

An Experimental Investigation on Short and Long Fiber Reinforcement in Tampico and Palymira Reinforced LLDPE Produced by Rotational Moulding Process.

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Research Article

Keywords: Rotational Moulding, Natural fiber composites, Vibration Damping, Mechanical Characterization, Morphological Characterization

Posted Date: May 28th, 2021

DOI: <https://doi.org/10.21203/rs.3.rs-554781/v1>

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Abstract

Rotational moulding is a powder processing technology widely used to manufacture hollow plastic products. It is a well-known process used for the manufacturing of large containers, water tanks, fuel tanks, refrigerated containers etc. The present work aimed at the incorporation of Tampico and Palmyra fibers as reinforcement in Linear Low-Density Polyethylene (LLDPE) matrix. The effect of fiber length on various properties like mechanical, morphological and vibration damping characteristics of the composites were investigated. The fibers were initially treated with 5% NaOH solution for better reinforcing efficiency and to improve the degradation temperature. Thermo Gravimetry Analysis (TGA) reported that a sufficient increment in degradation temperature is achieved by NaOH treatment on fibers. The short fiber composites considered for the study has obtained better mechanical strength than long fiber reinforced LLDPE composites. The results proved that fiber length has less effect on the vibration damping characteristics of the composites, but the percentage of fiber content is a significant factor for improving the natural frequency of the composites.

Introduction

Rotational moulding is a well-known technique used to produce hollow non-metallic stress-free products. The process is also known as roto moulding or roto casting. The process involves heating the polymer powder in a closed mould which is rotating bi axially inside a heating chamber or furnace[1]. Theoretically, it is possible to rotomould any plastic which melts while heating but practically most of them degrade during rotational moulding. The thickness of the finished product depends upon the weight of the polymer powder fed into the mould. The mould is rotated biaxially at a very slow speed, and then it is cooled to room temperature either by natural cooling or induced cooling[2]. The strength of the product, surface roughness and sintering efficiency depends upon the quality, size and shape of powder used for processing. Since there is no external force applied, the product obtained is relatively stress-free as compared to other polymer processing techniques.

Recent advancements in the rotational moulding are the development of composite hollow products. Natural fiber finds its application as a good reinforcement in polymer composites due to its better strength and also the biodegradable nature[3]. Studies reported that the mercerization treatment done on natural fibers leads to better tensile properties[4]. The use of natural fibers as reinforcement has some vital advantages like cost reduction, weight reduction, bio degradable nature etc., supports the usage of natural fibers than synthetic fibers now a days[3], [5]. Earlier studies reported on banana and abaca fibre-reinforced polyethylene (PE) composites prepared with 5% fiber addition results in improved elastic modulus and reduced impact strength as compared with unreinforced PE[6]. Cabuya and Sisal fibers were used as the potential reinforcement to High-Density Poly Ethylene (HDPE) with a fiber length of 5 mm and reported that Cabuya fiber composites show better impact strength than sisal fiber composites[7]. The optimum fiber content for better mechanical properties was estimated to be 5 wt% for long fibers of 5mm length. Fiber addition beyond that optimum composition drastically reduced the mechanical properties. Natural fiber finds its application as potential reinforcement in polymer

composites due to its complicated structures, better mechanical strength, and biodegradable nature. Many research works extended the investigation towards the biodegradability, moisture absorption, vibration damping and thickness swelling of natural fiber reinforced polymer composites (NFRPC)[8], [9]. Luffa fiber reinforced epoxy composites were investigated and found that the addition of fiber increases the moisture absorption and hence due to this increased moisture absorption, the mechanical properties were decreased[10]. Many researchers recommended the use of bamboo fibers since it possesses better tensile strength and flexural strength compared to other fibers[10]–[14]. The use of filler particles such as Rice Husk in LLDPE matrix has been recently reported with the addition of nano silica and nano clay[15]. The Rotational moulding process with natural fiber as reinforcement has limited number of literatures reported. The primary focus of the present work is to investigate the mouldability of natural fiber reinforced polymer composites produced by rotational moulding process. Two novel fibers were identified as a potential reinforcement in LLDPE matrix. Reinforcement with Palmyra fibers and Tampico fibers were not yet reported in LLDPE matrix and hence these fibers are considered for the present work. Palmyra fibers are medium coarse fibers available in southernmost regions of India whereas Tampico fibers are imported from Mexico. The present work investigates the mouldability as well as the mechanical, morphological, and vibration damping properties of palmyra and Tampico fiber reinforced LLDPE by rotomoulding process.

Materials And Methods

Table 1
Palmyra and Tampico fiber Composition.

Fiber Compositions	Palmyra Fiber	Tampico Fiber
Extractives %	6.4	45.34
Lignin %	18.54	3.6
Holocellulose %	68.5	17.72
Fiber Diameter (mm)	0.4	0.25

Fiber treatment

Tampico and palmyra fibers were collected and chopped to the specified length with the help of a chaff cutter. The chopped fibers were manually cleaned in distilled water and dried exposed to sunlight for 8 hours after rinsing with the help of a centrifuge. The dried fibers were collected and immersed in 5% NaOH solution for 24 hours to remove the lignin and extractives on the surface of fibers. Then it is dried in sunlight for two days to remove the moisture content. The stearic acid is applied to ensure uniform fiber dispersion and to eliminate agglomeration. The palmyra and Tampico fibers chemical compositions are shown in Table 1.

Preperation of composites

The composites were prepared using a lab model rotational moulding machine with a mould dimension of 32cm X 32cm X 34cm. The charge weight is calculated for a mould thickness of 5mm. The metered quantity of LLDPE powder with different fiber content is fed to the stainless-steel mould and rotated biaxially. Silicon spray is applied gently over the inner mould surface in order to remove the final product easily. Both Tampico and palymira fibers were added at different wt% of 5, 10 & 15. The LLDPE powder along with the fibers was dry blended in a blending machine for a few minutes. The speed ratio of the rotomoulding machine was set as 1:4. An electric motor attached to the major arm ensures the driving torque. The heating phase of 30 minutes is followed by fan cooling until room temperature is achieved [16]. The heat is transferred to the mould, then to the composite powders. Cooling is achieved with the help of a high-speed fan which continues for about 30 minutes after processing time. The composite designations are indicated in table No 2.

Table 2
The composite designation

Matrix	Fiber	Fiber Type	% Weight of fiber	Designation
LLDPE	NIL	NIL	0	LLDPE
LLDPE	Tampico	Long	5	TL1
LLDPE	Tampico	Short	5	TS1
LLDPE	Tampico	Long	10	TL2
LLDPE	Tampico	Short	10	TS2
LLDPE	Tampico	Long	15	TL3
LLDPE	Tampico	Short	15	TS3
LLDPE	Palymira	Long	5	PL1
LLDPE	Palymira	Short	5	PS1
LLDPE	Palymira	Long	10	PL2
LLDPE	Palymira	Short	10	PS2
LLDPE	Palymira	Long	15	PL3
LLDPE	Palymira	Short	15	PS3

Mechanical Characterization

Estimation of mechanical properties such as tensile strength, flexural strength, impact strength and hardness is essential for the novel composite developed. The specimens are prepared as per ASTM standards with the help of a water jet cutting machine in order to avoid excessive pressure and

delamination of fibers. Tensile strength was determined as per ASTM D638. Samples are cut from all the six sides of the composite cube. The values obtained are averaged to obtain the tensile strength of the composite product. ASTM D 790 standards were followed for investigating the flexural strength of the composites. Instron Universal testing machine was used for both the analysis. Shore D Hardness values were measured by taking the average of 8 different indentation points. The toughness of the composites was evaluated from the impact strength determined as per ASTM D256.

Morphological Characterization.

A Scanning Electron Microscope (SEM) is used to determine the fibre-matrix adhesion and interaction. TESCAN- VEGA3 machine was used for the characterization. JEOL-JFC 1600 auto fine coater is used for gold sputtering over the exposed surface. Sputtering enhances the electron interaction with the sample surface thereby initiating a conductive layer over a non-conducting polymer. Fractured surfaces were analysed to determine the fibre-matrix adhesion and to investigate micro-macro cracks and void formation inside the matrix.

Vibration damping characteristics.

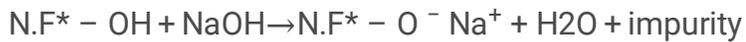
Experimental Modal Analysis (EMA) is conducted to determine the natural frequency of the composites. Figure 1 shows the schematic of the experimental set up for EMA. For conducting the vibration analysis, ASTM E756 procedure is followed. The specimen is clamped in cantilever position at one end and kept free at the other end. An accelerometer of miniature size is attached with the specimen to acquire the displacement signals. An impact hammer with rubber bush attached is used for the excitation of the cantilever beam. The signals from the accelerometer are analysed with the help of a computer to determine the natural frequency of the samples.

Results And Discussions

FTIR Spectroscopy

FTIR analysis was carried out to analyze the effect of NaOH treatment on Tampico and palmyra fibers. The interaction of natural fibers with functional groups can be analysed with the help of FTIR graph. The infrared (IR) spectrum stretches from 12800cm^{-1} to 200cm^{-1} . These wavenumbers are subclassified as near, middle, and far spectrums. The region of middle to far in the IR spectrum comprises of wave number 4000cm^{-1} to 200cm^{-1} which is usually considered for the study of natural fibers. Untreated Palmyra Fiber (UPF) and Untreated Tampico Fiber (UTF) were compared with the spectral images of Treated Palmyra Fiber (TPF) and Treated Tampico Fiber (TTF). It can be observed from Fig. 2 that the peak at 1449cm^{-1} and 1433cm^{-1} for UPF and UTF respectively were removed for TTF and TPF. This implies the fingerprint region with C-O, C-N or C-C bonds were eliminated by NaOH treatment done on fibers. The peaks indications in UTF and UPF between 500 and 750cm^{-1} confirms the presence of

cellulose which is absent in the spectra of treated fibres. The reaction of natural fibre (N.F*) with NaOH is expressed as[1]



Thermo Gravimetric Analysis (TGA)

TGA is conducted using Perkin Elmer STA 6000 Thermo - Gravimetric Analyzer with a resolution of 0.1µg. The experiment is conducted in a controlled atmosphere containing nitrogen. Heating rate is selected as 10-degree Celcius per minute starting at room temperature to 950 °C. The nitrogen gas is supplied at the rate of 20ml/minute. An alumina crucible supported by a precision balance is used to hold the powdered specimen weights 5mg. TGA is done in order to characterise the materials by measuring the change in mass as a function of temperature. Treated and untreated fiber specimens were investigated and TGA – DTG curve has been plotted as shown in the Fig. 3. The first stage of degradation for Tampico fibers (Fig. 3a) was improved from 232 °C to 251 °C for NaoH treated fibers. The second stage of degradation was found to be shifted to 362 °C from 343 °C for treated Tampico fibers compared with untreated one. A similar hike in degradation temperature is observed for palymira fiber also (Fig. 3c). The derivative thermogravimetric (DTG) curve plotted for Tampico and palymira fibers are shown in Fig. 3 (b & d), respectively. From the DTG curve it can be inferred that the degradation peak is obtained at a higher temperature for treated fibers than untreated fibers. This proves that the fiber treatment done with NaOH was effective.

Mechanical Characterization

Effect of fiber content on tensile strength

Tensile properties of all the 12 composites were conducted as per ASTM D638 standards and compared with unreinforced LLDPE. From Fig. 4, it can be observed that Tampico fiber composites showed better tensile properties than palymira fibre-reinforced composites. It was found that by the addition of fibers in the form of long fibers, the tensile properties of TL3 composite was decreased to 14% as compared to TL1. At the same time the introduction of short fibers enhanced the tensile strength of TS3 composite to 18.25MPa compared with 16.5 MPa of TS1. A similar trend was observed for palymira fiber reinforced composites also. Tensile strength of PL3 is decreased by 19% compared to PL1, but the inverse effect was observed in the case of short palymira fiber composites. During tensile deformation, the short fibers were able to impart better adhesion with the LLDPE matrix, and the long fibers shows debonding and poor interfacial properties with LLDPE matrix. Since the process in a low shear process with no external pressure is applied while manufacturing the composites, the void formations will be more as compared with other polymer processing techniques. The void formation inversely affects the tensile properties sine the crack formation is initiated through the voids.

Effect of fiber content on Flexural strength

Flexural strength is the stress experienced in the material at the yield moment. It is measured as per ASTM D 790 and plotted in Fig. 5. Flexural strength shows a similar trend as that of tensile strength results. The reinforcement with short Tampico fiber at 15wt% shows a percentage increase in flexural strength of 22.5% whereas short Palmyra fiber at 15wt% gives an increment of 20%. Both TL3 and PL3 composites marked poor flexural strength compared to other composites. While processing the composites, the long fibers are randomly oriented and found to be agglomerated at the corners of the cubical mould. But the short fibers were evenly distributed to all the sides of the mould. This is due to the process characteristics. Rotational moulding is a powder processing technology, and long fibers incorporation reduces the slushing effect required for perfectly cooked mould. This is the reason for the reduction in flexural strength when processing with long fibers and an increase in strength when processing with short fibers.

Effect of fiber content on Impact Strength

ASTM D 256 standards were followed for the investigation of impact strength of the composites. A significant hike in impact strength was inferred when comparing long and short fiber composites, as shown in Fig. 6. On 5% fiber addition, TS1 marked a percentage increase of 5.5% in impact strength than TL1. But on 15% fiber addition, an increase of 65% in strength was observed. The same trend was observed in the case of Palmyra fibre-reinforced composites also. PS1 marked a percentage increase of 6% than PL1. But on 15% fiber addition, the percentage hike improved to 41.3% for PS3 compared to PS1. The powdered LLDPE has better bonding with short fibers than long fibers thereby resulting in homogeneous reinforcement than long fiber composites. The number of fiber particles per square centimetre in extrados of mould for short fiber composites is more compared with long fiber composites. This is the reason for achieving high impact strength for short fiber composites than long fiber composites.

Effect of fiber content on Hardness.

Hardness values of all the 12 composites were estimated and compared with unreinforced LLDPE are shown in Fig. 7. It can be inferred that by the addition of natural fibers, the hardness values of the composites increased for both long and short fiber reinforcement. Even though the percentage increase is very marginal, the hike in hardness values promises the surface properties are improved by the addition of natural fibers. There is an improvement of 3.5 and 2.5 shore D points for TS3 and PS3 respectively compared with a hardness value of 58 obtained for Untreated LLDPE. The surface treatment done on fibers were effective in improving the mechanical properties of the composites. But the hardness values depend on the surface quality of the product. In rotational moulding, the bubble formation is found on the surface of the composite product also. The void formation is more in rotational moulding process compared to other polymer processing techniques. No external pressure is applied to remove the air bubbles entrapped in this process. But it can be concluded from the hardness results that the surface void formation is less by the addition of natural fibers.

Morphological Characterization.

The fractured surface of tensile test specimens was analysed morphologically in order to determine the fibre-matrix adhesion and interaction. The SEM images of PL1, PS1, PL3 and PS3 samples are included as a,b,c and d respectively. In Fig. 8 (a) it is clearly visible that the fiber and matrix bonding is improper, and the presence of voids and microvoids are also visible. But when considering the PS1 samples, the fibre-matrix bonding is perfect. The fibers surface seems to be peeled off during the application of tensile force and still the matrix fiber adhesion seems to be perfect. This is the reason for improved mechanical strength observed for PS1 when compared with PL1. Figure 8 (c) & (d) depicts the images of fractured surfaces of PL3 and PS3 composites, respectively. The fibre-matrix adhesion and bonding are better in PS3 composites where matrix crack, void formations etc. like defects are not visible at all. But the defects like fiber agglomeration and debonding of fibers can be observed in Fig. 8 (c) which represents PL3 composite. This is the reason for the decrement in mechanical properties obtained when long fibers are further considered upto 15 wt %. Since rotomoulding is a powder processing technique, the fibers can perform better when it is added in powdered form. This can be clearly inferred from the SEM images of PS1 and PS3 composites.

In the Fig. 9 (a), it can be observed that the matrix resin fails to penetrate into the bundle of fibers which leads to the fiber matrix debonding and finally results in poor mechanical properties. Even though the random fiber dispersion is achieved for TL3 composites, the matrix crack and void formations lead to a drastic reduction in mechanical strength when compared to TL1 and TL2 composites. The Fig. 9 (b) indicate the strong interfacial bonding of Tampico fibers with LLDPE matrix, which results in improved mechanical properties of TS3 composites.

Vibration Damping characteristics

The frequency at which the composite material resonates is termed as its natural frequency. The aim of incorporating the fibers in the present study is to enhance the natural frequency of the composites, thereby achieving better stiffness. The most commonly used experimental modal analysis technique is hammer testing method. Here the composite bar is excited with an impact hammer attached with force transducer at a number of points, and the response is measured at a single point. A fast fourier transform analyser is used to generate a frequency response function (FRF) as output. The peak indications in the FRF curve represents the natural frequency of the composites. Figure 10 represents the Frequency response curve of Tampico fiber reinforced LLDPE compared with that of unreinforced LLDPE. As the percentage of fibers increases, the natural frequency of the composites also found to be increased for both short and long fiber composites. It is clear from the results that the length of fibers is not significant, but the percentage of fibers is the significant parameter. The natural frequency of TL3 composites has obtained an increase of 131% compared to unreinforced LLDPE, whereas TS3 composite shows an increment of 120% when compared to LLDPE. The results conclude that fiber length plays an insignificant role in EMA results for Tampico fiber composites.

When considering the palymira fiber composites, the results obtained was much better compared to that of Tampico fiber composites results, as shown in Fig. 11. PL3 composites obtained an increase in natural frequency of 166% when compared to unreinforced LLDPE whereas PS3 composites could mark an

increase of 135%. The results conclude that the fiber, when included as long fibers, shows better vibration damping properties than short fiber composites. Also, Palymira fibers are suggested as a potential vibration damping material for natural fiber reinforced polymer composite structures produced by rotational moulding process.

Conclusions

The present work investigates the mouldability of LLDPE with palymira fiber and Tampico fiber as reinforcement and successfully incorporated these fibers under three different weight percentages. The mechanical properties and vibration damping properties of unreinforced LLDPE is compared with short fiber and long fiber reinforced LLDPE, and the results are summarized as follows.

The FTIR results showed that the NaOH treatment done on fibers were effective in removing the excess dirt and cellulose, thereby reducing the hydrophobic nature of fibers. This results in improving the mechanical properties of the composites. The thermal degradation of both treated and untreated fibers were examined using thermogravimetric analysis and the results shows that the degradation temperature can be effectively improved by proper NaOH treatment on fibers. The tensile properties of the composites were improved by the addition of short fibers. By increasing the fiber content, the long fibers find difficult to adhere with the matrix, thereby decreasing the tensile strength. There is an increment of 30% in tensile strength reported for PS3 compared to PL3. In the case of Tampico fiber reinforced composites, the TS3 composite shows an increase of 50% in tensile strength when compared with TL3 composite. Flexural properties also depict a similar trend as that of tensile properties. The PS3 composites obtained a flexural strength of 17.96MPa which is 38% more than that of PL3 composites. The better adhesion of short fibers with LLDPE matrix, as shown in morphological studies, helps to achieve this increment in flexural properties. The TS3 composites have better flexural strength than TL3 composites as an increment of 44% is observed in the results. The fiber content and fiber length have a significant role on the impact strength of the composites prepared by rotational moulding process. Short fiber composites have better impact properties compared to that of long fiber composites. By the addition of long fibers from 5–15% by weight, the impact strength is found to be decreased in the case of long fiber reinforcement and found to be improved in the case of short fiber composites. The reinforcement as both short and long fibers has no significant impact on the hardness values. Since the fiber addition only up to 15wt% is considered for the study, the hardness values are found to be almost same as that of unreinforced LLDPE. The vibration damping experimentation results are most significant in the present study. There is a significant hike in the natural frequency of the composites considered for the study when compared to the unreinforced one. Thus, it can be suggested that the palymira and Tampico fiber reinforced rotomoulded products are a better alternative for automotive-related parts since the natural frequency of the product is improved, which leads to minimal vibrations.

Declarations

Funding:

No funding was received to assist with the preparation of this manuscript.

Conflicts of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Availability of data and material

Any data related to the present work may be disclosed under request.

Code availability

Not applicable

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Figures

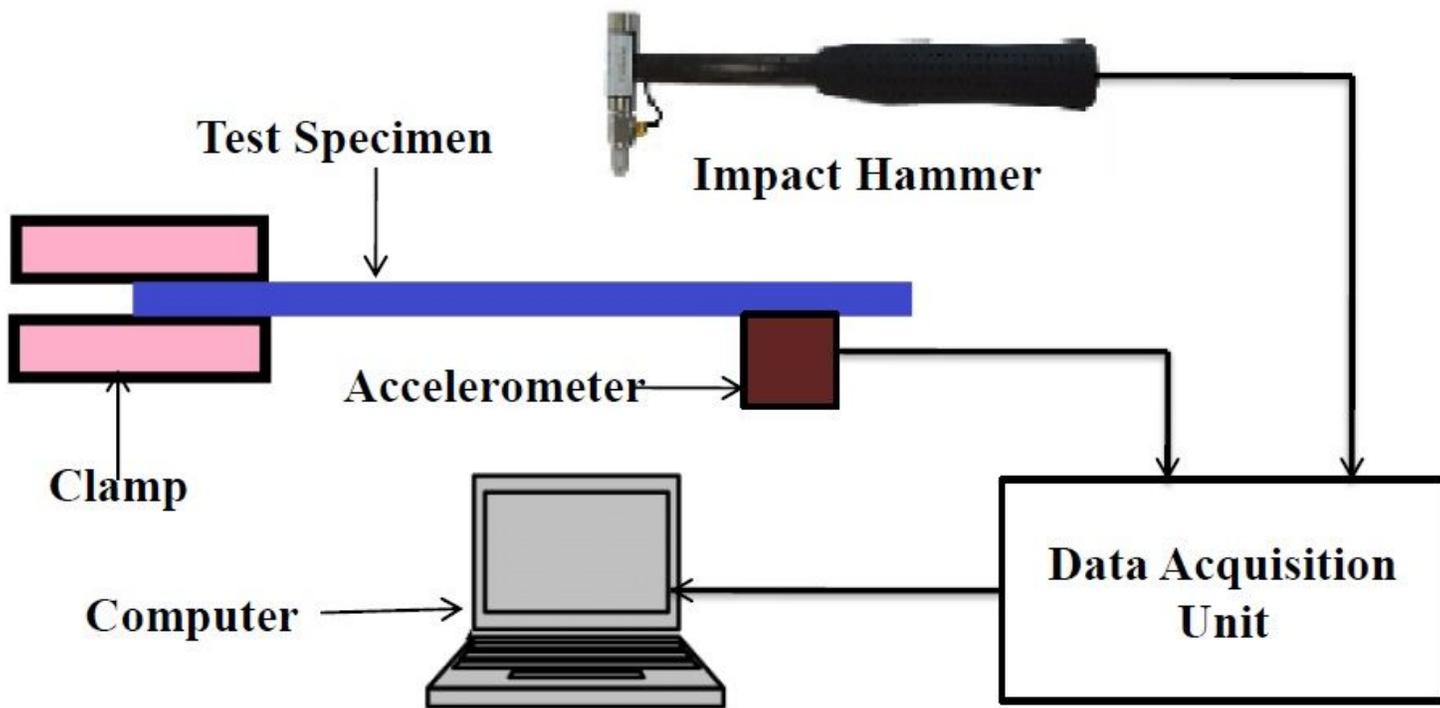


Figure 1

The experimental setup for the estimation of Vibration damping characteristics.

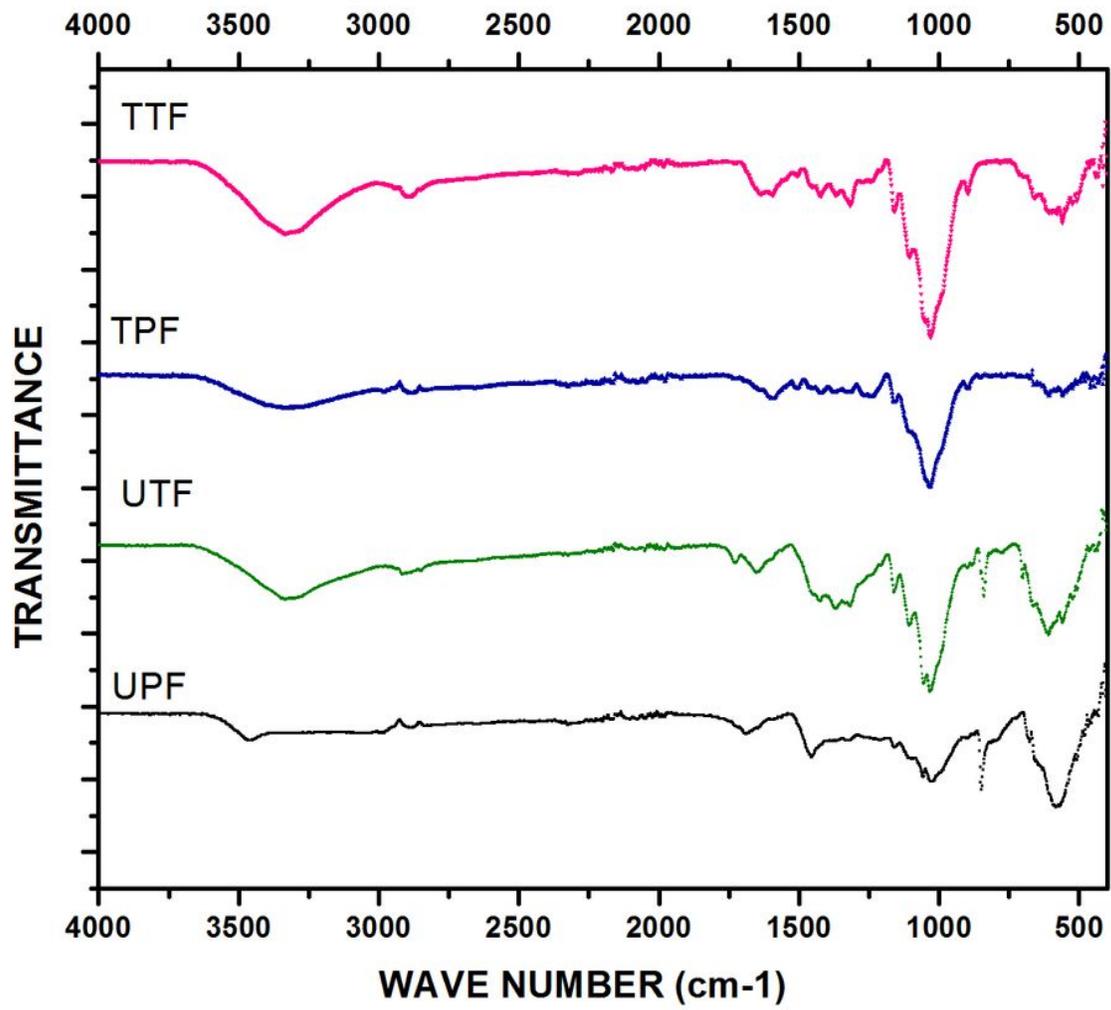


Figure 2

FTIR results of treated and untreated palmyra and Tampico fibers

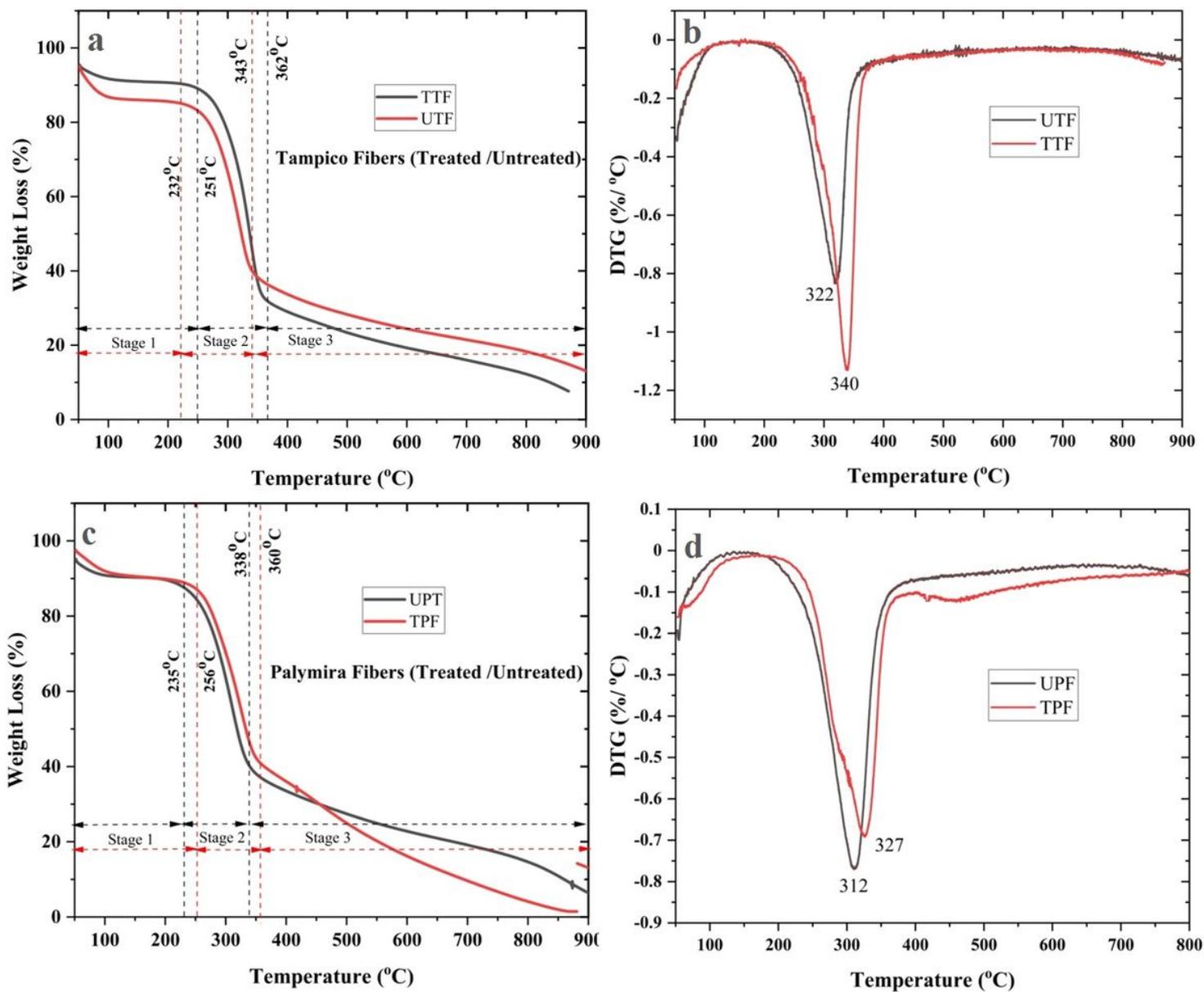


Figure 3

(a) TGA curve for tampico fibers (b) DTG curve for tampico fibers (c) TGA curve for palmyra fibers and (d) DTG curve for palmyra fibers

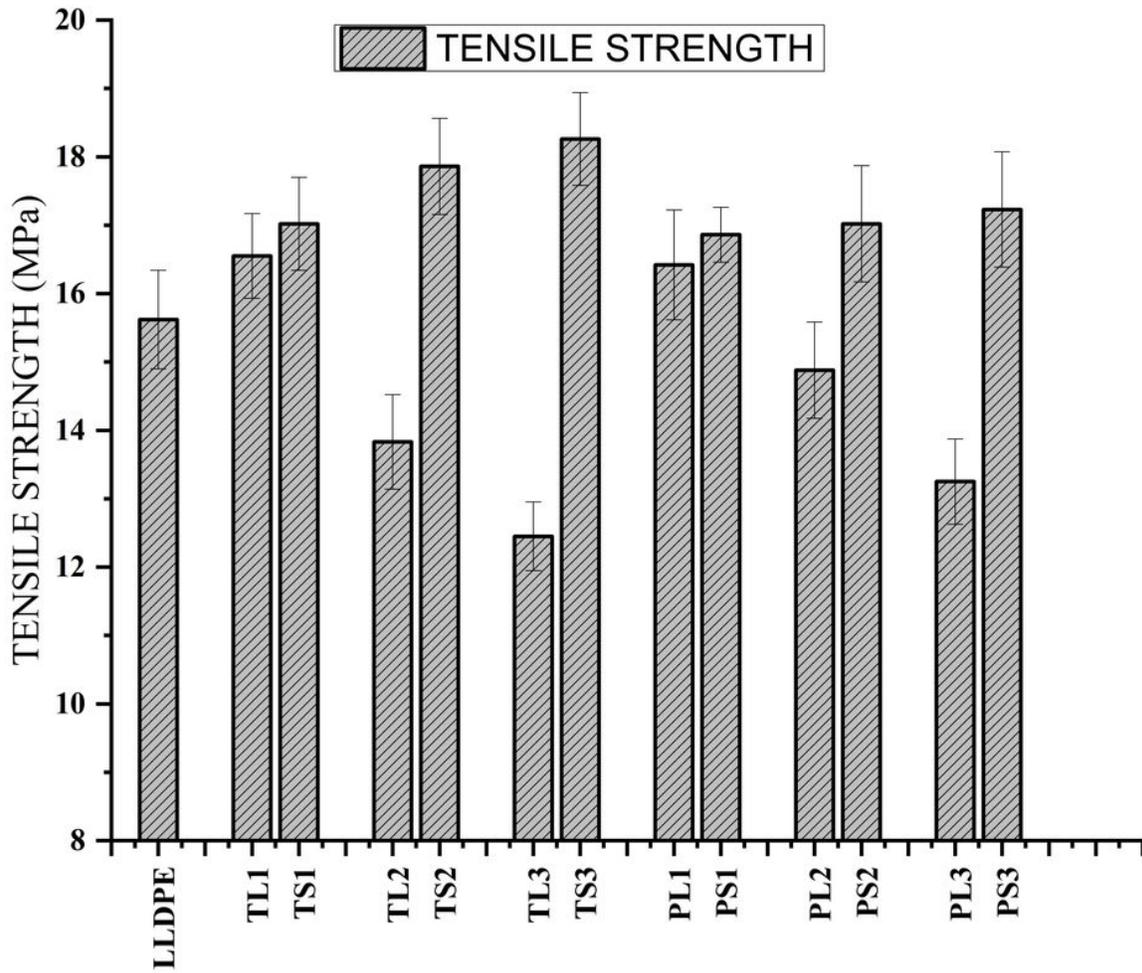


Figure 4

Effect of fiber content on the tensile properties of the composites.

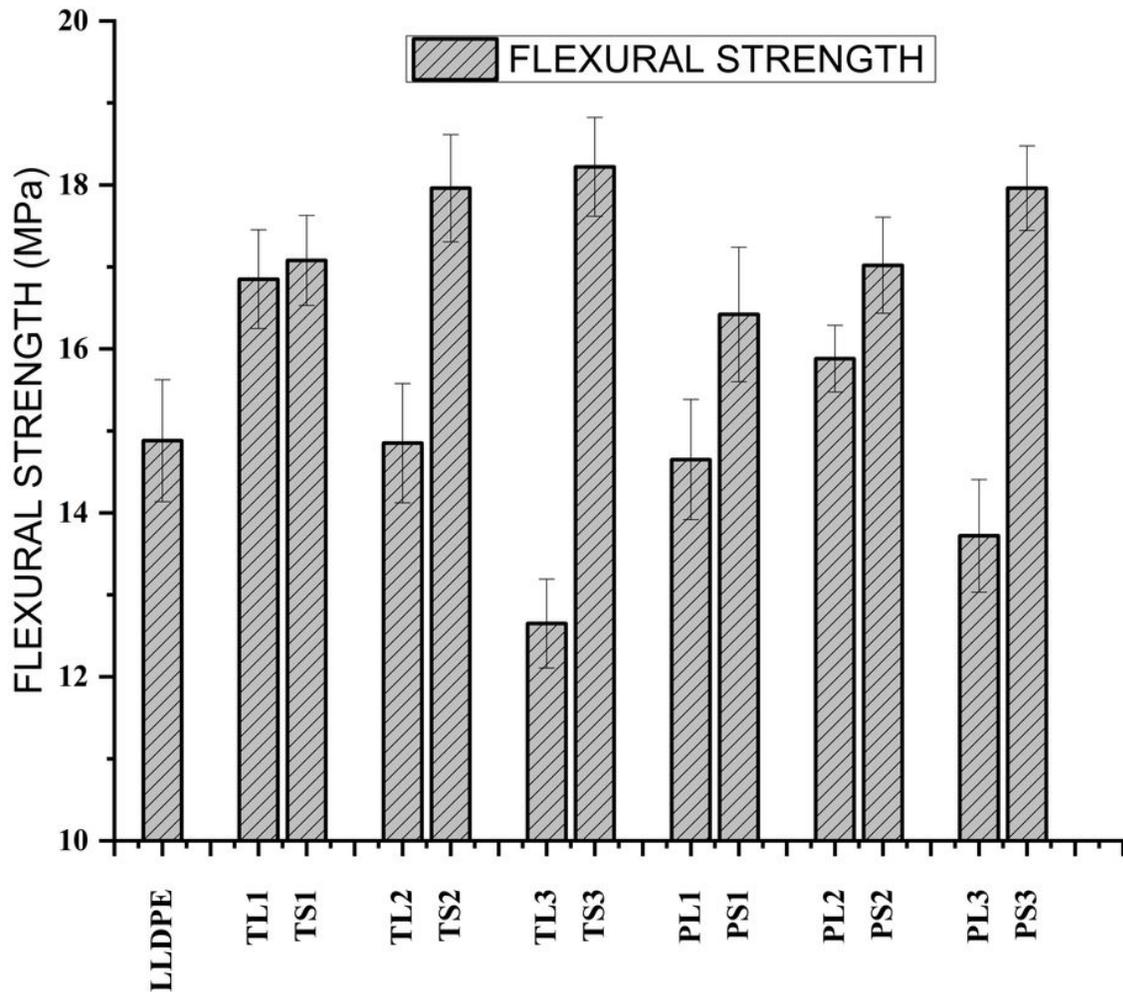


Figure 5

Effect of fiber content on the flexural properties of the composites

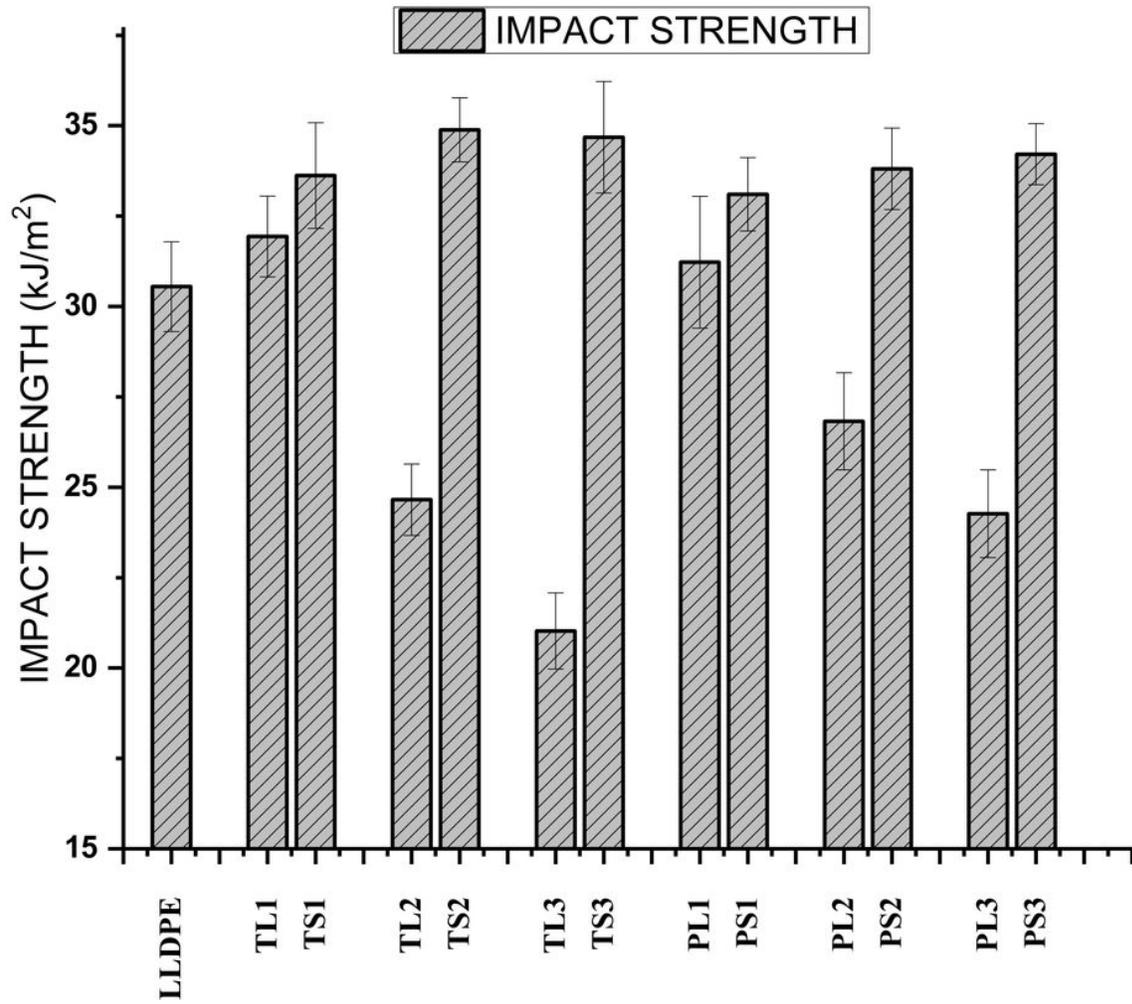


Figure 6

Effect of fiber content on the impact properties of the composites.

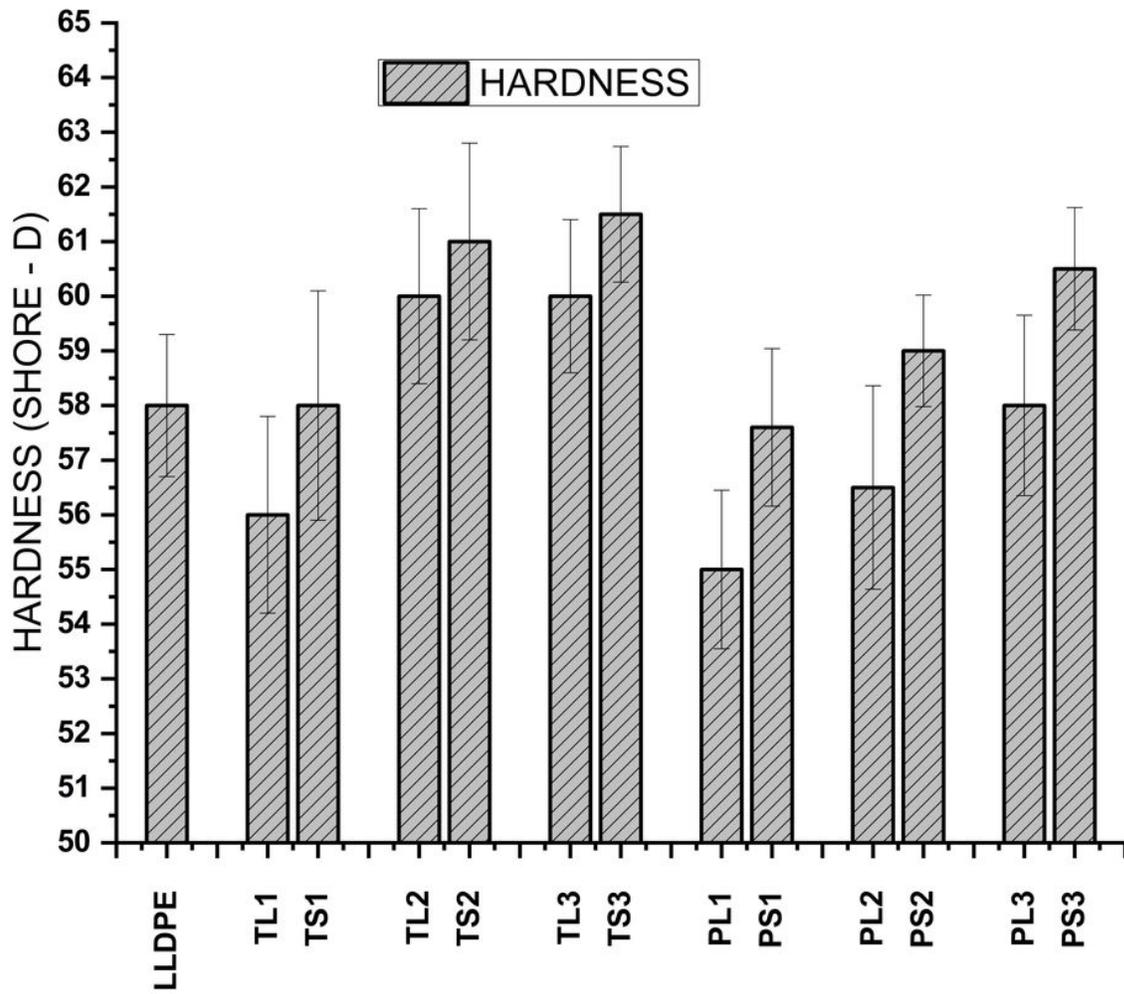


Figure 7

Effect of fiber content on hardness of the composites.

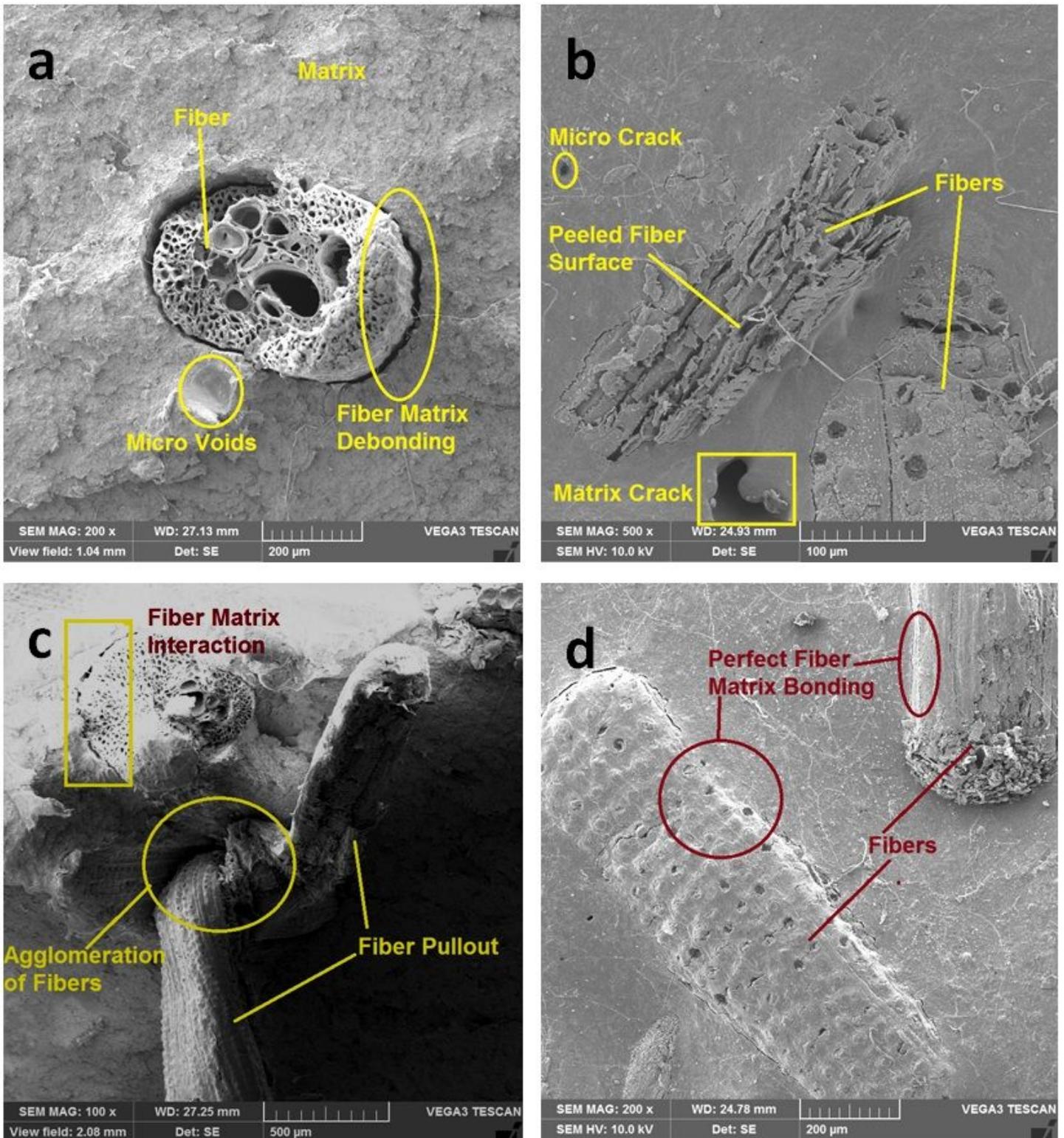


Figure 8

The SEM images of the fractured surfaces of PL1, PS1, PL3 and PS3 composites after the tensile test.

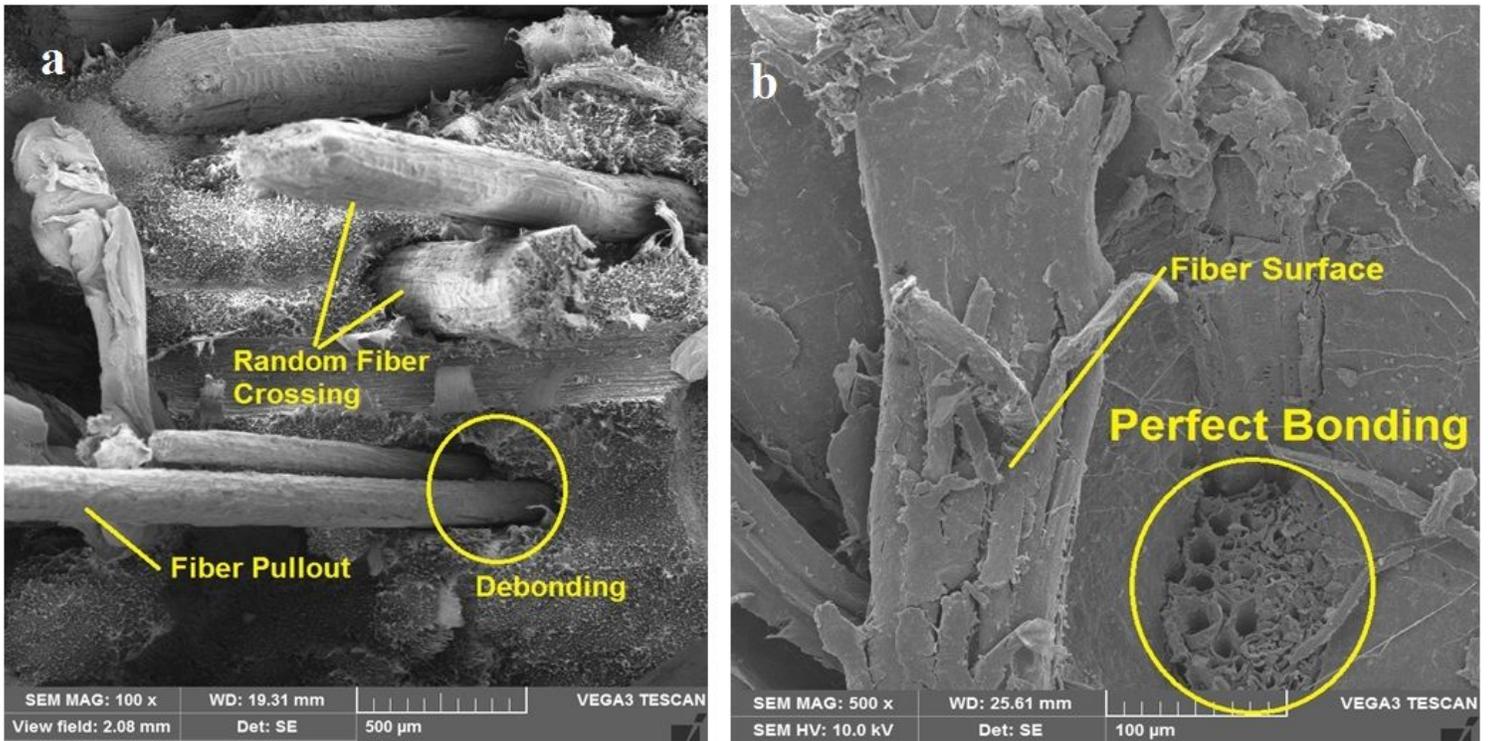


Figure 9

The SEM images of the fractured surfaces of TL3 and TS3 composites after the tensile test.

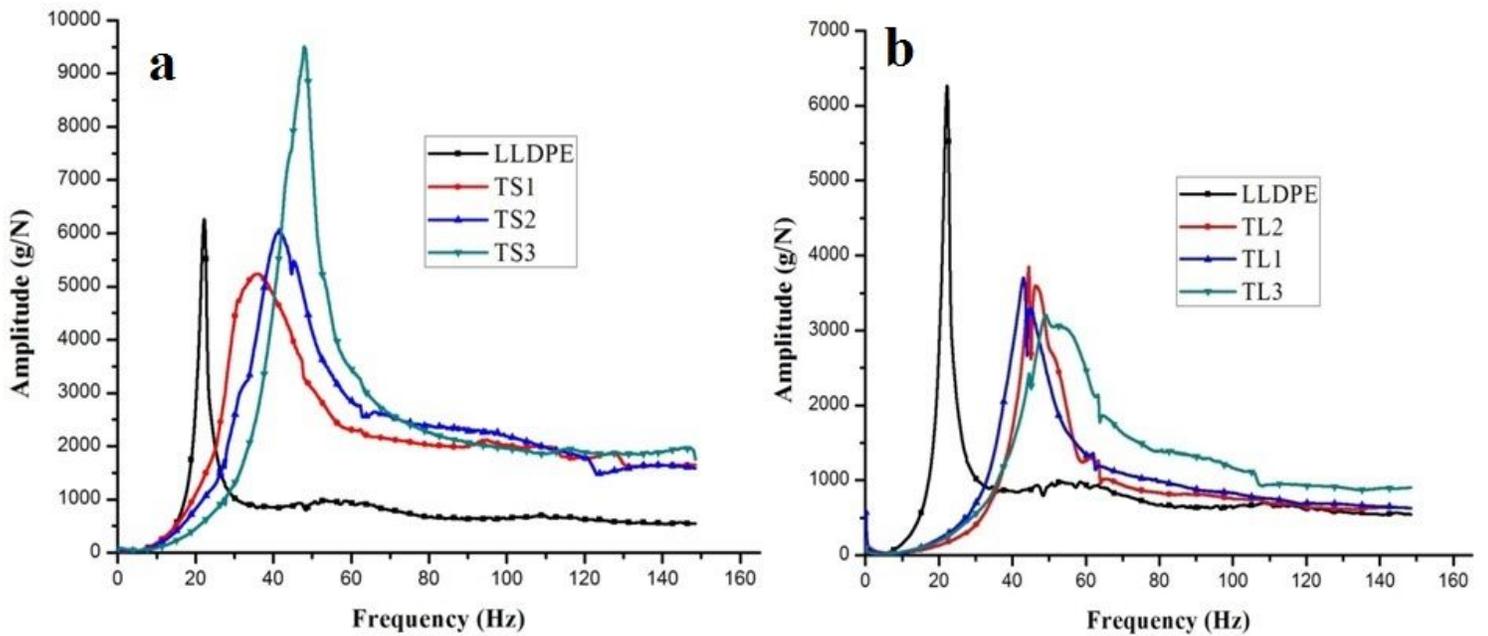


Figure 10

Effect of fiber length and weight percentage on Natural frequency of the composites (a) Short Tampico fiber and Unreinforced LLDPE (b) Long Tampico fiber and Unreinforced LLDPE

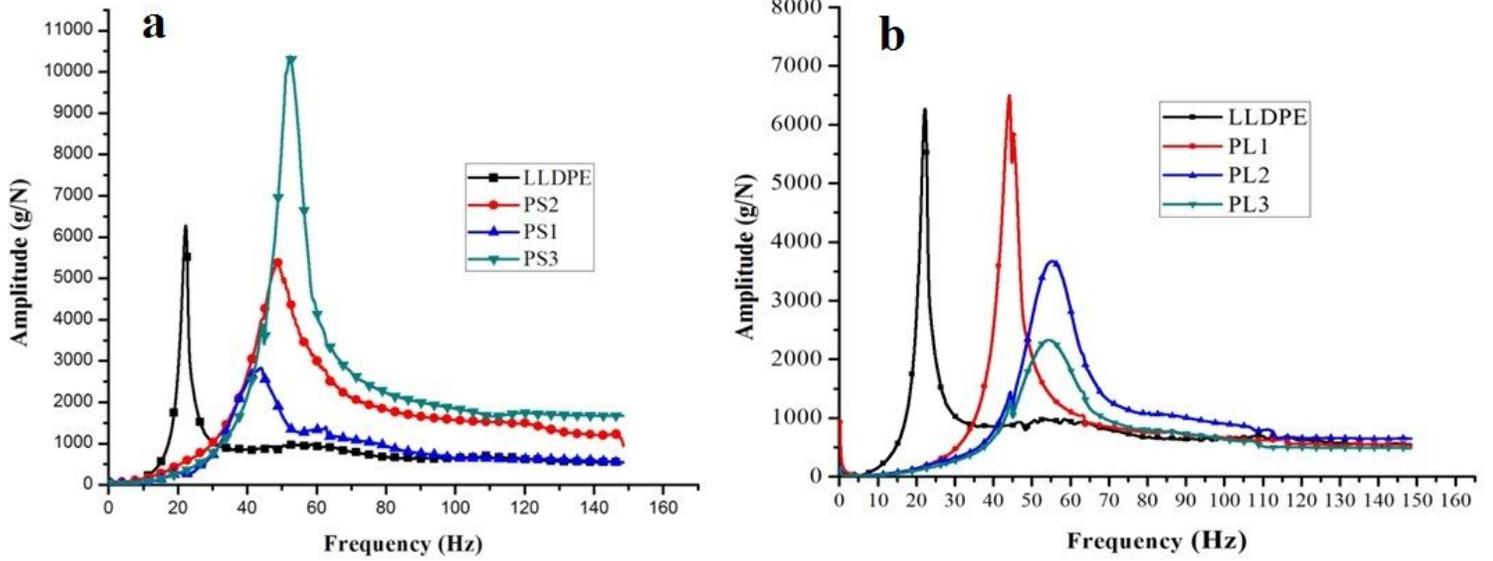


Figure 11

Effect of fiber length and weight percentage on Natural frequency of the composites (a) Short Palmyra fiber and Unreinforced LLDPE (b) Long Palmyra fiber and Unreinforced LLDPE