

Characterization and mechanical property of Trinidad coir fibers

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ABSTRACT: Coir fibers were retted in distilled water (DW) and saline water (SW) for up to 12 weeks. Fibers had diameters of 0.16 mm to 0.56 mm, gauge lengths (GL) of 20 mm and 50 mm, and loaded at strain rates of 5, 20, 40, and 60 mm/min. Tensile strength, Young's modulus, and strain at break properties were evaluated and the results statistically analyzed using analysis of variance (ANOVA). For non-retted fibers, as the gauge length decreased, the tensile and strain at break increased by 14% and 42%, respectively, while the stiffness increased by 33% for larger gauge lengths. As the fiber diameter decreased, the tensile strength increased from 48.45 MPa to 134.41 MPa for 50 mm gauge length fibers. X-ray diffraction (XRD) was used to calculate the crystallinity index (CI) of the coir fibers. Secondary electron microscopy was used to assess the fiber surface and fractured area. Although the chemical composition was different, the properties of Trinidad coir fibers were in-line with coir fibers from other parts of the world making them an ideal material of choice for composites. © 2016 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2016**, *133*, 43692.

KEYWORDS: biodegradable; fibers; mechanical properties; recycling

Received 28 September 2015; accepted 15 March 2016

DOI: 10.1002/app.43692

INTRODUCTION

Natural fibers are being utilized and considered in the composite automotive, aerospace, and general consumer industries due to the recent drive to use more sustainable, green, and environmentally friendly materials. Certain engineering projects now consider the use of Life Cycle Assessments (LCA) for the end of life of components and place importance on recyclability of the design, which makes natural fiber indispensable.¹ Natural fibers are lightweight and lower the amount of carbon dioxide emitted during their production (growth) and manufacturing as compared to their synthetic counterparts. They also help reduce the environmental impact on the final product disposal. For example, various natural plant materials evolve from industrial waste by-products such as; hemp, flax, barley, coir, jute, and wheat.² The idea of transforming a waste material into a value added product has gained momentum worldwide.

Coconuts are from the coconut palm fruit *Cocos nucifera* and produce coir fibers from the coconut husk. The customary method to extract the coir fibers from the husk is done by soaking or retting the fibers in ponds and lagoons. More recently, enzymes have been added to retting tanks to reduce the retting period from months³ to days.⁴ Retting enables easier removal of the fibers from the coir pith (or coir dust) which is the corky, softer material surrounding the fibers, and acts to hold them together in the husk. After the fibers are softened, a decorticator

separates the fibers from the husk mechanically or they can be extracted manually by beating the husk. Depending on the age of the coconut, fibers can be white (unripe) or brown (ripe). When compared to mature brown fibers, white fibers are relatively smoother, finer, and weaker,⁵ hence for materials engineering, brown fibers are utilized more than white fibers.

Coconuts are found in tropical regions where the main producers and exporters of the raw coir fibers and its products are India and Sri Lanka, followed by Thailand, Vietnam, the Philippines, and Indonesia with the Caribbean sharing a less than significant portion of this market.^{2,6} About 55 billion coconuts are harvested annually in the world and of that amount, less than 15% of the husk fibers are recovered for use such as in ropes, doormats, flower pots, geotextiles, coastal defense structures, and composites, while the rest is abandoned as waste.² The annual production of coconuts in the Trinidad and Tobago is approximately 16,500 tons.⁷ In general, for the coconut by-product industry in the Caribbean and elsewhere to become economically viable, there is a need for wider product development. Growth geography of natural plant materials may affect their strength properties. Coir fiber properties are dependent on plant species, regional climate, soil type, and water supply in addition to the husk extraction method and harvest time.⁸ It is anticipated the location may affect the growth process and the quality of the fibers. Globally, mechanical property studies have been conducted on coir fibers, however, the literature indicates

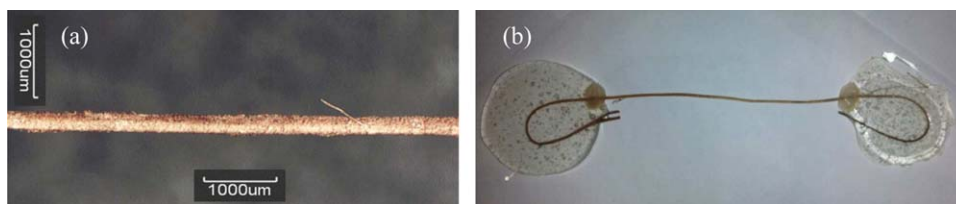


Figure 1. Coir fiber images (a) typical coir fiber section showing surface profile and (b) digital image of tabs for mounting coir fibers into tensile grips. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

limited material characterization/evaluation on Caribbean coir fibers. For example, a study conducted on single coir fibers from Brazil obtained mechanical properties of tensile strength, Young's modulus, and strain at break in the range of 118–143 MPa, 1.3–2.7 GPa, and 25–60%, respectively.⁹ Another study performed on coir fibers originating from Vietnam achieved properties of 186–343 MPa, 4.2 GPa, and 26–64%, respectively.¹⁰ Similarly, coir fibers from India had mechanical properties of 112–161 MPa, 4–5 GPa, and 18–43%, respectively.¹¹

The present study assessed selected properties of Caribbean brown coir single fibers after retting in two mediums; distilled water (DW) and saline water (SW) for 1, 2, 4, 8, and 12 weeks periods and compared against non-retted fibers. The tensile strength, tensile Young's modulus, and strain at break were evaluated for different fiber diameters, gauge lengths (GL), and strain rates. X-ray diffraction (XRD) was used to investigate the crystallinity index (CI) of retted and non-retted fibers. Fractured surfaces of the fibers were evaluated using the scanning electron microscope (SEM). Finally, selected fiber properties were compared against coir fibers grown in other regions of the world.

EXPERIMENTAL

Material Preparation

Brown coir fiber husks were obtained from Cedros Atlantic Waters Limited (Trinidad). Retted and non-retted fibers were extracted from the husks manually. The as-received coir fiber husks were treated by soaking in two liquid mediums; distilled water (DW) and a saline water (SW) to simulate seawater retting, while the non-retted fibers were removed from the husk by beating with a wooden mallet. In general, the salinity of seawater is on average 34 grams per kilogram or 34 parts per thousand (ppt) and was replicated in this study. To gain insight on the retting process, the retting period was evaluated for 1, 2, 4, 8, and 12 weeks. At the end of each week, the coir fibers were removed from each medium, rinsed with distilled water, dried at ambient temperature for 48 hours, and manually separated into individual fibers.

The fibers were arbitrarily chosen from the husks for both unretted and retted fibers ensuring the fibers were not damaged. The fibers were separated into bundles by gauge lengths (GL) of 20 mm and 50 mm. The lengths of the single fibers were measured in accordance to ASTM D5103-12. The diameter of the coir fibers were measured using a stereo microscope (SZ-40, Olympus) at a magnification of 67× and digital images were captured using a camera (Moticam 2300, Motic) and image analyzing software (Motic Image Plus 2.0). The average of six

locations along the fiber length was taken. A typical coir fiber section is shown in Figure 1(a).

X-ray Diffraction Analysis

The crystallinity of cellulose from non-retted and retted coir fibers was evaluated with an X-ray diffractometer, Philips X'Pert PRO diffractometer with Cu-K α radiation ($\lambda = 1.54 \text{ \AA}$), a current and voltage of 40 mA and 45 KV, respectively. X-ray diffraction (XRD) spectra were collected over a 2θ angle of 5–40°. The fibers were initially dried and made into a powder using a pestle and mortar. The crystallinity index or the degree of crystallinity of the coir fibers was estimated from the XRD results based on the empirical equation proposed by Sarikanat *et al.*¹²:

$$\text{CI (\%)} = [(I_{002} - I_{\text{am}}) / I_{002}] \times 100 \quad (1)$$

where I_{002} (002 peak) characterizes the maximum peak intensity corresponding to both crystalline and amorphous materials at a diffraction 2θ angle of 22–22.7°. I_{am} represents amorphous materials for cellulosic fibers with the lowest intensity at 2θ angle of 18–18.5°. This formula can be used as an approximation for comparing the crystallinity of natural fibers after retting treatments.

Coir Fiber Chemical Composition

The chemical composition (cellulose, lignin, and hemicellulose) of the coir fiber without the soft corky material was determined by acid digestion using an Ankom A200 Fiber Analyzer (Ankom, Macedon, NY, USA). The content of the cellulose, lignin, and hemicellulose were calculated from the difference in acid detergent fiber (ADF), neutral detergent fiber (NDF), and acid detergent liquid (ADL) using Ankom A200 Method 5, Method 6, and Method 8, respectively. Each assay was run in triplicates.

Scanning Electron Microscopy (SEM) Analysis

The surface morphology and fractured surface of the coir fibers were examined using an FEI Quanta 650 FEG scanning electron microscope (SEM). SEM micrographs were taken at various magnifications using an accelerating voltage of 20 kV. The samples were placed on aluminum stubs, fixed with carbon tape, and further sputter coated with carbon to make them electrically conductive.

Tensile Tests

Tensile behavior were conducted on single fibers using a Zwick/Roell tensile testing machine (Z020, Zwick/Roell Group) with a 20 kN load cell and a strain rate of 20 mm/min according to the ASTM D3379-89. For ease of calculating the cross-sectional area, the diameter of the fibers was assumed to be constant. For each condition, the average of five coir fibers tested. To gain an

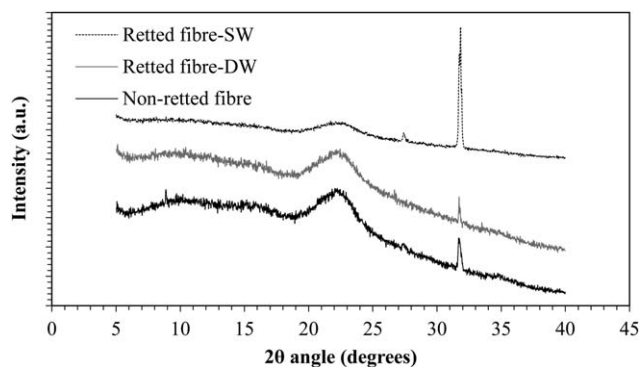


Figure 2. X-ray diffraction of non-retted and retted coir fibers from Trinidad.

understanding of the effect of the strain rate during a tensile test, four strain rates; 5, 20, 40, and 60 mm/min were evaluated on 0.30 mm diameter fibers with gauge lengths of 50 mm.

The fine coir fibers were mounted individually into the tensile grips using epoxy tabs as shown in Figure 1(b). The epoxy used was Devcon 5-minute epoxy (ITW Devcon, Danvers, MA). In order to make the tabs, the gauge lengths were first measured and two drops of LePage White general purpose glue were placed at either ends. The glue acted to block any accidental epoxy that may 'leak' onto the coir fiber. To increase the bonded surface area and prevent slippage in the epoxy tabs, the fiber ends were curled and taped to the piece of paper prior to applying the epoxy. The epoxy was allowed to cure for 48 hours.

Statistical Analysis

Statistical analysis was performed using one-way analysis of variance (ANOVA) using Microsoft Excel 2010. The F-test was used with a level of significance of 0.05, which is a confidence level of 95%. ANOVA determined if any significant differences existed between the groups over the retting time periods. In this study, the properties of tensile strength, Young's modulus, and strain at break were measured at different weeks of retting, where the retting period is the only variable that is changed throughout the test program. The outcomes will help recognize if the retting treatments provided a statistical improvement in mechanical properties of these coir fibers.

RESULTS AND DISCUSSION

X-ray Diffraction Analysis

The XRD powder patterns of non-retted and DW and SW retted are given in Figure 2. The profiles and low resolution are comparable to other coir fiber XRD results.^{3,13} The crystalline peaks occur at a 2θ angle of approximately $22\text{--}22.7^\circ$ and $15.8\text{--}16^\circ$ and are representative of cellulose crystallographic planes I_{002} and I_{101} , respectively.¹³ The non-retted coir fiber had a reduced crystallinity peak intensity which is attributed to the crystalline cellulose micro-fibrils being embedded in large amounts of lignin, hemicellulose, and pectin materials. After 12 weeks of retting in DW or SW, increases in peak intensities were not visible which suggests these substances remained. The coir fibers in this study were obtained on the coastal estate of a coconut farm. Salt from the ocean water and air have been drawn into the coconut fibers

Table I. Chemical Composition of Untreated Raw Coir Fibers (wt %)

Cellulose	Lignin	Hemicellulose	Pectin	Region
48.6 ± 0.4	30.4 ± 0.6	8.2 ± 0.3	-	Present work
36.0-43.0	41.0-45.0	0.15-0.25	3.0-4.0	India ³
44.2-54.2	35.4-43.4	1.5-2.5	-	India ¹³
43.4-53.0	38.3-40.8	14.7	3.5	Brazil ¹⁴
49.5-50.5	35.2-36.2	20.1-20.7	-	Brazil ¹⁵
32.7	59.4	7.9	-	Jamaica ¹⁶
32.7	42.1	22.6	-	Thailand ¹⁸

during their growth. This is confirmed by the XRD results of the diffraction peak at a 2θ angle of 31.7° which is indicative of the mineral salt, sodium chloride (NaCl) with the (002) crystallographic plane. The fiber retted in SW shows a higher diffraction peak possibly from the higher salt content in the retting medium due to a lack of proper rinsing. The fiber retted in DW has a slightly smaller peak than the natural coir fibers indicating that some of the salts may have leached out.

The microstructure of natural fibers contains cellulose microfibrils surrounded by a matrix of lignin, hemicelluloses, and pectin. The cellulose is composed of high molecular weight well-ordered crystalline chains, which provide strength to the fibers, while lignin, hemicelluloses, and pectin are amorphous with chains that are not well ordered. The amorphous materials have much lower molecular weights and do not provide strength to the fibers but act to adhesively bond the cellulose microfibrils. At the micro-structural level, cell morphology, and amounts of each polymer elements can be affected by climate (location), water supply, soil nutrients, temperature, age, and coconut specie. Typical chemical composition of coir fibers from different tropical regions have been reported by several authors and are given in Table I along with the composition of the coir fibers in the current study. Based on these previous studies, the major constituents of coir fibers have a general range of; cellulose (33-54%), lignin (35-59%), and hemicellulose (1.5-23%). The coir fibers from Trinidad do not have an identical composition as any of the reported coir fibers. For instance, the cellulose content is close to that of coir fibers from Brazil¹⁴ and India,¹³ the lignin content is similar to Brazil¹⁵ and India,¹³ while the hemicellulose content matches that of Jamaica.¹⁶ This study reinforces the fact that growth location

Table II. Crystallinity Index of Coir Fibers

Material	$I_{(002)}$ ($2\theta = 22.5^\circ$)	$I_{(am)}$ ($2\theta = 18.5^\circ$)	CI (%)
Non-retted fiber ^a	2729.03	2139.73	21.59
Retted fiber-DW ^a	2377.33	1904.86	19.87
Retted fiber-SW ^a	2080.98	1642.20	21.09
India	5407.93	4568.76	15.52 ¹³
India	2482.37	1881.61	24.20 ¹⁹
Brazil	873.42	611.98	29.93 ²⁰

^aPresent work-coir fiber from Trinidad.

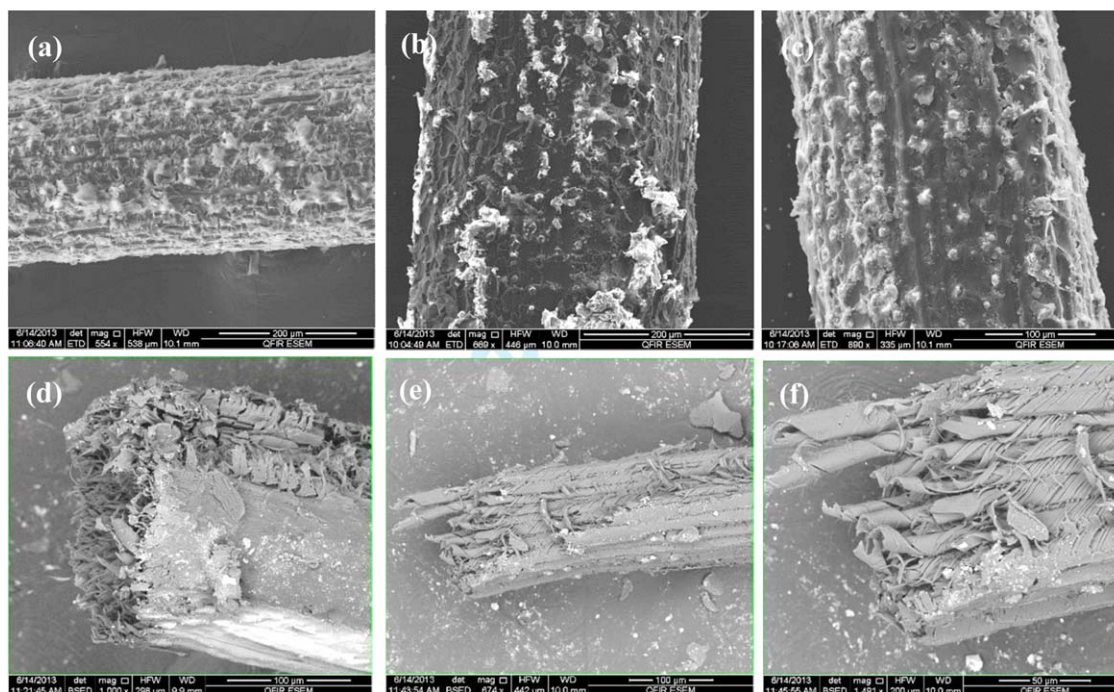


Figure 3. SEM coir fiber images (a) non-retted surface, (b) retted in distilled water (DW) for 12 weeks, (c) retted in saline water (SW) for 12 weeks, (d) fractured surface-retted in DW (e) retted in SW, and (f) magnified fractured surface. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

may affect the properties of single coconut fibers. The cellulose content is the source for fiber tensile strength. Lignin surrounds and protects cellulose and acts to increase the stiffness and toughness of the fiber.¹⁷ It also enhances durability with regards to decay in moist environments. Fibers with elevated cellulose contents can be used in applications requiring high strength while fibers with lower lignin contents can be used where high modulus and toughness is needed.

The CI coir fibers from different regions are given in Table II. Compared to the non-retted fibers in this study, the crystallinity index of coir fibers retted in DW and SW decreased by 8% and 2%, respectively. The XRD peaks of the non-retted fiber results were corroborated to these results in Figure 2. The lack of increase in crystallinity index for the two retting treatments indicated an increase in cellulose content from removal of the lignin did not occur. Coir fibers with a higher quantity of lignin tend to have a reduce tensile strength but improvement in toughness.

To compare the Trinidad coir fiber CI, coir fibers XRD patterns from other studies were used to obtain the I_{002} and I_{am} intensities at the 2θ angles of 22.5° and 18.5° , respectively. Data points were extracted directly from the XRD patterns using the Plot Digitizer 2.6.2 software. The results show coir fibers obtained from different geographical areas can have different amounts of cellulose and lignin contents as shown in Table II. The CI values from this work are in agreement with the literature. The lower CI reported value of 15.52% was increased with a chemical alkali treatment by reducing the lignin content and increased the cellulose component. After the treatment the cellulose increased from 39.3% to 50.5%.¹³ However, this study

did not use chemical treatments but only conventional, environmentally friendly methods employed in the Caribbean.

Scanning Electron Microscopy

Typical SEM micrographs of coir fibers are shown in Figure 3. Figure 3(a) shows the as-received non-retted fiber, while the fibers retted in DW and SW for 12 weeks are shown in Figure 3(b,c), respectively. The untreated fiber sample was covered with a waxy layer of substance that could have included impurities, lignin, and pectin.²¹ This uneven surface is also visible in the retted fibers. In general, coir fibers treated with chemicals such as an alkaline treatment remove lignin on the surface of the fibers have shown to have a smoother pitted surface,²²

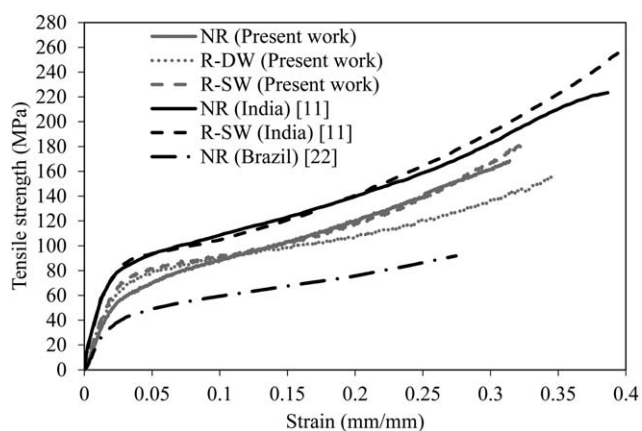


Figure 4. Stress–strain diagram for non-retted and retted coir fibers from various tropical regions [Non-retted (NR), Retted (R), Distilled Water (DW), and Saline Water (SW)].

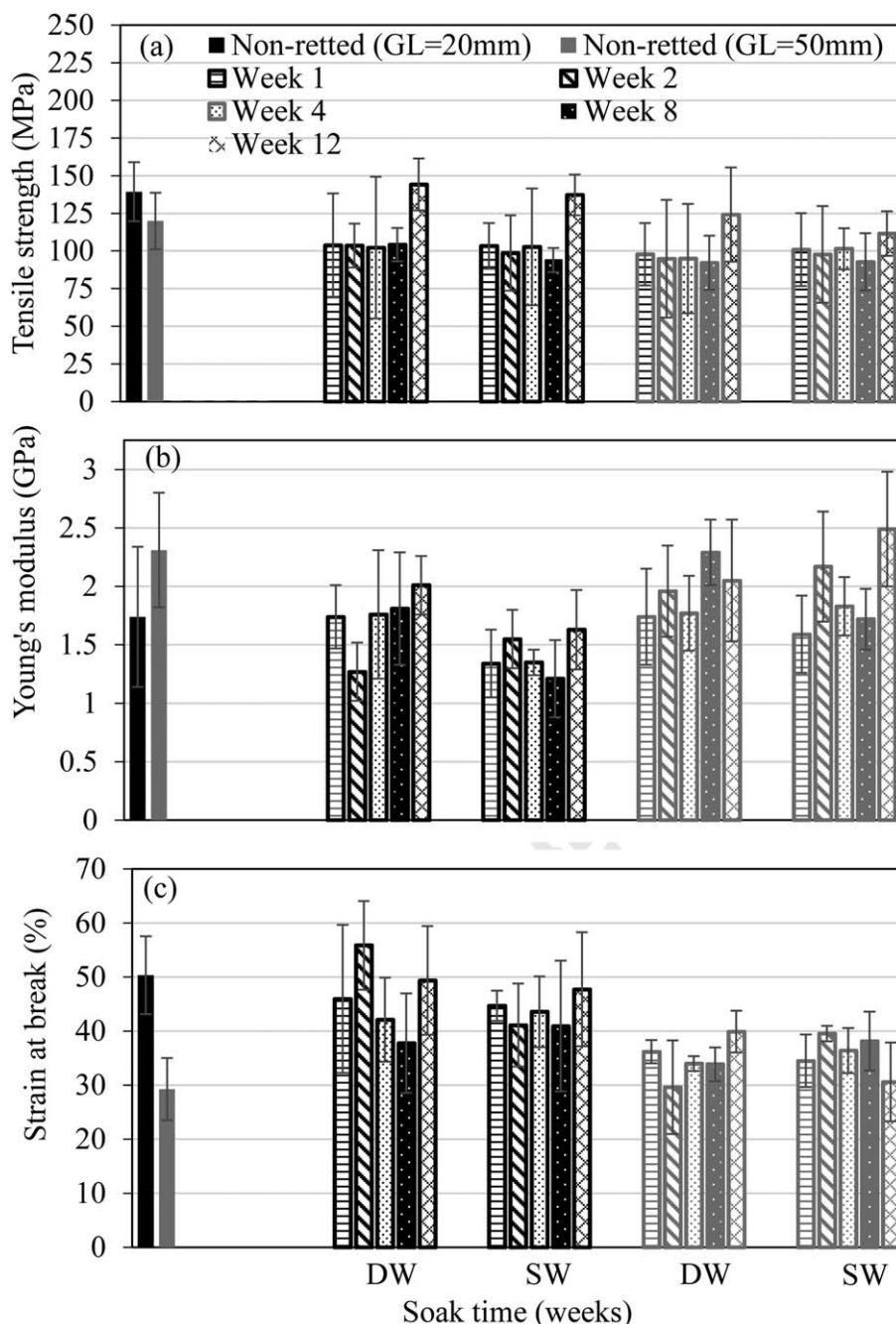


Figure 5. Results for (a) tensile strength, (b) Young's modulus, and (c) strain at break as a function of soaking time in distilled water (DW) and saline water (SW) for gauge lengths (GL) of 20 mm and 50 mm.

however, this was not visible on water-retted fibers. The retting treatments did not appear to have an effect on the coir fibers, perhaps longer retting times are required.

After loading to failure at a strain rate of 20 mm/min, the fractured surface of coir fibers retted in DW and SW are shown in Figure 3(d,e), respectively. Coir fibers are constructed of helical cellulose microfibrils as shown in the fractured surfaces. These helical spirals grow at angles between 30–51°⁹⁹ to form circular longitudinal cells within the coir fiber structure and are used for water and nutrient transport. When the fibers are positioned

in a tensile state, these spirals unravel and align with the load axis as shown in the magnified image of Figure 3(f). This phenomenon is known as strain hardening. As the spirals untwist and re-orient themselves into a straightened position, the tensile strength, and modulus increases. At the fractured ends of the coir fibers, neck regions were observed similar to that found in polymers.

The main reason for retting was to remove the fibers from the husk. The study showed the retting treatments had an advantage to not adversely affect the tensile properties of the coir

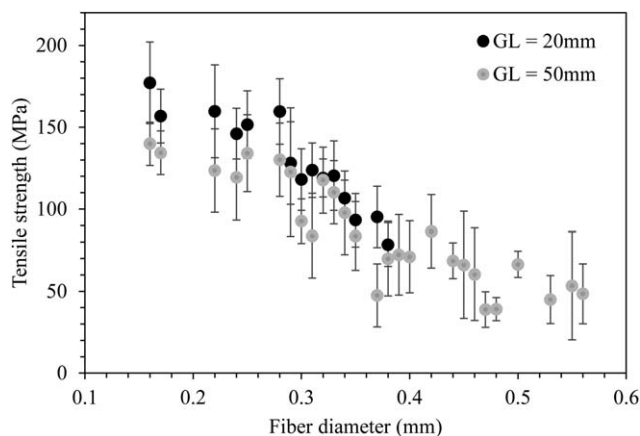


Figure 6. Tensile strength as a function of diameter for non-retted coir fibers.

fibers. The results were similar to other coir fibers in other parts of the world. After studying single fibers, it is anticipated to further evaluate single fiber mats and composite materials for potential product development in the Caribbean's.

Tensile Properties

A typical stress–strain behavior of non-retted and 12 week retted single coir fibers from Trinidad are given in Figure 4 for diameter of 0.25–0.30 mm, strain rate of 20 mm/min and gauge length of 50 mm. For comparison purposes, on the same graph the results of a representative coir fiber with diameter of 0.25 mm and GL of 50 mm originating from India was non-retted and retted in saline water for up to 25 weeks and tested at 20 mm/min.¹¹ Similarly, a Brazilian coir fiber with diameter of 0.25 mm and gauge length of 50 mm was tensile tested at a strain rate of 20 mm/min.²² The Plot Digitizer 2.6.2 software was used to replicate the graphs. The profiles were comparable to other coir fiber tensile tests conducted for different parameters^{3,9,11} and were independent of retting condition. At the beginning of the test, the coir fibers deformed elastically with a high linear slope up to the yield strength of the material. At this point, the coir fibers began to exhibit a non-linear transition from elastic to plastic behavior. After the start of plastic deformation, the slope dropped with a steady linear increase up to failure. Prior to failure, a concave curvature formed with increasing strain indicating the strain hardening phenomenon. For the two studies with non-retted and retted stress–strain curves in Figure 4, at low strains below 2.5%, the coir fiber curves nearly overlap with one another and deviate slightly as the strain increases. This low variation in the elastic and plastic regions suggests the modulus is not affected by DW or SW retting. From the area under the stress strain curve, coir fibers show good toughness as observed from the combination of high strength and ductility.

Tensile Strength

The tensile strengths for non-retted coir fibers and fibers retted in DW and SW for 1, 2, 4, 8, and 12 weeks are given in Figure 5(a). Two GL of 20 mm and 50 mm were tested with diameters ranging from 0.20–0.30 mm. The results show high standard deviations, which are typical of natural plant fibers. During

growth or removal of the husk from the nut, surface defects can occur. Material defects/imperfections at the cellulose microfibril level are difficult to control and are partly responsible for the large variations in mechanical properties. However, the values are in agreement with other tested single coir fiber tensile strength studies.^{9,11}

The non-retted coir fiber strengths for gauge lengths of 20 mm and 50 mm were 139 ± 19.53 MPa and 120 ± 18.76 MPa, respectively which are in accordance with literature mechanical properties. For example, untreated coir fibers from Brazil were tested with a diameter of 0.225 mm, strain rate of 20 mm/min, and gauge length of 20 mm and produced an average tensile strength, 142.40 ± 70.20 MPa.⁹ Another study reported coir fibers from Brazil with a gauge length of 50 mm, average fiber diameter of 0.250–0.411 mm that had a mean tensile strength of 76 ± 15 MPa.²² Similarly, coir fibers from Thailand with a gauge length of 50 mm, fiber diameter of 0.226–0.637 mm, had a mean tensile strength, 123 ± 34.7 MPa.¹⁸ The untreated fiber strengths reported in the literature are comparable to that of Trinidad. In the current study, non-retted coir fiber tensile strengths decreased by 14% as the gauge length increased from 20 mm to 50 mm. Comparing identical gauge lengths, a study from Brazil showed a decrease of 34% for coir fibers,¹¹ while a work from coir fibers in India conducted at gauge lengths from 5 mm to 25 mm decreased in tensile strength by 17%.⁹ Differences could be attributed to fiber defects, coconut species, location of growth, and age of the fiber.

Coir fibers retted from 1 to 8 weeks in DW or SW, did not increase the tensile strengths for both gauge lengths. Coir fibers with gauge lengths of 20 mm and soaked in DW and SW decreased in tensile strength by 25–26% and 26–32%, respectively compared to the non-retted fibers. Similarly, gauge lengths of 50 mm decreased by 18–23% and 15–22%, respectively. A similar behavior was observed in a study conducted on coir fibers from India.²³ Tensile strengths for fiber diameters of 0.25–0.30 mm and retted for 9 days in tap water decreased by 9%. After 12 weeks of retting, the change in tensile strength was minimal. Fibers retted in DW increased in tensile strength by about 4%, while a decrease of 1–7% occurred after retting in SW for gauge lengths of 20–50 mm and diameters ranging between 0.20–0.30 mm. Coir fibers from India with a gauge length of 50 mm and diameters of 0.25 mm and 0.30 mm were retted in SW for 6 months and achieved an increase in tensile strength of 13% and 3%, respectively.¹¹ Variation in the results may be due to the retting times. As shown in Figure 5(a), fibers retted for 12 weeks in either medium are superior to those retted for 1 and 8 weeks. Perhaps longer retting periods may slightly improve their tensile strengths. The results indicate the retting technique used to remove the fibers from the coconut husks did not adversely affect the tensile strengths.

Young's Modulus

The Young's modulus of the non-retted and retted coir fibers are given in Figure 5(b). The non-retted fibers with diameters of 0.20–0.30 mm and gauge lengths of 20 mm and 50 mm had a Young's modulus of 1.74 ± 0.60 GPa and 2.31 ± 0.49 GPa, respectively. This was an increase in modulus of 33%. The

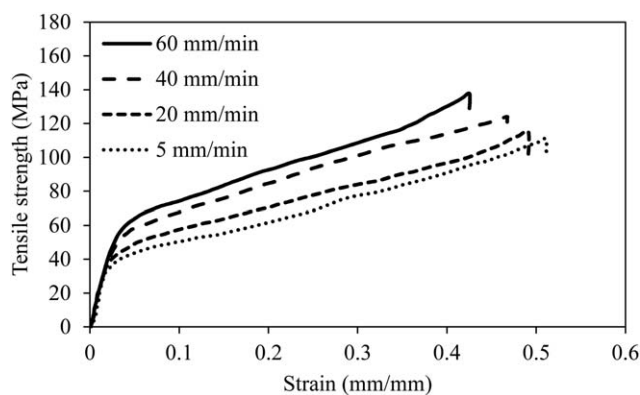


Figure 7. Stress–strain diagram for non-retted coir fibers loaded at different strain rates.

modulus was observed to increase as the gauge length increased for both non-retted and retted fibers. A similar phenomenon occurred for a study on coir fibers from Brazil with diameters of 0.225 mm where the modulus increased by 115% when gauge lengths were increased from 5 mm to 25 mm. The authors determined the improvement in modulus was due to a greater amount of lignin in longer fibers, which contributes to a more rigid and stiffer fiber.⁹ Studies reported similar Young's modulus for un-retted coir fibers from different geographical regions. For example, an earlier study⁹ reported the Young's modulus for Brazilian coir fibers to be 2.39 ± 0.87 GPa for a gauge length of 20 mm and diameter of 0.225 mm when pulled to failure at a strain rate of 20 mm/min. Another study reported coir fibers from Brazil with a gauge length of 50 mm and fiber diameter of 0.250–0.411 mm had Young's modulus of 2.1 ± 0.3 GPa.²² Similarly, coir fibers from Thailand with a gauge length of 50 mm, fiber diameter of 0.226–0.637 mm, had a modulus of 2.29 ± 0.47 GPa.¹⁸ After 12 weeks of retting, there was no significant improvement in modulus for either gauge lengths tested. Retting in different mediums for different amount of times did not show any apparent trend in modulus. This could possibly be due to the high standard deviations obtained, which can have a tendency to affect the accuracy of the results. Similar to the tensile strength results, the modulus was not affected by retting in DW and SW for up to 12 weeks. This suggests the retting method was useful in separating the fibers from the husk but did not affect their stiffness properties.

Strain at Break

The strain at break of the non-retted and retted coir fibers are given in Figure 5(c). The gauge length had an impact on the amount a coir fiber can stretch before it breaks. For a gauge length increase from 20 mm to 50 mm, the non-retted fibers had a strain at break of $50.35 \pm 7.19\%$ and $29.26 \pm 5.75\%$,

respectively for a reduction of 42%. Comparable results were achieved from two separate coir fiber investigations from Brazil. One study looked at 50 mm gauge length and diameter of 0.250–0.411 mm coir fibers where the authors reported a strain at break of $29.0 \pm 5.0\%$.²² Another study reported a strain at break of $28.60 \pm 13.60\%$ for diameters of 0.225 mm, strain rate of 20 mm/min, and gauge length of 20 mm.⁹ The same tendency for the strain at break to decrease with increase in gauge length was observed in previous works conducted on untreated coir fiber. For instance referring to the same gauge lengths as the current study, the strain at break decreased by 40%.¹¹ Another work increased the gauge lengths from 5 mm to 25 mm, where a strain at break decrease of 47% was observed.⁹ Retted fibers in DW and SW exhibited comparable strain at break behaviors as the non-retted fibers for both gauge lengths and all retting times. Coir fibers from the Trinidad had relatively high strain at break values, which would make them acceptable for situations requiring high impact toughness. An earlier study¹¹ did not observe any significant differences in mechanical properties between non-retted and SW retted coir fibers for 6 months. In a similar way, a study conducted on retting of coir fiber in saline water for 6–10 months was able to remove pectin, pentosan, fat, and tannin contents, however the retting treatment did not affect the contents of cellulose and lignin.²⁴ From these observations it can be acknowledged that no significant differences in strength or stiffness are anticipated to take place between non-retted and retted coir fibers. If required, additional chemical treatments such as silane, acetylation, and alkalization may be used to improve the mechanical properties but is outside the scope of this research.

Change in Gauge Length

The tensile strength as a function of diameter for non-retted coir fibers are shown in Figure 6 for two gauge lengths of 20 mm and 50 mm. The fiber diameters ranged from 0.16 mm to 0.56 mm and were loaded at a rate of 20 mm/min. For both gauge lengths, the tensile strengths decreased as the fiber diameter increased. This trend was similar to the results of another study on coir fibers from Brazil⁹ where the authors tested fiber diameters in the range of 0.04 mm to 0.40 mm, gauge length of 20 mm, and strain rate of 5 mm/min. However, in an earlier study, non-retted fibers were observed to increase in tensile strength for diameters of 0.1 mm to 0.2 mm and further become constant for diameters of 0.2 mm to 0.45 mm, although with relatively high standard deviations.¹¹ Over all the tensile strength of non-retted fibers was highest for fiber diameters in the range of 0.16 mm to 0.28 mm and gauge length of 20 mm.

For the same diameter value, the results show for an increase in gauge length from 20 mm to 50 mm, the linear trend was for a reduction in tensile strength. A decrease in mechanical

Table III. ANOVA Results (0.05 Level of Significance) for Tensile Strength of Coir Fibers Retted in Distilled Water with 20 mm Gauge Lengths

Source of variation	SS	Df	MS	F	Fcrit
BG	8707.414	5	1741.483	1.210062	2.740058
WG	2,7344.19	19	1439.168		
Total	3,6051.6	24			

Table IV. ANOVA (0.05 Level of Significance) Analysis of Mechanical Property Results for Coir Fibers Retted and Non-Retted from 1 to 12 Weeks

Gauge length (mm)	Distilled water		Saline water	
	F	Fcrit	F	Fcrit
Tensile strength				
20	1.21	2.74	1.85	2.81
50	0.656	2.74	0.909	2.77
Young's modulus				
20	1.66	2.71	1.37	2.71
50	1.70	2.74	3.26	2.74
Strain at break				
20	0.984	2.68	0.354	2.71
50	1.46	2.64	2.70	2.71

properties with increase of fiber diameter can be caused by an increase in the probability of defects inherent to natural fiber materials but more importantly to the microfibril angle. For example, a study found natural Sisal fiber to have a microfibril angle of 10–22°, which produced a tensile strength of 568–640 MPa, modulus of 9.4–15.8 GPa, and a strain at break of 3–7%. The same study compared coir fiber with a much larger microfibril angle of 30–49° which generated a tensile strength of 106–175 MPa, modulus of 4–6 GPa, and a strain at break of 17–47%.²⁵ This would indicate higher microfibril angles produce weaker fibers in terms of lower tensile strength, lower modulus, but higher strain at break.

Coir fiber diameters are not uniform along their length. For instance, one end of the fiber has a larger diameter and progressively decreases thereafter. A study conducted on a spruce tree branch measured the microfibril angles of the wood cells near the trunk and further away at the branch tip. The authors found the microfibril angle to change significantly along the branch length. At the trunk where the diameter was the largest, the microfibril angles were the highest. Further along the branch, the angles decreased continuously to the tip, which infers stiffening of the branch. The gradual decrease in microfibril angle along the length of the branch corresponded to a stiffening of the wood material.²⁴ This gradual change in microfibril angle may be a way for nature to maximize its efficiency. It is thought that coir fibers may grow in the same manner but further investigations are required in this area.

Change in Strain Rate

Non-retted coir fibers with average diameters of 0.30 mm and gauge length of 50 mm were loaded at strain rates of 5, 20, 40, and 60 mm/min. as shown in Figure 7 to evaluate the visco-elastic response of the coir fiber material. The result illustrate the stress–strain behaviors were different for different strain rates. As the strain rate increased, the tensile strength increased and the strains reduced. In natural fibers, when loading is rapid, the fibers behave elastically and the load is shared by the crystalline regions resulting in higher tensile strengths. At low strain rates, the fibers behave as a viscous liquid and load is shared

among the amorphous regions,²⁶ which can be noticed by the higher strains. Up to about 40 MPa, the Young's modulus was the same at all strain rate levels, which indicated the fiber stiffness was not affected by an increase of strain rate.

Statistical Analysis

A typical ANOVA one-factor result from Excel is given in Table III. The nomenclature is as follows: between groups (BG), within groups (WG), sum of squares (SS), degree of freedom (Df), mean square (MS), F (F-test statistic), and Fcrit (critical value). The F-test determines if significant differences between groups occurred over the range of retted times. If F is greater than Fcrit, the null hypothesis is rejected and the results have a significant difference in mean value. This would suggest the retting treatments were not equally effective. If F is less than Fcrit, the null hypothesis cannot be rejected and the results are not significantly different in mean values.

The ANOVA results are shown in Table IV showing the differences between the tested samples for tensile strength, Young's modulus, and strain at break. The analysis has confirmed that for almost all tests, the F is less than Fcrit. The results suggest the coir fibers at the two gauge lengths tested, did not have a significant increase or decrease in tensile strength, Young's modulus, and strain at break. Thus, the coir fiber properties measured did not improve due to a retting treatment in either distilled water or saline water. However, one exception was the Young's modulus of the 50 mm gauge length fibers, retted in saline water, where F was greater than Fcrit. This means that the coir fibers had a statistically significant change in stiffness as the retting time increased. To clarify this result, a further ANOVA analysis was conducted on the Young's modulus for this gauge length without taking into account the 12-week group. Therefore, the analysis included the un-retted fiber group with fibers retted for 1, 2, 4, and 8 weeks. Interestingly, the F and Fcrit values were 2.84 and 2.96, respectively, indicating no significant change in the stiffness. Perhaps the Young's modulus increases considerably beyond 12 weeks of retting in saline water, but should be verified in a future study. Overall, the retting treatments did not significantly change the tensile strength, Young's modulus, and strain at break of the coir fibers with 20 mm and 50 mm gauge lengths.

CONCLUSIONS

Tensile strengths and modulus were not affected by the retting treatments for both gauge lengths tested which suggests these are appropriate methods to remove the coir fibers from the husks. The strain at break was higher for fibers with gauge lengths of 20 mm. The tensile strength for non-retted fibers was better for short gauge lengths of 20 mm and small diameters ranging from 0.16 mm to 0.28 mm. When the strain rate was increased, the tensile strengths improved and the strains decreased. The X-ray diffraction identified the crystalline peaks of cellulose, the main constituent of natural based fibers. It was also used to calculate the crystallinity index to verify and determine if retting improved the cellulose content. The result indicated lignin removal and an increase in cellulose did not occur. The coir fibers from Trinidad did not have an identical

composition as other coir fibers reported which indicated the growth location may be important. From the scanning electron microscope images, the waxy surface generally found on coir fibers was not removed by retting in either water mediums for 12 weeks. The fractured surfaces showed signs of strain hardening from the uncoiling and untwisting of the microfibrils. Statistical analysis using ANOVA suggested the retting treatments in distilled water and saline water for up to 12 weeks did not have a significant statistical difference in tensile strength, Young's modulus, and strain at break. Therefore, the retting treatments did not affect the mechanical properties of the coir fibers.

ACKNOWLEDGMENTS

The authors would like to acknowledge the support of the Government of Canada through the Emerging Leaders in the Americas Program (ELAP), the support of the Natural Sciences and Engineering Research Council of Canada (NSERC) Discovery Grant (418729-2012 RGPIN) for the financial support of this study. Also Queen's University (Canada) and The University of the West Indies (St. Augustine) for their laboratory support.

REFERENCES

1. Ahmad, F.; Choi, H. S.; Park, M. K. *Macromol. Mater. Eng.* **2015**, *300*, 10.
2. Ramamoorthy, S. K.; Skrifvars, M.; Persson, A. *Polym. Rev.* **2015**, *55*, 107.
3. Varma, D. S.; Varma, M.; Varma, I. K. *Text. Res. J.* **1984**, *54*, 827.
4. Basu, G.; Mishra, L.; Jose, S.; Samanta, A. K. *Ind. Crop. Prod.* **2015**, *77*, 66.
5. Harish, S.; Michael, D. P.; Bensely, A.; Lal, D. M.; Rajadurai, A. *Mater. Character.* **2009**, *60*, 44.
6. Deka, H.; Varghese, T. O.; Nayak, S. K. *Polym. Compos.* **2015**. DOI: 10.1002/pc.23529.
7. Food and Agriculture Organization of the United Nations-FAO. Database Agricultural-Production-Crops-Coconuts, **2014**. Available from: <http://faostat3.fao.org>.
8. Khalil, H. A.; Hossain, M. S.; Rosamah, E.; Azli, N. A.; Saddon, N.; Davoudpoura, Y.; Islam, M. N.; Dungani, R. *Renew. Sustainable Energy Rev.* **2015**, *43*, 1006.
9. Tomczak, F.; Sydenstricker, T. H. D.; Satyanarayana, K. G. *Compos. A* **2007**, *38*, 1710.
10. Defoirdt, N.; Biswas, S.; De Vriese, L.; Van Acker, J.; Ahsan, Q.; Gorbatikh, L.; Van Vuure, A.; Verpoest, I. *Compos. A* **2010**, *41*, 588.
11. Kulkarni, A. G.; Satyanarayana, K. G.; Sukumaran, K.; Rohatgi, P. K. *J. Mater. Sci.* **1981**, *16*, 905.
12. Sarikanat, M.; Seki, Y.; Sever, K.; Durmuşkahya, C. *Compos. B* **2014**, *57*, 180.
13. Abraham, E.; Deepa, B.; Pothan, L. A.; Cintil, J.; Thomas, S.; John, M. J.; Anandjiwala, R.; Narine, S. S. *Carbohydr. Polym.* **2013**, *92*, 1477.
14. Satyanarayana, K. G.; Guimarães, J. L.; Wypych, F. E. *Compos. A* **2007**, *38*, 1694.
15. Ramires, E. C.; de Oliveira, F.; Frollini, E. *J. Appl. Polym. Sci.* **2013**, *129*, 2224.
16. Jústiz-Smith, N. G.; Virgo, G.; Buchanan, V. E. *Mater. Character.* **2008**, *59*, 1273.
17. Satapathy, S.; Kothapalli, R. V. *J. Appl. Polym. Sci.* **2015**, *132*, DOI: 10.1002/app.42237.
18. Muensri, P.; Kunanopparat, T.; Menut, P.; Siriwanayotin, S. *Compos. A* **2011**, *42*, 173.
19. Rajini, N.; Jappes, J. W.; Rajakarunakaran, S.; Bennet, C. *Chin. J. Polym. Sci.* **2013**, *31*, 1074.
20. Carvalho, K. C. C.; Mulinari, D. R.; Voorwald, H. J. C.; Cioffi, M. O. H. *BioResources* **2010**, *5*, 1143.
21. Chandy, M.; Sarma, U. S.; Latha, M. S.; Kumar, K. A.; Kumar, K. S. *Int. J.* **2015**, *3*, 140.
22. Silva, G. G.; De Souza, D. A.; Machado, J. C.; Hourston, D. *J. Appl. Polym. Sci.* **2000**, *76*, 1197.
23. Nazareth, S.; Mavinkurve, S. *Int. Biodeterior.* **1987**, *23*, 343.
24. Rajan, A.; Senan, R. C.; Pavithran, C.; Abraham, T. E. *Bio-process. Biosyst. Eng.* **2005**, *28*, 165.
25. Satyanarayana, K. G.; Sukumaran, K.; Mukherjee, P. S.; Pillai, S. G. K. *Metall.* **1986**, *19*, 389.
26. Tomczak, F.; Satyanarayana, K. G.; Sydenstricker, T. H. D. *Compos. A* **2007**, *38*, 2227.