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# COMPARATIVE ASSESSMENT OF PHYSICO-CHEMICAL AND MECHANICAL PROPERTIES OF ALKALI AND FUNGAL TREATED *MUSA PARADISIACA* L. FIBER

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**ABSTRACT**

The current study deals with the investigation on the properties of lignocellulose fibre from the stem of *Musa paradisiaca L.* The *Musa paradisiaca L.* fibres were extracted from its pseudo stem by mechanical process. The extracted fibres were treated with five different concentrations of NaOH and two different fungal cultures (*Aspergillus niger and Aspergillus cereus)*. The treated fibres were analysed for physical, chemical and mechanical properties as per standard procedure with raw banana fibre. From the chemical investigation, lignin content of the Raw Banana fibre (15.98%) was decreased in the fibre treated with 25% of NaOH (13.56%), *Aspergillus niger* (11.40%) and *Aspergillus cereus* (12.54%). Mechanical properties of treated fibre were shows progressive results in comparison with raw banana fibre. In conclusion, the investigation proved that banana fibre treatment with the NaOH and *Aspergillus spp* has a significant effect on the physiochemical properties.

**Keywords**: lignocellulose fibre, Mechanical process, NaOH, *Aspergillus niger and Aspergillus cereus,* physiochemical properties

## INTRODUCTION

Natural fibers are flattering a substitute and attractive for synthetic fiber, due to their renewability, recyclability gradeability, cost effective and eco-friendly. Banana is currently cultivated in 129 counties around the world. It is the fourth most important global stable food crop. Globally the production of banana fruit is 26.2 million among this, India contributing 14.7% production of banana fruit from 7.1lakhs hectares of banana crop cultivation area. *Musa paradisiaca* L. commonly known as banana belongs to the Musaceae family. The banana plant is a tree-like perennial herb. The aerial parts of the parent plant die down to the ground after the cultivation (Elanthikkal *et al.,* 2010). Billion tons of stems and leaves are thrown away annually. Such wastes are available sources for fibers which lead to reduction of synthetic fibers.

Synthetic fibres are widely used as reinforcing materials in polymeric composites because of their high specific strength, light weight, and durability. However, they do have some downside, which include high cost, non-biodegradable nature, and high energy consumption, leading to problems such as environmental pollution, skin irritation, and abrasion processing equipment ( Manimaran, *et al.,* 2018). The banana fibers are good moisture absorbent, highly breathable, quikly dry with high tensile strength. The matrix in which the cells are embedded in the fiber had a role in deciding the tensile strength of the fiber.

Banana pseudo stem fibre is a lignocellulosic material mainly consists of polysaccharides with cellulose microfibrils (50-60%) embedded with hemicelluloses (25-30%), lignin (12-18%), pectin (3-5%), water soluble material (2-3%), wax and fat (3-5%) and ash (1-1.5%) (Mohiuddin *et al.*, 2014). Banana causes a considerable amount of cellulosic-based waste, which is lignocellulosic in nature (Manimaran *et al.,* 2018a). This agricultural waste holds on the soil causing more environmental complications in banana farming regions. High holocellulose content and low lignin are indicated by chemical analysis of banana pseudo-stem fibre compared with some other non-wood fibre resource. Fibre having 20-30% of lignin content, it is difficult to spin in textile processes. Therefore, lignin needs to be removed by the process of degumming (Vishnu Vardhini, *et al*., 2015).

Banana fibre had a very limited application and was primarily used for making items like ropes, mats, and some other composite materials. With the increasing environmental awareness and growing importance of eco-friendly fabrics, banana fibre has also been predictable for all its good qualities and now its application is increasing in other fields too such as apparel garments and home furnishings (Tholkappiyan-2016).

The softened banana fibres have application as reinforcement in polymer composites, packaging, automobiles, interiors and storage devices (Mageshwaran Vellaichamy 2017). Enzymes modify the fibre parameters with desired properties. They are bio-active compounds or catalyst which acts on regulation of various biochemical reactions in living tissues and cells (Silva, *et al.,* 2002). Among the enzyme's pectinases have great biotechnological potential with involvement in many industrial processes including processing of fibres (Indrani Sarma, *et al.,* 2012). Pectinase enzymes are sourced from different genera of bacteria, fungi, yeast and some actinomycete are reported among which fungi the maximum producer of secondary metabolites and extracellular enzymes are including pectinases. Production of Pectinases involving fungal species has been reported earlier actinomycetes *Aspergillus flavus, Aspergillus sp, Penicilluim italicum, Penicillium viridicatum, Penicillium roqueforti, Penicillium expansum* and Pectolytic moulds. Fungal treatment of fibres is an eco-friendly method for improving fineness property of banana fibre.

Many researchers have been carried out so far, for the characterization of banana fibre for their utilization in various fibre-based industries like textile, automotive and pulp and paper industries etc. Even though they are used for textile and pulp industries, the lignin content of the fibre affects the quality of the end product. So, various chemical treatments have been used by the industries for the removal of lignin content. But the usage of these chemicals in large quantity has resulted in hazardous effluent disposal problem and is also high cost and unsafe to workers. Therefore, safer and environmentally sound technologies are necessary for the removal of lignin content of the fibre that enhances the quality of the fibre. For this bio-softening process was carried out, where the microbes are used to remove the lignin content of the fibre. In his background, the present study was focused to improve and compare the effectiveness of softening of banana fibre by chemical and biological treatment methods.

## MATERIALS AND METHODS

**Collection and extraction of banana fiber**

The banana pseudo stem was collected from the representative village in Coimbatore district of Tamil Nadu, India. The fibre was extracted from the middle layers of banana pseudo-stem with the help of a manual extracted or a semi- automatic machine called Raspador, having a drum speed of 700 – 800 rpm. The Raspador machine has a scrapping plate in front of a drum (14” diameter), fitted to a carbon steel angle blade (12”). The leaf sheath was passed through the machine one by one. The obtained fibres were thoroughly washed with water to remove dirt and pulp left to it that are stuck together and allowed to dry. The fibres were then combed through an army of large nails hammered on block of wood. The fibres were gathered and straightened out.

The collected raw banana fibers were very coarse (140 Denier) and have more lignin content in nature. Subsequently the removal of lignin content from the fiber surface has done by retting process for 2-3 weeks. After the retting treatment the banana fibers have been subjected into chemical and biological treatments to reduce the fineness (rigidity).

**Chemical treatment on raw banana fiber**

Alkalization treatment was carried out through the immersion of the raw banana fibres in a sodium hydroxide (NaOH) solution of different percentages like 5%, 10%, 15%, 20%, 25% and 50% and it was kept overnight to reduce the fiber rigidity level. After the treatment fibres were washed with distilled water until a neutral pH was reached and dried in an oven at 60°C for 12 hr after drying, this dried banana fiber treated with the softener, prepared by the combination of castor oil (4-6%), Aloe Vera (4-6%), Cotton seed oil (4-6%) and emulsifier (2.5%), for 1hr.

**Softening of banana fiber by biological treatment**

**Microorganisms**

The 2g of sterilized banana fibers cut into 30cm length were used for biological treatment. The two cellulolytic pure fungal strain cultures, *Aspergillus niger* and *Aspergillus terreus* were obtained from microbiology laboratory of PSG hospital, Coimbatore. The cultures were grown in Potato Dextrose broth at 30±1 for a period of 48hrs under static condition. The cultures were preserved in Potato Dextrose agar slant at $4^0$C. The anaerobic consortium maintained in microbiology laboratory of PSG hospital Coimbatore was used for anaerobic treatment.

**Biological treatment**

In biological treatment, cultures of *Aspergillus niger* and *Aspergillus terreus,* were treated at for softening of banana fibres shown in figure 1.

Figure 1. Softening of Banana fibre by biological treatment (Fungal cultures)

**Culture treatment**

Five percent inoculums of 48hr old culture of *A. niger* and *A. terreus,* were transferred separately into a flask containing sterile 100ml of mineral medium (pH 6.0) containing 0.5% glucose and incubate in rotary shaker (150rpm) at 30° C for 24 h. One gram of mechanically extracted banana fibre was aseptically placed into the flask with the help of sterile forceps. The flaks were further incubated for 72 h (T4) at room temperature under stationary condition. After treatment, the fibres were air dried, combed and analyzed for physicochemical and mechanical characterization.

**Physical properties of treated and untreated banana fibre**

The physical properties namely length, diameter, density, linear density, moisture absorption was quantified as follows. The surface morphology and diameter were measured using a polarised microscope (Lieca make) with a magnification of ×100 and interfaced with a PC. The diameter of 10 randomly sampled fibres was measured microscopically at five different positions and the average was calculated.

**Density** (TAPPI, 1980): The density of the fibre is determined using standard methods. The fibre sample was conditioned for 24h at 65% relative humidity and 25°C before carrying the density test. 2g of fibre sample was immersed in toluene in a calibrated glass tube 15 (10ml measuring cylindrical and the value of toluene displaced was equal to the fibre in the solution.

Density = mass / volume

**Linear density (ASTM D 1577)**

The linear density is measured using standard ASTM standard. The density of fiber determined was at standard

**Table: 1** Weight Losses of Banana Fibres treated with NaOH

| S.No | Concentration of NaOH | Weight before alkalization(g) | Weight after alkalization(g) | Weight loss (%) | Conditions (%) |
|---|---|---|---|---|---|
| 1 | Raw banana fiber (control) | 25 | 25 | 0.050 | Temp=35°c Time=24 hrs |
| 2 | 5% | 25 | 23.50 | 1.50 | |
| 3 | 10% | 25 | 21.00 | 4.00 | |
| 4 | 15% | 25 | 18.75 | 6.25 | |
| 5 | 20% | 25 | 17.50 | 7.50 | |
| 6 | 25% | 25 | 16.50 | 8.50 | |

**Table-2** Physical properties of fibre

| S.No | Sample | Hygroscopicity (%) | Density (g/cm³) | Linear density(denier) |
|---|---|---|---|---|
| 1 | Raw banana fibre | 10.15 | 0.66 | 12.76 |
| 2 | Softened with *Aspergillus niger* | 12.15 | 0.57 | 11.62 |
| 3 | Softened with *Aspergillus terreus* | 11.95 | 0.52 | 10.87 |
| 4 | Softened with 25% of NaOH | 9.50 | 0.55 | 12.10 |

**Table 3.** Colour of raw and treated banana fibres

| S.No | Sample | Colour |
|---|---|---|
| 1 | Raw banana fibre | Reddish yellow |
| 2 | Softened with *Aspergillus niger* | Light yellow |
| 3 | Softened with *Aspergillus terreus* | Golden yellow |
| 4 | Softened with 25% of NaOH | Reddish orange |

**Table: 4** Chemical properties of banana fibre

| S.No | Sample | Cellulose (%) | Hemi-cellulose (%) | Lignin (%) |
|---|---|---|---|---|
| 1 | Raw Banana fibre | 61.15 | 23.45 | 15.98 |
| 2 | Softened with *Aspergillus niger* | 59.14 | 26.13 | 11.40 |
| 3 | Softened with *Aspergillus terreus* | 59.02 | 25.86 | 12.54 |
| 4 | Softened with 25% NaOH | 60.50 | 24.52 | 13.56 |

**Table:5** Effect of mechanical properties in raw and treated banana fibres

| S.No | Treatments | Peak load (g) | Mean Breaking Elongation Dl at Fmax(%) | Tenacity (g/den) | Youngs modulus Emod(MPa) | Mean breaking strengthFmax (MPa) |
|---|---|---|---|---|---|---|
| 1 | Raw Banana Fibre | 528 | 1.6 | 1.07 | 132.0 | 2.84 |
| 2 | *Aspergillus niger* | 445 | 2.9 | 2.77 | 114.0 | 4.89 |
| 3 | *Aspergillus terreus* | 460 | 2.5 | 2.33 | 106.0 | 3.58 |
| 4 | Softened with 25% NaOH | 474 | 7.2 | 1.71 | 73.5 | 3.01 |

atmospheric conditions and measured in deniers. The fibre samples were cut using standard length (L) template (5-10 cm) and weighed (w) in grams. The denier (D) of the fibre sample is calculated using the formula,

$D = (9000 \times W) / (L \times N)$ Where N is the number of fibres in the sample.

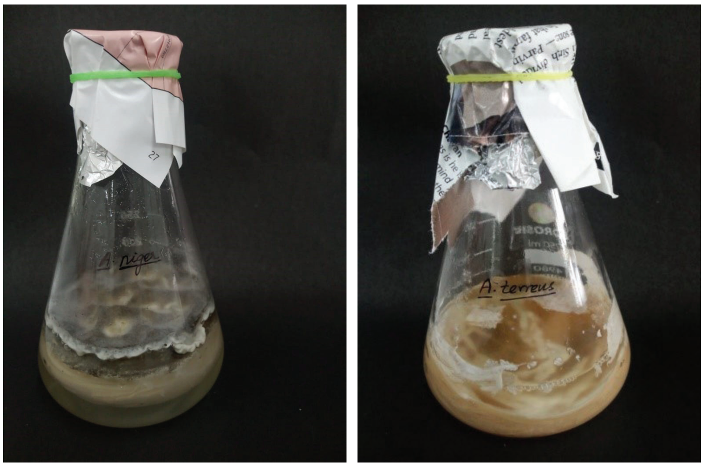
**Moisture regains (%) - (Hygroscopicity) (ASTM – 1997, D 2654 – 76)**

It is defined as the amount of water present in a specimen expressed as a percentage of its dry mass. The fibre sample was conditioned for 24h at 27±2°C and the weight (g) was taken (L). The conditioned fibre sample then dried at 105°C in oven for 4h and the weight was taken (w). Percentage of moisture regain was calculated according to the formula,

Moisture regains (%) = $L – W \times 100/L$

Where, L = Initial weight (g), W= Final weight (g).

**Tensile properties-** The tensile strength of untreated and treated banana fibres was measured according to the ASTMD 3822 – 01 standard test method for tensile properties of single textile fibres. The test was performed with 1kN load cell at a crosshead speed of 5 mm/min, and approximately 20 fibres were tested for each sample, at 10mm gauge length in Zwick tensile tester.




*Aspergillus niger* *Aspergillus terreus*

**Figure 1.** Softening of Banana fibre by biological treatment (Fungal cultures)

**Colour-** The colour of treated and untreated banana fibres was determined by visual method.

**Determination of Chemical Composition in treated and untreated banana fibre**

**Determination of Lignin Content -** Two grams of extracted fibre sample were placed in a flask and 15 mL of 72% sulphuric acid was added. The mixture was stirred frequently for two and half hours at 25°C followed by addition of 200 mL of distilled water to the mixture. Then the mixture was boiled for next two hours and cooled. After 24 h, the lignin was transferred to the crucible and washed with hot water repeatedly until it becomes acid free. The collected lignin was dried at 105°C, cooled down in a desiccator and weighed. The drying and weighing were repeated till constant weight was achieved, and the lignin decomposition was calculated.

**Determination of Cellulose and Hemicellulose Contents** - Two grams of holocellulose were placed in a beaker and 10 mL of sodium hydroxide solution (17.5%) was added. The fibres were stirred by glass rod so that they could be soaked with sodium hydroxide solution vigorously. Then sodium hydroxide solution was added to the mixture periodically (once in every 5 min) in 30 min and the mixture temperature was kept at 20°C. About 33 mL of distilled water was added in the beaker and kept for an hour. The holocellulose residue was filtered, transferred to the crucible and washed with 100 mL of sodium hydroxide (8.3%),200 mL of distilled water, 15 mL of acetic acid (10%) and again water successively. The crucible with _-celluloses was dried and weighed. The content of hemicelluloses of banana fibre was calculated from the following equation:

Hemicelluloses = Holocellulose - α- Cellulose

**Evaluation of mechanical properties of treated and untreated banana fibre**

The individual fibre was tested under Universal Testing Machine having 10 kg load scale capacity, guage length 50mM and cross head speed 10 mm/min. Ten individual fibres were tested and the weight of ten fibres were noted for each treatment. The peak load and peak elongation of each fibre was noted. The mechanical properties such as breaking tenacity (g/tex) and young's modulus (kgf/mm) were also recorded by using zwick using standard ASTM (ASTMD 3822-01) methods. The raw untreated fibre was considered as control.

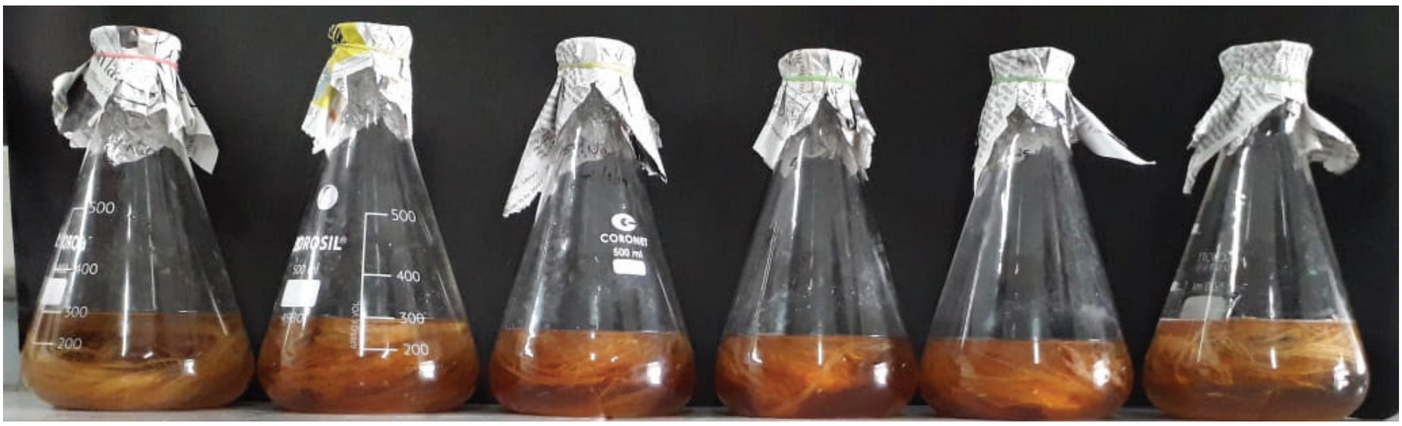
## RESULTS AND DISCUSSIONS

**Softening process by alkaline treatment using Sodium hydroxide (NaOH)**

The banana fibres treated with the five different concentrations of NaOH shown in figure 2, where tested along with the untreated fibre. The percentage of weight loss of the fibres represents the removal of the non-cellulosic components in the fibres. The highest percentage of weight loss (8.5%) was observed in banana fibre treated with 25% of NaOH (Table-1). which indicates the best softening nature of banana fibre compared with other treatments. The weight of the banana fibre was decreased with increases concentration of the alkali (Tholkappiyan, 2016).

**Subjective analysis**

To select the best soft nature of banana fibre by touch and feel method both treated and untreated banana fibre was given to 25 members (Ankita Shroff *et al*., 2015). Among twenty-five members 15 members selected the banana fibre treated with 25% of NaOH. In biological treatment ten members selected the banana fibre treated *Aspergillus*



control 5% 10% 15% 20% 25%

**Figure2.** Softening of Banana fibre by using Sodium hydroxide (NaOH)

*niger*. Based on this result the best softened fibre was selected for further screening.

**Physical properties of Banana fibre**

The density of fibre is an important parameter for its application in the preparation of light weight composites (Table - 2). The increase in density is due to the removal of the low denser region such as lignin and hemicelluloses and higher denser region of cellulose content (Vishnu Vardhini *et al*., 2016). The density of the raw banana fibre was 0.66g/cm$^3$ and softened banana fibre was 0.55g/cm$^3$ and 0.57 g/cm$^3$. These values are lower than the value of E-glass materials i.e., 2.5 g/cm$^3$ reported by (Saechtlinng, 1987). Therefore, the banana fibre with higher density will be excellent material for light weight composites whereas the lower density fiber is most suitable for textile application.

The Linear density shows the fineness of the fibre. The linear density of softened and raw banana fibre was from 12 to12.76 denier. It was closer to the maximum value of cotton 3 to 8 denier, linen 1.7 to 17.8 denier and hemp 3.2 to 20 denier (Batra, 1998). The density and linear density of softened banana fibre decreased compared to the raw banana fibre.

Cellulose molecules with hydrogen bond formation capacity make them to absorb water readily. The percentage of moisture content of treated and raw fibre ranged from 9.50% to 12.15% it was based on the chemical contents of the banana pseudo stem fibres. The moisture regains of the treated banana fibre in the present study reveals the closer value of the hemp and kenaf 8% to12% and 9% to 10.05% respectively. The fiber treated with *A.niger* regains the highest percentage of moisture content (12.15%) compare with other two different treatments. Increase in the moisture regain of the fibers, are more suitable for the textile applications due to is more hydrophyllic nature.

**Colour**

The colours of treated and untreated banana fibres are shown in Table 3. It shows that there is significant change in colour. The treated fibre indicates that the fibres have become darker which was evident from the visual observation. The fibres are towards redder side as the values of ‘a’ are positive and the redness has also increased. The increase in the ‘b’ value shows that the fibre is in the yellower side and the yellowness has increased. Also, there is an increase in the hue value.

**Chemical properties of Banana fibre**

Cellulose influences the major characteristic for deciding the quality of the fibre. Cellulose is the main structural component that provides strength and stability to the plant cell walls. Cellulose enzyme is produced by cellulolytic microorganism and it is able to degrade the cellulose materials and assumed as greater importance in modifying low grade roughage (Garg and Neelakandan, 1982). Filamentous fungi are considered to be the most important group of microorganisms for the production of plant cell wall degrading enzymes in solid state fermentations. In this study, the two fungal strains *A, niger* and *A. terreus* were screened for plant fiber cell wall degrading enzymes (Shazia Rehman., *et al.,* 2014). The amount of cellulose in fibre responsible for the utility of the fibre for various applications. The cellulose content of the raw banana fibre and softened fibre ranged from 59.14% to 61.15%. The value was closer to the jute 64.4% and flax 64.3% but comparatively lower than the cotton 82.7%. Hence it can be a good alternative for jute and flax fibres (Table-4). Several studies on fiber composition and morphology have revealed that cellulose content and microfibril angle tend to control mechanical properties of cellulosic fibers (Biswas *et al.*, 2001).

Hemicellulose in plants usually acts as filler between the cells. The hemicellulose content of softened banana fibre and raw banana fibre ranged from 23.45% to 26.13%, it was higher than jute 12% and cotton 6%.

Higher lignin content makes the fibre tough and stiff. Lignin provides the plant tissue and individual fibres with comprehensive strength. Lignin content of softened banana fibre and raw banana fibre ranged from 11.40% to 15.98%. As lignin content decreased below 0.78% tenacity continue to decrease (Preethi *et al.,* 2013). In the percent study the lowest lignin content was observed in fibre treated with *Aspergillus niger* (11.40%). The percentage of the cellulose and lignin content of banana fibre treated with *A. niger* and *A. terreus* were lower than the 25% of NaOH treated fiber, whereas the percentage of hemicellulose content of fibres treated with *A. niger* and *A. terreus* was higher than the alkaline (25% of NaOH) treated fiber. Thus, the variation in cellulose and lignin content were determinants of fiber quality, could be of value in assigning fiber to specific end uses. Removal of lignin should have increased the moisture regain to a higher level. Removal of hemicelluloses has decrease the moisture regain value cellulose region may have become more accessible to the moisture for absorption (Vishnu Vardhini *et al*., 2016).

**Mechanical properties of banana fibres**

The mechanical properties such as the initial modulus (YM), ultimate tensile strength (UTS) and percentage elongation are evaluated as a function of fibre. Compared to raw fiber, the improved tenacity and tensile strength was observed to be around 2.77(g/den) in *A.niger* treated fiber the improvement in the tensile strength of raw fiber compares to that with 25%NaOH and fungal treated fibers range in between 2.84 and 89Mpa.The increase in tensile strength is associated with the decrease in fiber diameter because of the loss of hemicelluloses in the fibers due to alkali and fungal treatments as shown in Table-3. Furthermore, under alkali treatment with 25%NaOH, and fungal treated fibers, the percentage of cellulose content of the fibres increases to produce high tensile strength of the fiber. Tensile strength or tenacity depends on the fibre diameter, fibre length and test speed. Increase in tenacity will be due to the removal of lignin content, resulting in the compaction of microfibrils, thereby decreasing the tensile strength in softened banana fibre (Jayapriya and

Vigneswaran, 2010).

The Young's modulus of softened banana fibre was decreased when compared to the raw banana fibre. At the same time improvement in the elongation of softened banana fibre indicates that the softening process did not weaken the quality of the banana fibre Table-5. The banana pseudo-stem fiber has good modulus of elasticity, tensile strength and stiffness which makes promising material. This improvement is useful to enhance the spin ability of the fibre and increase the weavability of the yarn.

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## CONFLICT OF INTEREST

The author declares no conflict of interest.

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