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Research Article

Effect of Curing on the Tensile and Flexural Performance of Fully Biodegradable Corn Starch/Areca Frond Composites

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Composites have monopolized the automotive, construction, and packaging industry. Their high strength to weight ratio has made them an integral part of numerous engineering applications. In this study biodegradable matrix is combined with areca frond fibres for developing composites for low strength structural applications. Areca frond fibres were extracted and treated with sodium bicarbonate to improve the surface characteristics. Hand lay-up and compression moulding techniques were used to fabricate composites having unidirectional fibre orientation. The specimens prepared were exposed to varied environments, namely, sunlight, OTG oven, steam oven, and hot air oven, for curing and the results were analyzed to best suit the implicated requirements. Scanning electron microscopy was used to observe the changes in surface characteristics of the frond fibres after treatment. Tensile and flexural strength of starch based/areca frond reinforced composites were evaluated according to ASTM standards. Test results revealed that composites cured in a steam oven resulted in improved tensile and flexural strength compared to other curing environments.

1. Introduction

Composites have been used by mankind since prehistoric times. Straw reinforced clay bricks, bamboo shoots reinforced mud walls, and swords made out of laminated metals are some of the evidences for it [1]. Composites have their ability to be formed into any desired shape with excellent dimensional stability and high strength to weight ratios. They are cost effective substitutes for materials requiring high maintenance. Customized surface finish can be obtained with good resistance to heat, chemicals, and corrosion. In addition it is also resistant to creep and has exceptional electrical insulation when required [2, 3]. Natural fibre reinforced biodegradable polymers have great scope in the engineering field. Their development is independent of petroleum based products and so it makes a good green material [4]. Agriculture crop residues can be used as reinforcement materials to improve mechanical strength and heat resistance. In the past several decades, biodegradable products have experienced a revival, with an exponential increase in production having high commercial value.

Srinivasababu et al. [5] explored the possibility of using okra as reinforcement for polyester resin and noticed that the tensile strength and modulus of untreated and treated samples were found to be higher than the pure polyester specimens. Nishino et al. [6] carried out a study on kenaf fibre reinforced PLA composites. They observed that the composite possesses superior mechanical and thermal properties. Paralleled to this, Ochi [7] demonstrated the tensile and flexural strengths of kenaf reinforced composites. Many such composites fabricated display their potential as an alternative to traditional composites and have become an integral part of the electrical, medical, and construction industry.

Swamy et al. [8] in their study determined the strength of areca nut fibres treated with alkali which exhibited excellent resistance to moisture compared to wood based boards making them a very promising material in the packaging industry. Zhang et al. [9] studied the synthesis and characterization

(a) (b) (c)

Figure 1: (a) Areca palm, (b) areca frond, and (c) dried areca frond.

of polyvinyl alcohol (PVA), nanohydroxyapatite (HA), and natural silk composite hydrogel using repeated freezing and thawing method based on Taguchi technique. Results showed that the formula of polyvinyl alcohol, nanohydroxyapatite, and silk composite hydrogel with PVA 15%, HA 2.0%, and silk 1.0% mass percentage was the best. Qiao et al. [10] studied composites made from poly(ethylene-co-vinyl alcohol), polypropylene carbonate, calcium carbonate, and starch fabricated using melt blending. The authors observed an increase in tensile strength with increase in starch content.

Heckadka et al. [11] used Taguchi technique to study the flexural strength of the areca frond fibres reinforced with starch, methyl cellulose, resorcinol, and glycerol based matrix material using hand lay-up/compression moulding technique. The authors reported that glycerol as plasticizer had maximum effect on the flexural strength of the composites.

Majdzadeh-Ardakani and Nazari [12] investigated the effects of the ingredients, namely, starch, poly(vinyl alcohol), and clay, to understand clay intercalation and mechanical properties.

Starch and cellulose based polymers have been used in the past for furniture, structural panels, and gardening equipment. Green composites result from a combination of a biodegradable polymer and biodegradable fillers, usually biofibres. Their production is generally based on renewable resources and their waste can be managed in an eco-friendly way in terms of lower emissions and energy consumptions [13]. These incentives have pushed researchers to dwell deep into the possibilities of tapping the raw materials available in abundance to us and shifting from the synthetic trend.

Present work is aimed at developing unidirectional laminates fabricated with areca frond fibres and corn starch based matrix to evaluate their characteristics based on different curing methods. Varied curing techniques are adopted and their tensile and flexural strengths are determined. The laminates obtained display immense potential in the packaging industry as well as for low strength applications including stationaries.

2. Experimental Details

2.1. Materials. The areca frond fibres used in this study were obtained from local plantations. Areca frond is a part of the tree which covers bunch of areca nuts. These trees belong to family Palmaceae. Coastal Karnataka is the main source for these fronds. The palm tree measures around 20 m in height with a trunk diameter of around 0.4 m [14]. The fronds resemble a boat hull and have dimensions of around 0.25 m in width and 1.5 m in length and have a depth of 0.1 m. The areca palm tree, raw frond, and dried fronds are shown in Figure 1. The matrix ingredients were corn starch, water, and vinegar as base materials, methyl cellulose and resorcinol as binders, and glycerol as plasticizer. Table 1 shows the detailed specifications of the matrix ingredients.

2.2. Fibre Extraction and Treatment. Areca fronds were soaked in water for about seven days in a tank as shown in Figure 2. The pH of water was monitored on regular basis using digital pH meter to dispose it safely. For precautionary measures water was changed after every two days.

The leaves became soggy in nature after soaking from which the fibres were extracted using metal wire brush by moving it in the longitudinal direction. After the extraction of individual strands the membrane covering it was extracted using bare hands to get untreated fibres. Fibres obtained were treated with sodium bicarbonate ($NaHCO_3$) to get rid of fleshy membrane and impurities to enhance their physical properties. The treatment was carried out for duration of 8 hours. Solutions with three different molarities, namely, 0.3 M, 0.5 M, and 1 M, were used. During the course of the treatment, pH of the solution was monitored and recorded at regular intervals.

(a) (b)

Figure 2: (a) Fibre treatment. (b) Treated fibres.

Table 1: Specifications of the matrix ingredients.

Name	Description
Base material specification	
(a) Corn starch	Edible maize starch
Manufacturer	Manibhadra Food Products, Hubli, Karnataka
Ingredients	25% amylose and 75% amylopectin
(b) Water	Normal tap water/potable water
(c) Vinegar	Edible cooking vinegar
Manufacturer	Aditi Foods Private Ltd., Sangli, Maharashtra
Ingredients	Acetic acid and water
Binder specification	
(a) Methyl cellulose	300–560 cps purified
Manufacturer	Merck Specialities Private Ltd., Mumbai
Ingredients	Cellulose, sodium hydroxide, methyl chloride
(b) Resorcinol	Recrystalline extra pure
Manufacturer	Merck Specialities Private Ltd., Mumbai
Ingredients	Assay, chloride, sulfate, water, and sulfated ash
Plasticizer specification	
(a) Glycerol	98% purified
Manufacturer	Merck Specialities Private Ltd., Mumbai
Ingredients	Assay, acid, alkali, chloride, sulfate, water, and sulfated ash

Table 2: Average diameter of areca frond fibres.

Description/molarity	Untreated	0.3 M	0.5 M	1 M
Diameter (mm)	0.415	0.382	0.363	0.349

2.3. Preparation of Test Samples of Areca Fibres and Composite Laminates. Treated areca frond fibres were cut to a length of 60 mm. Diameter of the fibres was measured using a digital micrometer nearest to 0.001 mm at different locations on a single fibre. Measurements were taken on fifty fibres to arrive at an average. The average diameters of the fibres are tabulated in Table 2.

Table 3: Curing environments and parameters.

Curing environment	Curing parameters	
	Temperature (°C)	Duration (h)
Sunlight	38	8
OTG oven	80	4
Steam oven	80	4
Hot air oven	80	4

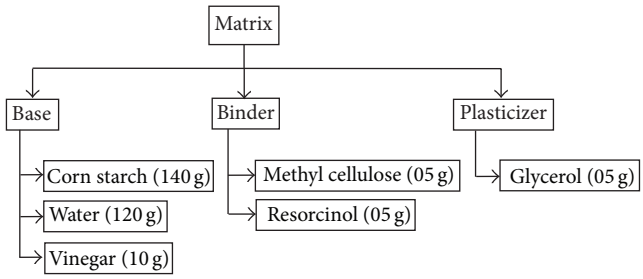
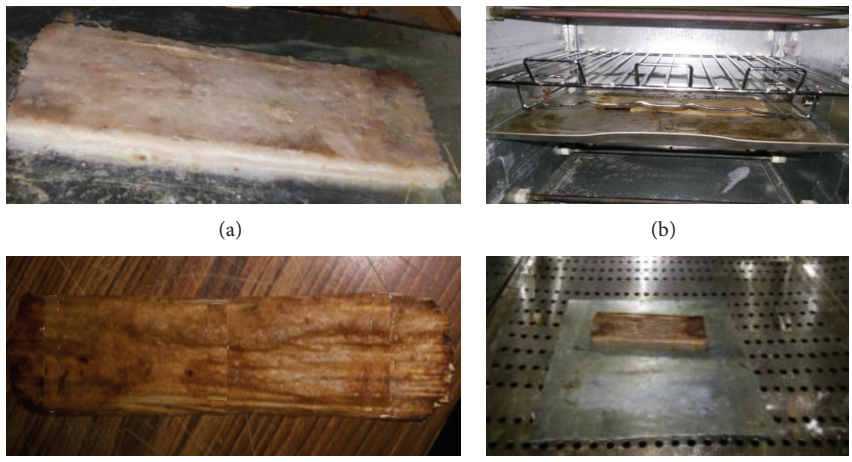


Figure 3: Ingredients of the biodegradable matrix.

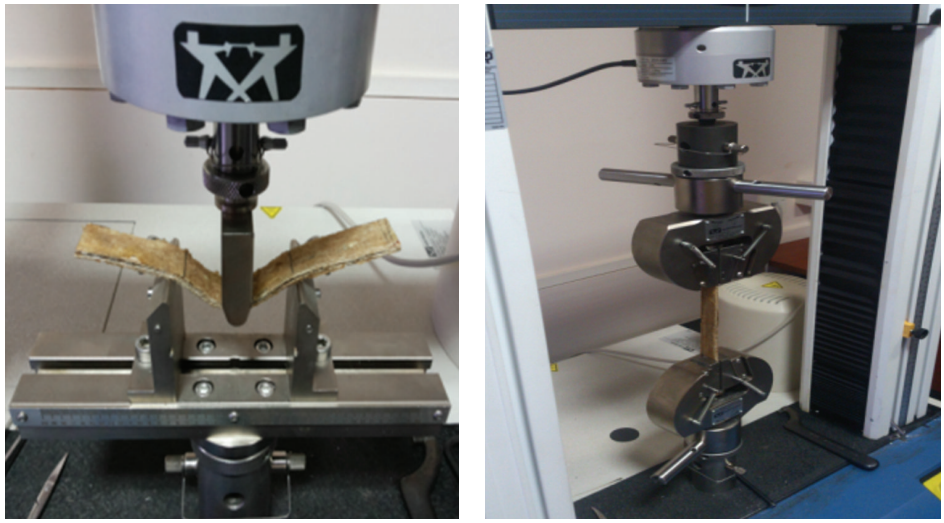
Figure 3 shows the basic ingredients of the matrix. The matrix ingredients were weighed to the nearest 1 mg. The weighed constituents were mixed in a vessel using a magnetic stirrer. Plasticizer and binders were added and stirred for 20 minutes. The mix is then transferred to a bowl and heated at 140°C. Heating is continued till it transforms to a semisolid state. The fibres are kept aligned in a single direction in a mould of 250 mm × 250 mm dimension. The matrix mix was transferred to the mould and spread evenly using a roller to facilitate escape of air from the matrix to prevent voids in the composite. Compacting can be achieved using several techniques. In the present work, in-house fabricated pneumatic press was used for compaction of the unidirectional laminate [15]. The mould is coated with a releasing agent to prevent sticking of the composite. The composites were next cured in four various environments, namely, sunlight, OTG oven, steam oven, and hot air oven. The average thickness of all the laminates was 4 mm. Table 3 presents details of curing and Figure 4 presents the laminates cured in different environment.



(a) (b)

(c) (d)

Figure 4: Laminates cured in different environments: (a) sunlight, (b) OTG, (c) steam, and (d) hot air.



(a) (b)

Figure 5: Test setup. (a) Tensile test and (b) flexural test.

2.4. Mechanical Properties of Fibre and Composites. Tensile test of single fibre was evaluated on Universal Testing Machine (UTM) of Instron Make (model 3366). The tests were conducted according to ASTM C1557 [16] at a constant crosshead speed of 1 mm/min and gauge length of 30 mm [17]. Fibres from all the categories, namely, untreated, 0.3 M, 0.5 M, and 1 M, were tested and, to arrive at a mean, five fibres from each category were selected. Composite specimens for tensile test were cut from the cured laminates to a size of 250 × 25 × 4 mm. The tests were conducted on Instron UTM at a constant crosshead speed of 2 mm/min according to ASTM D3039 [18]. Tensile strength was obtained from the data acquisition computer which recorded the load and displacement. Flexural tests were conducted to determine the bending strength of the laminates. Procedure A (three-point bending) of ASTM D790 [19] was adopted and the tests were carried out at a constant crosshead speed of 2 mm/min on Instron UTM. The dimension of the flexural test specimen was 78 × 13 × 4 mm. Five specimens of each curing condition were tested. The test setup is presented in Figure 5.

2.5. Scanning Electron Microscopy (SEM). The surface of the areca frond fibres was examined using Zeiss Make scanning electron microscope (model: Zeiss EVO 18, Germany). Prior to analysis of areca frond fibres, samples were coated with a thin layer of silver by ion sputtering to prevent charging of the specimen. An accelerating voltage of 15 kV was used. The SEM micrographs were used to assess the effectiveness of treatment and to decide the suitable concentration of the treating medium.

3. Results and Discussion

3.1. Effect of Treatment on the Surface Characteristics of Areca Frond Fibres. SEM images of the fibres are shown in

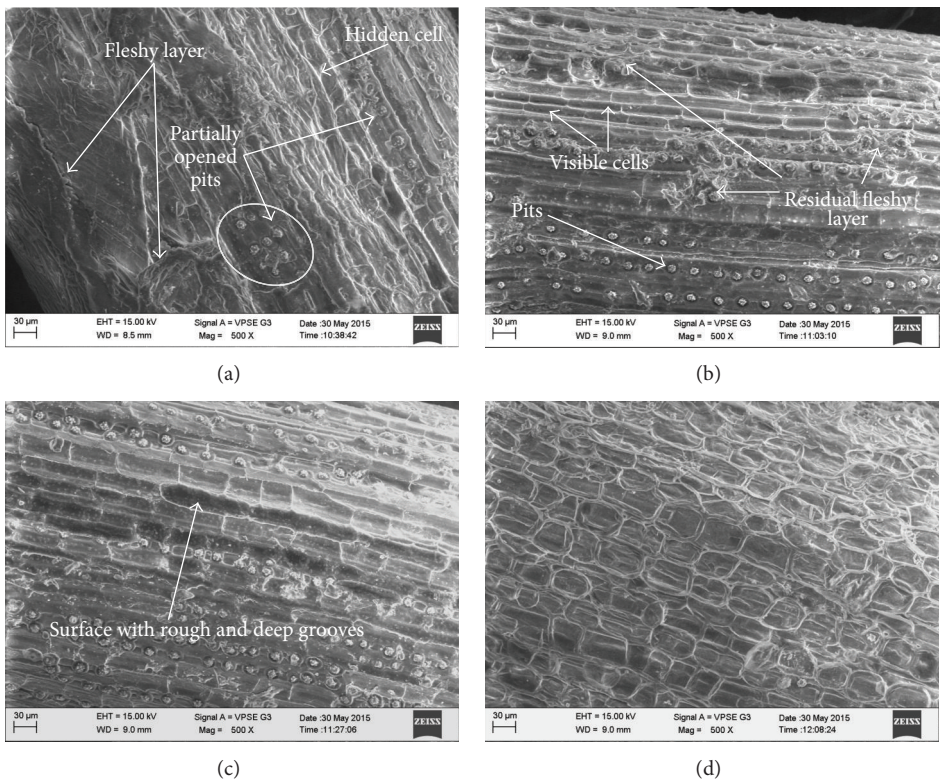


Figure 6: SEM micrographs of (a) untreated fibre, (b) fibre treated with 0.3 M $NaHCO_3$, (c) fibre treated with 0.5 M $NaHCO_3$, and (d) fibre treated with 1 M $NaHCO_3$.

Figures 6(a)–6(d). Figure 6(a) shows the micrograph of the untreated areca frond fibre. From this micrograph, it can be seen that a fleshy layer covers the fibre beneath which lies cells and pits aligned along the axis of the fibre. Treating the fibres helps in removing this fleshy layer thereby exposing the cells and pits making the surface rough. Research work in the past [20–22] has proved that rough surface of the fibre, free from any oily, fleshy, or waxy layer, improves the surface interaction between it and the matrix. Figures 6(b)–6(d) show the surface of the treated fibres. From these micrographs, it is evident that as the concentration of the treating solution increases, surface roughness of the fibre increases forming grooves. This is due to removal of the fleshy material that covers the fibre. With removal of fleshy material, pits and cells become more prominent. Treating with 1 M solution of sodium bicarbonate caused excessive removal of material from the fibre surface resulting in disappearance of pits leaving behind a well-connected network of cells with decreased wall thickness. Excessive removal of material weakens the fibre making it unsuitable for use.

3.2. Tensile Properties of Areca Frond Fibres. Table 4 presents the tensile strength of areca frond fibres. It was observed that the tensile strength of untreated fibre was greater than that of treated fibres. Higher concentration of sodium bicarbonate (1 M) reduces the strength by about 47% and 36% when compared to concentrations of 0.3 M and 0.5 M, respectively. Among the treated fibres, the highest tensile strength of 63.54 MPa was observed for fibres treated with 0.3 M solution while the lowest tensile strength was noticed for fibres treated with 1 M solution. However, untreated fibres exhibited higher tensile strength (67.86 MPa) than the treated fibres. This is due to the fact that treatment leads to weakening of the fibres as a result of removal of the outer layer along with impurities and fleshy matter. Considering the surface characteristics and strength aspects, fibre treated with 0.5 M solution was selected for this study.

Table 4: Tensile strength of areca frond fibres.

Description/molarity	Untreated	0.3 M	0.5 M	1 M
Tensile strength (MPa)	67.86	63.54	58.93	43.22

Table 5: Tensile properties of composites.

Curing environment	Maximum load (N)	Maximum tensile extension (mm)	Average tensile stress (MPa)
Sunlight	1260.63	2.48	12.60
OTG oven	1803.39	2.73	18.03
Steam oven	2431.17	4.10	24.31
Hot air oven	2103.54	3.44	21.03

3.3. Tensile and Flexural Properties of the Composites. The variation in tensile strength is shown in Table 5 and Figure 7.

Table 6: Flexural properties of composites.

Curing environment	Maximum load (N)	Maximum flexural extension (mm)	Average flexural stress (MPa)
Sunlight	34.79	14.24	16.06
OTG oven	48.07	6.34	22.19
Steam oven	73.99	8.29	34.15
Hot air oven	60.82	12.10	28.07

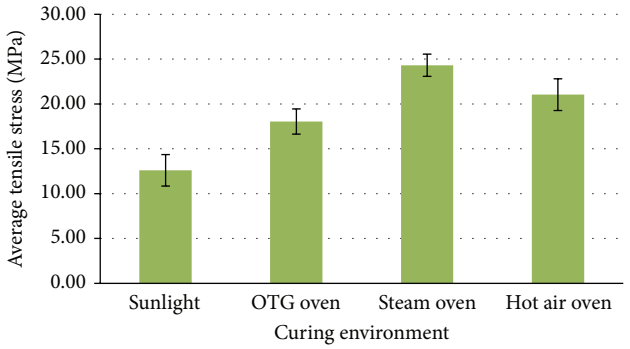


Figure 7: Variation of tensile stress with curing environment.

It is seen that the best tensile strength of 24.31 MPa was obtained for laminates cured in steam oven. The increase in tensile strength when compared to laminates cured in sunlight, OTG oven, and hot air oven was of the order 93%, 35%, and 16%, respectively.

Table 6 and Figure 8 show the variation in flexural strength. Laminates cured in steam oven resulted in maximum flexural strength of 34.15 MPa. Compared to steam oven cured samples, laminates cured in sunlight, OTG oven, and hot air oven resulted in inferior results. Steam oven cured samples were 112%, 54%, and 22% stronger in bending than sunlight cured, OTG oven cured, and hot air oven cured laminates. Flexural strength results of steam cured laminates show an improvement when compared to composites cured in similar conditions using sodium carbonate treated fibres [11].

Improved strength in case of steam oven cured laminates was due to uniform moisture removal from the composites during curing resulting in minimal distortion and shrinkage. Poor strength of sunlight cured laminates may be attributed to lower curing temperature, excessive matrix shrinkage due to nonuniform raise, and drop in atmospheric temperature and prolonged curing duration resulting in disintegration and softening of material.

4. Conclusion

Sodium bicarbonate concentration in the treatment solution affects the surface and strength of the areca frond fibre. Lower concentration of sodium bicarbonate had minimal effect on the strength of the frond fibres. Increase in concentration to 1 M led to increase in surface roughness but weakened the fibre. Steam curing resulted in improved tensile and flexural strength of the composite when compared to other curing

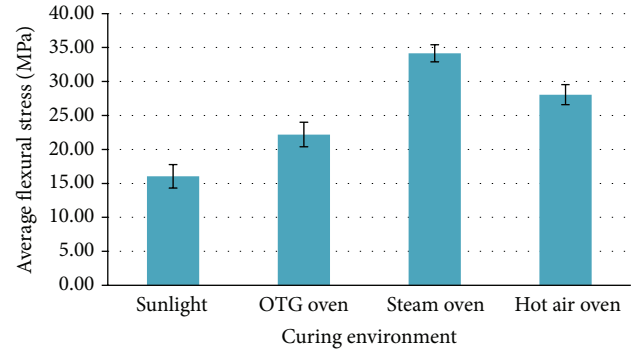


Figure 8: Variation of flexural stress with curing environment.

environments. Sunlight curing resulted in lower tensile and flexural strength of the composites. From the results, the strength of the composites followed the order of steam curing > hot air oven curing > OTG oven curing > sunlight curing.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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