Free Vibration and Mechanical Characteristics of Palmyra Palm Leaf Stalk Fibers Reinforced Unsaturated Polyester Composites

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The mechanical and free vibration behaviors of unsaturated polyester matrix composites reinforced with 10-, 50-, and 150-mm-long palmyra palm leaf stalk fibers were examined. The fibers were alkali-treated before being used as reinforcement, which improved fiber-matrix interfacial strength, while reducing their hydrophilic character. The hand layup process followed by compression molding technique were used to produce the composites. Experiments were conducted to determine the tensile, flexural, impact, and vibration characteristics following the required ASTM standards. The results demonstrated that the most effective adhesion with the matrix was achieved with 50-mm-long fibers. To identify the properties of free vibration, a fast Fourier transform analyzer was used. Longer fibers offered slightly higher natural frequencies in the composites. To understand the mechanism of fracture, specimens that had been subjected to tensile testing were analyzed using scanning electron microscopy. Developing engineering applications with effective vibration-damping capabilities for a sound absorption potential compared to other lignocellulosic fiber composites may be achieved using palmyra palm leaf stalk fibers-reinforced composites.

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INTRODUCTION

During the last decades, interest has been spread on expanding the range of natural lignocellulosic fibers able to replace or hybridize with synthetic ones in polymer composites; such an approach can offer advantages such as reduced costs, especially when the fibers are byproducts or waste, low density, and sometimes have high specific strength (Sanjay *et al.* 2016). However, as hydrophilic materials, a characteristic that depends on their cellulose content, natural fibers soak up moisture and may soften over time and with use (Mahalingam *et al.* 2023). As hierarchized composites of lignin and cellulose, natural fibers present a viscoelastic behavior during loading, which in turn results in a significant absorption of vibrations: despite this, the studies on free vibration characteristics on natural fiber composites are still quite limited. Vibration studies have a particular importance in view of the potential application of natural fiber composites in the automotive sector. It is no surprise that the most diffuse lignocellulosic fibers, mainly extracted from the plant bast, such as flax and hemp, have been tested with the purpose to identify damping factors and variability of vibration characteristics, *e.g.*, in car door panels (Jawale *et al.* 2024).

Rajesh *et al*. studied the free vibration properties of banana/sisal hybrid composites, indicating that alkali treatment increased their stiffness, hence their vibration frequency (Rajesh *et al.* 2016). It had been suggested in particular that sodium hydroxide produces a full lattice modification from cellulose I to cellulose II, unlike other alkalis, therefore hindering the interaction of fiber with water; however the amount of NaOH applied needs to be limited in order not to damage the fiber structure (Venkateshwaran *et al.* 2013).

In recent years, the application of leaf fiber composites has been proposed in the automotive industry, first and foremost pineapple leaf fibers (PALF), which received specific attention (Sangilimuthukumar *et al.* 2020). From the natural frequency and damping study of PALF/unsaturated polyester composites, three specific frequencies were calculated, defined as Mode 1 (bending), Mode 2 (twisting), and Mode 3 (secondary bending) (Senthilkumar *et al.* 2019). In the case of phoenix leaf (date palm) composites, the natural frequency (Mode 1) was revealed to be between approximately 24 and 32 Hz, depending on the fiber length (10 to 50 mm) (Rajeshkumar and Hariharan 2014).

Palmyra palm leaf fibers have been considered for use in composites in a number of previous studies: in particular, it has been amongst the first leaf fibers to be considered as a waste added in a hybrid with fiberglass (Velmurugan and Manikandan 2007). Laminates with up to 60 wt% and five layers of palmyra continuous uni-directional fibers were fabricated in unsaturated polyester resin reaching the significant value of 160 MPa tensile strength, though limited by an elongation to failure not exceeding 4.4% (Manikandan *et al.* 2004). A more recent investigation concentrated on the considerable advantage obtainable for micro-hardness and wear resistance following the addition of a limited amount of palmyra fibers (up to 14 wt%) to an epoxy matrix (Biswal and Satapathy 2018).

In this study, unsaturated polyester matrix was reinforced with 30 wt% palmyra palm (*Borassus flabellifer*) leaf stem fibers, originating from southern India. In addition to the mechanical (tensile, flexural, impact) performance, the vibration characteristics of these composites were also studied and reported.

EXPERIMENTAL

Materials

The Palmyra palm tree’s leaf stalks can be seen in Fig. 1. A sharp-edged knife was used to remove the thorns from the leaf stem. Additionally, the stalks of the leaves had their skins peeled off by hand. The stems of leaves were retted in fresh water for 20 days after the thorns and skin had been removed. After retting, the leaf stalks were gently hammered with a wooden mallet to separate the individual fibers. Thoroughly whipping and washing in water were used to eliminate many unwanted compounds from the split fibers. The split fibers were dried in the sun to remove any remaining moisture. To make a composite, the dried fibers were first trimmed to size. Company Covai Seenu of Coimbatore, Tamil Nadu, India, supplied the chemicals used in fiber treatment, including sodium hydroxide pellets, sulfuric acid, unsaturated polyester resin, cobalt naphthene (an accelerator), and methyl ethyl ketones (a catalyst).

Fig. 1. (a) Leaves of palmyra palm (*Borassus flabellifer*); (b) Extracted leaf fibers

Alkali Treatment

The fibers were soaked by being completely immersed for 30 min in a 5% NaOH solution. After this time, the fibers were taken out and first rinsed with diluted sulfuric acid to get rid of any unreacted alkali remaining on the surface of the fibers and of the waxy materials attached to it. No further compatibilizer was used in this study. Following this, further rinsing cycles of 5 min each with distilled water and tap water, respectively, were carried out. Finally, the fibers were dried out by placing them for 1 h in a pre-heated oven with temperature set at 60 °C.

Composite Fabrication

The composite plates were produced using a die set with approximate dimensions 260 x 170 x 5 mm^3 made of steel. To compress the treated fibers, the lower die was loaded with them in the desired configuration (random or aligned), and then the higher die was put on top serrating it tightly, hence compressed hydraulically with an approximate pressure equal to 0.5 bars. At the end of this phase, a fiber mat was extracted by opening the die.

The matrix was created by combining a low viscosity orthophthalic unsaturated polyester resin with accelerator and catalyst. The resin was purchased at Herenba Ltd., Chennai, India. A 2% of methyl ethyl ketone peroxide (MEKP) was used as a curing agent and cobalt naphthenate (0.5%) as a catalyst. Polyester resin exhibits good mechanical as well as resistance to degradation and low weight. After positioning the fiber in the bottom die, the matrix was transferred within the die set. Following curing, which required a 90 minutes' cycle at ambient temperature, the composite plate of dimensions 250 x 165 x 3 mm^3 could be easily removed by cleaning the die and using the mold release agent. Once again, hydraulic compression was used to insert and compress the top die.

Fig. 2. Composite plate

After 12 h of loading, the die was released, and the composite plates were removed to cure at room temperature. The manufactured plates' vibration test samples were cut. Figure 2 shows the composite plate that was made and the samples that were cut. Information on the matrix and fiber used in the production of the composite may be found in Table 1.

Table 1. Composite Configurations

Nomenclature	Fiber Length (mm)	Fiber (wt%)	Matrix (wt%)	Description
10APPLSFC	10	30	70	Alkali-treated PPLSF with 10 mm fiber reinforcement
50APPLSFC	50	30	70	Alkali-treated PPLSF with 50 mm fiber reinforcement
150APPLSFC	150	30	70	Alkali-treated PPLSF with 150 mm fiber reinforcement

Mechanical Properties

Using standard testing procedures, tensile, flexural, and impact performance of the three categories of composites manufactured from alkali-treated palmyra palm leaf stalk fibers (10APPLSFC, 50APPLSFC, and 150APPLSFC) were examined. For the purpose, a minimum of five valid tests per composite category and per loading mode was acquired.

An Instron universal testing apparatus was used with a crosshead speed of 5 mm/min to submit specimens measuring 165 x 19 x 3 mm^3 according to ASTM D638-03 (2012) requirements for the tensile test. The same Instron universal testing equipment was used to analyze the flexural properties of specimens measuring 127 x 12.7 x 3 mm^3, with a crosshead speed of 2 mm/min, in accordance with ASTM D790-03 (2017). In accordance with ASTM D256-23e1 (2023) standards, specimens measuring 165 x 13 x 3 mm³ were used in the impact testing procedure carried out by the Izod impact testing equipment to determine the impact strength.

Vibration Test

The prepared samples of 165 x 20 x 3 mm³ were subjected to free vibration tests using an FFT analyzer, orienting the samples in the longitudinal direction, though the short fibers format of the reinforcement does not ensure real unidirectionality. The signals from the samples were received from the accelerometer through the data acquisition device controller. The composite sample was placed on a 5 mm thick poly(vinyl chloride) (PVC) soft foam to create free-free condition for the specimen and the vibration was produced by using an impact hammer at the free end. The accelerometer, glued with the beam to pick up the vibration signals, was placed at 10 different locations at 15 mm intervals, at each location the first three natural frequencies were measured, and their average value has been reported. The FFT analyzer was used to conduct a free vibration test on the produced samples. The data collection device's controller received the accelerometer's sample signals. A soft form was used to produce a free-free state for the specimen, and then an impact hammer was used at the free end to apply the impact on the composite sample. To detect vibration signals, the accelerometer was attached to the beam. In this research, the first three natural frequencies were averaged across 10 tests, with the accelerometer being positioned at different positions and impacts being applied at random locations over the surface of the board were considered. The set-up used for the test, including the supporting foam, is depicted in Fig. 3.

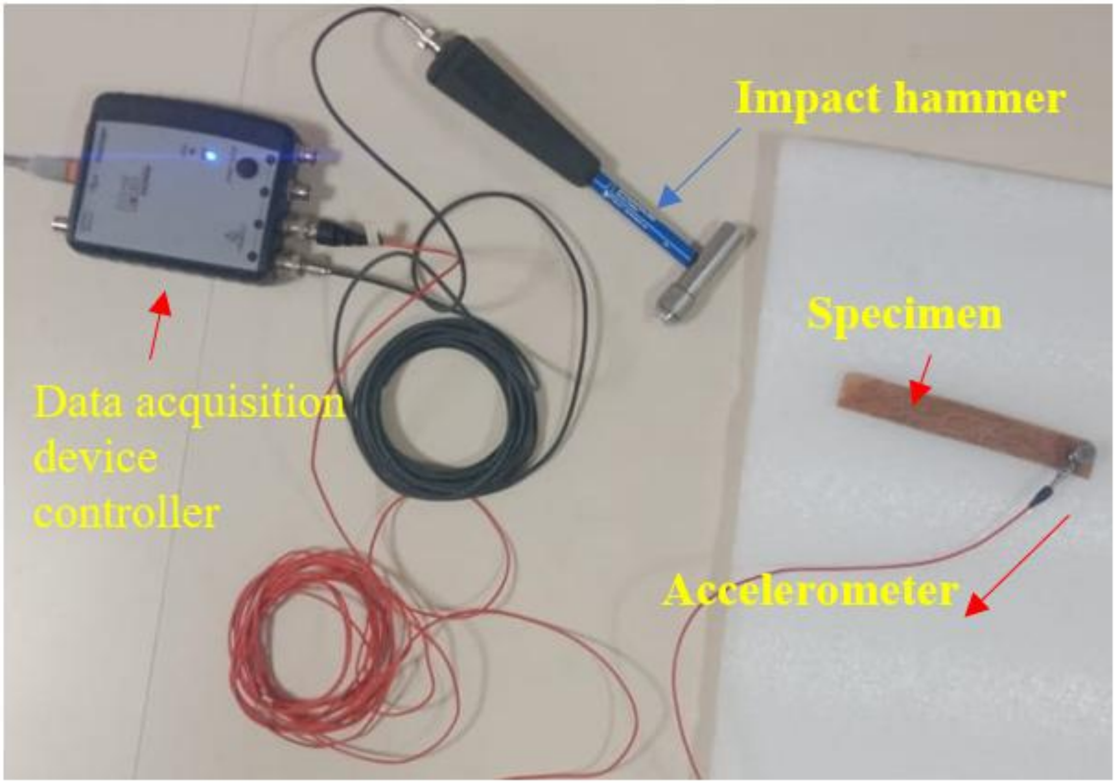

Fig. 3. Free vibration test set-up

Scanning Electron Microscopy (SEM)

A scanning electron microscope, specifically the Carl Zeiss model EVO MA 15 (Carl Zeiss GmBH, Jena, Germany), was used for the purpose of analyzing the tensile fractured PPLSF composite samples. Electron high tension (EHT) equal to 5 kV was used, with a working distance (WD) equal to 8.2 mm. Before being carefully attached to an aluminum stub using double-sided tape, each specimen was carefully coated with a very tiny coating of gold of around 10 nm thickness to protect them from electric charge interference during analysis. The SEM micrographs were created using conventional secondary electron imaging techniques.

RESULTS AND DISCUSSION

In Table 2 the first three modes of vibration, namely bending, twisting, and secondary bending of the various palmyra leaf stems fibers composites are reported. As expected, a larger shift of the frequencies was observed by the bare addition of the fibers. Vibration behavior may also be correlated with the fact that palmyra fibers are reported as being ductile structures with relatively low crystallinity, not exceeding 50%, and a high microfibrillar angle (Sarasini *et al.* 2017). As a matter of fact, early theoretical prediction on microfibrillar angle for palm fibers, such as oil palm empty fruit bunches (OPEFB), did confirm high values for it, in the specific case 42° (Sreekala *et al.* 1997). It is also suggested by Jonoobi *et al*. (2019), that the larger microfibrillar angle does not allow palm leaf fibers to have a comparable strength with other fibers *e.g.*, from agave, such as sisal.

Some further increase of the frequency with the fiber length was observed, consistent from 10 mm to 50 mm, and less so from 50 to 150 mm. It has been noticed elsewhere (Kumar *et al.* 2014), that whenever the natural frequencies would not increase with fiber length, this would be related to a possible agglomeration of fibers in the composite, possibly connected to a weaker matrix-fiber interface. For the bending resonance mode, as reported in Table 3, palmyra fiber composites do compare effectively in most cases, one exception being luffa fibers, which have nonetheless a level of porosity not attainable into most other natural fibers (Adeyanju *et al.* 2021).

Table 2. Natural Frequency Modes of Vibration of Different PPLSF Composites

Composite	Natural Frequency (Hz)		
	Mode 1	Mode 2	Mode 3
Neat resin	15	34	114
10APPLSFC	85	100	585
50APPLSFC	90	120	860
150APPLSFC	98	160	775

In Fig. 4, tensile, flexural, and impact performance of the three different composite configurations is reported. The results clearly indicate the superiority of the one realized using 50-mm-long fibers. Fracture surfaces, as obtained in Fig. 5, show that a sounder interface was obtained with this fiber length, whereas for 10-mm-long fibers pull-out appeared more frequent and for 150-mm-long ones a higher roughness of the contact surface with the matrix might have proved detrimental for the properties.

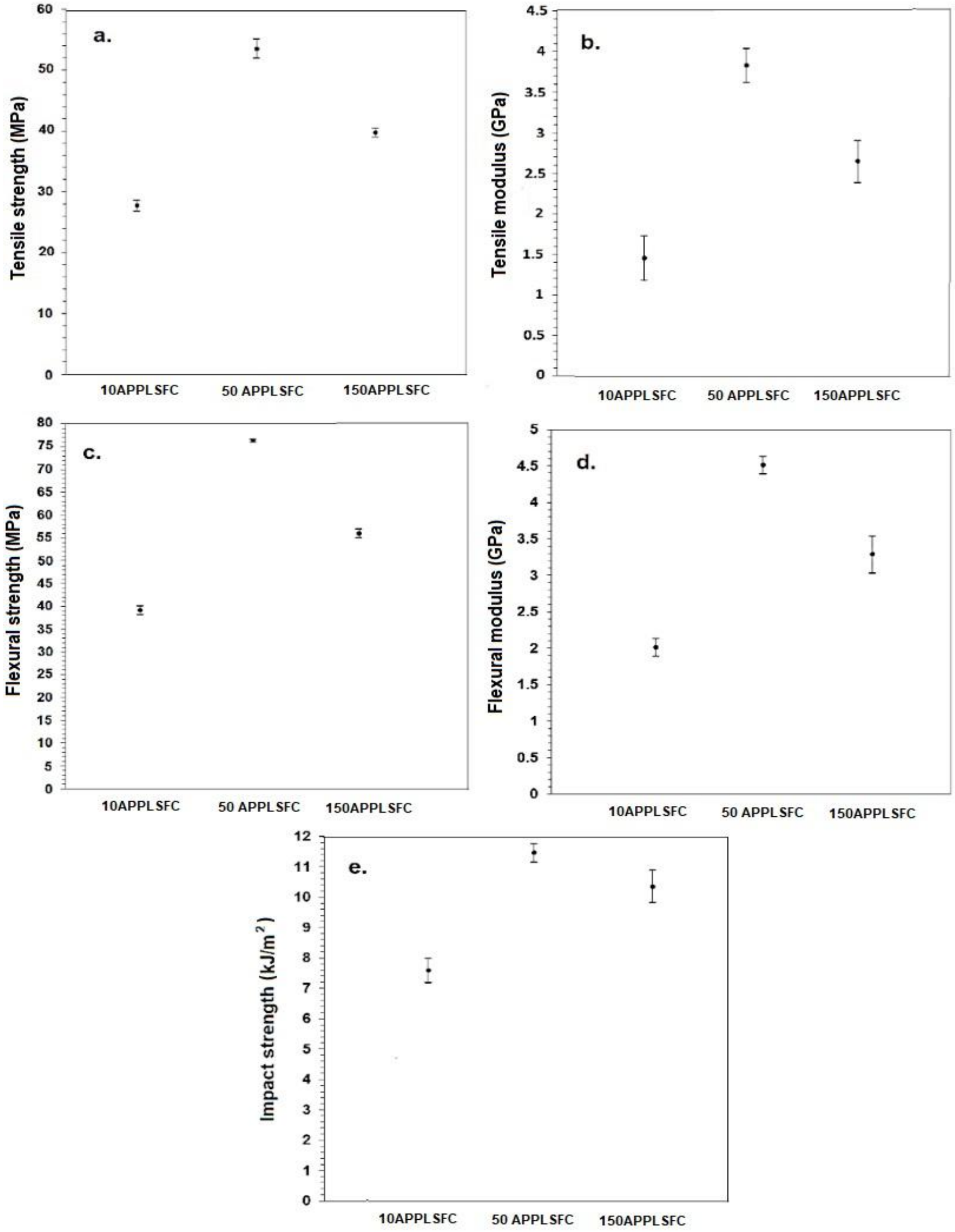

Fig. 4. Mechanical properties of various palmyra leaf stem fiber composites: (a) Tensile strength; (b) Tensile modulus; (c) Flexural strength; (d) Flexural modulus; (e) Impact strength

Table 4 indicates how PPLSF composites compared against a few other ones with similar quantity of fibers. The findings suggest that palmyra composites achieved a suitable performance, especially in the case of 50-mm-long fibers. A single study on palmyra fibers with 7 mm length indicated comparable properties (Muruganrama *et al.* 2022). In the case of comparison with other fibers, two factors need to be considered, which are the origin of the fibers, such as from the plant leaf, and the amount of fibers introduced in the composite, when declared, *i.e.*, close or equal to 30 wt%, such as in the materials examined in this study. A potential match for palmyra can be with sansevieria composites, as from

Sreenivasan *et al.* (2014), where the length of fibers used was comparable, yet the latter appeared superior.

Table 3. Mode 1 Frequency of Vibration of Different Natural Fiber Composites in Unsaturated Polyester Matrix

Natural Fibers in the Composite	Amount (wt%)	Mode 1 Natural Frequency (Hz)	Reference
PPLSF	30	85 to 98	This study
Sisal or banana	30 to 50	26 to 32	Kumar *et al.* (2014)
Sansevieria cylindrica/Coconut sheath and hybrids (three layers)	-	28.5 to 48.2	Bennet *et al.* (2015)
CBC–Coconut/Banana/Coconut sheath	-	35.4	Senthilkumar *et al.* (2016)
Luffa	40	67 to 110	Kalusuraman *et al.* (2019)
Banana (three layers)	-	31.3	Senthilkumar *et al.* (2018)
Coconut sheath (three layers)	-	21.9	Senthilkumar *et al.* (2018)
Sisal (three layers)	-	23.2	Senthilkumar *et al.* (2018)

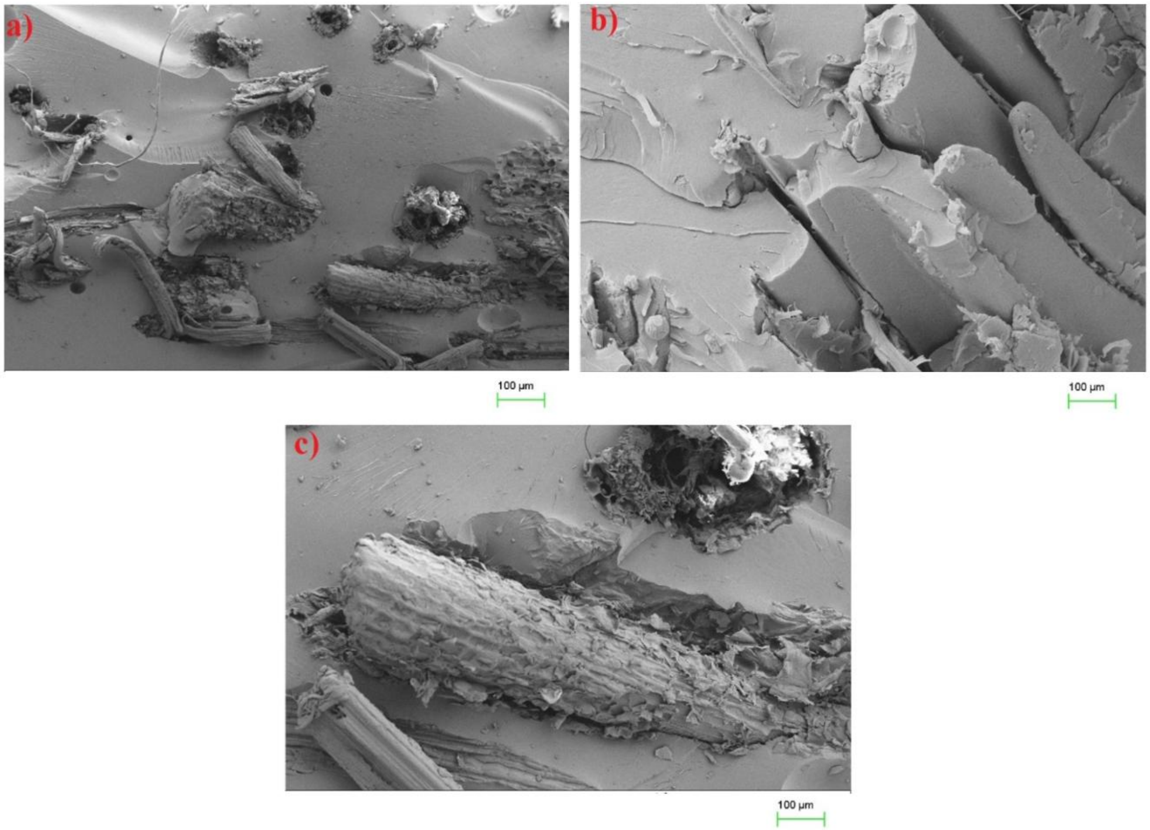

Fig. 5. Scanning electron microscopy image of tensile fracture surfaces of: a) 10APPLSFC; b) 50APPLSFC, and c) 150APPLSFC

Table 4. Tensile, Flexural, and Impact Properties of Different Natural Fiber Composites

Composite Fibers	Amount of Fibers (wt%)	TS (MPa)	TM (GPa)	FS (MPa)	FM (GPa)	IS (kJ/m^2)	Reference
10APPLSFC	30	27.52	1.45	39.36	1.97	7.54	Present work
50APPLSFC	30	54.22	3.83	76.03	4.47	11.36	Present work
150APPLSFC	30	40.31	2.61	55.43	3.23	10.25	Present work
APPLSF (7 mm)	30	36.13	2.13	71.35	4.81	10.21	Muruganrama *et al.* 2022
Banana	40	108.42	-	71.28	-	86.2	Ramesh *et al.* 2014
Olive leaves (2 to 3 mm)	30	-	-	33.75	0.8	58	Al-Oqla 2021
Pineapple (30 mm)	30	52.9	2.29	80	2.8	24.2	Devi *et al.* 1997
***Sansevieria* (30 mm)**	40	75.75	11.02	82	3.1	92	Sreenivasan *et al.* 2014

(Tensile strength = TS; Tensile modulus = TM; Flexural strength = FS; Flexural modulus = FM; Impact strength = IM)

From the above results, it is possible to notice that 50-mm-long palmyra fibers achieved a performance suitable for structural applications both in terms of vibration and mechanically. Another possible reference is about other palms from which leaf fibers of some length can be obtained and applied to composites. These can be considered the direct competitors for palmyra. Literature investigation suggests that the main difference is considering palm fibers from composites according to the main product the palm yields. As a result, reviews are available on the use of oil palm (Shinoj *et al.* 2011), date palm (Ghori *et al.* 2018), and sugar palm (Asyraf *et al.* 2021) in composites. However, the number of palm species that have been investigated for potential introduction in a composite material is steadily increasing, and specific studies are more recently available. In addition, Khalasa fiber from Qatar, offers potential fiber length from fibers in the region of 200 mm, with fiber tensile strength in the region of 48 MPa and Young's modulus around 2.1 GPa. In this context, palmyra can find its position and regarding vibration behavior, can be potentially introduced in structures, which are most recently of interest for natural fiber composites, such as wind turbine blades (Batu *et al.* 2020), on account of its quite high first mode.

CONCLUSIONS

1. Palmyra fiber composites achieved vibrational and mechanical performance results comparable with that of other similar materials with a close amount of fibers, *i.e.*, 30 wt%, as their reinforcement.
2. The highest performance was obtained by introducing 50-mm-long fibers, both in terms of vibration behavior and of mechanical properties. Measurements indicated an average first natural frequency of 90 Hz; an average tensile strength and modulus of 54 MPa and 3.8 GPa, respectively; an average flexural strength and modulus of 76 MPa and 4.5 GPa, respectively, and an average impact strength of 11.4 kJ/m^2.

3. Increasing the fiber length to 150 mm did not prove beneficial in increasing the interfacial strength with the polymer matrix. It is possible that this is due to the alkali treatment, yet in general the influence of defects over the fiber structure was much higher.
4. The results indicate that these fibers were able to compete with others as for composite reinforcement, in particular with those extracted from various palm species, which are increasingly investigated over the last years.

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Data Availability Statement

Data are available on request from the authors.

Declaration of Conflicting Interests

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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