



Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) Analysis of Chemically Treated Bagasse Fibre

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Authors' contributions

This work was carried out in collaboration between both authors. Author BD designed the study. Author JTO performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors JTO and BD managed the analyses of the study. Author JTO managed the literature searches. Both authors read and approved the final manuscript.

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ABSTRACT

Bagasse is a natural fibre obtained as a by-product of sugarcane milling process. As with other natural fibres it has the drawback of being hydrophilic; this research seeks to explore the use of chemical modification on the fibre surface as a possible remedy to this. The chemicals used were sodium hydroxide, acetic acid, acrylic acid, and potassium permanganate. The treatments were carried out for 3 hours at 70°C except for sodium hydroxide treatment which was done at room temperature using 2wt% concentration. The fibre analysis revealed that the potassium permanganate treatment was most effective in reducing the OH group visible at a peak around 3400 cm⁻¹. Also the peaks showing lignin, pectin, and hemicellulose at 1250-1260 cm⁻¹, 1600-1650 cm⁻¹, and 1720-1750 cm⁻¹ respectively were also removed which were all present in the untreated fibre. The SEM studies revealed that the roughness of the fibre was imparted by the effect of the

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various chemicals to varying degrees with potassium permanganate treated fibre becoming rougher as compared to the untreated fibre and other chemically modified fibres.

Keywords: Bagasse; fibre; sugarcane; hydrophilic; chemical modification.

1. INTRODUCTION

Over the years, there has been a progressive increase in the use of natural fibres as compared to synthetic fibres particularly in the production of composites. This is as a result of several issues such as global warming, high cost of processing synthetic fibres, biodegradability, decomposition, waste management, and disposal issues [1,2]. Advantages of natural fibres include low density, low cost of production, recyclability and environmental friendliness amongst others [1,3-5]. Despite the many desired characteristics of natural fibres, they are hydrophilic which is a limitation for its full potential in composite production as it enhances the intake and retention of water, fibre pull-out, and debonding if used without treatment [2,6-8]. In order to rectify this problem, physical or chemical modification of the fibre surface is employed. Physical modification includes heat, plasma, and corona treatment, each of which involves heat and ray transmission at high temperature. The presence of OH and polar groups in cellulosic natural fibres makes them hydrophilic. Heating cellulosic fibres at high temperature causes changes in its physical and chemical properties. Previous studies have shown that thermal modification of cellulosic fibres results in removal of the lignin, pectin wax and hemicellulose as well as reduction in hydroxyl group and increase in carbon-carbon double bonds [2,8-13]. These modifications are done in order to maximize and obtain the best possible properties in the fibres for use as reinforcement or fillers. The physical methods are friendlier and cheaper but have lower efficiency than their contemporary chemical treatments [1,2].

The large amount of hydroxyl group in cellulose gives natural fibre hydrophilic properties and when used to reinforce hydrophobic matrices; the result is reduced compatibility, fibre/matrix interaction, interphase resistance and increased moisture absorption which promotes debonding in composites over time [14-16].

This research used four chemicals in its chemical modification as opposed to the usual one or two. These are: sodium hydroxide, acetic acid, acrylic acid and potassium permanganate. Of these four

chosen, sodium hydroxide is the most commonly used; several reasons accounts for this, they include; low cost, availability, ease of handling and good safety profile [3,8,17].

2. MATERIALS AND METHODS

2.1 Materials

The sugarcane was obtained from a farm in Hunkuyi village of Zaria through the plant department of the faculty of Agriculture, Ahmadu Bello University, Zaria-Kaduna state, Nigeria and was of the variety NG57-258. Bagasse fibre has the following characteristic: crystallinity between 63% and 68%, fibre fineness between 25tex and 35tex, moisture regain lies between 13% and 18%, tenacity between 12 g/tex and 18 g/tex, breaking elongation between 2.5% and 3.5%, torsional rigidity is high between 95 dyne-cm² and 330 dyne-cm² and flexural rigidity between 0.015 gcm² and 0.032 gcm² [17].

Already standardized sodium hydroxide, acetic acid, acrylic acid, and potassium permanganate were supplied by Steeve-Moore Chemicals and Laboratory Equipments Enterprise Zaria-Nigeria.

2.2 Methods

2.2.1 Fibre extraction

The juice was extracted from the sugarcane leaving the residue behind, from which the pith (inner part) was separated from the rind (outer part) manually. The rind was then subjected to hot water treatment (material:liquor ratio 1:50) at a temperature of about 90°C for 1 hour in order to remove dirt, soil, and colouring matter. This process also softened the rind making it fluffy which made it easier for the bagasse to be separated from the rind. This was followed by sun drying for 1 hour, drying at room temperature for 24 hours and finally oven drying.

This process of fibre extraction was as described by Asegakar and Joshi [17].

2.3 Fibre Treatment

The processed bagasse fibres was then divided into five portions (each weighing 2 grams), one

was left untreated designated as UTF (untreated fibre) while all the other portions were chemically treated with sodium hydroxide, acetic acid, acrylic acid, and potassium permanganate designated as SHTF, AATF, ACTF, and PPTF respectively. The choice of concentration and time for treatment were based on literature review which revealed that at this concentration, good results were obtained [1]. Other works varied between concentration, temperature, and timing [8,12,15,17].

2.3.1 Sodium hydroxide treatment

2 g of the fibres were soaked in 2wt% concentration of sodium hydroxide at ambient conditions [18,19] for 3 hours followed by thorough washing in distilled water to neutralize pH and finally drying at room temperature then oven drying for 6 hours at 50-60°C.

2.3.2 Acetic acid treatment

2 g of Bagasse fibres (portion 3) was treated with 2% acetic acid at a temperature of 50°C for 90 minutes followed by sodium hydroxide treatment to neutralize pH for 10 minutes. The fibres were then first sundried and oven dried at 50-60°C.

2.3.3 Acrylic acid treatment

The fourth portion of the fibres were soaked in 1% solution at room temperature for 3 hours; soaked in sodium hydroxide solution for neutralization then washing with distilled water, sundried and then oven dried for 6 hours at 50-60°C.

2.3.4 Potassium permanganate treatment

2wt% potassium permanganate for 3 hours at 70°C was used in treating the last portion of the fibres, this was seen to impart colour unto the fibre thus thorough washing was carried out to remove colouring matter as much as possible. The fibres were further soaked in water for 60 min to facilitate colour removal.

2.4 Testing

2.4.1 FTIR analysis (ASTM E2224-10)

Assessment of the fibres functional groups before and after the fibre treatment was carried out using Fourier Transform Infrared Spectroscopy (FTIR NICOLET iS10). This revealed the functional groups present and to

what extent they were available particularly after treatment. This is with particular reference to the hydroxyl (OH) group which enhances moisture absorption in the cellulosic fibres. The dried treated fibres were made into powdery form and mixed with KBr to form a disc which improved the transparency of the fibres.

2.4.2 SEM analysis

The morphological characteristics of fibres were studied by microscopic analysis using the Scanning Electron Microscopic (SEM, Phenom type Model: Pro X). SEM is a surface analytical technique used to generate magnified topographical images of a material surface 20x to over 100,000x. In this study, the analysis was done at a magnification of 1000x [Fig. 2].

The hair fibre to be studied is first made electrically conductive by ion sputtering with platinum. The examination is typically done along the fibre axis. SEM studies for fibres have been carried out to examine the degree of micro damage resulting from combing, brushing, bleaching, perming, and heating from curling ions, UV exposure, and other chemical treatments.

The SEM uses a focused beam of high energy electrons to generate a variety of signals at the surface of the solid specimens. The field emission cathode in the electron gun of the SEM provides narrower probing at low as well as at high electron energy, resulting in both improved spatial resolution and minimized sample charging and damage. The signals that derive from electron sample interaction reveal information about the morphology of the material making up the sample; they include, texture, chemical composition, crystalline structure, and orientation of materials making up the sample.

3. RESULTS AND DISCUSSION

3.1 FTIR Analysis

The analysis revealed that the OH group which enhances the absorption of water was reduced in all samples. Fig. 1a shows the spectrum of the untreated fibre, which reveals the peaks of all unwanted elements – OH group, pectin wax, hemicellulose, and lignin. Fig. 1b shows the spectra for Sodium hydroxide treated fibre where the peaks corresponding to vibrations of bonds typical of lignin, pectin, and hemicellulose are absent except for the OH group seen at a peak

of 3421.30 CM^{-1} . Observations from Fig. 1c (AATF), the OH peak at 3420.98 CM^{-1} , the pectin peak at 1734.30 CM^{-1} is also present, a peak at 1636.59 CM^{-1} indicates the presence of hemicellulose and a peak at 1259.34 CM^{-1} shows the presence of lignin. This result will definitely affect its use in a composite as it can still absorb water. Acetic acid have been used previously for fibre modification and a good result was obtain, its negative result here may be due to the fibre type (bagasse) and furthermore the variety of the fibre used [8,19,20].

Table 1 shows the wavenumbers in which peaks were observed and their respective functional groups representative in each sample.

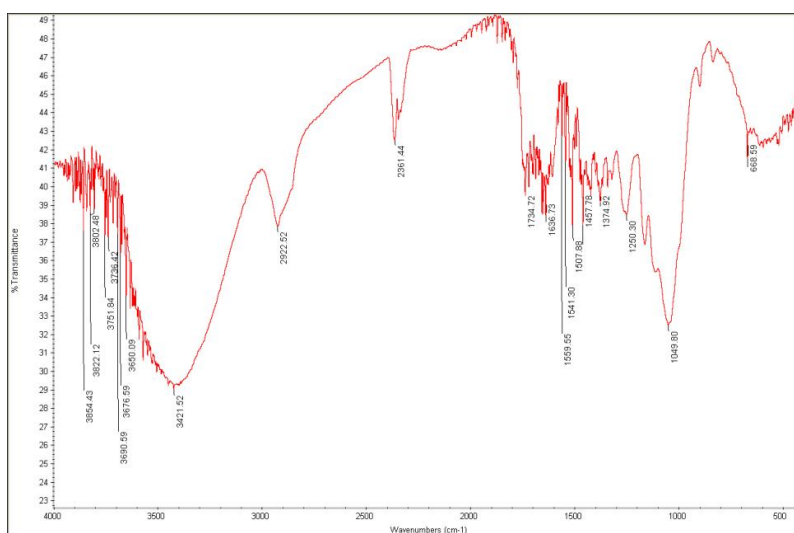
From Fig. 1d, we see that acrylic acid has its OH peak visible at 3421.58 CM^{-1} and lignin peak at

1254.59 CM^{-1} , but there no peak showing pectin and hemicellulose which indicates their absence. It is observed that on the potassium treated fibre all the peaks were removed (Fig. 1b). This shows that the potassium permanganate treatment had very positive effect on the fibre and is thus promising for use as reinforcement in Fibre-Reinforced Polymer (FRP) composite.

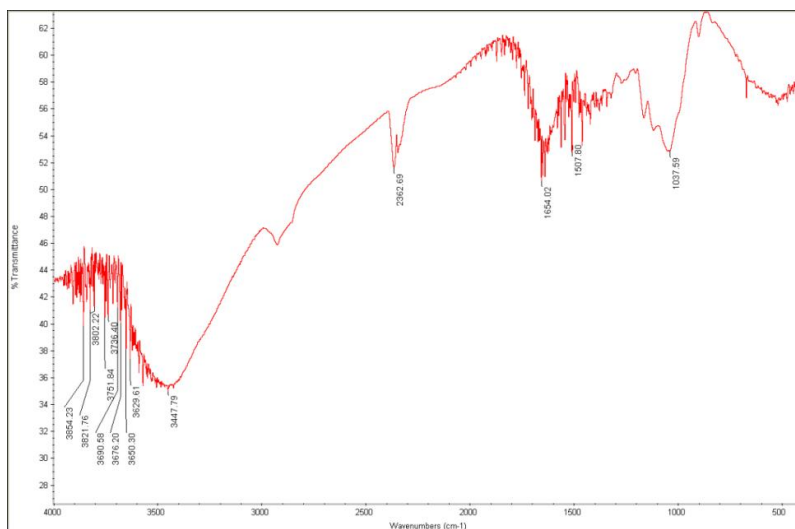
However, looking at Fig. 1f, a peak around 3629.48 CM^{-1} is visible indicating the presence of water from the hygroscopic KBr. Thus it could be inferred that the OH peaks as seen in the treated samples was influenced by unavoidable experimental errors. However, all these were found in the untreated fibre with lignin peak at 1250.30 CM^{-1} . Pectin peak at 1734.72 CM^{-1} , hemicellulose at 1636 CM^{-1} , and OH group at 3421.52 CM^{-1} .

Table 1. summarized wavenumbers for various functional groups on the samples

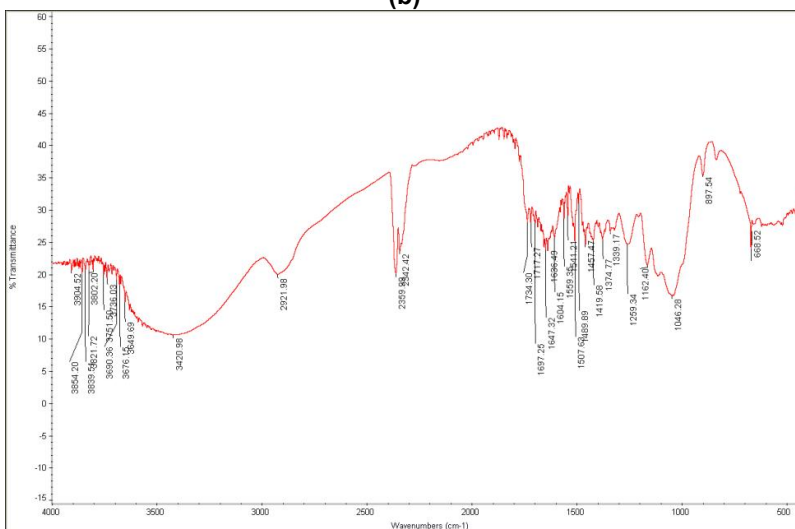
Band wavenumber (cm^{-1})	Associated chemical group	Untreated bagasse	Sodium hydroxide treated bagasse	Acetic acid treated bagasse	Potassium permanganate treated bagasse	Acrylic acid treated bagasse
3200- 3650	-OH stretching	Dominant	Low	Low	Very low	Low
1250-1260	Lignin peak	Predominant	Removed	Reduced	Removed	Quite dominant
1600-1650	Hemicelulose peak	Predominant	Removed	Reduced	removed	No change
1720-1750	Pectin wax	Present	Removed	No change	Removed	Removed



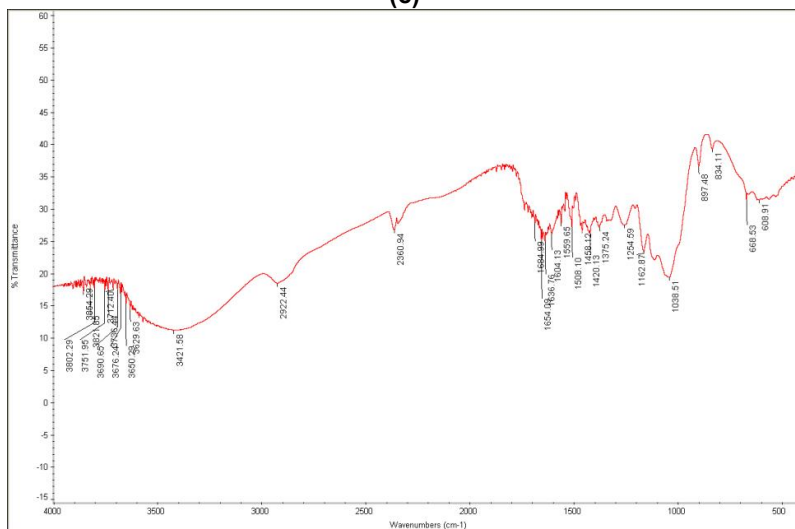
(a)



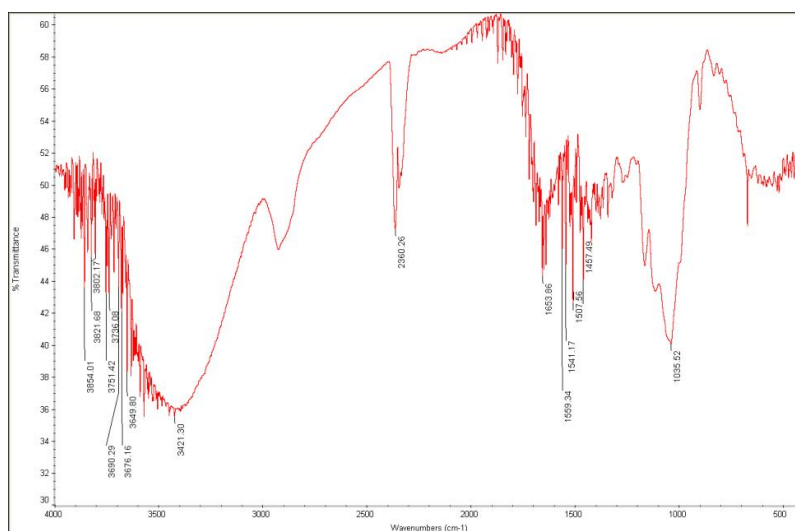
(b)



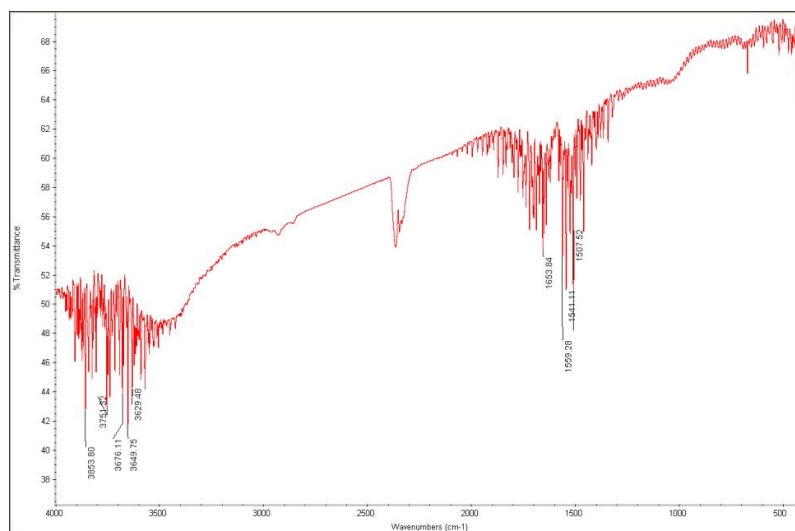
(c)



(d)



(e)



(f)

Fig. 1. FTIR spectra of (a) UTF, (b) PPTF, (c) AATF, (d)ACTF, (e) SHTF, and (f) KBr

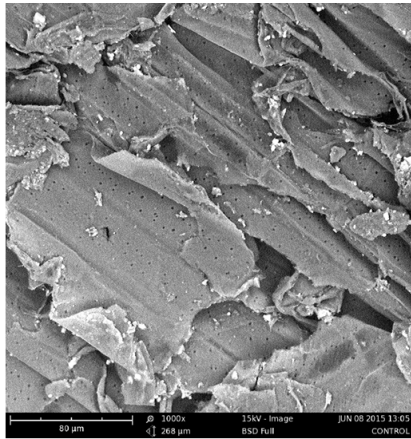
3.2 SEM Analysis

This reveals the changes that have occurred within the fibre morphology with respect to roughness, alignment, and flake removal due to the chemical effect. Using Scanning Electron Microscope (SEM), back scattered images were obtained at a magnification of 1000x as shown in the plates in Fig. 2.

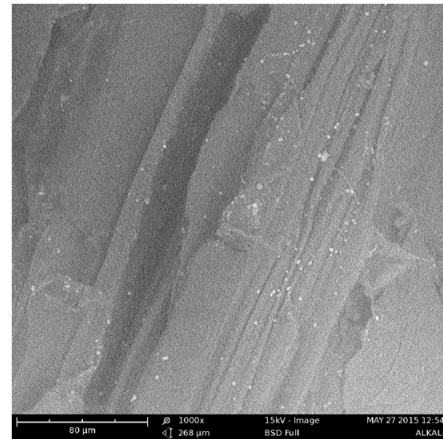
Fig. 2a shows a micrograph for the untreated fibre which is seen to be rough on the surface even before treatment. This is most likely due to the rigorous process of preparing the fibres as stated by previous works [9,19], the however the

alignment required is not evident. In Fig. 2b the fibres were seen to have become aligned but it is smoother than the UTF (Fig. 2a).

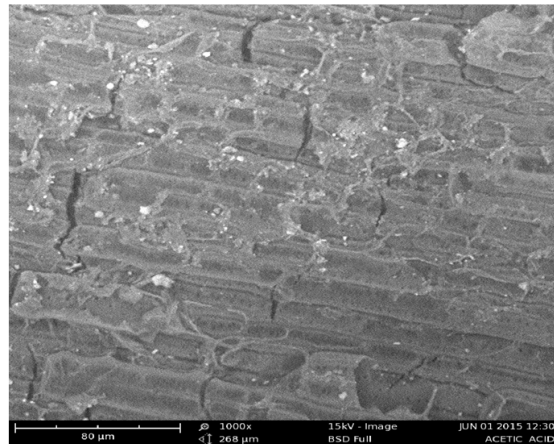
In Fig. 2c, the flake-like (the dominant white thin-like patches) structure indicating the presence of waxy substance is still visible, while in ACTF (Fig. 2d) cracks can be seen which may be due to air spaces or voids within the fibre created during pretreatment [9,18,19]. However, in Fig. 2e (PPTF) there is an adequate combination of alignment and roughness which is good for compatibility in composites. The fibres also got split after chemical treatment. [20].



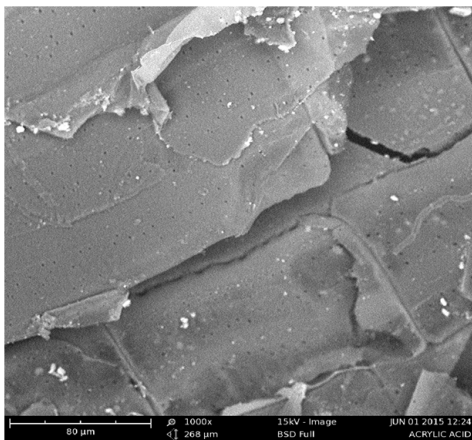
(a)



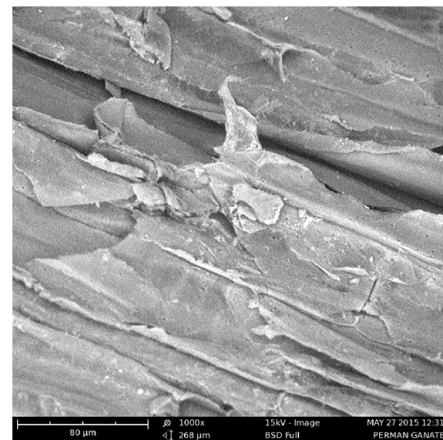
(b)



(c)



(d)



(e)

Fig. 2. SEM micrograph of (a) UTF, (b) SHTF, (c) AATF, (d) ACTF, and (e) PPTF at magnifications of 1000x

4. CONCLUSION

From the results and analysis above, Potassium Permanganate succeeded in removing more polar groups (OH) and other waxy substances such as pectin, hemicellulose and lignin responsible for the fibre hydrophilic nature than any other chemical used in the study. A good combination of roughness and alignment which is vital for compatibility and adhesion when fibres are used in composites was observed on the potassium permanganate treated fibre. However, others (that is, the sodium hydroxide, acetic acid, and acrylic acid treated fibres) could be used as fillers particularly in applications where strength is not of great importance.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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