one more naturally occurring bast fiber - nettle - which is hardly used for making biocomposites. As known, nettle is a cellulosic plant fiber which is abundantly available in tropical wasteland areas around the world. The nettle family, *Urticaceace*, contains around 500 species. Of them, *Girardinia diversifolia* produces nettle fiber. There is not much scientific information available on this fiber in literature. However, it is known that this fiber is very strong, but rigid and inextensible [8]. Very recently, it was investigated for reinforcement with polyester [9], polypropylene [10,11], and poly(lactic acid) matrices [12]. The work of Mahendrakumar et al. [9] reported on the development of fibrous composites by placing the nettle fibers randomly in polyester resin. The composites were evaluated for tensile, flexural, and compression properties. It was suggested by looking at the mechanical performance of the composites that they could be considered to be a candidate material for making precision machine tools. The work carried out by Paukszta et al. [11] dealt with a polypropylene composite reinforced with nettle fibers by injection molding process. The tensile properties of the composite were compared to those of polypropylene composites reinforced with other bast fibers such as hemp and kenaf. The reinforcements of polypropylene matrix with nettle and other bast fibers were not

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found to bring any significant difference in tensile strength, breaking elongation, and Young's modulus.

In a separate work conducted by Bajpai et al. [10], a nettle fiber mat was reinforced with polypropylene matrix to prepare a fibrous composite. The composite was exposed to different environments such as river water, diesel oil, freezing, soil, and sunlight to examine the changes in weight and tensile strength. It was found that there was an increase in weight of the composite in all the environments except on exposure to sunlight and the maximum weight gain was observed in river water environment. On the other hand, the tensile strength of the composite was found to reduce under all the environments. While the maximum reduction was observed in sunlight and river water environments, the minimum reduction was noticed when the composite was buried in soil. The work done by Fischer et al. [12] reported on a biocomposite prepared from nettle fibers as reinforcement and poly(lactic acid) as matrix. In this work, the tensile strength, Young's modulus, and flexural modulus of the biocomposites were determined. It was concluded that nettle had in principle the same potential as other bast fibers to act as reinforcement in poly(lactic acid). However, a large difference was reported for the mechanical properties of nettle fibers as compared to those of biocomposites. There was no attempt made to improve the mechanical properties of the biocomposites by chemical treatment on nettle fibers. Also, the performance of the biocomposites was not evaluated in terms of dynamic mechanical analysis, thermogravimetric analysis, and biodegradability. It is necessary to examine these behaviors of biocomposites when the intended application is automotives [7,13,14].

In this work, an attempt was made to prepare poly(lactic acid) biocomposites reinforced with nettle fibers in different weight proportions using carding and compression-molding processes and evaluate their static and dynamic mechanical properties, thermogravimetric behavior and biodegradability with a view to automotive dashboard panel application.

## 2. Material and methods

### 2.1. Materials

In this work, the Himalayan nettle (*Girardinia diversifolia*) filaments of 1 m–2 m length, were procured from Uttarakhand Bamboo & Fiber Development Board in India. The nettle filaments as procured were cut into staple fibers of 55 mm ± 1 mm length, followed by cleaning mechanically and sorting manually. The fineness of the nettle fibers was determined as 24.61 denier ±1.19 denier and the tensile strength, breaking elongation, and initial modulus of the nettle fibers were found to be 479.86 MPa ± 17.57 MPa, 2.60% ± 0.10%, and 21.61 GPa ± 0.82 GPa, respectively [15]. The nettle fibers were alkali treated and for alkali treatment, easily obtainable EMPARTA® Sodium hydroxide pellets (low chloride) with assay (NaOH) more than 97% were used. Further, in this work, poly(lactic acid) fibers of 55 mm length and 6 denier fineness were used to form a matrix in the biocomposites. The tensile strength, breaking elongation, and initial modulus of poly(lactic acid) fibers as procured were 226.24 MPa ± 9.93 MPa, 23.72% ± 1.67%, and 7.45 GPa ± 0.29 GPa, respectively [16]. The glass transition and melting point temperatures of poly(lactic acid) were 68 °C and 160 °C, respectively.

### 2.2. Alkali treatment of nettle fibers

An attempt was made to process the nettle fibers as procured on a laboratory-based miniature carding machine (Make: Trytex, India) for preparation of fibrewebs. This attempt was, however, not successful as it resulted in significant fiber breakage. This was probably associated to the high stiffness and low extensibility of nettle fibers [8]. In order to make them processable on a carding machine, it was necessary to improve their mechanical properties. Alkali treatment was therefore carried out on nettle fibers. This was done by treating the cleaned staple nettle fibers with alkaline (NaOH) solution in a laboratory-based batch process. The optimum alkali treatment conditions, reported elsewhere [17], were followed for alkali treatment. In one batch, 40 g of cleaned nettle fibers were immersed into 2000 ml of alkaline solution with an alkali concentration of 10%. The fibers were treated at 61.5 °C temperature for 0.5 h. The treated fibers were extracted from the solution, squeezed, washed with distilled water and neutralized with 10% acetic acid. The fibers were then washed with distilled water and squeezed to remove excess water. Afterwards, they were manually opened to single fiber stage. This was followed by drying in a hot air oven at a temperature of 30 °C for 48 h. The dried fibers were kept under standard testing atmospheric conditions as stipulated in ASTM D1776–04 standard. The fineness of the alkali-treated nettle fibers was 24.52 denier ±0.78 denier and the tensile strength, breaking elongation, and initial modulus of the alkali-treated nettle fibers were 626.15 MPa ± 22.79 MPa, 4.43% ± 0.11%, and 20.16 GPa ± 0.96 GPa, respectively [17].

### 2.3. Preparation of biocomposites and pure poly(lactic acid) film

The alkali-treated nettle fibers were blended with poly(lactic acid) fibers as homogeneously as possible by manually in five different blend proportions (w/w) (10/90, 25/75, 50/50, 75/25, 90/10). The fiber blends were fed to the laboratory-based miniature carding machine (Make: Trytex, India). The machine had a feed roll running at 1.04 rpm, cylinder at 60/175/400/600 rpm, and doffer at 2.07 rpm. It delivered parallel-laid fiberwebs consisting of nettle and poly(lactic acid) fibers with five different blend proportions. The fiberwebs were processed through a laboratory-based compression molding machine (Make: Carver, USA) for 8 min, keeping the temperature at 160 °C and pressure at 6 bar. Afterwards, the samples were cooled down using air and water flow and finally removed from the machine when the temperature fell down to 40 °C. Thus prepared biocomposites were kept under standard atmospheric conditions before any testing was carried out. In the second route, the fiberwebs were directly processed through the compression molding machine under the same process conditions as followed in the first route. This was followed by keeping the biocomposites under standard atmospheric conditions before any testing was carried out. In addition to the five biocomposites stated above, a film was prepared from 100% poly(lactic acid) fibers following the same method as stated above. The nominal density and thickness of the biocomposites as well as the film were kept at 1000 kg/m$^3$ and 3 mm, respectively, for determination of mechanical properties.

### 2.4. Characterization of biocomposites and pure poly(lactic acid) film

#### 2.4.1. Morphology

The morphology of the biocomposites was investigated using a Zeiss scanning electron microscope (EVO 18) with resolution of 2 nm at 30 kV, accelerating voltage of 30 kV, and field of view of 6 mm. Also, the morphology of the biocomposites was examined using a light microscope (Make: Leica, Model: VZ 80 RC).

#### 2.4.2. Determination of physical properties

The biocomposites and poly(lactic acid) film were tested for density and thickness. The density and thickness were determined in accordance with ASTM D3039/D3039M-14. To measure the

density and thickness, five samples were tested and the average of five readings was calculated.

*2.4.3. Determination of fiber orientation*

The biocomposites prepared directly from fiberwebs by running the cylinder at four different angular speeds (60 rpm, 175 rpm, 400 rpm, and 600 rpm) were evaluated for the planar orientation of the constituent fibers. To determine the orientation of fibers in the biocomposites, the scanning electron microscopic images of the biocomposites were taken. The images of the fibers were digitized in the form of coordinates of many points using an image processing system. The observed fibres were then divided into many short segments and the direction of each fibre segment was determined and transferred to the respective class interval of 5° width. The fiber orientation histograms were based on around 1500 readings of angle of fiber inclination from the longitudinal axis (direction of applied load) of the biocomposites.

*2.4.4. Determination of mechanical properties*

Further, the biocomposites as well as the pure poly(lactic acid) film were tested for tensile, flexural, and impact properties. The tensile properties of the biocomposites and the PLA film were determined using an universal tensile tester (Make: Tinus Olsen and Model: H5KS) in accordance with ASTM D3039 standard. For tensile test, five samples, each of 200 mm × 25 mm size, were randomly selected and the test speed was kept at 2 mm/min. The average of five readings was taken to determine the mean tensile strength and mean breaking elongation, and mean Young's modulus of the biocomposites and the film. The flexural properties of the biocomposites and the film were determined using the Autograph universal tensile tester (Make: Shimadzu, Model: AG-IS) according to the ASTM D790 standard. An average of five readings was taken to determine the mean flexural strength of the biocomposites and the film. To determine the impact properties of biocomposites and the film, the pendulum impact tester (Make: Tinius Olsen and Model: Impact 104) was used and ASTM D256-10 standard was followed. Here again, an average of five readings was taken to determine the mean impact strength of the biocomposites and the film.

*2.4.5. Thermogravimetric analysis (TGA)*

The thermogravimetric analysis of the biocomposites and the film was carried out using a thermogravimetric analyzer (Make: PerkinElmer and Model: TGA 4000). The biocomposites and film samples of 4 mg–8 mg weight were loaded on the sample pan. The test was carried out under the temperature ranging from 50 °C to 800 °C at a constant heating rate of 10 °C per minute in a nitrogen atmosphere.

*2.4.6. Dynamic mechanical analysis (DMA)*

Also, the biocomposites and the film were tested for their dynamic mechanical analysis. This was carried out on a DMA Q800 machine at a frequency of 10 Hz. The samples of dimensions 17.5 mm × 13.05 mm × 1.9 mm were tested in a 3 PB mode using a frequency scan from room temperature to 200 °C at a heating rate of 5 °C/min. This test was successfully completed with all the biocomposites, except one which was prepared from 90% nettle and 10% poly(lactic acid). This biocomposite failed the test due to its limpness.

*2.4.7. Soil burial test*

The biocomposites were examined for biodegradability using a soil burial test. This test was carried out with a series of flower pots containing Miracle-Gro® compost soil. As reported, this type of compost soil required 50% less watering [18]. The composite samples of 100 mm × 200 mm size were buried under the compost soil at a burial depth of 12 cm–15 cm from the surface of the soil to ensure aerobic degradation [19]. The flower pots containing the compost soil and composite samples were kept at a room temperature (30 °C ± 5 °C) and covered with plastic film in order to avoid any evaporation of water from the surface of compost soil. The moisture content of the soil was measured at regular intervals using a moisture meter (Make: Smiledrive) and maintained at 40%–50% by sprinkling water whenever required. As reported, this humidity was optimal for microbial activity [20]. Biodegradation was estimated by monitoring the tensile strength of the composites as a function two different burial times viz. 20 days and 45 days. The tensile strength of the composites was determined in accordance with ASTM D3039 standard. The buried samples of composites were dug out after the required time, air-dried for one day, oven-dried for one day at 60 °C temperature and cleaned thoroughly before testing for their weight and tensile strength. The loss of weight was calculated by dividing the difference in weights before and after burial to the weight before burial and expressed as percentage. In a similar manner, the loss of tensile strength was calculated.

## 3. Results and discussion

### *3.1. Role of carding process in deciding tensile strength of biocomposites*

In this work, an attempt was made to establish the role of carding process in deciding the tensile strength of biocomposites prepared from nettle and poly(lactic acid) fibers. For this, a set of biocomposites was prepared with equal weight proportion of nettle and poly(lactic acid) fibers by running the cylinder of the carding machine at four different angular speeds (60 rpm, 175 rpm, 400 rpm, and 600 rpm). Thus prepared biocomposites were evaluated for their tensile strength. The results are shown in Table 1. It can be observed that the tensile strength of the biocomposites increased with the increase of cylinder speed. However, during carding operation, it was noticed that the width of the fiberwebs shrunk tremendously when the cylinder speed was too high. The width-wise shrinkage of the fiberwebs was calculated by the ratio of the change in width of the fiberwebs to the original width of the fiberwebs and expressed as a percentage for different levels of cylinder speed in Table 1. It can be observed that the width-wise shrinkage of the fiberwebs increased with the increase of cylinder speed, but this effect was found to be too high at very high levels (400 rpm and 600 rpm) of cylinder speed. Therefore, in order to have a balance between the tensile strength and the width-wise shrinkage, it was decided to run the cylinder at 175 rpm for the manufacturing of biocomposites.

To find out a reason for the increase of tensile strength of the biocomposites increased at higher cylinder speed, the scanning electron microscopic images of these biocomposites were taken. They are displayed in Fig. 1. These images were studied in accordance with the method described in Section 2.4.2 to obtain the fiber orientation histograms. The experimental histograms of fiber orientation for different levels of cylinder speed are displayed in Fig. 2. Here, an attempt was made to explain these histograms in the light of a theoretical model proposed by Neckář and Das [21]. As per this model, the probability density function of fiber orientation takes the following form

$$f(\psi) = \frac{1}{\pi} \frac{\eta}{\eta^2 - (\eta^2 - 1)\cos^2(\psi - \alpha)} \quad (1)$$

where $f(\psi)$ denotes the probability density function of angle

**Table 1**
Structural and tensile characteristics of biocomposites as a function of cylinder speed.

| Cylinder speed, rpm | Tensile strength, MPa | Width-wise shrinkage, % | Measure of preferential intensity of fiber orientation, 1 | Modal direction, degree |
|---|---|---|---|---|
| 60 | 42.23 ± 0.75 | 0.36 ± 0.13 | 1.82 | 2.39 |
| 175 | 50.82 ± 0.82 | 1.28 ± 0.43 | 1.97 | 2.44 |
| 400 | 53.32 ± 0.44 | 26.67 ± 0.68 | 2.12 | 2.43 |
| 600 | 57.54 ± 0.56 | 42.15 ± 0.55 | 2.25 | 2.45 |

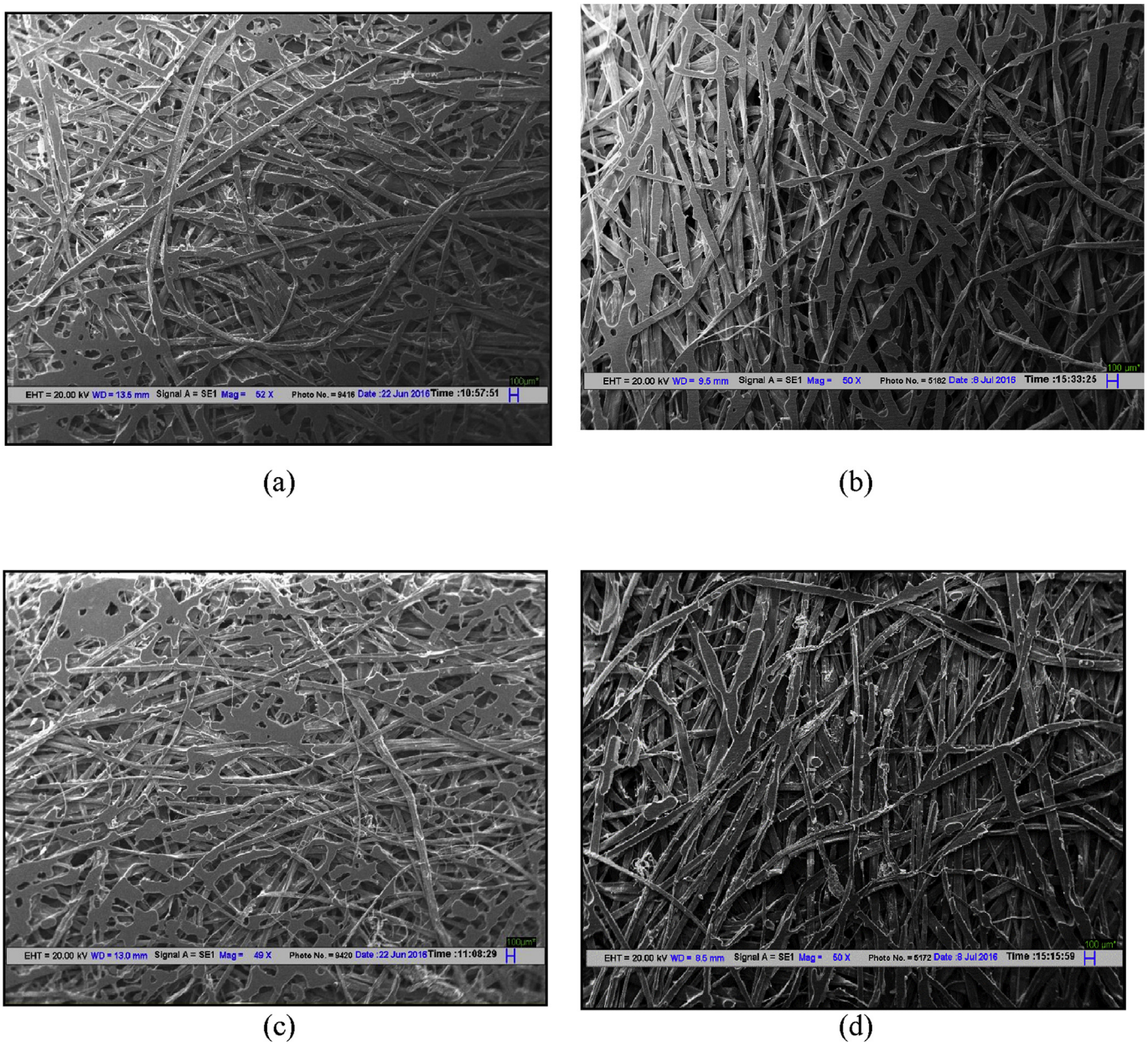


**Fig. 1.** Scanning electron microscopic images of biocomposites prepared with equal weight proportion of nettle and poly(lactic acid) fibers at cylinder speed of 60 rpm (a), 175 rpm (b), 400 rpm (c), and 600 rpm (d).

$\psi \in \langle -\frac{\pi}{2}, \frac{\pi}{2} \rangle$ of inclination of fibers from the longitudinal axis (direction of applied load) of the composites and $\eta$ is a measure of preferential intensity of fiber orientation along the modal direction $\alpha$, which was taken from the longitudinal axis of the composites. The physical meaning of intensity of fiber orientation $\eta$ and modal direction $\alpha$ are given below. If all the fibers in a biocomposite are lying parallel to the longitudinal axis of the composite then $\eta$ tends to infinity and $\alpha$ is equal to zero. But, such idealized composite structures are never achieved in reality. In practice, the fibers always follow a continuous angular distribution and the modal angle may deviate from 0°. This happens due to the inherent characteristics of composite forming processes and the influences of the

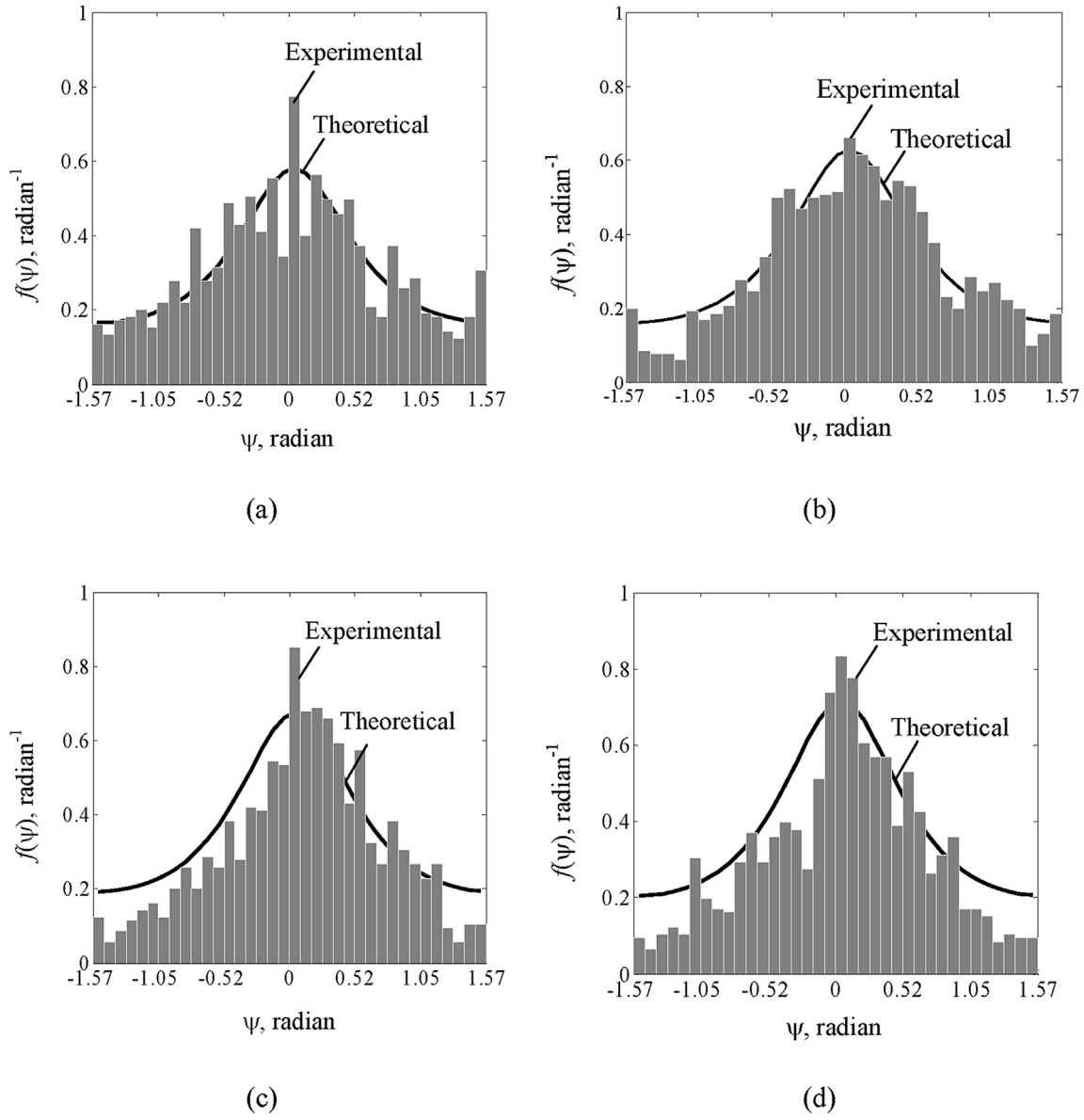


**Fig. 2.** Fiber orientation distributions biocomposites prepared at keeping cylinder speed as (a) 60 rpm (b) 175 rpm (c) 400 rpm and (d) 600 rpm.

surrounding fibers and the machine elements while forming the composites. The numerical values of η and $\alpha$ were obtained from the experimental data using a standard statistical regression technique. The results are reported in Table 1. It can be seen that the modal angle was too small and it did not practically change with the increase of cylinder speed, however, the measure of preferential intensity of fiber orientation increased with the increase of cylinder speed. This was an indicative of alignment of more fibers along the longitudinal axis of the composites which resulted in higher tensile strength of the biocomposites at higher cylinder speed.

### *3.2. Mechanical and physical properties of biocomposites and PLA film*

The mechanical properties of composites are very, if not the most, important for their applications in automotive dashboard panels. It was reported by Sapuan et al. [22] that the automotive dashboard panels should exhibit a tensile strength of at least 25 MPa and a Young's modulus of at least 2.3 GPa. Further, it was also reported by Ali et al. [14] that the PDS (product design specification) values for automotive door panels should exhibit a tensile strength of at least 22 MPa, a Young's modulus of at least 1.7 GPa, and a density of at least 980 kg/m$^3$. Table 2 reports on the density, tensile, flexural, and impact properties of the biocomposites and the pure poly(lactic acid) film prepared in this work. It was found that the density of the biocomposites decreased with the increase of nettle fiber content. It can be observed that the tensile strength, elongation-at-break, Young's modulus, flexural strength, and impact strength of the biocomposites increased initially with the increase of nettle fiber content and decreased afterwards. The optimum nettle fiber content in the biocomposites was found to be 50 wt%. The increase of the mechanical properties of the biocomposites was attributed to the reinforcement provided by the fibers to the matrix and the decrease of the properties was due to poor adhesion between reinforcement and matrix because of insufficient availability of the reinforcing nettle fibers in the biocomposites. Interestingly, the biocomposite prepared with equal weight proportion of nettle and poly(lactic acid) met the above-

**Table 2**
Mechanical and physical properties of biocomposites.

| Weight percentage of nettle and PLA fibers | Density kg/m$^3$ | Tensile strength, MPa | Elongation-at-break, % | Young's modulus, GPa | Flexural strength, MPa | Impact strength, kJ/m$^2$ |
|---|---|---|---|---|---|---|
| 0:100 | 1066.84 ± 15.17 | 2.21 ± 1.03 | 0.83 ± 0.21 | 2.21 ± 0.07 | 10.51 ± 1.89 | 1.95 ± 0.02 |
| 10:90 | 1046.79 ± 17.23 | 15.54 ± 0.45 | 1.42 ± 0.06 | 2.37 ± 0.05 | 30.42 ± 1.70 | 4.54 ± 0.16 |
| 25:75 | 1036.72 ± 19.21 | 36.70 ± 1.30 | 1.80 ± 0.08 | 3.11 ± 0.05 | 34.70 ± 0.93 | 16.54 ± 0.42 |
| 50:50 | 825.59 ± 12.79 | 50.82 ± 0.82 | 2.45 ± 0.10 | 3.34 ± 0.04 | 36.89 ± 0.98 | 28.76 ± 0.77 |
| 75:25 | 570.95 ± 11.94 | 27.04 ± 1.17 | 1.80 ± 0.09 | 2.33 ± 0.06 | 13.72 ± 0.72 | 14.31 ± 0.85 |
| 90:10 | 536.72 ± 12.68 | 15.92 ± 0.45 | 1.11 ± 0.06 | 2.19 ± 0.04 | 2.62 ± 0.25 | 3.34 ± 0.74 |

mentioned criteria for automotive dashboard panel applications exceedingly well. Fig. 3 depicts the microscopic images of tensile failure of the biocomposites prepared with 10%, 50%, and 90% nettle fibers. It can be noticed that as the fiber content in the matrix increased the dominant tensile failure mechanism shifted from fiber breakage to fiber slippage. A similar trend was observed in case of elongation-at-break and Young's modulus of the biocomposites. As reported by Rahman et al. [23], Haque et al. [24,25], and Karim et al. [26], the partially separated microspaces, created during tensile loading, put a barricade to the stress propagation between the fiber and the matrix, and the degree of this obstruction increased as the fiber content increased. Consequently, this resulted in higher stiffness of the biocomposites. The maximum obstruction took place at equal weight proportion of nettle and poly(lactic acid) fibers. However, when the nettle content was too high (90%), the biocomposite was too limpy and exhibited very low flexural strength. Further, at a higher nettle fiber content, the frequency of fiber agglomeration increased which resulted in creation of regions of stress concentration requiring less energy for crack propagation, which is also responsible for reduction in impact strength at higher nettle fiber content. Furthermore, it can be observed that the mechanical properties of the pure (poly lactic acid) film were inferior to those of the biocomposites. It can be therefore stated that the reinforcement of nettle fibers to the matrix resulted in improvement of the mechanical properties of the matrix. Further, Fig. 4 displays a series of SEM images of cryo-fractured biocomposites prepared with 10 wt% (a), 25 wt% (b), 50 wt% (c) and 75 wt% (d) nettle fibers. It clearly indicates the homogenious distribution of fibers and matrix in the biocomposites. It can be noticed that as the fiber weight percentage increased beyond 50 wt% of nettile fibers, the fiber agglomeration increased in the structure. This resulted in generation of more failure points thus tensile strength decreased with higher fiber weight percentage. A similar observation was reported by El-Shekeil et al. [27] and Shukor et al. [28] in case of kenaf fiber reinforced poly(vinyl chloride)/thermoplastic polyurethane composite and PLA/Kenaf/APP biocompostes respectively.

### 3.3. Thermogravimetric analysis of biocomposites and PLA film

Thermal stability is one of the key requirements of fibrous composites for their application in automotives. The thermal degradation behavior of the composites is very important as it might cause the mechanical properties of the composites to decrease, thus effectively rendering the composite products useless.

The thermogravimetric curves, illustrating the changes in weight of the biocomposites prepared with nettle fibers of 10 wt%, 25 wt%, 50 wt%, 75 wt%, and 90 wt% and the pure poly(lactic acid) film as a function of increasing temperature, are displayed in Fig. 5. As shown, all the thermogravimetric curves started with a small loss of weight indicating evaporation of absorbed moisture, followed by a single-stage decomposition resulting in a large loss of weight. The initial weight loss was found to be smallest with the pure (poly lactic acid) film and it increased with the increase of nettle fiber content in the biocomposites. A tentative reason for this behavior is given below. Being a natural fiber, nettle had a higher moisture regain than poly(lactic acid), hence the biocomposites prepared with higher amount of nettle fibers contained higher amount of moisture. Hence, on drying, the biocomposites prepared with higher amount of nettle fibers would be expected to show higher weight loss. The lowest temperatures at which the onset of weight losses were seen in the biocomposites prepared with 10%, 25%, 50%, 75%, and 90% and the pure poly(lactic acid) film were 315.3 °C, 321.73 °C, 324.66 °C, 326.33 °C, 328.54 °C, and 312.83 °C, respectively. This indicated that the initial decomposition in the biocomposites took place at a higher temperature than that happened in pure poly(lactic acid) film. Further, the initial decomposition temperature was higher at higher content of nettle fibers in the biocomposites. On further increase of temperature, the

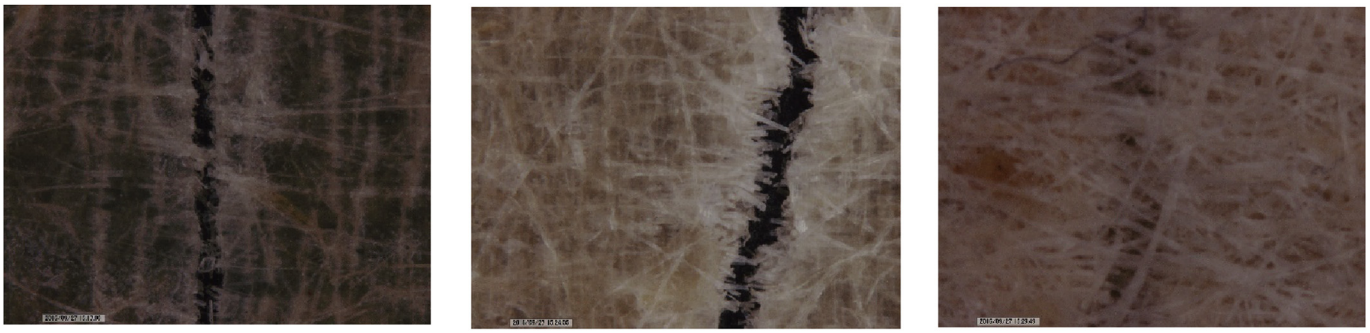


**Fig. 3.** Microscopic images of tensile failure of biocomposites prepared with 10 wt% (a), 50 wt% (b), and 90 wt% (c) nettle fibers.

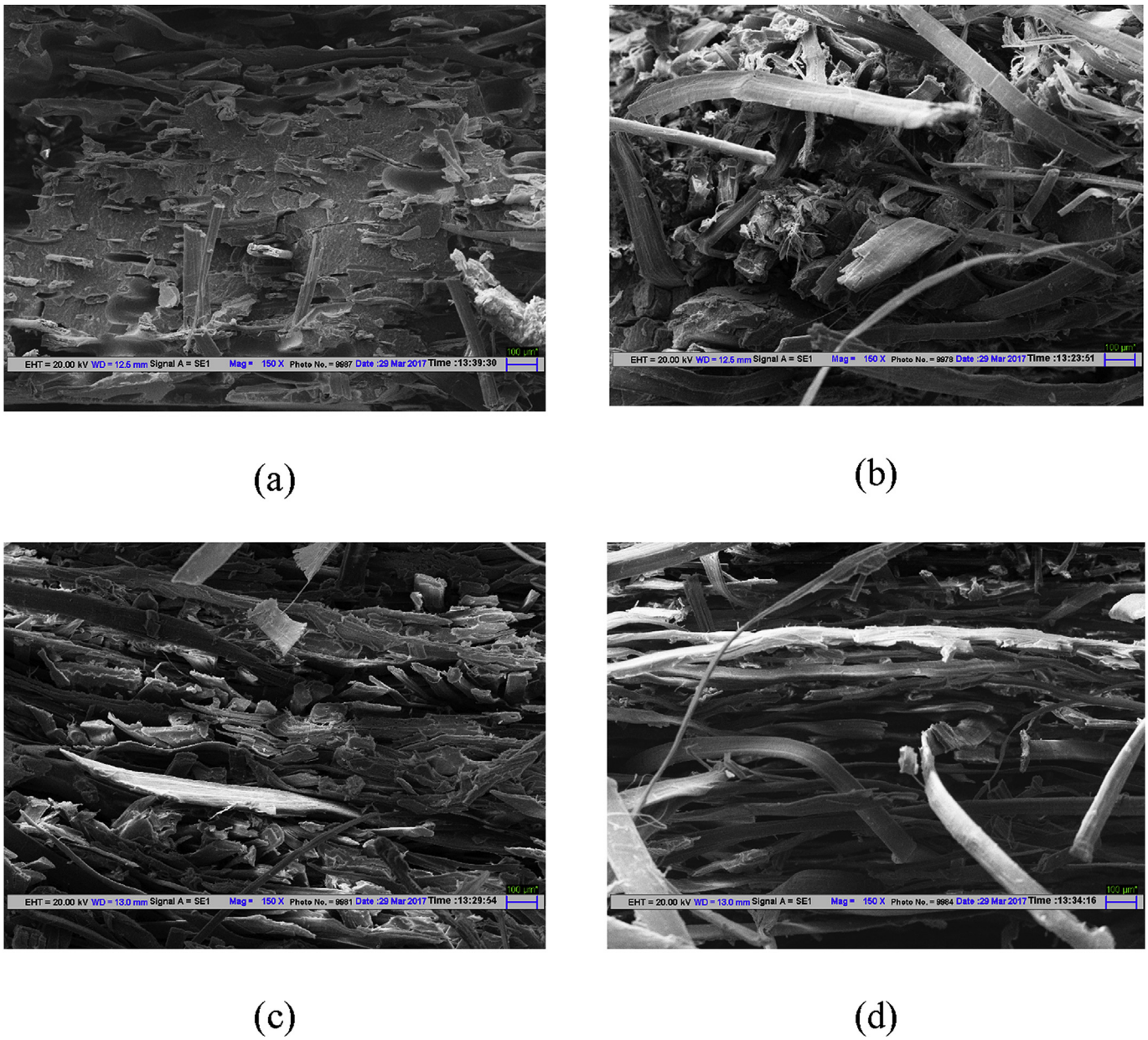


**Fig. 4.** SEM images of cryo-fractured biocomposites prepared with 10 wt% (a), 25 wt% (b), 50 wt% (c) and 75 wt% (d) nettle fibers.

biocomposites as well as the film exhibited significant decomposition, which was indicated by the steep slope of the thermogravimetric curves. This could be assigned to the decomposition of cellulose [29,30] which was very high (~86%) in nettle fiber, as reported by Netraveli and Pastore [30]. As the steepness of the curves decreased with the higher content of nettle fibers, it can be stated that the rate of weight loss was higher with the lower content of nettle fibers. The temperatures of half decomposition (50% weight loss) exhibited by the biocomposites prepared with 10%, 25%, 50%, 75%, and 90% and the pure poly(lactic acid) film were 350 °C, 350.5 °C, 352 °C, 355.5 °C, 361.5 °C, and 351 °C, respectively. Once the temperature was raised to 400 °C, the biocomposites prepared with 10%, 25%, 50%, 75%, and 90% and the film lost around 96.08%, 91.71%, 86.85%, 77.31%, and 80.72%, and 97.59% of their initial weight. On further increase of temperature, a very small amount of weight loss was observed. This could be assigned to the decomposition of lignin [29], which was present in a small amount in lignocellulosic nettle fiber [30]. The thermogravimetric analysis inferred that the biocomposites were enough thermally stable and their thermal stability increased with the increase of nettle fiber content in the biocomposites.

### 3.4. Dynamic mechanical analysis of biocomposites and PLA film

The dynamic mechanical analysis of the biocomposites and the pure poly(lactic acid) film was carried out to examine their viscoelastic behavior against temperature, time, and frequency. This, in turn, would help to assess the performance of the biocomposites in automotive applications. The viscoelastic behavior was examined in terms of three parameters: storage modulus, loss modulus, and damping factor (tan δ).

Fig. 6 displays the storage modulus of the biocomposites and the pure poly(lactic acid) film at different temperatures. It basically depicts the energy stored in the biocomposites and the film as a

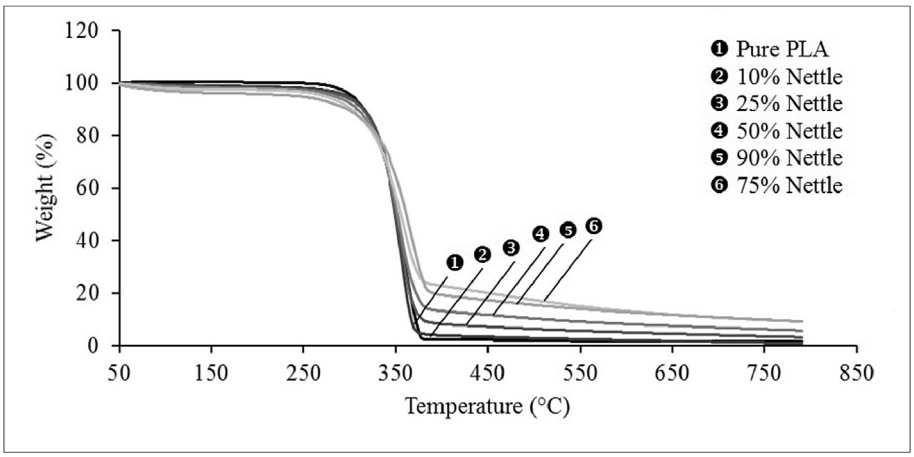


**Fig. 5.** Thermogravimetric analysis of biocomposites and pure poly(lactic acid) film.

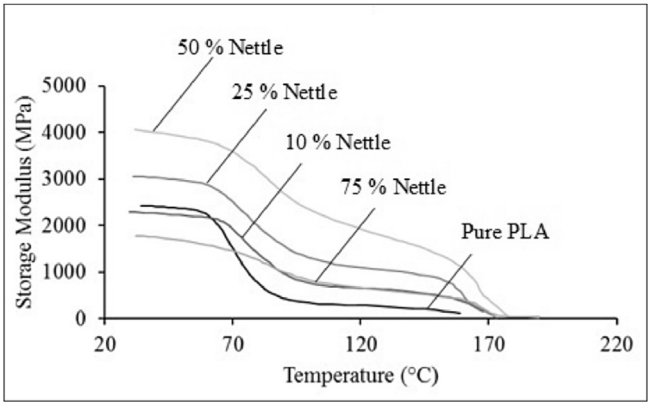


**Fig. 6.** Plot of storage modulus against temperature.

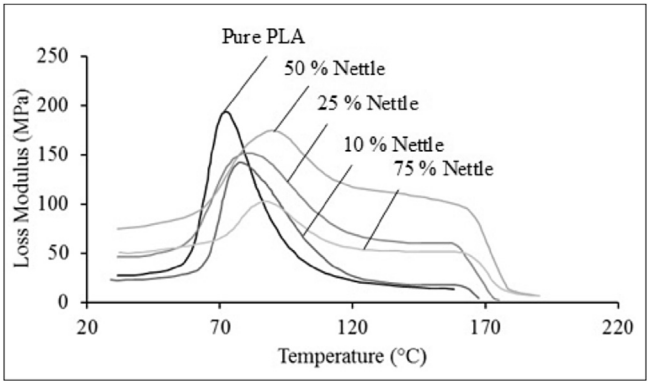


**Fig. 7.** Plot of loss modulus against temperature.

function of temperature. As expected, the storage modulus was found to decrease with the increase of temperature. The biocomposite prepared with equal weight proportion of nettle and poly(lactic acid) exhibited the highest storage modulus at all temperatures, followed by the biocomposites prepared with 25 wt%, 10 wt%, and 75 wt% nettle fibers, respectively. It was interesting to note that the addition of 50 wt% nettle fibers to poly (lactic acid) resulted in remarkable enhancement of storage modulus from 2.427 GPa of pure poly(lactic acid) to 4.024 GPa of the biocomposite at 35 °C temperature. Further, the rate of reduction in storage modulus with respect to temperature decreased with the addition of nettle fibers to poly(lactic acid). A similar behavior was observed in case of flax fiber reinforced high-density polyethylene (HDPE) composites [31] and PLA/kenaf/APP biocomposites [28]. This was attributed to the reinforcement disseminated by nettle fibers, causing the mobility of the polymer chains to reduce greatly with the addition of nettle fibers. A sudden fall of storage modulus happened at around 70 °C as the composites approached to their glass transition temperature that caused free molecular movement of the polymer chains.

Fig. 7 depicts the loss modulus of the biocomposites and the pure poly(lactic acid) film at different temperatures. It represents the dissipated energy of the biocomposites and the film as a function of temperature. It can be observed that the loss modulus of the biocomposites and the film initially increased with the increase of temperature and decreased afterwards. The maximum values of loss modulus of pure poly(lactic acid) and the biocomposites prepared with 10 wt%, 25 wt%, 50 wt%, and 75 wt% nettle fibers were observed as 193.75 MPa, 142.08 MPa, 151.77 MPa, 174.49 MPa, and 102.77 MPa respectively at temperatures 72.06 °C, 77.59 °C, 79.58 °C, 89.69 °C, and 86.69 °C respectively. It can be thus seen that the biocomposite prepared with equal weight proportion of nettle and poly(lactic acid) displayed the highest loss modulus among all the biocomposites studied in this work. As a higher value of loss modulus indicated a higher mobility of the polymer matrix that attributed to enhancement of toughness and energy absorption potency, the biocomposite prepared with equal weight proportion of nettle and poly(lactic acid) was the toughest and displayed the highest potential of energy absorption among all the biocomposites studied in this work. A similar trend was reported for PLA/kenaf/APP biocomposites [28] and flax fiber reinforced high-density polyethylene (HDPE) composites [31].

Fig. 8 shows the damping factor (tan δ) of the biocomposites and the pure poly(lactic acid) film at different temperatures. This behavior helps to obtain the glass transition temperature of the biocomposites and the film. The glass transition temperatures of the biocomposites prepared with nettle fiber content of 10 wt%, 25 wt%, 50 wt%, and 75 wt% were 90.41 °C, 91.62 °C, 94.12 °C, 90.82 °C, respectively. At these temperatures the respective peak values of tan δ were 0.112, 0.090, 0.067, 0.100. It can be seen that the glass transition temperature increased but the peak tan δ decreased as the nettle fiber content increased till 50 wt% and then the glass transition temperature decreased but peak tan δ increased. Beyond the glass transition temperature, the transition of the biocomposites from glassy state to rubbery state was expected to take

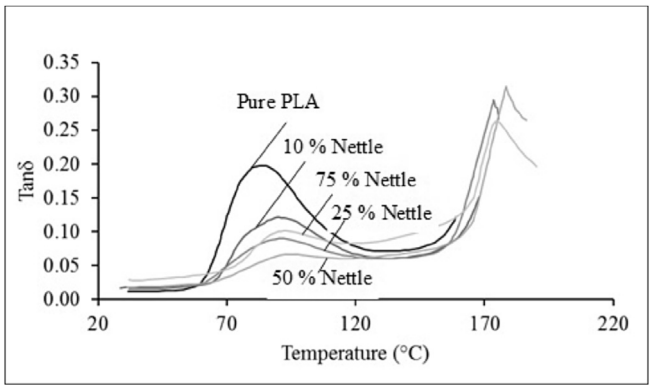


**Fig. 8.** Plot of damping factor against temperature.

place due to the high mobility of the molecular chains of the polymer. The pure poly(lactic acid) film exhibited a relatively sharp and intense peak centered at 83.55 °C, as there was no restriction to the polymer molecular chains at glass transition temperature. The higher peak tan δ value (0.198) of the film indicated that the pure poly(lactic acid) was more viscous as compared to the biocomposites.

The dynamic mechanical analysis of the biocomposites suggested that the optimum temperature range for their applications was ranging from 30 °C to 70 °C. Beyond 70 °C, the biocomposites reached to a rubbery state where their performance could be diminished.

### 3.5. Biodegradability of biocomposites

The biodegradability of automotives and their components is a growing concern in the world today. As the biocomposites developed in this work were intended to have automotive dashboard application, it was necessary to evaluate the biodegradability of the biocomposites prepared from nettle and poly(lactic acid) fibers.

Fig. 9 displays the loss of weight and loss of tensile strength of the biocomposites after burial in soil for 20 days. The biocomposites prepared with nettle fiber content of 10 wt%, 25 wt%, 50 wt%, 75 wt %, and 90 wt% exhibited loss of weight as 3.65%, 7.59%, 12.78%, 19.67%, and 24.93%, respectively and loss of strength as 37.11%, 84.33%, 85.23%, 91.33%, and 97.29%, respectively. Clearly, as the nettle fiber content increased, the biocomposites experienced higher weight loss and higher strength loss. Though the loss of weight seemed to be linear, the loss of strength appeared to be non-linear. The non-linearity of loss of strength is arising because of the relatively low loss of strength showed by the biocomposite prepared with 10 wt% nettle and 90 wt% poly(lactic acid). It was reported by Rashdi et al. [32] that the increase of fiber weight percentage in composites led to the decrease in the tensile properties of the specimens buried under soil. It was observed that the structure of this biocomposite after burial in soil was relatively less open as compared to those prepared with higher nettle fiber content. Further, it was observed that there was not enough residue of biocomposites left for measurements of weight and tensile strength after 45 days of burial in soil, except for biocomposite prepared with 10 wt% nettle fibers. This biocomposite exhibited a weight loss of 6.57% ± 0.72% and strength loss of 88.24% ± 1.58%. It is therefore evident that nettle showed higher biodegradability than poly(lactic acid). Probably, this was due to a greater effect of preferential hydrolysis in case of nettle as compared to poly(lactic acid), where the polymers were broken down into smaller units by micro-organisms. It was reported by Sapuan et al. [33] that the tensile strength of kenaf fibre reinforced thermoplastic polyurethane composites decreased after the soil burial test.

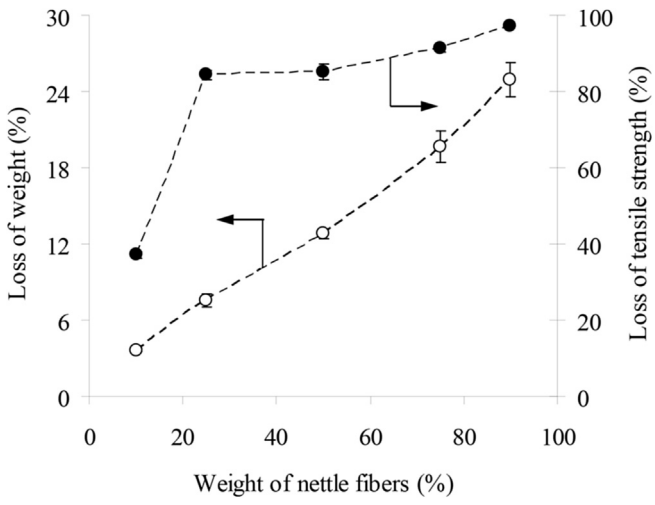


**Fig. 9.** Weight loss and strength loss of biocomposites after burial in soil for 20 days.

## 4. Conclusions

A series of fibrous biocomposites, prepared by using alkali-treated nettle fibers and poly(lactic acid) fibers in different weight proportions and employing carding and compression-molding processes, was evaluated for static and dynamic mechanical properties, thermogravimetric behavior, and biodegradability. The role of carding process in determining the tensile strength of the biocomposites was analyzed. It was observed that at higher cylinder speed, the tensile strength of the biocomposites increased, but at the cost of higher width-wise shrinkage of fiber-webs. The increase in tensile strength at higher cylinder speed was attributed to the preferential alignment of more fibers along the longitudinal axis (direction of applied load) of the biocomposites. The tensile, bending, and impact properties of the biocomposites initially increased with the increase of nettle fiber content and decreased afterwards. The maxima of these properties were obtained at equal weight proportion of nettle and poly(lactic acid) fibers. The thermogravimetric analysis inferred that the biocomposites were thermally enough stable and their thermal stability increased with the increase of nettle fiber content. The dynamic mechanical analysis suggested that the biocomposites were extremely good in terms of dynamic mechanical properties. Further, the biocomposites exhibited excellent biodegradability and their biodegradability increased with the increase of nettle fiber content. Overall, the biocomposite prepared with equal weight proportion of nettle and poly(lactic acid) showed high potential for automotive dashboard panel application.

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