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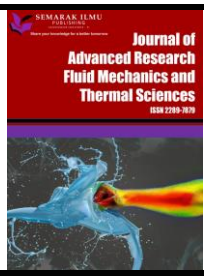

Effect of Thermal and Alkali Treatment on Morphological Analysis of Natural Bamboo Fibre

Nur Saadah Zainal[1], Zaleha Mohamad[1,*], Mohammad Sukri Mustapa[1], Fazimah Mat Noor[1], Nur Azam Badarulzaman[1]

[1] Department of Mechanical Engineering, Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia (UTHM), Batu Pahat, 86400, Malaysia

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ABSTRACT

This paper presents the physical and morphological properties of bamboo fibre derived from Schizostachyum brachycladum. The bamboo fibre was prepared through chemical treatment by sodium hydroxide (NaOH) and went through thermal treatment at 500°C. The changes and modifications of the surface of the fibre were evaluated by scanning electron microscopy (SEM). The surface of the fibre was rough and uneven at samples BF-0 and BF-1. This contributes to their amorphous portions. Sample BF-2, BF-3 and BF-4 showed smooth surface texture and firmness. This proved the fibre was in the transition to a crystalline state. The changes in internal structure changes during the process were successfully determined by Fourier to transform infrared spectroscopy (FTIR). Samples BF- 3 and BF-4 were a sample that underwent a high concentration of NaOH and was fired at 500°C during the fired process. The spectra show the high broad peak at 1436.95 cm^{-1} and 1435.59 cm^{-1} where the absorption of C-H bending took place. This proved the increase in the fibre content and a broken layer of hemicellulose and impurities components. The pattern performed by using x-ray diffraction (XRD) showed naturally bamboo properties at an amorphous state. The property behaviour could change by applying the alkali and thermal treatment.

1. Introduction

The resources found in nature have existed for billions of years. The wasting of these natural resources is crucial for discussion. The lignocellulosic materials are among the natural resources that have a wide range of applications in various technical fields, mostly in the construction field [1]. Lignocellulosic materials are typically made from fibres of plant origin, making them a natural, plentiful, and sustainable resource. These substances are mostly made of three chemical components which are cellulose, hemicellulose, and lignin. This, lead to the plant to its good strength, stiffness, and durability for its physical, mechanical and structural characteristics [2]. Bamboo is a naturally occurring composite material that is commonly required in building supplies, consumer items,

* *Corresponding author.*
E-mail address: zaleha@uthm.edu.my

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membrane supports, biopolymer films, and several other uses [3]. Researchers have researched the transverse dimensional alterations of bamboo of different elevations and species on a macro scale for the past few years. Three different types of cells—fibre, vascular, and parenchyma cells—make up the majority of bamboo. Fiber cells have sturdy multiple walls and thick walls with small lumens, whereas parenchyma cells have flimsy multiple walls and thin walls with large lumens [4]. As both glass and carbon fibres are difficult to biodegrade, using synthetic fibres has resulted in several environmental issues over the past decade [5]. Additionally, the development of synthetic fibres necessitates a significant amount of energy [6], which elevates the cost of both production and raw materials. Due to their benefits, such as their lightweight, renewability, biodegradability, low cost, minimal energy demands, vast availability, high strength, and elasticity modulus, natural fibre has been thought of as a synthetic fibre substitute [7,8]. Kenaf, jute, flax, sisal, coir, and bamboo fibre are among the plant fibres that are often explored [9-13]. Bamboo fibre has received particular attention among the various natural fibres because of its low density, high stiffness, high strength, and rapid growth of bamboo, which makes it readily accessible. The bamboo fibre cannot be extracted straight from the bamboo culm and must first undergo several treatments. Retting, steam explosion, alkali treatment, degumming, grinding, and crushing are a few of the techniques for removing bamboo fibre [14-17]. The extraction technique utilized has a direct impact on the fibres' strength and quality. Chemical treatment of the natural fibre can alter its surface state and chemical content to offer it the desired feature [18]. Sodium hydroxide (NaOH) [19,20], nitric acid and potassium chlorate (HNO3-KClO3) [21], sodium hypochlorite (NaClO), and benzoate [22] are a few of the chemicals frequently used to treat natural fibres. The most popular and reasonably priced chemical therapy is alkali treatment [23,24]. The interfacial adhesion between the polymeric matrix and the natural fibre is improved by the alkali treatment, which is an efficient way to change the fibre surface [25]. Alkali treatment is one of the chemical processes that are most frequently used to modify natural material fibres. This procedure uses an aqueous solution of sodium hydroxide (NaOH) to influence the structure and chemical build of plant fibres. Depending on the properties of the fibres and how they are utilized, the concentration, temperature, and period of action will change [26-42]. The removal of surface impurities and the prevention of fibrillation are both made possible by this treatment, which results in a fibrous material with a larger surface area and a smaller diameter, increasing the tensile strength of the fibres and the mechanical properties of the resulting composite [43]. Treatment affects the chemical composition and crystallinity index of the fibres.

This paper aimed to study the morphological properties of bamboo fibres. Additionally, this characterization of the natural bamboo fibre is influenced with and without having undergone any chemical and thermal treatment to see the variability. The investigation has been done using SEM, EDS, XRD and FTIR.

2. Material and Method

2.1 Materials

For Bamboo of species *Schizostachyum brachycladum* aged between three to four years was harvested in Selangor, Malaysia. The bamboo culms were split into splints in small sizes. Lignin is an aromatic polymer with an amorphous structure that functions as a binder on the plant, providing the fibres rigidity and guarding against microbial invasions [5]. Although lignin serves a variety of purposes in plants, it remains preferable to extract it from the plant fibres to ensure proper adherence of the composite components to the matrix. Combining thermal and chemical treatments, fibre extraction was carried out.

Bamboo strips were subjected to the alkaline treatment, a chemical process in which natural fibres are immersed in a predetermined concentration of aqueous sodium hydroxide (NaOH) at a certain time and temperature. The alkaline treatment modifies the surface of fibres by removing a certain rate of lignin, hemicellulose, wax, and oils covering the external surface of natural fibres.

2.2 Fabrication of Bamboo with Alkali Treatment

The process started with submerging strips of bamboo 2 cm broad and 5 cm long in a sodium hydroxide solution with a variety of concentration for a period of 60 mins with temperature of 80^{o}C. The chemical treatment process was undergone at different concentrations ranging from 0.1M, 0.5M and 1.0M. The treatment needs to repeat about 3 times and every treatment needs to change to a new solution. Table 1 shows the distribution group in this study.

After reaching the required immersed duration, the bamboo splints were removed and soaked with distilled water to dilute the alkaline content and to reduce the brittleness. Eventually, the bamboo splints were mechanically deliberated using a mill roller machine to obtain the fibre. The bamboo fibre produced was washed with running distilled water to eliminate the impurities (alkaline content and broken lignin) [44].

This process was followed by oven drying at a temperature of 60 °C for 1 hour. This process is important to gain a persistent mass. Moreover, the thermal treatment took place in a furnace at 500℃ for 2 hours with heating rate of 5℃/min [45]. Sieved process took place by passed through 63 µm mesh sieve. Lastly, all samples were characterized by using SEM, EDS, XRD and FTIR to find the phases and characterizations of bamboo fibre. Natural bamboo without chemical and thermal treatment (BF-0) and bamboo fibre with fired treatment but without chemical treatment (BF-1) have been used to see the different. The designation of these samples is summarized in Table 1.

Table 1
Designation of bamboo fibres at different treatment

Sample name	NaOH concentration treatment	Thermal treatment
BF-0	0	0
BF-1	0	500^{o}C
BF-2	0.1 M	500^{o}C
BF-3	0.5 M	500^{o}C
BF-4	1.0 M	500^{o}C

2.3 Morphological Testing

2.3.1 Scanning electron microscopy (SEM)

A scanning electron microscopy was performed to see the morphology of the bamboo fibre before and after treatment. The specimen surfaces were coated with a thin layer of gold before scanning. This process took place to obtain clear images and prevent charging of the surfaces. The process was conducted using FESEM (model JSM 6701F JOEL).

2.3.2 X-ray diffractometer (XRD)

An XRD was used to measure the mineralogical characteristics and phase index of bamboo fibre. The XRD was conducted by using an x-ray diffraction (XRD) system with the accelerating voltage and the current of 30 kV and 30 mA, respectively. The scanning was performed at a range of 5° to 90°

from 2 hours. The structural pattern of the samples was recorded and examined using the EVATM Software.

2.3.3 Fourier transform infrared spectroscopy (FTIR)

The Fourier transform infrared spectroscopy (FTIR) was used to detect the existence of organic compounds, O-H bonding and stretching in bamboo fibre. The model machine that has been used in this study was Jasco FTIR 4200 pulse Japan. The range was about 4000 cm-1 – 600 cm-1.

3. Result and Discussion

3.1 Scanning Electron Microscopy (SEM)

The scanning electron microscopy (SEM) of untreated and treated bamboo fibre is shown in Figure 1. The untreated specimen has a multifibrillar structure on its smooth surface, as seen in Figure 1(a) [44]. It was observed that the particle was in the existence hierarchical structure. The body is an elongated thin, in a form of a single thread or a thin flexible threadlike. The hemicellulose, pectin, wax, and other surface impurities in the layer of significant impurities with uneven distribution were likely the cause of the smooth fibre surface [46,47]. Poor tensile characteristics and poorer mechanical bonding with the polymer matrix were also consequences of smooth fibre surfaces [48]. The untreated fibres covered waxes and polysaccharides like lignin, hemicellulose, or pectins on their surface, giving the fibres a rough and uneven look. It shows the amorphous condition of the natural bamboo fibre. In the micrographs, it is impossible to observe the separation of individual fibres from the surface of the fibre bundle. The surface of sample BF-2 was found to be jagged and scabrous, as seen in Figure 1(b). This demonstrated that the effects of 500^{o}C temperature without chemical treatment are still restricted to crystallization [49].

The effect of the treatment on the lignin removal process can be observed. In Figure 1 (c), (d) and (e), it can be seen that by subjecting the material for a certain period of time to high concentrations of sodium hydroxide, the lignin and the hemicellulose are partially removed from the fibres. Recent research has demonstrated that the effect of the treatment on the roughness of the fibres, when treated with sodium hydroxide solution, relies on variables such as the concentration of the solution, the temperature, and the time of treatment. According to previous studies, treatments with concentrations over 7% can cause a stronger effect on the elimination of impurities, as indicated by the value of the average surface roughness of the fibres [50]. *Demir et al.,* [51] and confirm the efficiency of this method for exfoliating the surface of the fibres to increase their roughness. According to *F. Wang et. al.,* this situation was in accordance with fibre bundles on the radial direction of bamboo stem, which was caused by the variation of the self-construction properties of fibre cells, such as cellulose content, microfibril angle and crystallinity, on the radial direction of bamboo stem [52]. Even though, these images showed the surface not fully smooth and even, the transition of non-cellulose or impurities to eliminate on the fibre surface can be seen respectively.

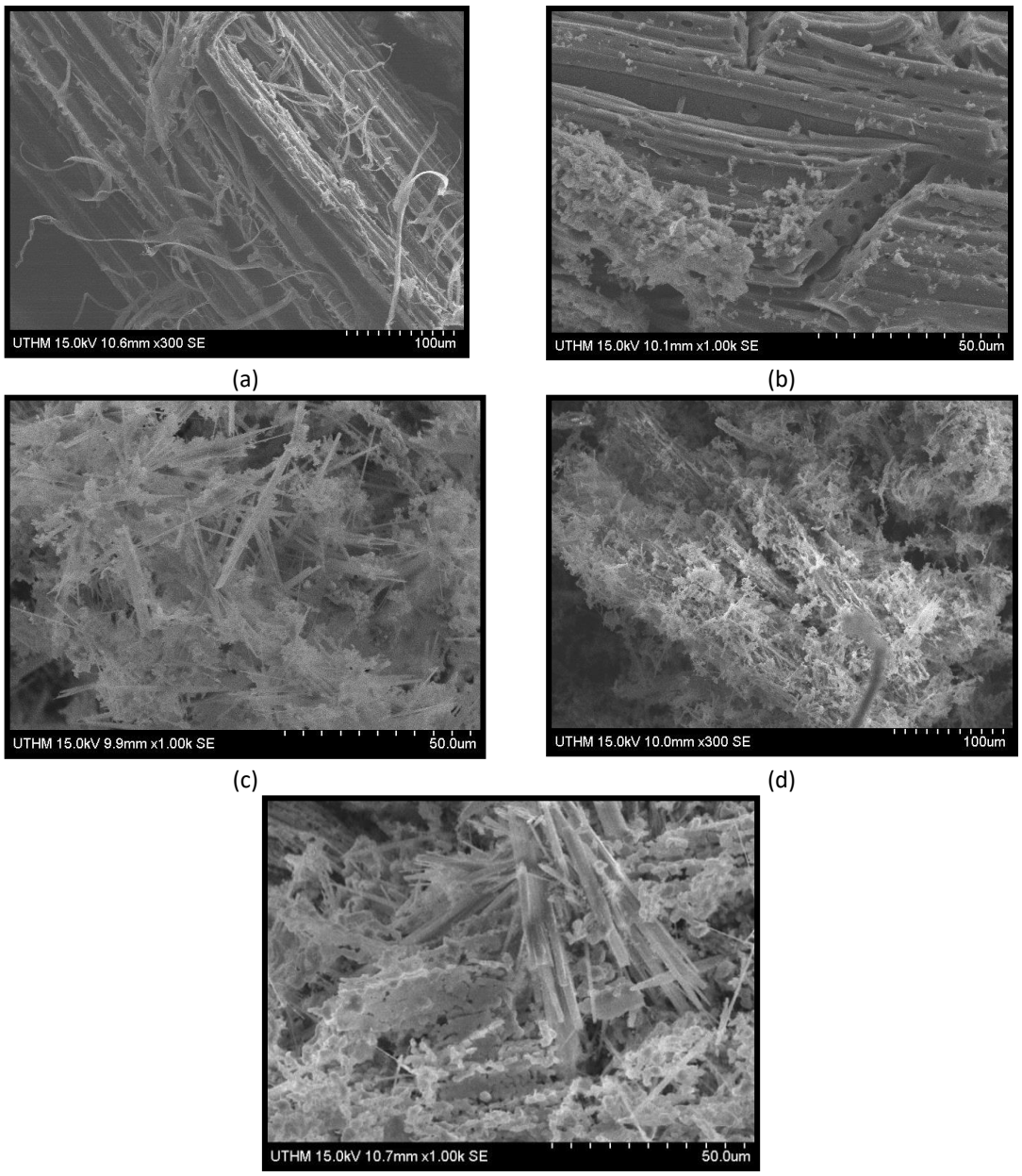


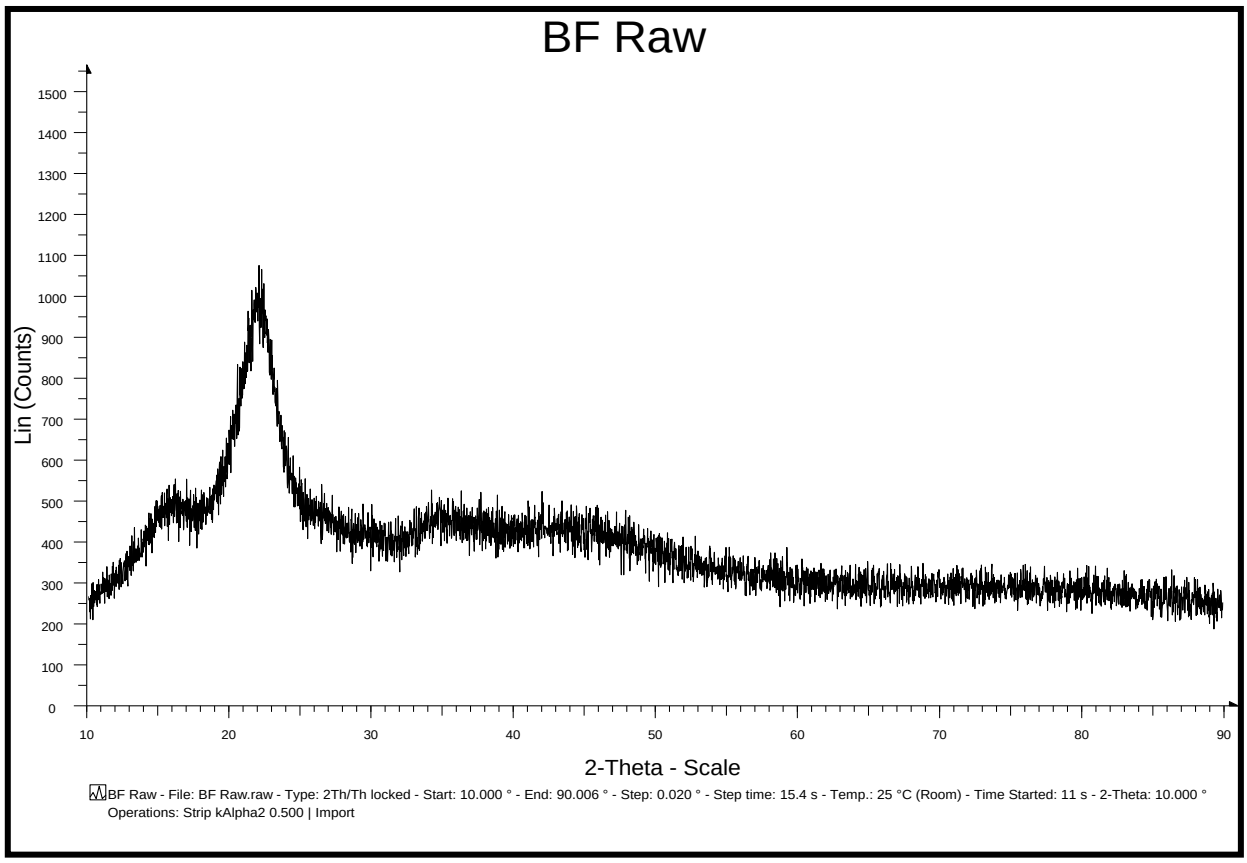
Fig. 1. SEM images of (a) BFR (b) BF-1 (c) BF-2 (d) BF-3 (e) BF-4

3.2 X-Ray Diffractometer (XRD)

Figures 2 show the pattern performed by XRD for natural bamboo fibre with various NaOH concentrations and thermal treatment. The important peak position of amorphous and crystalline parts of cellulose, and their corresponding crystallinity index values were analysed. Figure 2(a) indicated by single diffuse broad peak at about 22^{o} and this proved that the sample was at amorphous state. Figure 2(b) showed nearly the same pattern with broad peak of 22^{o} to 45^{o}. This is happened because there is no coalition of inclination of crystallites index along the fibre. Additionally, the non-crystalline part of cellulose, as well as the existence of amorphous chemicals in the fibre (lignins, pectins, and hemicelluloses), may be related to the low intensity of the peaks found in the diffractogram of the untreated fibres [53].

Figure 2(c), (d) and (e) show the pattern lead in the middle to the phase transition from amorphous state to crystalline state. The broad peaks showed the broad peaks in a range of 27^{o} to

56^{o}. The peaks indicated above were linked to the normal cellulose I lattice with the crystallographic plane or semi-crystalline structure. Both peaks corresponded to the distinctive peaks of cellulose, which was consistent with the information previously published on lignocellulosic fibre [45,50,51]. Meanwhile, the crystallinity index may improve with high temperature and high alkali concentration which could be correlated with bamboo delignification that produced microfibril with greater crystallinity [44]. The amount of cellulose in fibre bundles tended to rise from the inner to the outer portions, and the angle of the cellulose micro fibrils and the crystallinity also tended to fluctuate according to a certain rule on the radial direction of the bamboo stem [49]. According to the XRD data, alkali treatment can successfully eliminate the bamboo's amorphous component, leading to a high content of crystalline cellulose [44]. Additionally, the production of bio composites might benefit from fibres treated with relatively high crystallinity, which can increase their mechanical strength [54].



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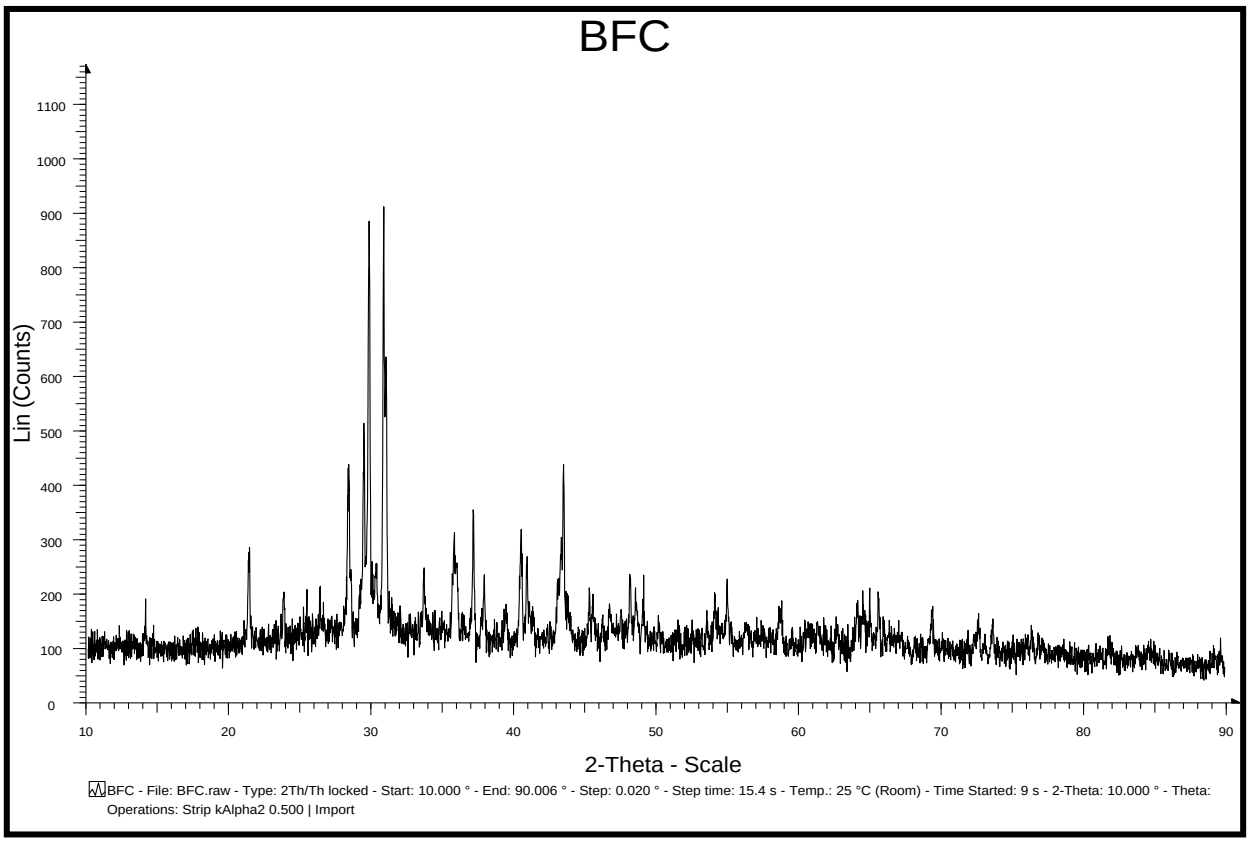
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2-Theta - Scale
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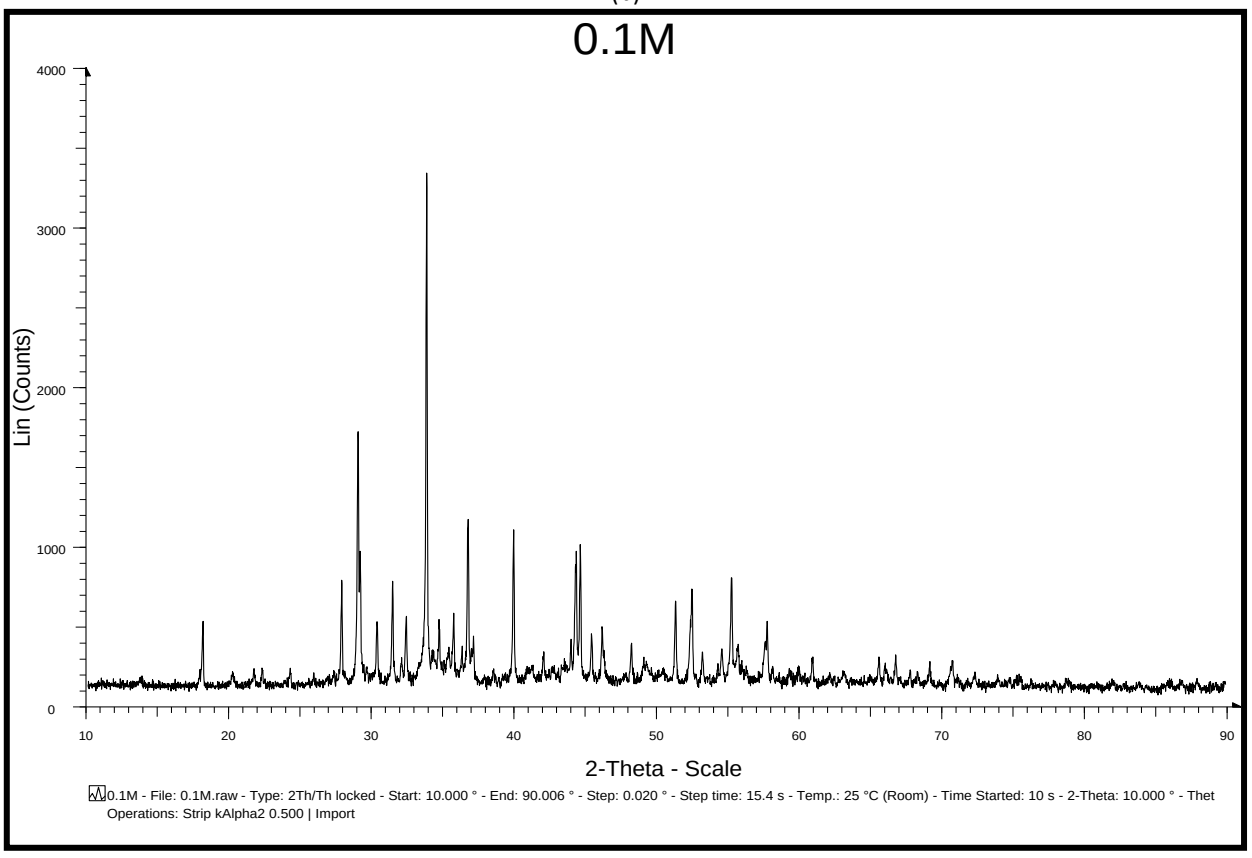
(b)


0.1M
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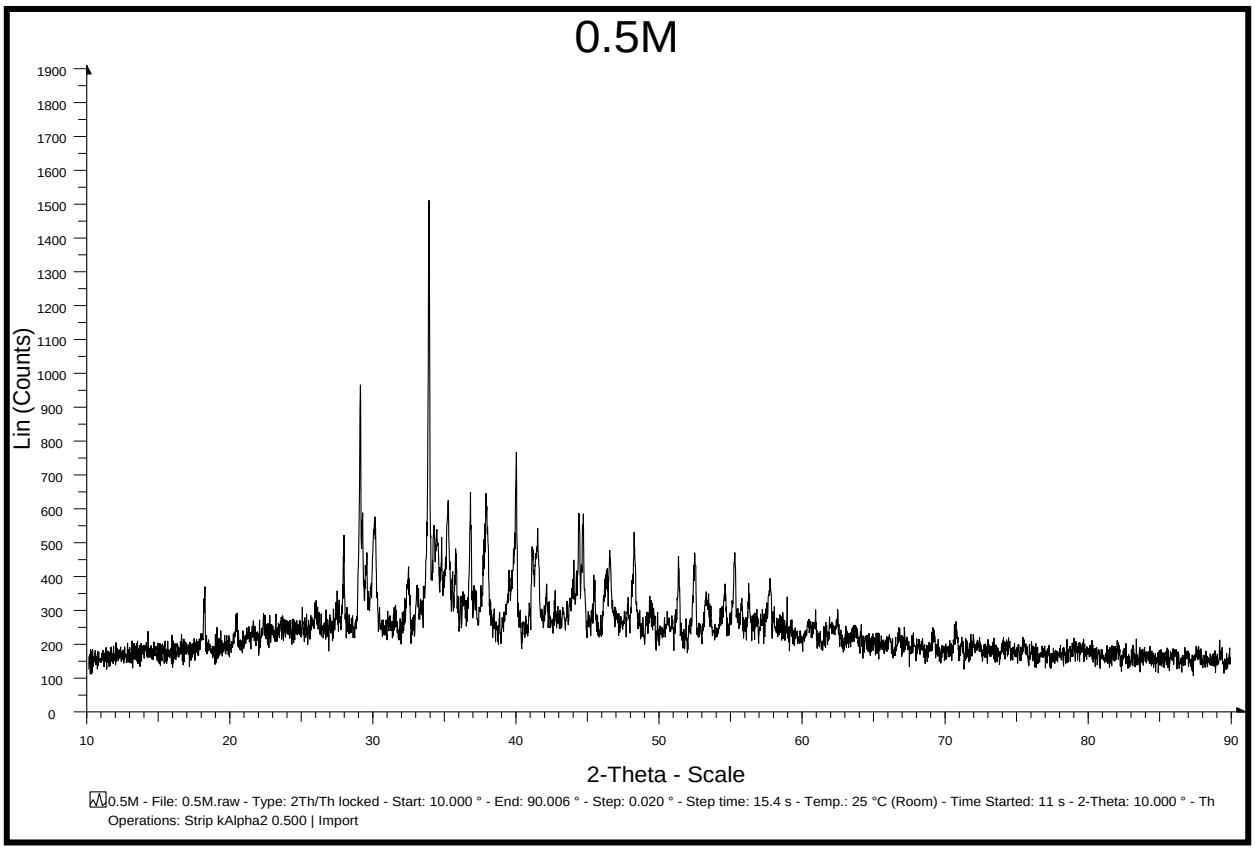
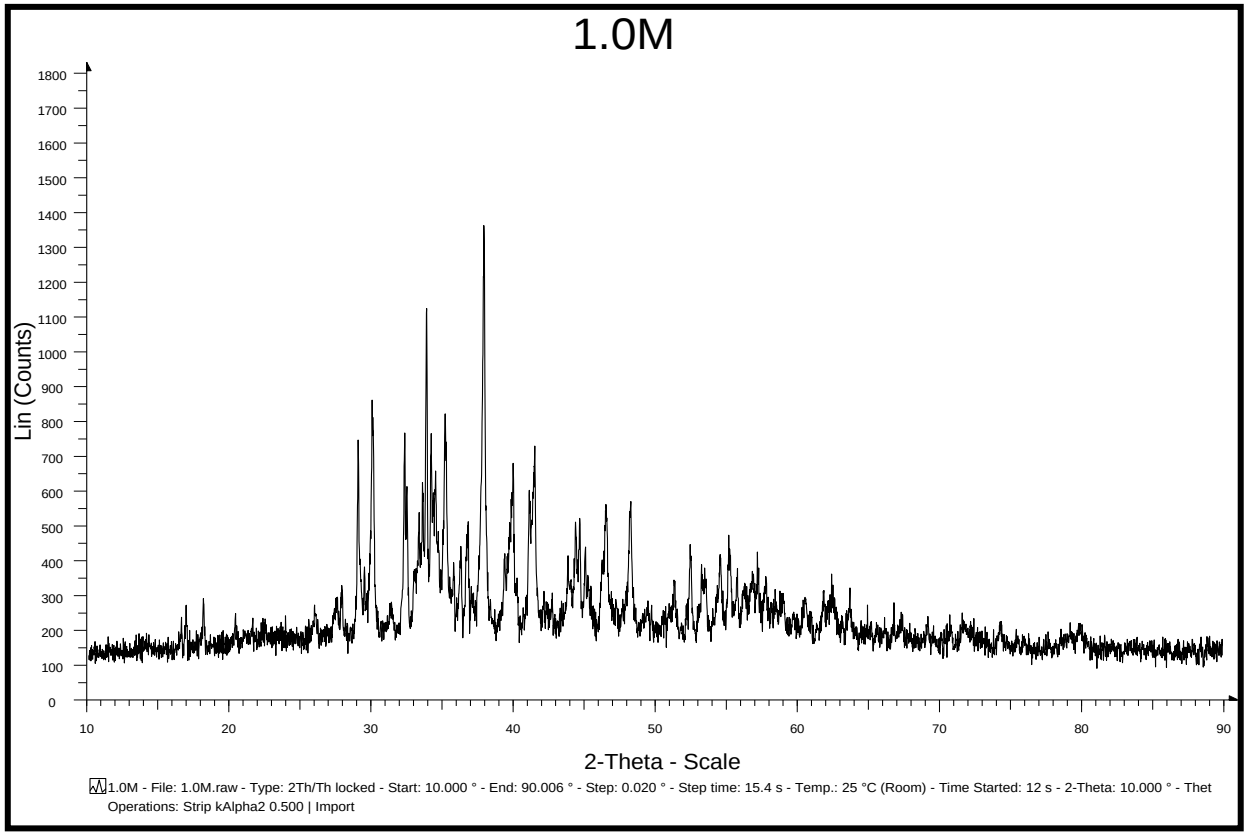
(c)



(d)



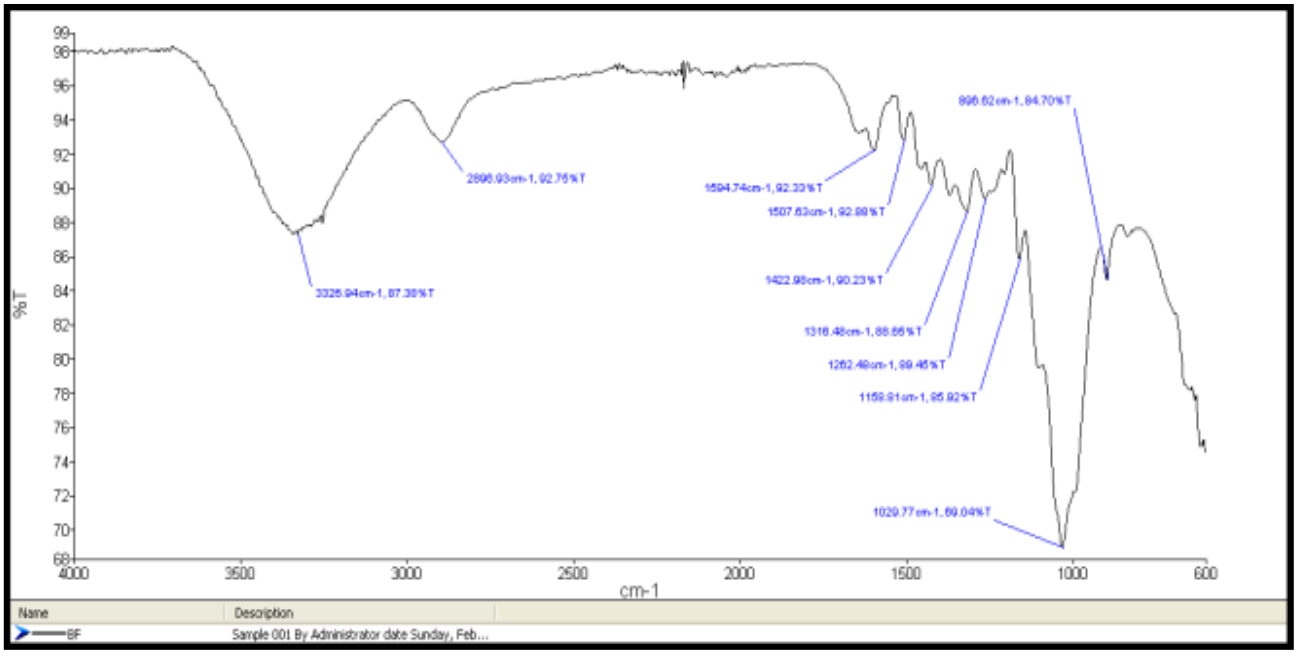
(e)

Fig. 2. XRD profiles of (a) BFR (b) BF-1 (c) BF-2 (d) BF-3 (e) BF-4

3.3.3 Fourier transform infrared spectroscopy (FTIR)

By Figures 3 show the FTIR spectra of natural bamboo fibre particles. Generally, FTIR was carried out to study the changes in structure behaviour in the material and to justify the material is pure cellulose. From the analysis, Figure 3(a) showed a strong band can be seen at 3326.94 cm^{-1}, which indicates an O-H stretching vibrations. This shows the reflectance of hydrophilic property [55,56]. The next peak was approximately 2896.93 cm^{-1}. This value represents the C-H stretching vibrations [57]. The stretching vibration of C-C can be seen when the peak at 1594.74 cm^{-1} verified the vibrations of the ester linkages in hemicellulose and lignin present in bamboo fibre [58]. The additional broad peak can be seen at 1029.77 cm^{-1}. This result is related to C-OH stretching vibrations, indicating the higher quality presence of cellulose [59]. Figure 3(b) approximately presents the strong bond at 2959.63 cm^{-1}. This condition indicates the O-H stretching which still contributes to the existence of lignin and cellulose though there is no chemical treatment. This shows the phase is still in an amorphous state. The broad slightly shifted to a low intensity where the peak was at 1402.70cm^{-1} which in this show the high presence of exposure of carbon and decreasing of impurities quantity.

Figure 3(c) was in the spectra of treated fibre where the vibration peak was at 1726.12 cm^{-1}. It showed the attribution of the stretching vibrations of unconjugated carbonyl groups C=O. Then the spectrum slightly changed to 629.99 cm^{-1}. This value represents the C-H stretching vibrations. The reduction could be related to the elimination of hemicellulose and lignin. Strong absorbance peaks in Figure 3(d) with a broad peak of 1028.21 cm^{-1}. This shows the sample of BF-3 was still attributed to C-O-C vibrations stretching. This result was supported by M. Rasheed et *al.,* where the asymmetric bending vibration referred to CH_3 [60]. The pattern then decreased and the spectra reduced at the peak of 871.61 cm^{-1}. A similar finding in Figure 3(e), the strong peak was seen peak at 1435.59cm^{-1} and highly reduced to 712.35 cm^{-1}. The increase progressively was along of fibre volume fraction. This situation had put these two samples at the absorption of C-H bending.



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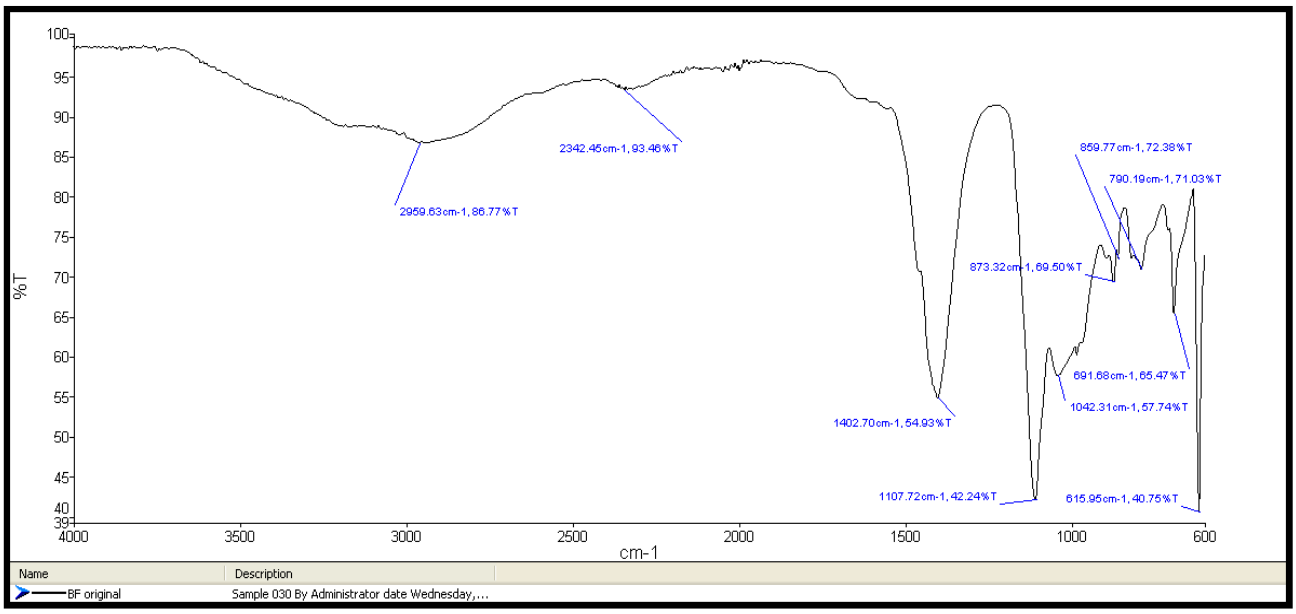
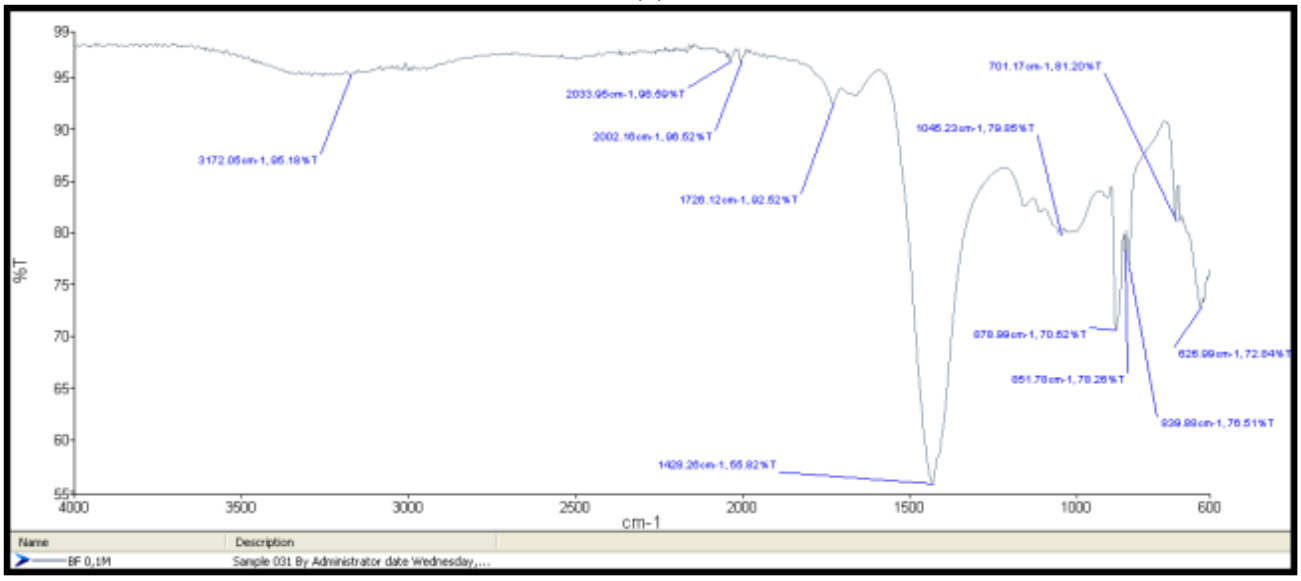

%T
cm-1
2959.63cm-1, 86.77%T
2342.45cm-1, 93.46%T
1402.70cm-1, 54.93%T
1107.72cm-1, 42.24%T
873.32cm-1, 69.50%T
859.77cm-1, 72.38%T
790.19cm-1, 71.03%T
691.68cm-1, 65.47%T
1042.31cm-1, 57.74%T
615.95cm-1, 40.75%T
Name
Description
BF original
Sample 030 By Administrator date Wednesday,...

(b)


%T
cm-1
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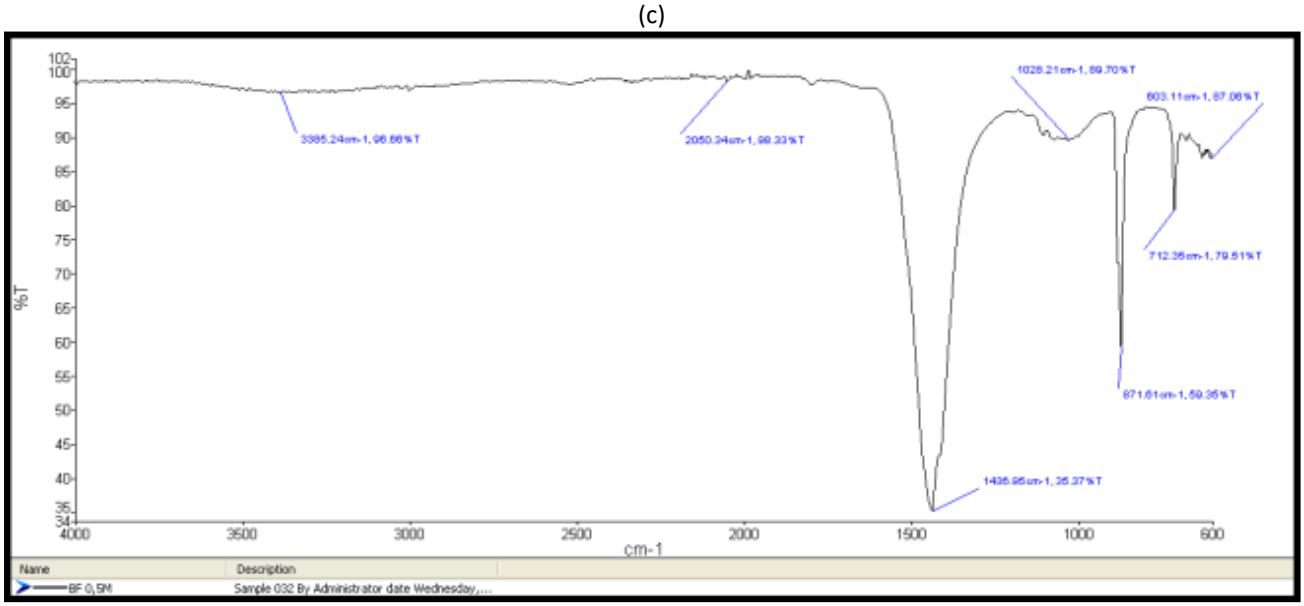
2033.95cm-1, 95.59%T
2002.16cm-1, 96.62%T
1726.12cm-1, 92.62%T
1428.26cm-1, 65.82%T
1045.23cm-1, 79.85%T
701.17cm-1, 81.20%T
678.99cm-1, 70.82%T
851.78cm-1, 78.26%T
626.99cm-1, 72.84%T
929.89cm-1, 76.51%T
Name
Description
BF 0,1M
Sample 031 By Administrator date Wednesday,...

(c)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1436.96cm-1, 35.37%T
1028.21cm-1, 89.70%T
803.11cm-1, 87.06%T
712.36cm-1, 79.51%T
871.61cm-1, 59.35%T
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Description
BF 0,5M
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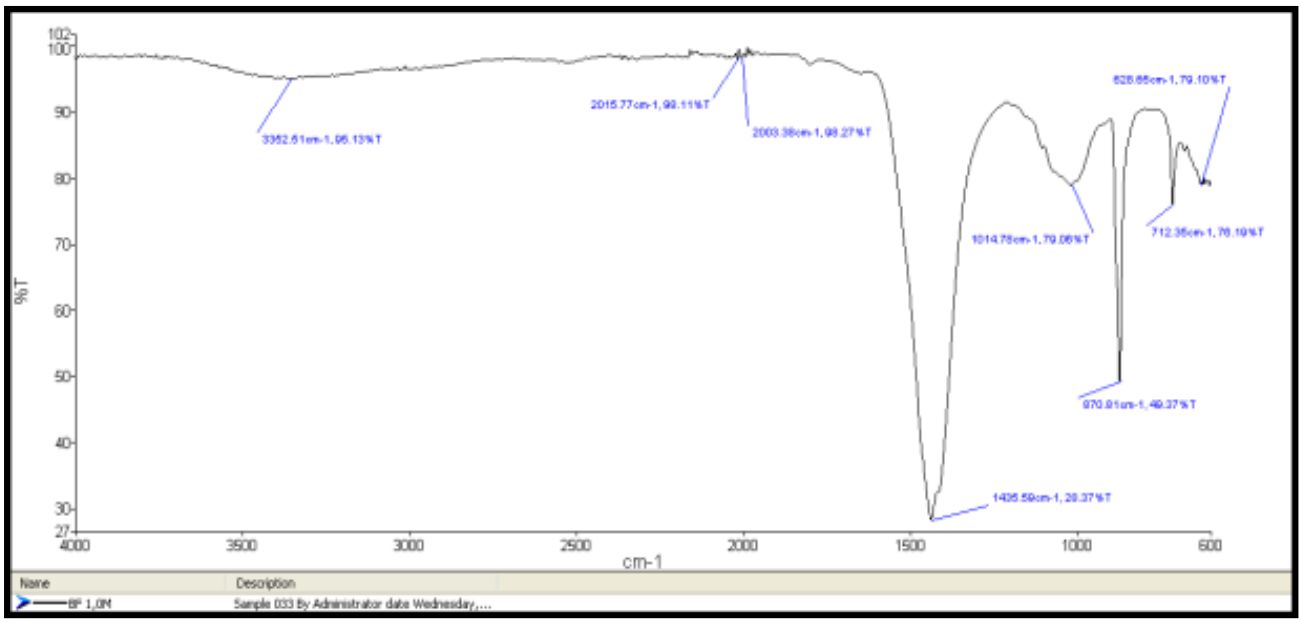
(d)



(e)

Fig. 3. FTIR spectra of (a) BFR (b) BF-1 (c) BF-2 (d) BF-3 (e) BF-4

4. Conclusions

In conclusion, bamboo naturally develops a multilayer structure from cellulose microfibrils with lignin and hemicellulose acting as an amorphous matrix. Impurities that were present in the fibre wall were able to alter under the impact of heat treatment and chemical alkali treatment. This adjustment has a significant influence and can aid in the assessment of physical, mechanical, biological, and morphological behaviour. This study shows that these two treatments can change the strength of absorption bands while producing robust cellulosic fibres with a high index of crystallinity and a smooth fibre surface. The results from SEM, XRD and FTIR from this study prove it respectively. Finally, with further in-depth research on the functional and nutritional qualities of the active components of bamboo, as well as ongoing development of extraction technology, effective chemical compositions of bamboo leaf will be more widely used in more fields. The development and usage of bamboo leaf resources has the potential to turn waste into treasure and increase the economic benefit of forestry production [61].

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