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Effect of ecofriendly coating and treatment on mechanical, thermal and morphological properties of sisal fibre

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The objective of the present work is to overcome the limitations of sisal fibre and to improve its performance by ecofriendly treatment (sodium bicarbonate) and ecofriendly coating [poly lactic acid (PLA)]. Mechanical, thermal, and morphological analyses of untreated, treated and treated + coated sisal fibres have been carried out and compared. Sisal fibres are treated with varying concentrations of sodium bicarbonate (5, 10, 15 and 20 % w/v) at varying soaking time periods (24, 48, 72, 96 and 120 h). PLA coating on sisal fibres is performed at a varying PLA concentrations (2, 4, 6 and 8% w/v). Scanning electron microscope and X-ray diffraction have been used for the examination of morphological and crystalline analysis respectively of untreated, treated and treated + coated sisal fibres. It is observed that the combined effect of treatment and coating leads to improvement in crystallinity, water absorption resistance, and mechanical and thermal properties of sisal fibres.

Keywords: Ecofriendly treatment, Mechanical properties, Polymer coating, Sisal fibre, Thermal properties, X-ray diffraction

1 Introduction

Natural fibres as substitute reinforcement have taken the attention of researchers over the last few years. Nowadays, preference of the use of natural fibre over man-made fibres in polymeric composites is given by the researchers due to their attractive properties like recoverability, biodegradability, ecofriendliness, vast availability, low cost, high specific properties and so forth[1-4]. Applications of their composites for medium strength are increasing and opening up new opportunities for various industrial applications such as packaging, interior parts of railway and automobile, furniture, marine, electrical and electronic parts, etc[5-8]. Despite many advantages, some drawbacks like more hydrophilicity with high moisture absorption, less durability and poor thermal stability are also found in natural fibres[9-12].

Surface modification methods such as physical treatments[13] (plasma, ultrasound, ultraviolet and so on) and chemical treatments[14] (silane, alkali, acetylation, benzoylation, sodium chloride and so on) were performed to improve the compatibility of natural fibres with polymer matrix and water resistance capacity. Among natural fibres, sisal has been most widely used fibre in the various applications, like mat, bags, ropes, paper, the textile, construction and automobile's interior parts on account of its acceptable properties[15]. A significant amount of research works has been reported on the material characterization of chemically treated sisal fibre and their composites[16-20].

In spite of chemical treatments, researchers are trying to enhance the performance of sisal fibres and their composites by an ecofriendly and economical treatment. Sodium bicarbonate, an ecofriendly chemical, is popular by name of baking soda and does not cause any harm to the atmosphere. It is being used in cooking, cleaning, and toothpaste as an ingredient[21]. It has also been used for medical applications; to reduce sour stomach, heartburn, and acid indigestion by neutralizing excess stomach acid. Chaitanya and Singh[22] studied the effect of sodium bicarbonate treatment on sisal fibre and its green composite. They performed the treatment at a constant concentration of 10% with the varying time period of 24, 72, 120 and 168 h. It was found that the treatment time of 72 h revealed the maximum tensile and flexural strength due to improved fibre strength and interfacial bonding afterwards, properties followed a declining trend because of excess removal of hemicellulose from the fibre's surface. A similar type of work was presented by Fiore *et al.*[23], wherein the effect of sodium bicarbonate treatment on

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properties of sisal fibre reinforced epoxy composite was studied. It was reported that 120 h is the optimum time at which properties were found good.

In present work, a cost-effective and ecofriendly treatment has been applied on sisal fibre and thereafter treated fibres are coated with PLA to enhance their further properties. Mechanical characterization of sisal fibres is performed through a single fibre tensile test. Water absorption behaviour and thermal stability of treated and coated sisal fibres are also studied.

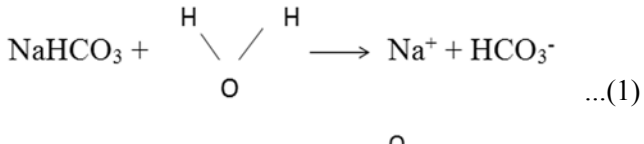
2 Materials and Methods

2.1 Materials

Sisal fibres were procured from Women's Development Organization, Dehradun, India. PLA biopolymer in pellet form, having a melting point of 145°C -160°C, was procured from Nature works, Chennai, India. Sodium bicarbonate in powder form was obtained from Uma Scientific Traders Shahganj, Leader Road, Prayagraj, India. The physical properties, mechanical properties and chemical composition of sisal fibre have already been reported[5].

2.2 Sodium Bicarbonate Treatment

Firstly, a solution of $NaHCO_3$ was prepared with varying concentration from 5% to 20% w/v and then sisal fibres were soaked into a solution from 24 h to 120 h at a constant interval of 24 h. The fibre soaked solution was stirred at regular intervals of 1 h during the treatment period. The fibres were removed from the solution at intervals of 24, 48, 72, 96 and 120 h, and subsequently washed under running distilled water to eliminate excess sodium ions. Treated sisal fibres were then dried at a temperature of 100°C for 8 h in a hot air oven. The reaction of sisal fibres with $NaHCO_3$ can be explained using the following equations:



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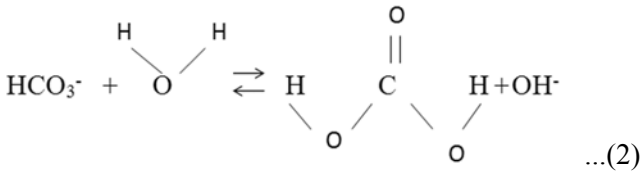

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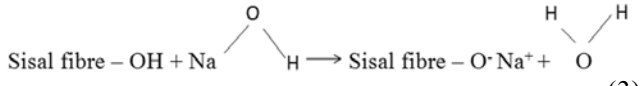

... (3)

Due to alkaline nature of aqueous solution of sodium bicarbonate, chemical reaction occurred during treatment leads to formation of hydroxide ion and carbonic acid as shown in Eqs (1) and (2). Na^+ and OH^-, obtained from Eqs (1) and (2), react with sisal fibre which is identical as alkali treatment, as shown in Eq. (3).

2.3 PLA Coating

Sodium bicarbonate treated sisal fibres were coated with PLA by wetting out the fibres with 2% w/v of PLA in chloroform solution. PLA was dissolved in chloroform at 60°C maintained by hot air oven and manual stirring at a constant interval.

2.4 Tensile Test

According to ASTM D 3822 standard, the tensile properties of sisal fibre were determined on Universal Testing Machine (model: Tinius Olsen H 10 K-L). Test was performed at a cross-head speed of 1 mm/min and with gauge length of 50 mm. Ten fibres of each untreated, treated and coated fibres were tested and their average values were reported.

2.5 Scanning Electron Microscope (SEM)

SEM is commonly used technique for morphological analysis of natural fibres and their polymer based composites. The effect of sodium bicarbonate treatment and PLA coating on the surface of sisal fibre was observed by ZEISS EVO Series Scanning Electron Microscope Model EVO15 after gold coating on them.

2.6 X-Ray Diffraction (XRD)

X-ray diffraction was performed to determine crystallinity of untreated, treated and coated sisal fibre using CuKα (λ = 1.54) radiation. The diffraction intensity was in the range of 5°–60° of 2θ with scanning speed of 0.02 °/s.

2.7 Thermogravimetric Analysis(TGA)

TGA was carried out using the instrument STA 6000 (Perkin Elmeris) for determination of thermal stability of the fibres. To measure the degradation of fibres with temperature, fibres were heated from 25°C to 600°C at heating rate 10°C/min in nitrogen atmosphere.

2.8 Water Absorption Behaviour

In order to obtain the percentage of water uptake, a pack of sisal fibres (1 g) was taken. The testing was carried out until the percentage of water uptake attain equilibrium at a constant interval of 24 h. Fibres were completely immersed into distilled water at room temperature. After a constant interval, the fibres were removed and wiped with a tissue paper to remove any

existing surface water. Thereafter, fibres were weighed using high accuracy (0.0001g) electronic weighing balance. The following equation was used to measure the percentage of water uptake of fibres:

$$\text{Water uptake (\%)} = \frac{x_2 - x_1}{x_1} \times 100 \quad \text{... (4)}$$

where x_1 is the weight before soaking into water (g); and x_2 , the weight after soaking into water (g).

3 Results and Discussion

3.1 Tensile Test

Effect of variation in treatment time and concentration of $NaHCO_3$ on the tensile strength of sisal fibres is presented in Fig. 1. It is observed that the tensile strength of sisal fibres increases with increase in concentrations of $NaHCO_3$ until 10% w/v and thereafter it is found to decrease. This improvement can also be credited to the fractional removal of amorphous hemicelluloses. The partial removal of hemicellulose leads to close packing of cellulose fibrils due to the formation of strong hydrogen bonds between them. This rearrangement of fibres increases homogeneity, resulting in the improved load bearing capacity among cellulose fibrils. After 10% w/v concentration, the tensile strength is found to decrease due to damage of fibre structure.

It can also be observed that there is a considerable effect of treatment time on the tensile strength of sisal fibres. Increasing and decreasing trends are seen in tensile strength of sisal fibres due to variation in treatment time. The tensile strength of sisal fibre is found to increase up to 96 h treatment time and then decrease due to excessive removal of hemicellulose content from the fibre's surface. A similar observation of a reduction in strength of the fibres due to the immoderate concentration of sodium bicarbonate has been reported by Fiore *et al.*[23]. Finally, it can be concluded that 10% w/v concentration and 96 h treatment time are optimal concentration and treatment time at which sisal fibre reveals the maximum tensile strength.

Effect of different concentrations of PLA coating on the tensile properties of treated sisal fibre with optimal concentration and treatment time is presented in Fig. 2. There can be observed a significant effect of PLA coating on the tensile strength of sisal fibre. The maximum value of tensile strength of treated sisal fibre is observed at 2 % w/v concentration of PLA coating which provides the higher strength to fibres. On the other hand, a decrease in tensile properties above 2 % w/v coating is observed due to delignification and degradation of cellulose chains during treatment.

From above results and discussion, it can be concluded that 10 % w/v, 96 h and 2 % w/v concentration of PLA coating could be considered as optimal, regarding the concentration, treatment time and coating concentration respectively. Therefore, the untreated sisal fibre (SF), $NaHCO_3$ treated fibre with 10 % w/v concentration and 96 h treatment time [SF (T1)] and with 2 % w/v of PLA coating on [SF (T1)] as [SF (T2)] are taken as optimal conditions for further analysis.

3.2 SEM Study

Figure 3 shows the SEM images of untreated, treated and coated sisal fibres. Figure 3(a) shows the

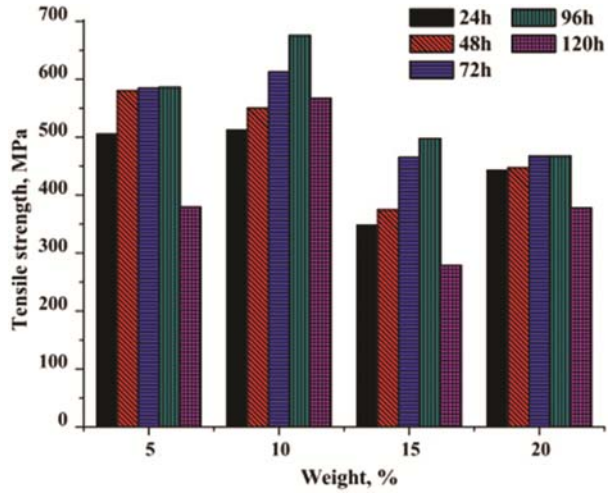

Fig. 1 — Effect of variation in treatment time and concentration of $NaHCO_3$ on tensile properties of sisal fibres.

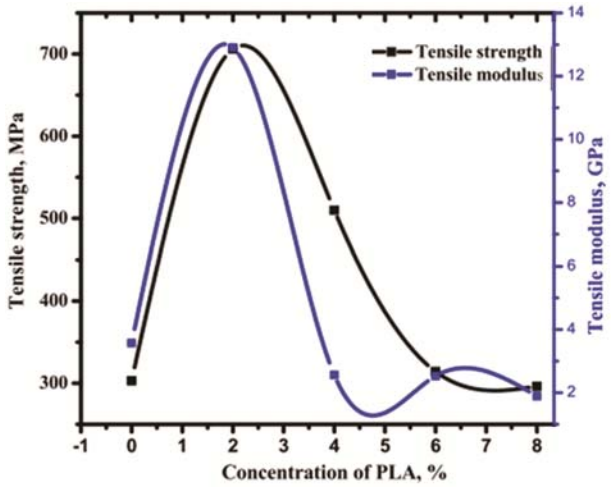

Fig. 2 — Effect of concentrations of PLA coating on tensile properties of treated sisal fibres

surface microstructure of untreated sisal fibre presenting fibre cells and impurities over the surface of the fibre. Figure 3(b) shows the SEM image of sodium bicarbonate treated sisal fibre. After sodium bicarbonate treatment, fibre fibrillation process starts due to partial removal of non-cellulosic content as hemicellulose and waxes from the fibre's surface lead to the development of smoother and cleaner surface than untreated fibre. The smooth surface can improve the interfacial bonding and compatibility with polymers. Figure 3(c) shows the SEM image of coated sisal fibre SF (T_2). In this case, it can be observed that the surface roughness is considerably increased in comparison to SF (T1), which can provide better interfacial bonding, thereby causing a uniform transfer of stress.

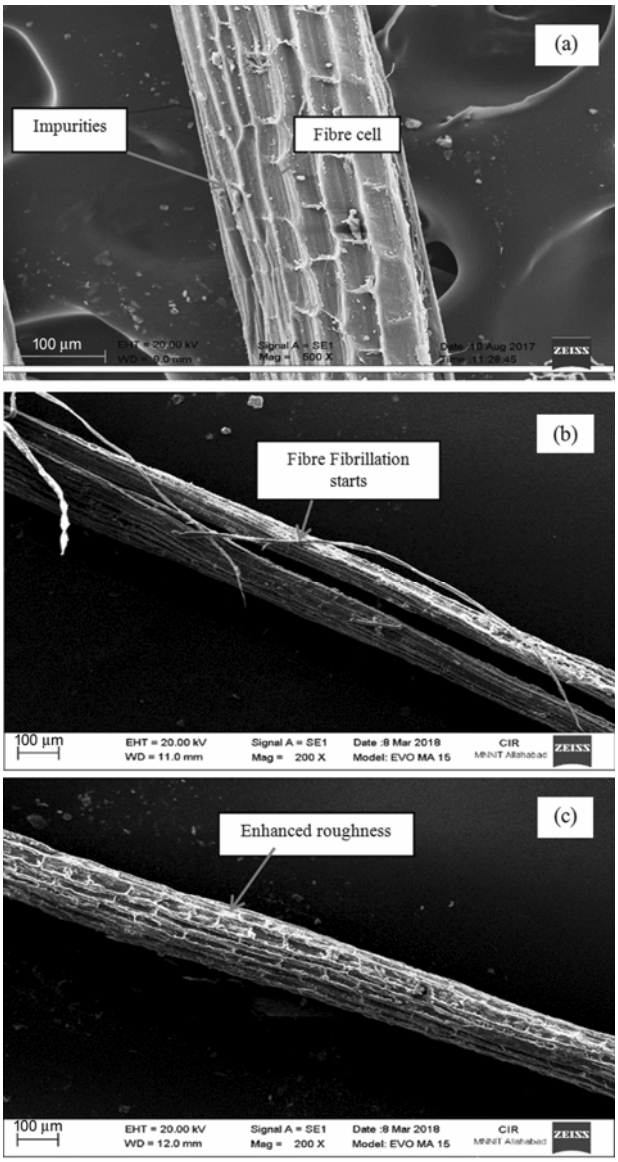

Fig. 3 — SEM micrographs of sisal fibres (a) untreated, (b) treated, and (c) treated and coated

3.3 X-Ray Diffraction

Figures 4 (a)–(c) show the XRD patterns of sisal fibres SF, SF (T1) and SF (T2) respectively. It can be inferred from the diffractograms that the major crystalline peak of each profile occurs at around $2\theta = 20\text{-}22^{o}$ with different crystallinity. It can also be noted that coated sisal fibre SF (T2) has the highest peak of intensity (5394 cps) at 22.28^{o} followed by SF (T1) and SF. Hence, it can be concluded that the degree of crystallinity index of the SF (T2) is greater than sisal fibres SF and SF(T1) because of PLA coating. Crystallinity index of SF (T2) is increased by 12 % as compared to SF(T1) due to elimination of amorphous phase of sisal fibre. The increase in the crystallinity index for SF (T2) leads to an

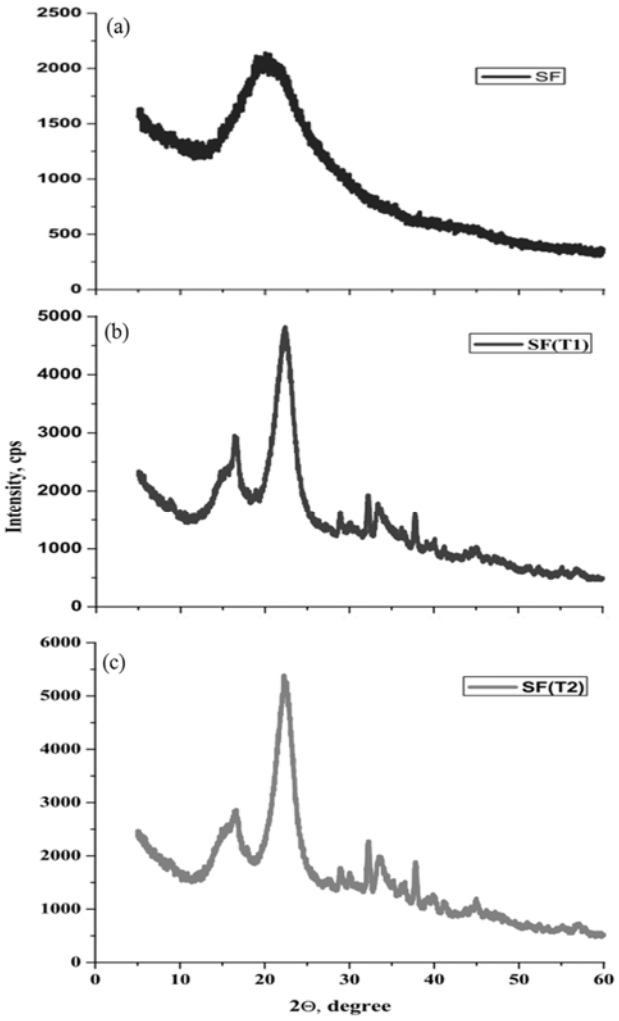

Fig. 4 — X-ray diffraction pattern of sisal fibres (a) untreated, (b) treated, and (c) treated and coated

improvement in tensile strength[24]. Sisal fibre SF (T2) can provide better interfacial bonding due to higher content of crystalline cellulose.

3.4 Thermogravimetric Analysis

Thermogravimetric curves show the change in percentage of weight of the fibres with increasing temperature (Fig. 5). Percentage of weight loss and their corresponding temperature is provided in Table 1. In case of all the fibres, initial weight loss (5%) occurs in the temperature range from 80°C -180°C which can be due to evaporation of moisture from the fibres[25]. Coated sisal fibre SF (T_2) possesses a higher temperature of initial weight loss as compared to SF and SF (T1). This may be due to the fact that PLA coating leads to higher crystallinity (as confirmed from X-ray diffraction analysis) and minimum moisture absorption. It can also be seen that treated fibre SF (T1) shows higher temperature of initial weight loss than untreated fibre because of sodium bicarbonate treatment, which improves the water resistance capacity.

The TGA curves of these fibres show three steps of degradation, namely (i) initial weight loss of 5% in the temperature range of 80°C - 180°C, (ii) major weight loss as 75% in temperature range of 416°C - 443°C, and (iii) final weight loss of above 90% in temperature range 550°C -600 °C. Major weight loss occurs due to the degradation of cellulose, hemicellulose and lignin[26]. It is also observed that coated sisal fibre has a higher temperature related to major weight loss followed by SF (T1) and SF. This may be due to PLA coating as it acts as a protective barrier which saves the fibres from the degradation. After major degradation, a higher temperature is required for subsequent degradation. At final stage, it can be observed that coated fibre SF (T_2) has the lowest weight loss at higher temperature than other sisal fibres SF and SF (T1), which shows its highest thermal stability.

3.5 Water Absorption Behaviour

The percentage of water absorption of sisal fibres SF, SF (T1), SF (T2) is plotted against the square root of soaking time (Fig. 6). The slope of this curve shows the capability of water uptake by the fibres. In initial stage, untreated sisal fibre has the highest percentage of water uptake. The increase in water absorption is due to the hydrophilic nature of sisal fibre owing to the presence of cellulose. On the other hand, treated fibre SF (T1) offers lower water uptake due to partial removal of hemicelluloses and lignin, which reduces the water uptake. The coated fibre SF(T_2) has the lowest water uptake due to PLA coating, which protects the fibres from water being observed. On increasing the soaking time, finally fibres reach to saturation stage. At saturation stage, highest percentage of water uptake (34 %) is shown by SF followed by SF (T1) and SF (T2). Coated sisal fibre SF (T2) has 70% and 62% lower water uptake than those of SF and

Table 1–Percentage weight loss of pure and modified sisal fibres at different temperatures

[Initial weight loss 5 % and major weight loss 75 %]

Fibre	Initial wt. loss temperature, ^{o}C	Major wt. loss temperature, ^{o}C	Final wt. loss temperature, ^{o}C
SF	78.66	416.74	550.73
SF(T1)	111.85	442.47	650.27
SF(T2)	183.84	443.19	650.74

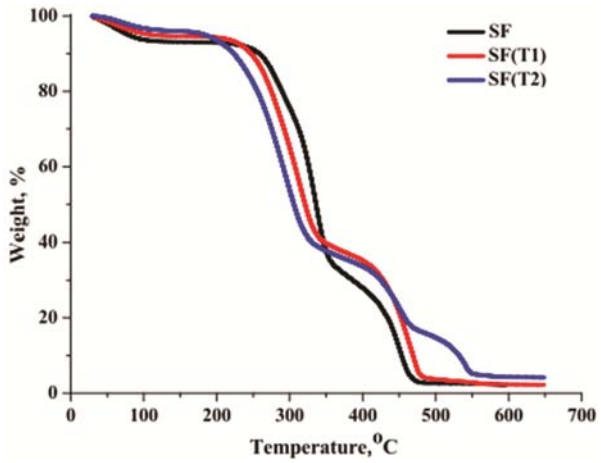

Fig. 5 — Variation in weight with temperature of pure and modified sisal fibres

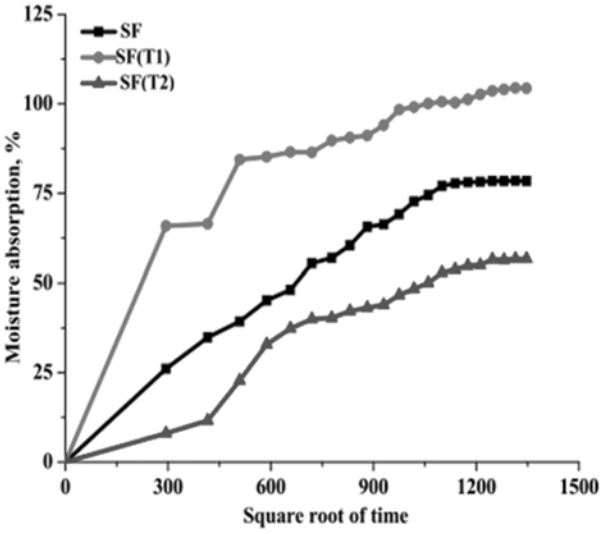

Fig. 6 — Variation in water uptake as a function of square root of time of pure and modified sisal fibres

SF (T1) respectively. At saturation stage, the lowest water uptake of SF (T2) can be due to higher crystallinity produced by PLA coating.

4 Conclusion

The present research work deals with the economic and ecofriendly sodium bicarbonate treatment and coating on sisal fibres. The outcome of the result suggests that a strong sisal fibre can be developed by biopolymer coating with improved properties. Following inferences are drawn:

4.1 Fibres surface modifications by sodium bicarbonate treatment and PLA coating are found successful in enhancement of the properties.

4.2 It is observed that the treated fibres show notable improvement in tensile properties if compared to untreated one. A further increment in tensile properties is also observed after coating.

4.3 Thermal stability, water resistance, crystallinity of sisal fibres are also improved by sodium bicarbonate treatment and PLA coating.

4.4 Ecofriendly treatment then coating technique can be considered as an effective method to enhance the performance of the fibres, and hence their composite may be used for high strength and outdoor applications.

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