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Study on the Performance of Bamboo Fibre Modified with Different Concentrations of Sodium Hydroxide and Chlorine Containing Agents

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Abstract

In this study, the bamboo fibre bundles (CAN retted fibre bundles) have been treated with sodium hydroxide at different processing conditions, in combination with potassium hydroxide and chlorine containing agent sodium chlorite solution. The purpose of this study was to develop efficient method for the extraction/loosening of bamboo fibres for further textile applications, therefore, lignin content, tensile strength, weight loss, moisture content, whiteness and yellowness indices were measured. Lignin content analysis of the extracted fibre bundles showed that there was a remarkable reduction in lignin content after these treatments. Scanning electronic microscopy of the treated bamboo fibre bundles showed removal of short elementary fibres from their surfaces in appropriate amount which further improved their overall physical properties of treated bamboo fibres.

Keywords: Bamboo fibres; Scanning Electronic Microscopy (SEM); Fourier Transform Infrared Spectroscopy (FTIR); X-Ray Diffraction (XRD)

Introduction

To get rid of the natural amorphous colloidal substances (e.g., hemicellulose and lignin from bamboo) and obtain fibre from phloem tissue, mechanical, chemical and biological methods are accessible in literature [1-3]. Chemical method is the most commonly used method for opening of fibre bundles and considered as the important key procedure for textile fibre extraction for further wet processing. Alkaline solutions like caustic soda, sodium tri-phosphate, sodium sulphate, sodium carbonate, sodium hydrogen phosphate and sodium silicate are usually used for this purpose. Alkalis support breakage of lignin in the fibres without attacking the cellulose. Amid various alkalis, caustic soda is one of the mainly explored for opening and loosening of bamboo fibre bundles. In this study, the bamboo fibre bundles (CAN retted fibre bundles) have been treated with sodium hydroxide at different processing conditions and in combination with potassium hydroxide. The fibres have also been treated with sodium chlorite solution. The purpose of this study was to develop efficient method for the extraction/loosening of bamboo fibres for further textile applications. An optimum method for extraction of fibres from the bundles has been standardized here and the extracted fibres were further characterized by calculating weight loss, whiteness and yellowness indices, lignin content measurement, tensile strength, Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD) and Scanning electron microscope (SEM).

Materials and Experiments

Materials

Raw culm of *Bambosa vulgasis* was harvested from Botanical Garden of Guru Nanak Dev University, Amritsar. All the chemicals used in this investigation were of AR grade and were purchased from Merck Ltd., Hi-media Labs, Bombay (India).

Methods

The fibre bundles were retted by chemical assisted natural retting (CAN) technique followed by scouring with 15 g/L Na_2CO_3 and subsequently treated either of the following ways:

Method A: The bamboo fibres were firstly soaked into a mixture of 17.5% (owf) NaOH and 17.5% (owf) KOH solution with a material to liquor ratio of 1:40 at ambient temperature for 1 h followed by washing at 40°C for 15 min, neutralization (2-3 g/L acetic acid) and drying.

Method B: The bamboo fibres were immersed into solution 0.1 N NaOH with a fibre to liquid ratio of 1:40 at 35°C for 72 h followed by washing at 40°C for 15 min, neutralization and drying.

Method C: The bamboo fibres were treated with 0.7% (owf) $NaClO_2$ solution (Sodium chlorite solution) with a fibre to liquid ratio of 1:40 at 90°C for 1 h at pH-4 (using acetic acid and sodium acetate as buffer). After that it was followed by an antichlor treatment with 2% (owf) $NaHSO_3$ solution (Sodium bisulfite) at room temperature for 15 min with material to liquor ratio of 1:40.

Method D: The bamboo fibres were treated with 20 g/L NaOH in the presence of 5 g/L Sodium tri phosphate solution ($Na_5P_3O_{10}$) with a material to liquor ratio of 1:40 at boil for 2 h followed by washing at 40°C for 15 min, neutralization and drying.

Method E (Rapid method): The bamboo fibres were treated with 150 g/L NaOH solution with a material to liquor ratio of 1:40 at 80°C for 45 min followed by washing at 40°C for 15 min, neutralization and drying.

Optimization of NaOH concentration (For method B)

The retted bamboo fibres were soaked into NaOH solution with different concentration (0.05 N, 0.1 N, 0.2 N and 0.3 N) with a fibre to liquor 1:40 at 35°C for 72 h followed by washing at 40°C for 15 min, neutralization and drying.

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Determination of fibre characteristics

Tensile testing: Breaking strength (Load N) of bamboo was tested using an Instron series IX fiber breaking strength machine in a constant 27°C temperature and 65% relative humidity using ASTM Method D-5035.

Weight loss: Fiber weight loss was calculated using eqn. (1):

$$\text{Weight loss } (\%) = w_i - w_f / w_i \times 100 \quad (1)$$

Where w_i is the initial weight of the raw bamboo fiber. w_f is the final weight of treated bamboo fiber.

Scanning electron microscopy observations: The deformation behaviour of a single elementary retted bamboo fibre bundle was investigated by scanning electron microscopy (SEM) using Quorum Q150RES (Supra 55- CARLZEISS) equipment (EHT=10.00 KV, Signal A=In Lens). All specimens with longitudinal-section were coated with a thin layer of gold to avoid electrical charging.

Chemical analysis: The chemical composition of the original bamboo culm (without outmost layer) and retted bamboo fibre bundles was analysed for lignin contents by using TAPPI standard T250-um-85.

Whiteness and yellowness indices: The colorimetric properties of the retted fibers (D65 illumination, 10° observer) were determined using a Spectraflash 600 colorimeter (Data color International) [4].

Fourier transforms infrared spectroscopy (FTIR): IR spectra of the fibres were recorded by using FTIR; Make: PerkinElmer Spectrum; Model: Spectrum Two 92035, using KBr disks containing 1% finely ground samples. Eight scans were taken of each sample recorded from 4000 to 450 cm^{-1} at a resolution of 4 cm^{-1} in the absorbance mode.

X-Ray diffraction (XRD): Crystallinity and crystal size of raw bamboo fibres and optimally treated bamboo fibres were determined by wide angle X-ray diffraction (XRD; 2θ=0-60°), using a Panaltical expert pro using Cu-Kα radiation detector (λ=1.5406Å) Bamboo fibres were ground into powders as measuring samples. The scanning velocity was 50/minute, the voltage was 30 kV. The crystallinity expressed as the crystallinity index (Crl) was calculated as the percentage of crystalline material in fibre sample [5].

$$Crl = \frac{I_{002} - I_{am}}{I_{002}} \times 100$$

Where I002 is the maximum intensity of the (002) lattice diffraction ($2\theta \approx 22.1°$) and I_{am} is the intensity at $2\theta \approx 15.7°$.

$$D_{(hkl)} = \frac{k\lambda}{B_{(hkl)} Cos\theta}$$

The standard size of crystallites was determined from following equation.

Where (hkl) is the lattice plane, $D_{(hkl)}$ is the size of crystalline, K is the Scherrer constant (0.84), λ is the X-ray wavelength (0.94 nm), B_{hkl} is the FWMH (full width half maximum) of the measured hkl reflection, and 2θ is the corresponding reflection angle [6].

Results and Discussion

In lingo-cellulosic material, alkali plays an important role in separation of fibres. To obtain a satisfactory level of fibre extraction, either high concentration of alkali or high temperature treatment is required. In this section, the effects of different 5 different methods on physical properties of bamboo fibres were investigated. One of them was selected as best in terms of physical appearance and strength. That method was further optimized for sodium hydroxide concentration. The experimental results are shown in Table 1.

Chemical methods are based on the mechanism that the cellulosic fibrils and non-cellulosic substances have different stability in alkaline solution. It involves the soaking of bast fibres in aqueous alkaline solutions e.g., solution of sodium carbonate (Na2CO3), sodium hydroxide (NaOH) and potassium hydroxide (KOH) [7]. In this study, the different methods A, B, C, D and E led to the similar changes i.e., the separation of bundles from the culm. Table 1 presented the general physical changes during the processing. The selection of any particular chemical or combination of chemicals is related to the cost and efficiency involved [8]. Keeping this in view, the method A was designed in which a combination of NaOH and KOH was used. Since lignin and hemicellulose get dissolved after the alkali treatment, the pectin breaks and results in separation of fibres from the bundles [9-11]. Sodium chlorite acts as a bleaching and delignification agent for ligno-celluloses material [12]. By using sodium chlorite in Method C, lignin portion dissolves (lignin content 7.13%) while carbohydrates portions remain almost unaffected (weight loss 9.00%). The problem of this method is that acidified sodium chlorite solution is very reactive and causes liberation of large amount of chlorine gas which creates environmental pollution.

Methods D and E have been carried out at high concentration of alkali at temperature 80°C and 100°C respectively, leading to decrease in whiteness and yellowness indices along with the decrease in tensile strength. These two methods presented bundles of small fibres as well as into the degraded deepest layer. As these two methods D and E involve harsh reaction conditions causing fibre damage, they were not used for further processing of bamboo fibre in this study. An interesting result from method B observations revealed that fibres are not broken as this has been performed under controlled conditions for prolong hours. Visual examination of fibres extracted from these methods suggests that fibres of any length, appearance and strength could be obtained which is beneficial for downstream processing to meet the requirements for spinning. In method B, the greatest advantage is that it is a controllable process and the fibres obtained could be further used for textile applications.

During this study, it was observed that method B was the best option in terms of physical properties as comparison to methods B, C, D and E.

Method	Weight Loss (%)	Lignin Content (%)	Load (N)	Hunter Yellowness	Hunter Whiteness
CAN	5.0	9.00	20.07	54.13	17.28
A	9.42	8.96	11.87	64.71	2.19
B	16.87	8.04	12.6	64.58	15.46
C	9.00	7.13	13.5	52.99	14.02
D	19.0	8.27	10.5	70.19	-0.63
E	20.48	7.95	10.2	86.30	-8.96

Table 1: Comparison of physical properties of bamboo fibres extracted from method A, B, C, D and E.

Characteristics of treated bamboo fibres using five different methods

A detailed investigation in terms of lignin content, weight loss, tensile strength, whiteness index and yellowness index is carried out to determine the effects of different methods (A, B, C, D, and E). Table 1 shows the changes in physical properties of bamboo fibres after different treatments. It can be found that the lignin content (8.96%) for the method A treated fibres is higher than the fibres treated with other methods (B, C, D and E). In the fibre bundles obtained from methods B. C, D and E, the lignin content was remarkably reduced in comparison to the method A which clearly indicates dissolution of some parts of non-cellulosic substances from the fibre bundles. Table 1 also shows the maximum weight loss in case of methods D and E and resulting in the removal of noncellulosic substances. As shown in Table 1, treatment with methods A and C seems to have only limited effects on the extraction/fibrillation of bamboo fibres; however, they have good reduction in lignin content but with poor whiteness indices. Moreover, the method B and E at quite high alkaline concentration showed excellent weight loss along with loss in lignin content in adequate amount. As the method E was conducted at high temperature under highly alkaline conditions it resulted in high yellowness index and reduced tensile strength of the treated sample. Method B produced reasonable reduction in lignin content and weight loss with acceptable whiteness and tensile strength.

FTIR spectra of bamboo fibres

Fourier transform infrared spectroscopy is generally used for investigating the arrangement of constituents (elements) and the chemical changes in lingo-cellulosic fibres throughout the treatment. The FTIR spectra of bamboo fibres retted by CAN method, bamboo fibre treated with methods a, b, c, d and e are shown in Figure 1, respectively.

An observation of the residue removed from the bamboo fibres during the treatment with different methods was performed by FTIR (Figure 1), where a comparison was made with CAN retted bamboo fibre bundles with treated bamboo fibre bundles.

According to the infrared spectra of the untreated and treated fibres, the wave figures/numbers of the most important characteristics peaks have been observed. In the FTIR spectra, the peaks and the absorption intensity of the CAN retted bamboo fibres and fibres treated with method A were rather similar, representing an analogous structure of the samples CAN and a.

The peaks and the absorption intensity of the fibres treated with methods c and d were also found similar. Differences were observed at the range of 1700 cm^{-1} to 900 cm^{-1} in case of fibres treated with method B. The absorption at 3434 cm^{-1}, 3426 cm^{-1} and 3405 cm^{-1} were accredited to O-H stretch. The absorption at range 2932-2883 cm^{-1} and range 2891-2850 cm^{-1} were assigned to CH stretch in CH_2 and CH_3 groups, respectively.

The bands at 1605 cm^{-1}, 1506 cm^{-1}, 1425 cm^{-1} and 1345 cm^{-1} were related to skeleton stretching vibration of the aromatic rings and methoxyl C-H deformations and bending of lignin [13] which generally showed the existence of lignin in bamboo. The relative intensity of the bands appeared in the CAN retted bamboo fibres (Figure 1 CAN), and the fibres treated with methods a and e (Figures 1a and 1e) were found to be much stronger than those treated with methods b, c and d (Figures 1b, 1c and 1d). However, a weak signal in the zone 1506-1510 cm^{-1}, for alkali treated material indicates a reduction in the amount of lignin in the treated sample. CAN retted bamboo fibres and treated fibres with methods A, C, D also showed strong peaks at 1730-1737 cm^{-1}, which was assigned to unconjugated C=O stretching in acetyl group of hemicellulose [14]. No such absorption peak was observed for the

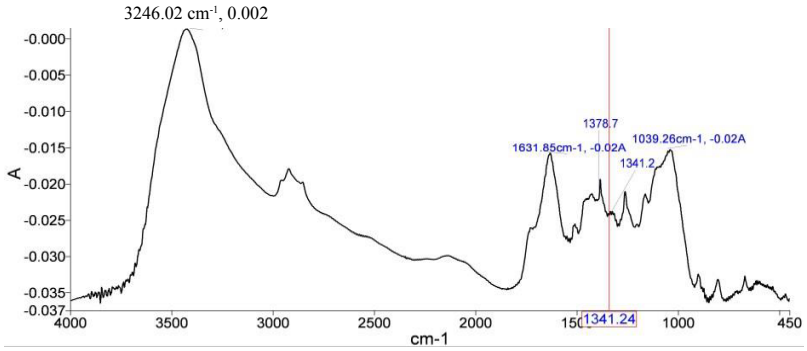


Figure 1: (CAN): FTIR of Chemical Assisted Natural retted bamboo fibres.

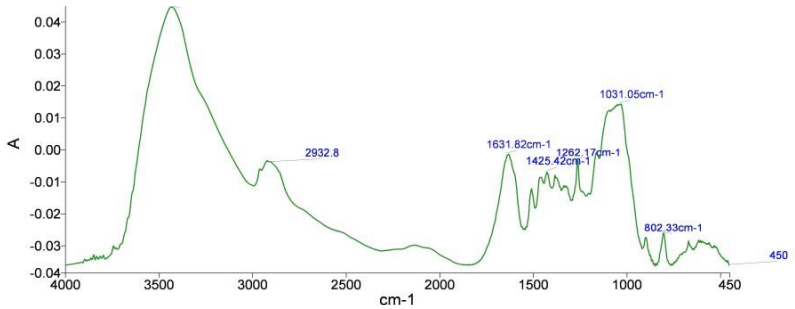


Figure 1a: FTIR of Chemical Assisted Natural retted bamboo fibres after treatment A [mixture of 17.5% (on the weight of fibres) NaOH and 17.5% (on the weight of fibres) KOH solution].

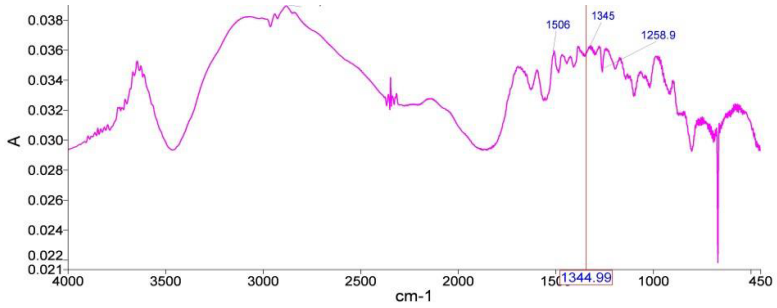


Figure 1b: FTIR of Chemical Assisted Natural retted bamboo fibres after treatment B (solution 0.1N NaOH).

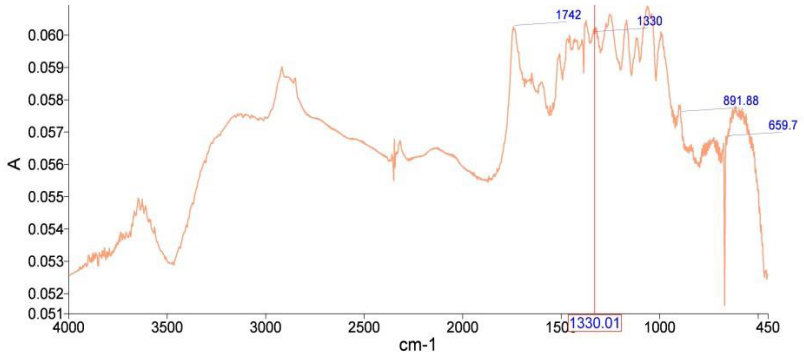


Figure 1c: FTIR of Chemical Assisted Natural retted bamboo fibres after treatment C [with 0.7% (on the weight of fibres) $NaClO_2$ solution (Sodium chlorite solution)].

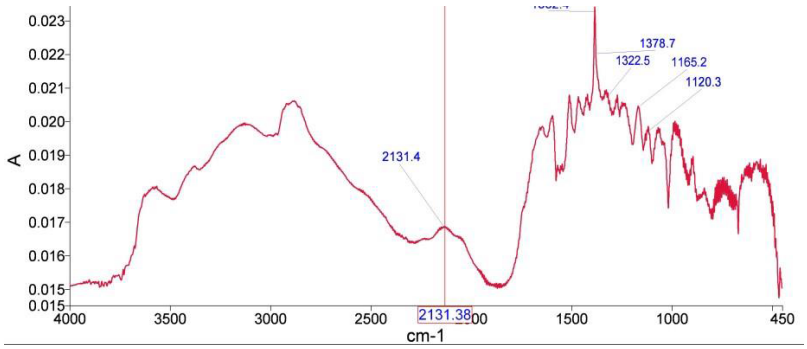


Figure 1d: FTIR of Chemical Assisted Natural retted bamboo fibres after treatment D [20 g/L NaOH in the presence of 5 g/L Sodium tri phosphate solution ($Na_5P_3O_{10}$)].

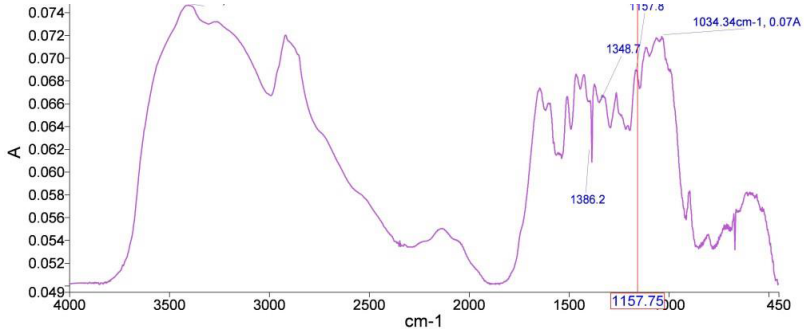


Figure 1e: FTIR of Chemical Assisted Natural retted bamboo fibres after treatment E (150 g/L NaOH solution).

method B treated fibres, indicating that deacetylation happened during alkali treatment at low temperature for prolonged hours. The precise intense peaks/bands in the range of 1072-1040 cm^{-1}, represented the presence of celluloses in fibres treated using method B. Thus, method B treated fibres had a higher concentration of cellulose. These annotations showed that CAN retted fibres were largely composed of lignin, hemicellulose and cellulose. It is suggested that the extraction or separation of fibres from fibre bundles can be carried out by removal of appropriate amount of these substances. It can be observed that all the bands mentioned were comparatively weaker for the fibres treated with alkali at low temperature for prolonged time (Method B).

XRD of bamboo fibres

Figure 2 showed the XRD pattern for untreated bamboo fibres and treated bamboo fibres. It was found that their patterns have the similar diffraction peaks, but with different intensity. Additionally, their crystallinity degree has been calculated by dividing the crystalline area by the total area formed by the curves in Figure 2. The crystalline degree has been obtained 7.5%, 93.9%, 53%, 71%, 73.6% and 92.6% for untreated (CAN) and treated (a, b, c, d and e) bamboo fibres respectively. Comparison of XRD spectra of CAN retted bamboo fibres and treated bamboo fibres using method a, b, c and d and e has been shown in Figure 3. Thus, when lignin and hemicellulose, both of which were amorphous in nature were partially removed that leads to an improved packing of cellulose chains, crystallinity would be better after different treatments. This analysis could be proved by the fact that treatment /swelling in sodium hydroxide at low temperature for long hours had higher crystallinity than CAN retted bamboo.

A huge amount of gum in the untreated CAN (Chemical assisted natural retting) bamboo fibres can be seen in Figure 4 (CAN). After treatment with methods A and D, the bamboo fibres were fiberized, however it was observed that the bamboo fibre bundles were joined with large amount of gum on surface (Figures 4a and 4d). On treatment using method b and e, the surfaces of the bamboo fibres were observed to have smooth surface (Figures 4b and 4e). From Fig. 4c, it can be observed that the fibres still remained in aggregated form. These pictures show that the fibres treated with methods b and e had more uniform geometry of fibrils arrangement than the fibres treated with methods a, c and d.

Conclusion

Natural bamboo fibres extracted by CAN retting technique were modified during the study and best processing parameters were optimized to improve the fibre quality for textile application. Reduced contents of non-cellulosic substances and improved fibre fibrillation after different chemical methods of modification were observed from

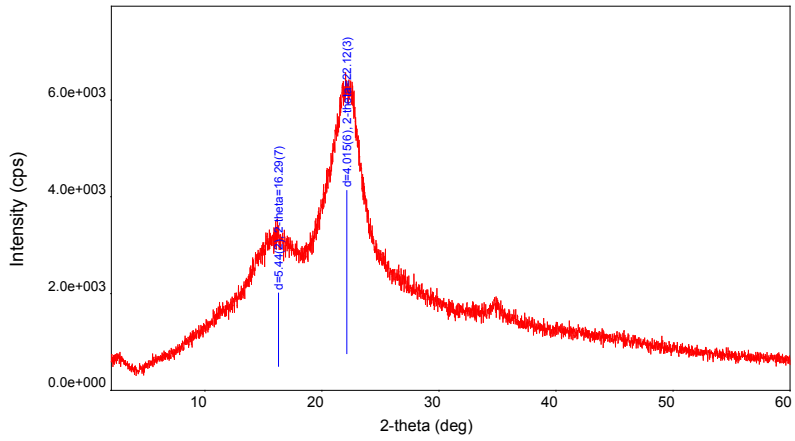


Figure 2: CAN. XRD of Chemical Assisted Natural retted bamboo fibres (CAN) without treatment.

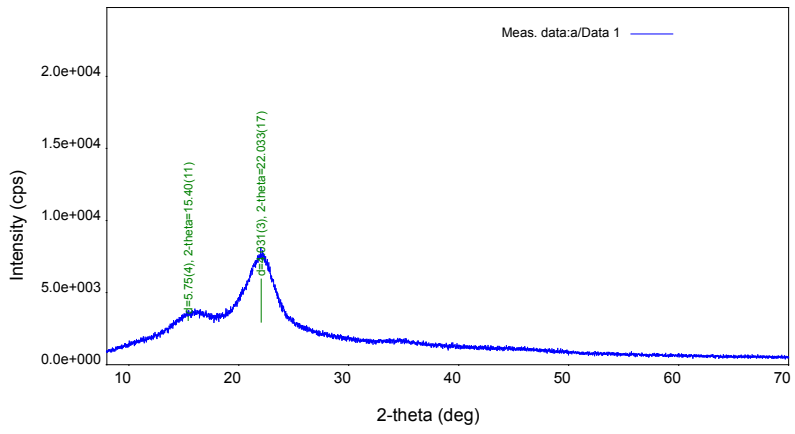


Figure 2a: XRD of Chemical Assisted Natural retted bamboo fibres after treatment A [mixture of 17.5% (on the weight of fibres) NaOH and 17.5% (on the weight of fibres) KOH.

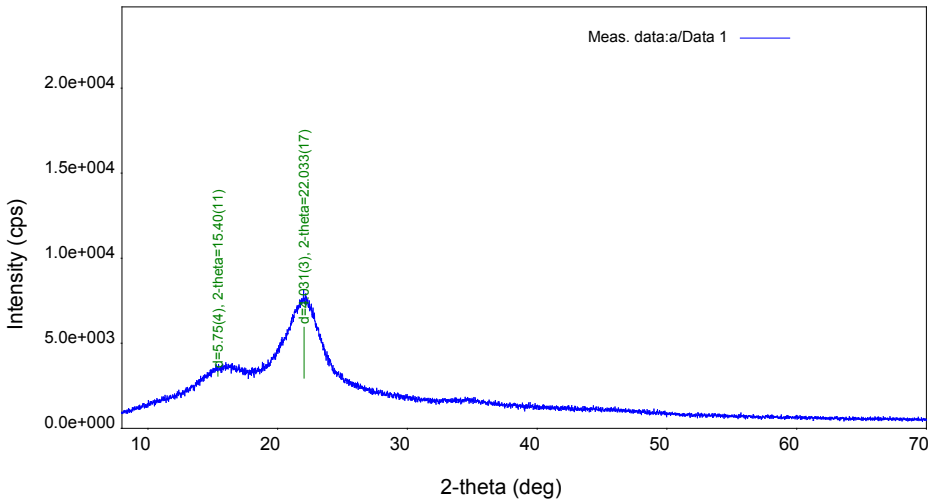


Figure 2b: XRD of Chemical Assisted Natural retted bamboo fibres after treatment B (solution 0.1N NaOH).

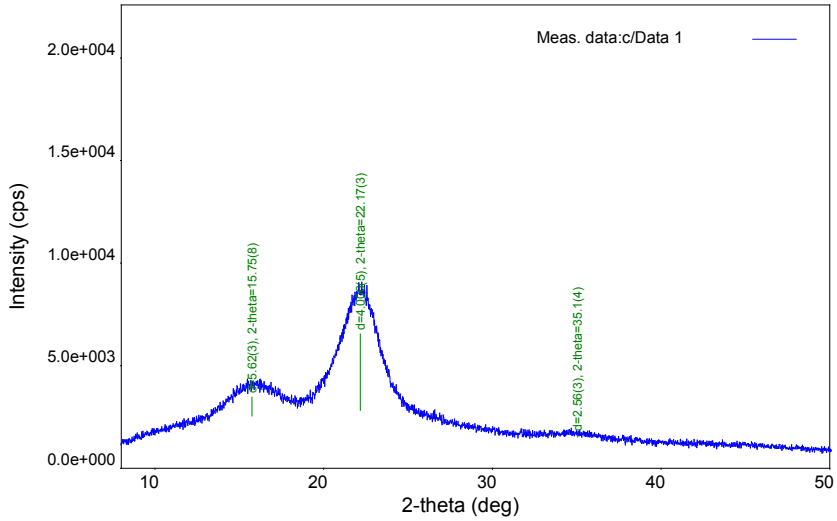


Figure 2c: XRD of Chemical Assisted Natural retted bamboo fibres after treatment C [with 0.7% (on the weight of fibres) $NaClO_2$ solution (Sodium chlorite solution)].

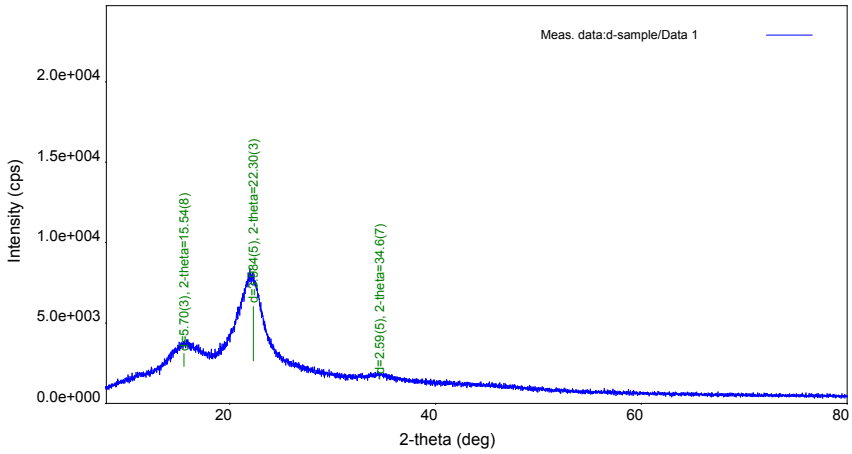


Figure 2d: XRD of Chemical Assisted Natural retted bamboo fibres after treatment **D** [20 g/L NaOH in the presence of 5 g/L Sodium tri phosphate solution ($Na_5P_3O_{10}$)].

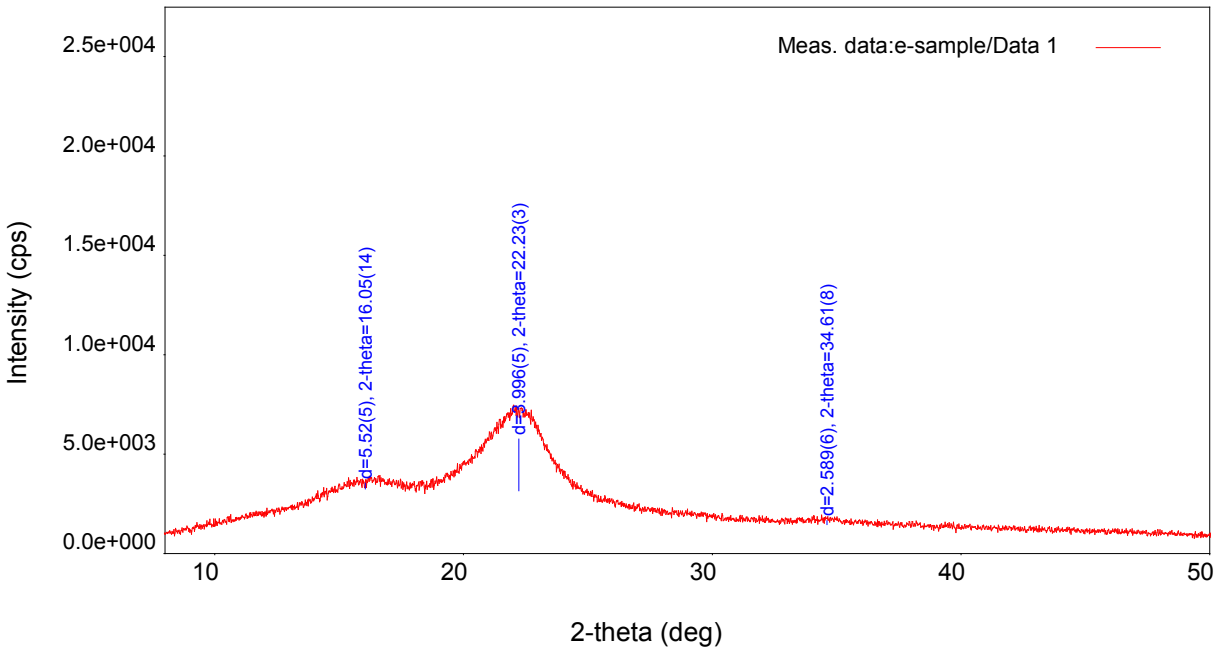


Figure 2e: XRD of Chemical Assisted Natural retted bamboo fibres after treatment **E** (150 g/L NaOH solution).

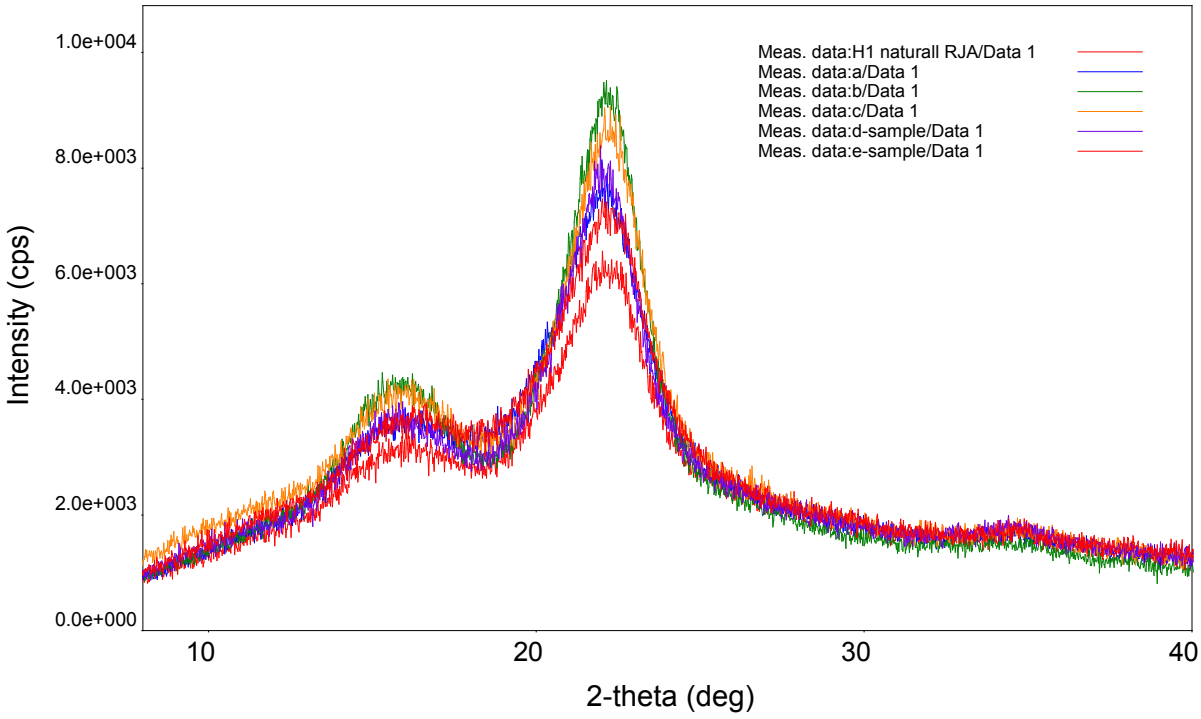
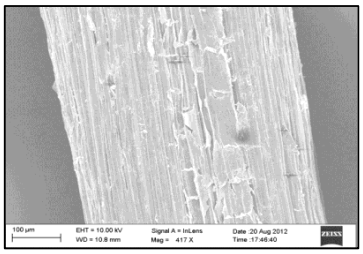
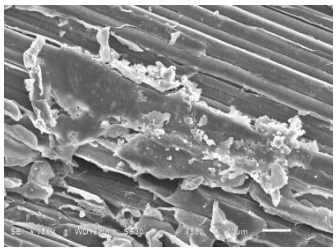


Figure 3: Comparison of XRD spectra of Chemical Assisted Natural retted bamboo fibres and treated bamboo fibres using method **A** (NaOH + KOH), **B** (0.1N NaOH), **C** (0.7% $NaClO_2$), **D** (20 g/L NaOH) & **E** (150 g/L NaOH).

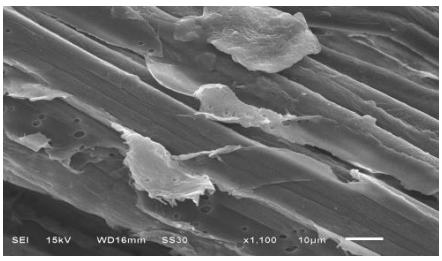
experimental results. Method B produced reasonable reduction in lignin content and weight loss with acceptable whiteness and tensile strength. From FTIR spectra, it can be observed that all the bands mentioned over were notable weaker for the fibres treated with alkali at low temperature for prolonged time (method B). Furthermore, from their XRD pattern, it was found that fibres had the similar diffraction peaks with different intensity. SEM pictures showed that the fibres treated with methods B and E had a more uniform geometry of fibrils



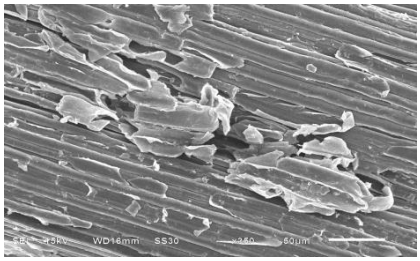
CAN (100 µm)



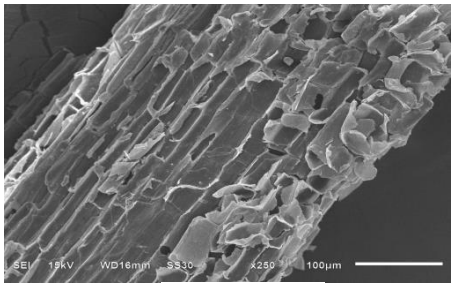
A (20 µm)



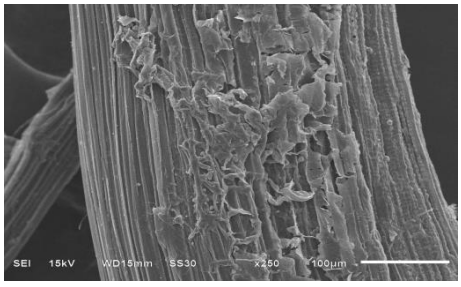
B (10 µm)



C (50 µm)



D (100 µm)



E (100 µm)

Figure 4: The longitudinal view of fractured surface of (CAN) CAN retted bamboo fibres and treated bamboo fibres with (a) Method A (b) Method B (c) Method C (d) Method D and (e) Method E.

arrangement than the fibres treated with methods A, C and D.

Hence it can be concluded that the chemical amendments as carried out in method B may propose an opportunity of using natural bamboo fibres for further textile applications.

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