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A systematic study on material properties of water retted *Sterculia* and *Bauhinia* fiber

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Abstract

Lignocellulose biomass forms an important component of traditional and next generation composite materials. To obtain desired properties, the biomass needs to be chemo–mechanically processed at different levels. The raw lignocellulose fiber obtained from sterculia villosa (Roxb.) and Bauhinia vahlii is traditionally believed to have high water stability; and therefore used in rural areas of South Asian regions to secure objects submerged under water. In this research, we systematically studied several material properties of raw Sterculia and Bauhinia fiber samples retted for 0, 20, 30 and 55 days (n=8). Water retting resulted in significant decrease in lignin and extractives content ($p<0.05$) and increase in cellulose content. Fiber bundle strength of Sterculia fiber increased with retting time ($R^2 = 0.7$) but Bauhinia fiber did not show significant change ($p>0.05$). Interestingly, water retting resulted in increased thermal stability in both fiber types. These findings suggested that the fiber studied have excellent water stability. The observed trend in mechanical and thermal properties could have resulted from crystallinity change and/or nominal fiber damage as supported by XRD and SEM imaging data; respectively. These findings suggested that Sterculia and Bauhinia fiber biomass could be an important component of biodegradable composite materials which are intended for high wetting and/or humid conditions.

Keywords
Lignocellulose, Water retting, Fiber processing, Cellulose fiber, Mechanical strength.

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1 Introduction

Lignocellulose biomass is one of cheap, environmentally friendly, and most abundant terrestrial biomass. The biomass can be obtained from leaf, seed, fruit, and bast portion of different plant species or other plant derived wastages following different processing methods [1–5]. Bast fibers are the soft woody fiber obtained from phloem tissues of plant stem. The fibers are usually long and strong and form an important components of next generation materials and finding applications in waste water treatment, biomedical applications, agro and automotive industries [6–10]. Cellulose

nano–composite fibers are also being explored in fabrication of fabrics having self-cleaning, antimicrobial, and UV-protection properties [11–13]. In natural state, bast fibers are cemented to the adjacent tissues by different gummy or non–cellulosic components such as wax, pectin, and lignin. To yield fiber of desired properties, the gummy components needs to be removed following different chemo-mechanical methods. Retting is one of the most important degumming methods. Several retting types are in practice such as chemical retting, enzymatic retting, water retting, dew retting, gel retting, ribbon retting, or their combinations [14–17]. Retting, in general, removes non-cellulosic components so that fiber bundles partially separate. To get fiber of optimal strength retting conditions needs to be optimized [1]. In water retting, fiber bundles are submerged in slow or stagnant water in a pond or tank normally for 2–3 weeks [15,18]. Ground or tap water contain anaerobic bacterial and or fungal colonies that produce enzymes capable of hydrolyzing gummy or fiber binding components such lignin and pectin with release of galacturonic acid and sugar as major by-products [1,19,20]. This results in partial removal of gummy components so that fiber bundles partially separate. The retting conditions, such as retting time, microbial type and load, and fiber type determine the fiber quality. Water retting results in putrid water and many pollutants. However, this is one of the cost effective methods and yields fibers having excellent length uniformity and strength [15, 21]. Retting can also be carried out by using enzyme such as pectinase, xylanases in controlled condition. Although enzymatic retting is faster (12–24 hours) and does not produce unnecessary pollutants, it results in fiber of low strength [1,18]. Retting can also be done mechanically using decorticator to break fiber to fiber bonding. Mechanical retting is rapid but results fiber of low quality. Also, high cost limits it application in resource limited settings. As a result, traditional water retting is still being explored as viable option for obtaining fibers of various lengths and excellent strength [22]. The fiber biomass obtained from *Sterculia villosa* (Roxb.) and Bauhinia vahlii plant species is believed to have excellent water stability; and therefore traditionally being used at local levels to secure objects submerged in water or in high humid conditions. It would be interesting to explore material properties of fiber water retted for different time period. In this study, two fiber types were retted in tank water for 0, 20, 30, and 55 days. Retting efficiency, change in content of major chemical components, fiber strength, and thermal stability of the fiber samples were systematically investigated. We also bridge the observed end properties with the morphological and crystallinity change. Finally, a brief discussion on the further implications of the research is provided.

2 Materials and methods

2.1 Materials

The *Sterculia Villosa* (Roxb.) and *Bauhinia Vahlii* plant stems (1 m long and 3 cm wide) were collected from Digam, Gulmi, Nepal (hilly region, 1250 masl). Bast fiber biomass was separated from the wet stem following traditional method. The stems were gently beaten around one end with a wooden log and pulled mechanically with hands. Outer cuticular layer was removed from biomass using a stainless steel knife (Figure 1). The samples were sun dried to remove excessive moisture and moved to lab for further study.

2.2 Methods

2.2.1 Water retting

Water retting was performed following literature reported methods with slight modifications [23, 24]. Bast fiber samples were divided into four groups of 100 g each. Samples were well rinsed with tap water and gently crushed to loosen the bundles. The samples were immersed in separate buckets containing 4 L of tap water (pH 7.2±1, conductivity 9± 1 μS). The samples were left open under natural conditions (temperature 20–30^{0}C and humidity 50–70%) for 20, 30 and 55 days. To minimize the excessive growth of microorganism and fouling, talk water was one third diluted in every 10 days. After each retting time, sample was washed with distilled water and oven dried at 105±2^{0}C. From the initial and final weights, weight loss% or retting efficiency was calculated. For convenience, Sterculia samples retted for 0, 20, 30, and 55 days hereunder named as S0, S20, S30, and S55 and the Bauhinia samples B0, B20, B30, and B55. Until further characterization these samples were stored in air tight plastic bag in dark. A simple schematics of experimental design used in this work is provided in figure 1.

2.2.2 Chemical analysis

Estimation of major chemical components viz. extractives, lignin, hemicellulose, cellulose and ash content in the water retted samples (S0, S20, S30, S55 and B0, B20, B30 and B55) was performed gravimetrically following the previously reported methods [25–28]. Triplicate measurements were made in all the samples for each chemical component. For estimation of lignin, 1.000 g of extractive free dry sample was treated with 72% H_2SO_4 (1:12.5 m/v ratio) for two hours at room temperature. The top solvent was removed with several

washings with distilled water. The residue was dried at 105 ^{0}C till constant weight was obtained. Form the known initial and final weights, lignin content was obtained. To determine the hemicellulose 1.000 g of extractive free dry biomass was boiled in 150 mL of 0.5 mol/L NaOH for 4 hours. The treated fiber content was washed several times with distilled water to neutral pH and oven dried at 105^{0}C until constant weigh was obtained. Hemicellulose content in % was obtained from the initial and final weights of the biomass. Finally, cellulose content was obtained by subtracting the collective content of extractive, lignin and hemicellulose from 100.

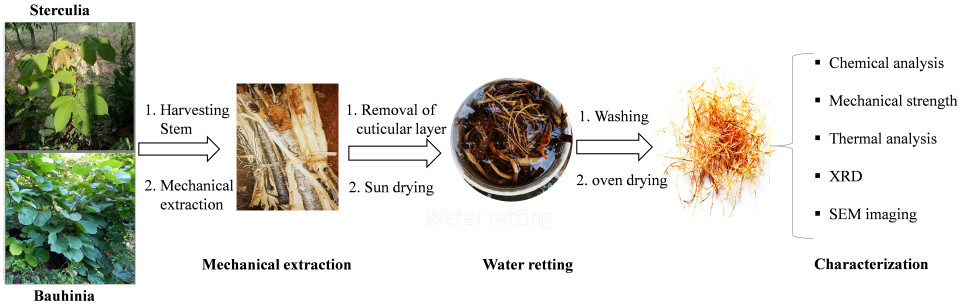

Figure 1: Schematic outline of the experimental design used in this work.

2.2.3 Mechanical strength of fiber

Water retted fiber samples were preconditioned for 24 hours at 23±1 ^{0}C and 65 % relative humidity [29]. A fiber bundle strength tester (TSI instruments) with a maximum load capacity of 7 kg was used to determine fiber strength. Flat fiber bundles were inserted into the Pressley Jaw (gauze length 15 mm) and then loaded into the tester. Load was applied at a continuous rate of 1 kg/sec until the fiber bundle breaks. The load at fiber break was recorded. The broken fiber pieces was removed from the Pressley Jaw and weighed in an analytical balance at nearest accuracy of 0.0001. The fiber bundle strength (breaking tenacity) in g/tex was obtained from known values of breaking load, grammage, and gauge length. To achieve statistical significant result, 25 measurements were taken for each sample type.

2.2.4 XRD, TGA, and SEM measurements

XRD data were measured in 2θ range 5–30^0 using a X-ray diffractometer (Rigaku, UK) The voltage, scan rate and step size were 40 kV, 0.02^0/min, 0.02^0; respectively. A monochromatic from Cu–K$_\alpha$ line (λ= 1.540 A) was used as X-ray source. Thermogrametric data of the fiber samples was measured using thermogravimetric analyzer (Perkin Elmer TGA–7). 7–10 mg of fiber sample was heated in an alumina crucible in nitrogen atmosphere (flow rate 80 mL/min) at the rate of 10 oC/minute. Data collection range, weighing accuracy and precision were 26-600 ^{0}C, 0.1 μg, and ± 2 ^{0}C; respectively. Scanning electron microscopic images were obtained using field emission electron microscope (Carl Zeiss, Supra 40VP) at the accelerating voltage of 15 kV. The collected images were imported in ImageJ (NIH, USA) for further analysis.

2.2.5 Statistical analysis

Descriptive statistical parameters such as mean, maximum, minimum, standard deviation and confidence intervals were calculated using Origin Pro (Origin lab, USA). To test the significance difference between the data, whenever relevant, t-test was performed.

3 Results and Discussion

3.1 Gravimetric analysis

The % weight loss in Sterculia fiber retted for 20, 30, and 55 days was found to be 10.5, 15.2, and 17.5 %; respectively. The corresponding numbers for Bauhina fiber were 4.1, 5, 8.1%; respectively. These data suggested that Sterculia fiber has higher retting efficiency than Bauhinia. Since retting conditions were same for both fiber types, the difference is retting efficiency could be linked to fiber origin; that is difference in chemical composition and fiber to fiber bonding.

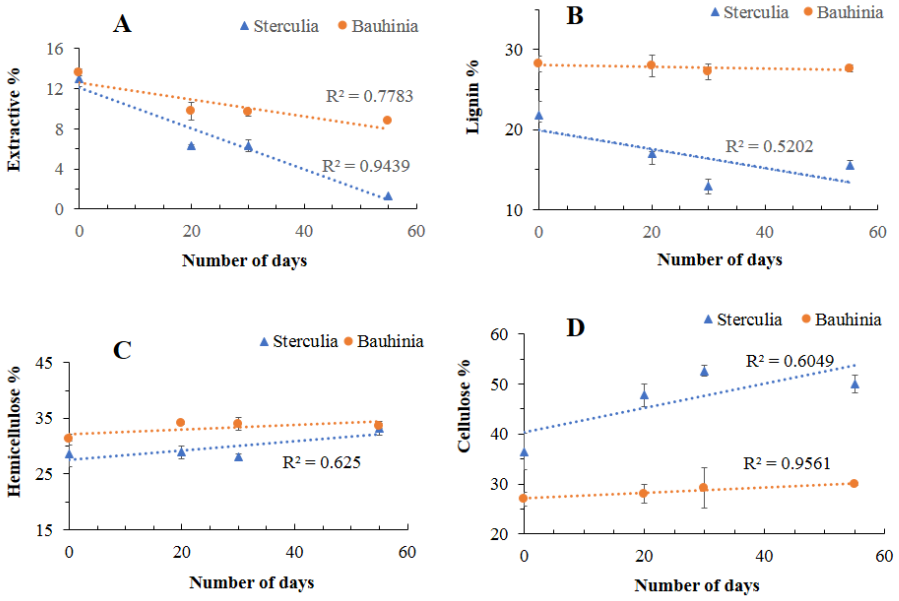

Figure 2: Analysis of major chemical components in the fiber samples. (A) Extractive. (B) Lignin. (C) Hemicellulose. (D) Cellulose. The blue and red data points correspond to Sterculia and Bauhinia; respectively. The dotted lines are the linear fit to the experimental data and R2 values greater than 0.5 are only shown.

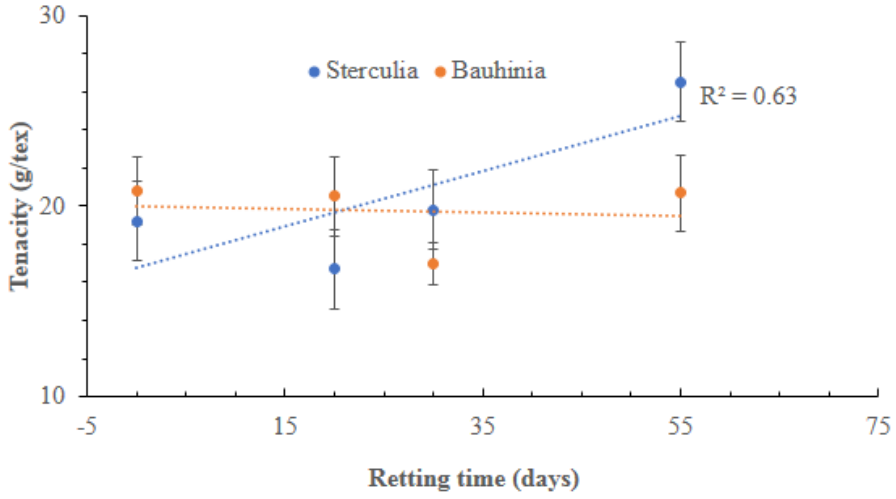

Figure 3: Mechnical strength of the fiber samples. The dotted lines are the liner fit to the experimental data points. The error bars are the 95% CI values to the mean (n=25.

The extractive, lignin, hemicellulose, and cellulose contents in the fiber samples for two fiber types in reported in figure 2. As expected, water retting resulted in decrease in extractive content with increase in retting time (Figure 2A). In the 55 days retting period, the extractive content decreased significantly from 13 to 1.3 % ($p<0.05$) in the Sterculia fiber. The corresponding decrease in Bauhinia fiber was from 13.6 to 8.7 %. The weight loss could have resulted from the removal of pectin and waxy materials due to antimicrobial action. In the 55 days period, a significant decrease in lignin ($p<0.05$) content from 21.9 to 15.5 % was also observed in Sterculia (Figure 2B). The weight loss in retting depends on type of microorganism, water composition, retting time, and fiber types [4, 19]. So it is not surprising to see the observed difference in weight loss in *Sterculia* and *Bauhinia* fibers. Hemicellulose content in both sample types did not change significantly (Figure 2D). In the 55 days retting period hemicellulose changed from 28.6 to 33.2 % in the Sterculia fiber. The corresponding

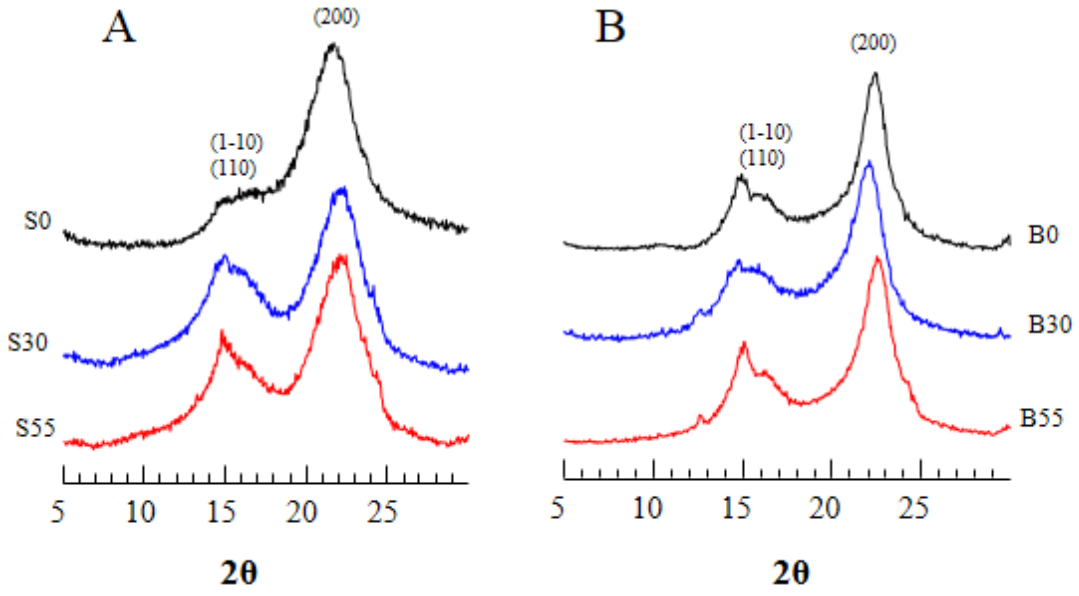

Figure 4: XRD data for the fiber samples. (A) XRD data of Sterculia samples retted for 0 (S0), 30 (S30) and 55 (S55) days. (B) XRD data of Bauhinia samples retted for 0 (B0), 30 (B30) and 55 (B55) days. Data are overlaid vertically for easy comparison. Numbers in parentheses indicate the reflection planes.

change in Bauhinia fiber was from 31.2 to 33.6 %. In the same time period, cellulose content in Sterculia fiber increased significantly (Figure 2C) from 36.5% to 50% ($p<0.05$). A nominal change from 27 to 30% was observed in Bauhinia fiber. We also measured ash content in fibers retted for 0 and 55 days. The corresponding values for Sterculia and Bauhinia were 6.7 and 4.7% and 6.2 and 4.4 %; respectively. The decrease in ash content could be due to partial loss of inorganic minerals and other impurities on water retting. As expected, ash change in ash content showed excellent negative correlation (r= -0.9) with extractive content.

3.2 Mechanical strength

The tenacity data for all the samples is provide in figure 3. In the 55 days period, the breaking tenacity of Sterculia fiber increased significantly ($p<0.05$) from 18.3±1.6 (mean ±95% CI) to 26.9±2.7 g/tex (R_2=0.73). In the same period, mechanical strength of Bauhinia fiber did not change significantly ($p>0.05$). The increase in fiber strength in Sterculia could be due to more loss in lignin from inter-fibrillar region that can lead in increased reorganization of cellulose chain thereby increasing crystallinity (later section) and strength [28].

3.3 XRD data

We measured XRD data of selected sterculia and Bauhinia samples (Figure 4A and B). The XRD data of all the samples resemble to that of typical cellulose material. Cellulose fiber in natural form contains amorphous and crystalline regions. A broad peak at 2θ value of 15.5^0 orginates form the crystalline planes (1-10) and (110) and the strong peak at $2\times$ value of $\sim 21.5^0$ from (200) plane. The broad contribuation underlying the crystalline peaks is known to orignate from amorphous scattering [30]. We used deconvolution method in the 2θ range of $5–30^0$ to get information on crystallinity index (CI) [30].

$$CI = \left(\frac{A_t - A_{am}}{A_t}\right) 100\% \quad (1)$$

where, Aam and At are the integrated intensity amorphous phase and total intensity of crystalline and amorphous phases; respectively. The crystallinity index in Sterculia samples retted for 0, 30, and 55 days was found to be 44%, 48% and 50%; respectively. A good positive correlation (r=+0.85) between crystallinity and strength was obtained. Crystallinity index increases on water retting is also reported for hemp fibers [31]. The increase in crystallinity index could be due to removal of lignin form inter–fibrillar region that can result in close packing of cellulose chains [28]. Crystallinity index in Bauhinia samples retted for 0, 30,and 55 days was found to be 55%, 54% and 54%; respectively.

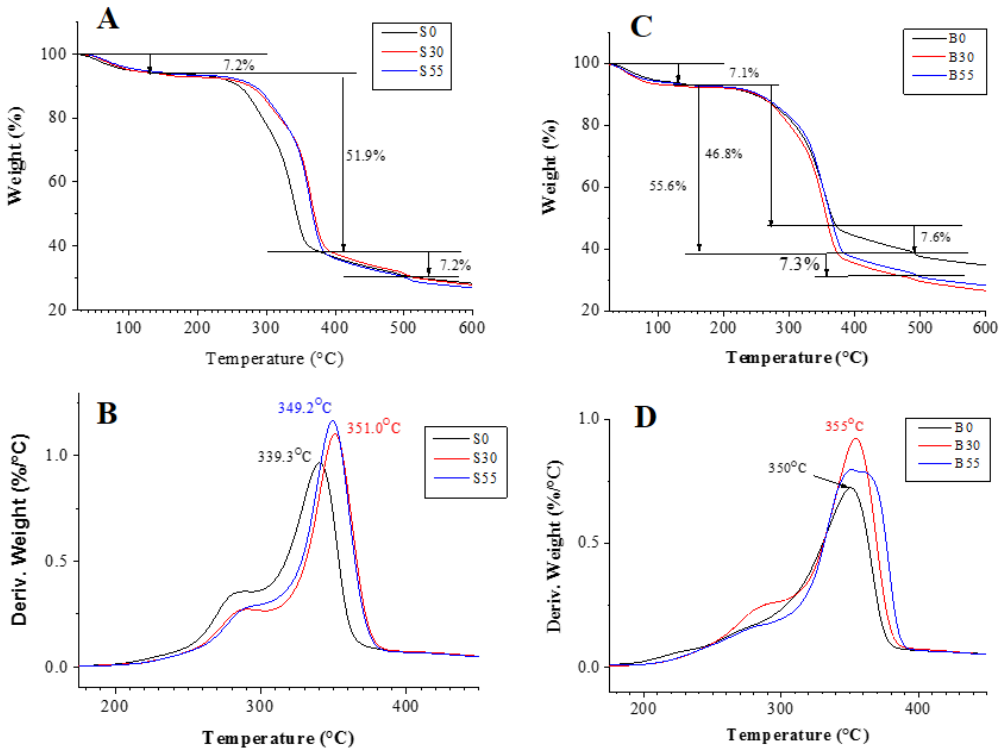

Figure 5: TGA and DTG data for the fiber samples. (A), (B) TGA and DTG for Sterculia water retted for 0 (S0), 30 (S30), and 55 (S55) days. (C), (D) TGA and DTG for Bauhinia water retted for 0 (B0), 30 (B30), and 55 (B55) days.

A poor positive poor correlation (r= +.5) was between the crystallinity change and strength was observed. This could be due to lower loss of lignin form inter–fibrillar region; consistent with data reported in figure 2B. A small peak shift in the XRD data could be due to change in relative contribution of amorphous and crystalline phases and or change in crystallite size.

3.4 Thermal properties

In both samples the weight loss below 100^0C (Figure 5A and C) is due to loss of moisture and or low volatile impurities [28, 32–34]. The weight loss between 100 to 300^0C is mainly due to hemicellulose; as hemicellulose is known to be less stable than cellulose (Figure 5A and C). The weight loss in 300–400^0C range is mainly due to degradation of glycosidic bonding in cellulose that creates organic molecules such alkenes and additional derivatives of hydrocarbons. The weight loss beyond 400^0C is mainly due lignin (5A and C) [35–37]. The main DTG peak for Sterculia shifts from 340^0C to 350^0C in water retted samples. This suggested that water retting results in increase in thermal stability (Figure 5B). It could be due increase of cellulose content and change in crystalline properties; as reported previously. The strong DTG cellulose peak in water retted Sterculia samples (S30 and S55) is consistent with increase of cellulose on water retting (figure 2D). In Bauhinia, DTG peak shifts from 350^0C to 355^0C in water retted samples (Figure 5C). This suggested that thermal stability of water retted Bauhinia does not change significantly on water retting.

3.5 SEM imaging

Imaging of selected samples (6 out of 8) was performed to see any change in fiber surface morphology. A close comparison of images of the Sterculia fiber retted for 0, 30, and 55 days (Figure 6A, B, and C) reveals that gummy materials are significantly removed from the fiber surface and fiber bundles are partially separated. This observation is consistent with the wet loss study. Similar change is observed in Bauhinia fiber (Frames D, E, and F).

4 Conclusion

To summarize, water retting efficiency and lignocellulosic content of Sterculia villosa and Bauhinia vahlii fiber retted for different periods was measured. In Sterculia fiber, weight loss increased significantly with retting time; which is consistent with reduction of lignin, ash and extractive contents. The change was less significant in Bauhinina fiber suggesting lower retting efficiency. In around

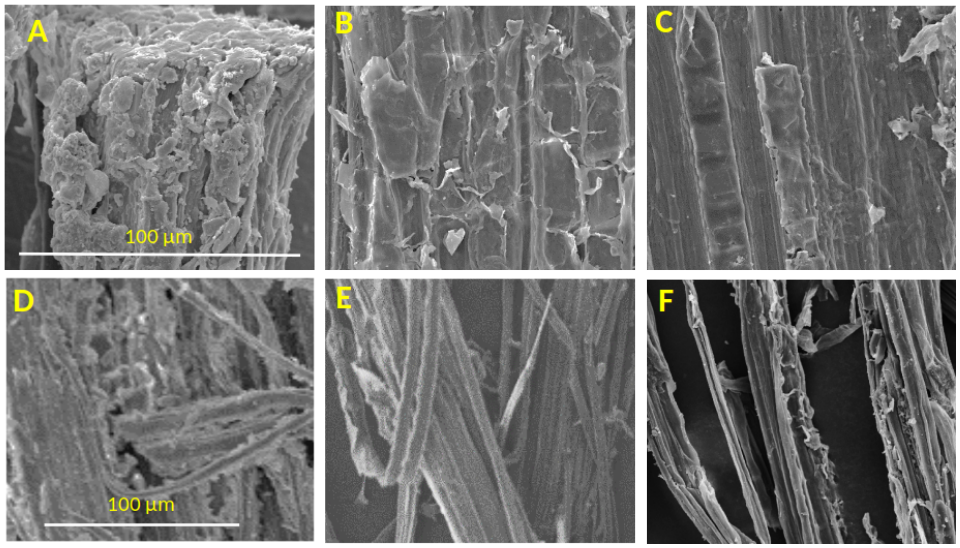

Figure 6: SEM images. A, B, and C are the images of Sterculia fiber retted for 0 (S0), 30 (S30), and 55 (S55) days; respectively. D, E, and F are the corresponding images for Bauhinia fiber. Scale bar of 100 μm are provided in A (also applicable for B and C) and D (also applicable for E and F).

2 months retting period, mechanical strength of Sterculia fiber increased significantly but that of Bauhinia did not change. Thermal stability of both fiber increased on water retting. These observations are also supported by XRD and SEM imaging data. These findings suggested that both fiber have excellent water stability and can be explored as an important component in fabricating cellulose based composite materials that are intended for high wetting or humid conditions.

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