



EXTRACTION AND CHARACTERIZATION OF CELLULOSE FROM AGRICULTURAL BIOMASS (SUGARCANE BAGASSE)

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ABSTRACT

The surplus in the amount of agricultural wastes leftover on the farmlands found in Song, Gombi, Mubi north, Mubi south, Michika, and Madagali local government areas of Adamawa state after each harvest season and the strong drive for sustainable resource utilization and alternative biodegradable and renewable packaging polymeric materials are the motivations to conduct this research study. Agricultural biomass management has been considered to be a vital strategy in the journey to accomplish natural resource management and utilization as well as in the maintenance of the quality of the environment. Agricultural residues like sugarcane bagasse, woodchips, sawdusts, rice husks, cotton linters, e.t.c can serve as sustainable, renewable, and inexpensive raw materials for the production of cellulose and its derivatives for the textile industry, bio-plastics, biomedical devices and in drug delivery as well as in agricultural mulch films. Cellulose was extracted from sugarcane bagasse by the alkali pre-treatment method with Sodium hydroxide (NaOH) at temperature range of 60°- 120°C and time duration of 1 hour to 2hours. This was followed by bleaching with 5% NaOCl and pH adjustment with 1% Hydrochloric acid and then washed with distilled water and dried in a hot air oven at 105°C until constant weight. The extracted cellulose was confirmed by X-ray diffraction and Fourier transform infra-red spectroscopy (FTIR) respectively. The cellulose yield for treated bagasse fibre was found to be 89.55±0.63%, hemicellulose as 6.14±0.81%, and lignin was 2.64±0.65%. Morphological analysis revealed that the average diameter of the cellulose was 12.03µm. The XRD diffraction showed that the untreated sugarcane bagasse has a crystallinity index of 31.76%, while the treated fibre (cellulose) has a value of 51.13%. FTIR spectra showed

a range of $3530\text{--}3050\text{cm}^{-1}$ which is ascribed to the free OH stretching vibration of the –OH group in the inter-molecular and intra-molecular hydrogen bonds in the cellulose molecules. The applied procedures was successful for extracting cellulose from sugarcane bagasse. The cellulose will be highly useful for the acetylation process to convert it to cellulose acetate (CA) for use as polymeric plastic, a modified derivative in drug delivery, reinforcement in biopolymer composites and for textile and fabric manufacturing.

Keywords: Cellulose, Sugarcane Bagasse, Biomass, Lignin, Biopolymer.

Introduction

Agricultural waste estimates are scarce, although it is widely assumed that it contributes an outsized share of total waste products within the developed world and even in the third world countries, it is an alarming figure in terms of statistics and data, (Saad et al, 2022). Increased quantities of agricultural crop wastes and agro- industrial by-products have naturally emerged from increased agricultural productivity. Globally, the rapid increase in agricultural wastes amounts has motivated many researchers and scientists to channel their energy and their studies towards using renewable natural fibre reinforced materials in a bid to reduce reliance on petroleum based polymeric materials. (Saad et al, 2022). The numerous distinguishing advantages of natural fibres over traditional ones include reduced dependence on non-renewable energy / materials sources, fewer pollutants, and greenhouse emissions as well as hlow cost, high toughness, low density, good specific strength properties, enhanced energy recovery and biodegradability (Saad et al, 2022; Thomas et al, 2021). Currently, there is an environmental pollution problem arising in part by packaging materials produced from non-biodegradable synthetic polymers made from petroleum. Nowadays, the potent desire for environment – friendly materials has encouraged the development of an industry (biomaterials) and the use of biopolymer for various applications (Maya and Sabu, 2008). Degradable polymers (plastics) have long been considered part of the solution to the environmental and waste management problems posed by extensive use of non- degradable polymeric materials such as polyethylene, polypropylene, polystyrene, whose raw materials are derived from petroleum resources. Therefore biodegradable polymers can make significant contributions to materials recovery and utilization of renewable resources (Davis & Song, 2006). Biopolymers from renewable materials are being preferred recently due to their safety, low production costs, and biodegradability. The development of biodegradable plastics from agricultural biomass has received more widespread attention particularly in EU countries where various sponsored researches under the EU horizon Pillar 2 project is currently ongoing. For example, the USA department of Agriculture’s bio-preferred program took the important step in promoting bio-plastics at the Federal procurement level (Mostafa et.al, 2015). In 2012, the two most influential commercial biodegradable and bio-

based polymers were polylactic acid (PLA) and starch-based polymers accounting respectively for 47% and 41% of total biodegradable polymer consumption (Petrova & Garner, 2014). However, despite the above developments, commercial bio-based plastics are still expensive and therefore there is need to research upon cheap alternatives that are also environment friendly. Various solutions employed to resolve plastic waste problems include recycling, re-use, incineration and biodegradation via biotechnology. Recycling of commercial plastics among other solutions is an important contemporary economic issue as regards the need to reduce the volume of plastics wastes and conserve the non –renewable petroleum resources from which most plastic raw materials are derived. Although the volume of plastics that are recycled is rapidly increasing each year, only less than 3% of all plastics are now recycled compared to about 20% of glass, 29% of paper, 16% of steel and 39% of aluminium. Part of the reason for this low recycling rate is that the total costs of producing recycled plastics including collecting and recycling is typically 20% higher than the cost of producing virgin polymeric plastics,(Fried,2005). An eco-friendly plastic materials based on cellulose acetate and its derivatives are suggested alternatives for food packaging applications, biomedical as well as in agricultural applications like mulching films due to its potential for bacterial degradation when buried under the soil. Cellulose acetate (CA) plastic and its derivatives can be used for producing ultra-filtration membranes, fibres, and some plastics tools (Cosimo,2013). Cellulose acetate has application in producing textile fibres for fabric production as well in polymeric composite reinforcement. Natural plastic like cellulose acetate is usually produced in a fluid form and therefore it is shaped easily and does not require a large amount of energy. This is to be compared with the conventional thermoplastics which is stored as granules and needs a massive amount of energy in order to be shaped by moulding, injection, or extrusion (Xiaoyun and Shuwen,2013). Due to the fact that raw materials have a high impact on the cost of bio-based plastics production, the use of low cost cellulosic raw materials such as sugarcane bagasse, wood chips, sawdusts, rice husks, corn cobs, flax, jute, cotton linters,e.t.c has become attractive therefore for industrial cellulose acetate(CA) production.

Sugarcane bagasse (SCB). Sugarcane bagasse is a fibrous material containing cellulose as its major component. It is produced in large quantities across the world especially in Africa, and South America (Brazil, Argentina, Colombia). Every year, there are massive quantities of sugarcane bagasse wastes left after the crushing process of sugarcane used for juice extraction and in the alcohol industry(Saad et al,2022). Sugarcane bagasse is used as a fuel for the sugar mill in the process of sugar production (e.g Dangote Sugar factory, Numan) as well as a raw material for electric power generation, fermented products, and paper production. More so various mechanical and chemical treatments has been developed to help extract cellulose fibres ,pure cellulose, cellulose nano-fibres, and cellulose nano-crystals from sugarcane bagasse(Mahmud & Anannya,2021). These extracted materials have diverse applications in

regenerated cellulose fibre and composite material production. Sugarcane (*Saccharum officinarum*) is cultivated in considerable quantities in tropical countries. In 2017, close to 1.84 billion tons of sugarcane were produced worldwide (FAOSTAT, 2018). It is used in sugar mills and alcohol mills in Brazil and Nigeria but it cannot be consumed entirely by those mills as about 30% pulpy fibrous residue is produced after being utilized in those mills (Michel et al, 2013., Chandel et al, 2012). These residues are called bagasse and is used in various applications including paper industries, as feedstock, and in biofuel production. Sugarcane bagasse is a ligno-cellulosic material and a form of agricultural waste product from which cellulose can be extracted for textile production. More specifically, this bagasse can also be used to reinforce polymer composites for creating totally new type of materials (Mahmud & Anannya, 2021).

Bagasse actually contains two types of fibres, the outside rind and inner pith. The outside rind portion contains longer and finer bundles of fibres, while the inner portion contain short fibres. Cellulose covers about one-third of the plant tissues of sugarcane. Raw unprocessed sugarcane bagasse (SCB) contains about 40-50% cellulose, and 25-35% hemicellulose. The rest contain lignin, wax, and ash (Mahmud & Anannya, 2021). Several methods can be employed to extract cellulose from ligno-cellulosic biomass materials. However, in this research study, alkali pre-treatment was adopted in which the temperature of the solution, duration time of contact between Sodium hydroxide and the SCB powder and the concentration of the alkali solution are considered to be the three most significant parameters that can potentially affect the yield of cellulose extracted from sugarcane bagasse.

Materials & Methods

Materials

Cellulose could be extracted successfully from many lignocellulosic materials such as cotton, rice husks, saw dusts, corn cob, sugarcane bagasse, e.t.c. In this study, the investigated cellulose was extracted from raw sugarcane bagasse from local farms, sugarcane vendors in Mubi and nearby towns and villages. These raw fibres were carefully prepared and treated to extract the maximum yield of cellulose as mentioned in the next section. A selection of analytical grade chemicals such as sodium hydroxide, hydrochloric acid, acetone, methanol e.t.c were all supplied from BDH and Sigma-Aldrich chemicals.

Methodology

Desilication of sugarcane bagasse

Raw sugarcane bagasse (SBC) contains a significant amount of sand picked up from the harvesting grounds. Bagasse was thoroughly washed with plenty of water for twenty minutes and this was repeated to get rid of high silica content and any dirt present. The wet bagasse was left to dry by exposure to strong sunlight for six (6) days and followed by drying in an oven for

24 hours at 90°C. Screening was performed to allow the silica to fall through the mesh. The desilicated bagasse was then subjected to the next step called milling of sugarcane bagasse(SCB).

Milling of Sugarcane bagasse

The dried sugarcane bagasse was grinded to particulate size and sieved with a mesh to get the finer particles. Bagasse fibres (before grinding) and bagasse fibres (after grinding) was labelled as seen in fig. 1 (a, b, c, & d) and fig. 2 (a, b, c & d)



(a)



(b)



(c)



(d)

Fig. 1: Samples of raw sugar cane bagasse



(a)



(b)



(c)



(d)

Fig. 2: Samples of grinded sugar cane bagasse.

Extraction of cellulose

The extraction of cellulose was carried out according to the method of Saad et al ,2022. This study used the alkali treatment method which used 0.5, 1.5, 3, and 5% NaOH. Hydrochloric acid with concentrations (0.2, 0.5 and 1%) necessary for neutralization was prepared. 200cm³ of NaOH solution was added to 10g of the dried milled bagasse powder. A magnetic stirrer was used to allow good mixing at an adapted temperature ranging from 60-90⁰ C at a constant stirring rate and at a different time ranging from 1-4hours. The alkaline treated samples were left to cool down at room temperature. The HCl was then added to adjust the pH range for the prepared samples from 8-8.5. The sample was filtered using filter paper and the cellulose solid residues was collected. The cellulose solid residue was left in the dryer for 12hours at 105⁰C to remove unwanted moisture until a constant weight is reached.

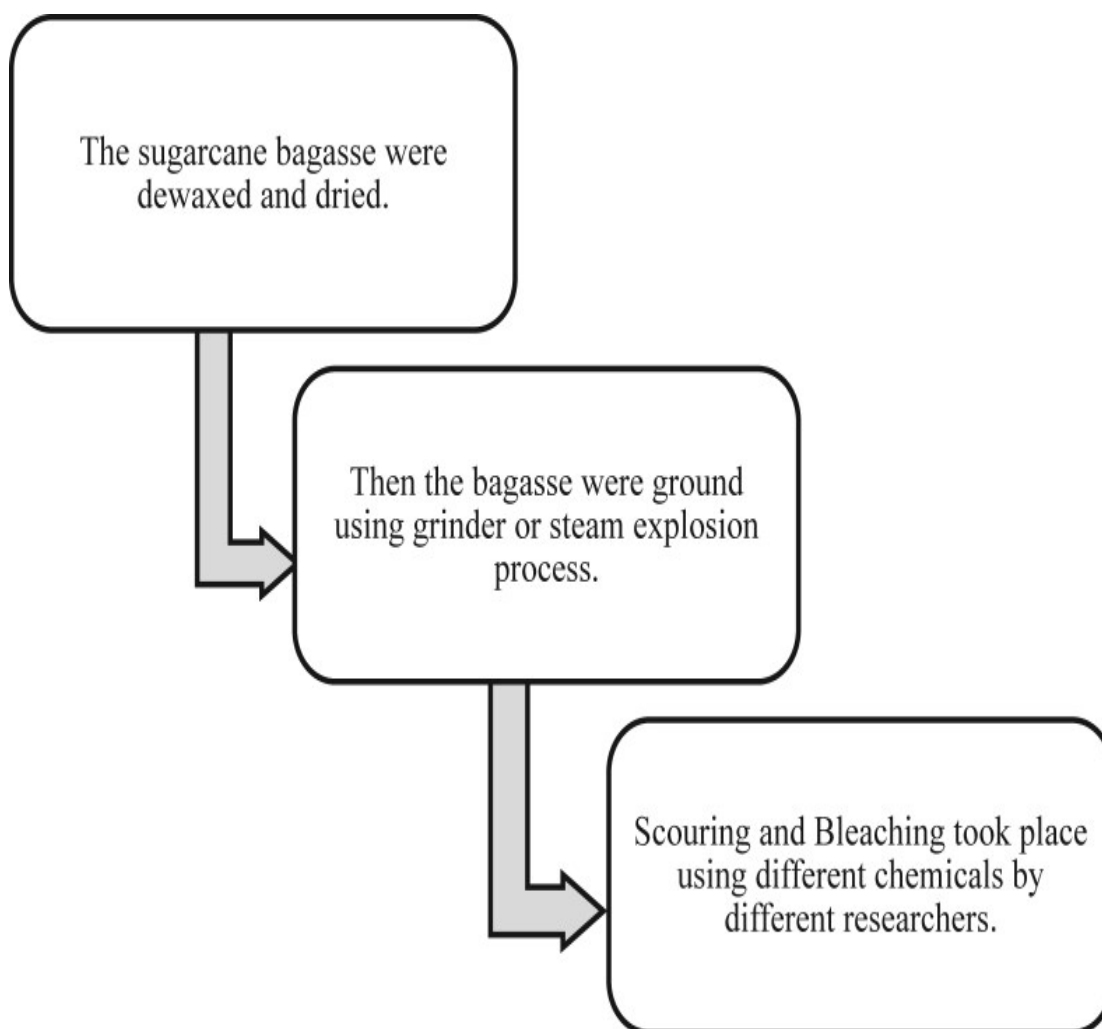


Fig. 3: Process flow diagram of cellulose extraction from sugar cane bagasse.

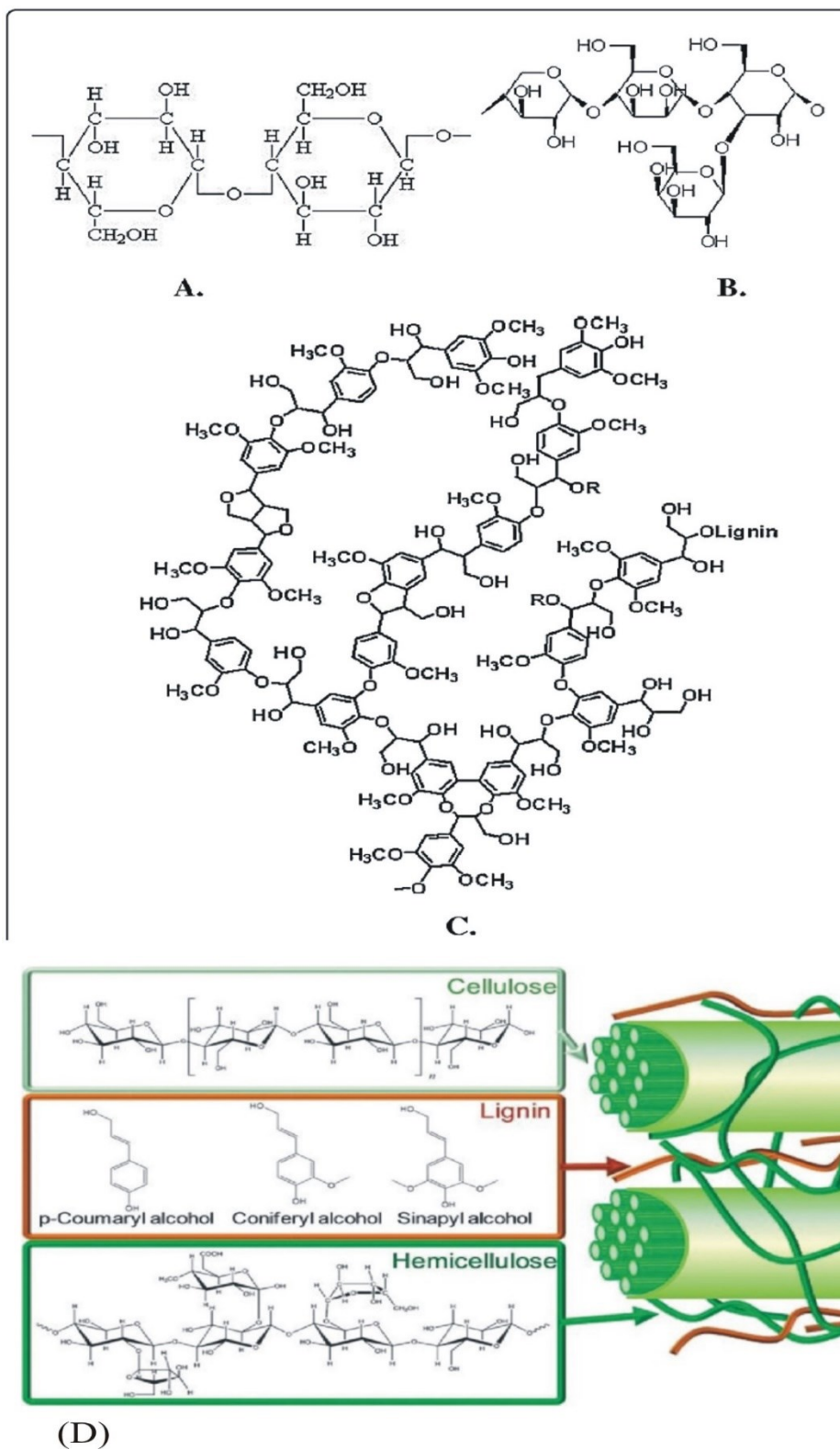


Fig. 4: Chemical structure of cellulose, hemicellulose, and lignin

Characterization of the extracted cellulose

The dried extracted cellulose was characterized by using the following analytical (physical and material) characterization techniques.

Infra-red spectroscopy (FTIR)

The raw untreated bagasse together with the extracted cellulose were dried and stored to be examined by Fourier transform infra-red analysis. The analysis was performed in the 4000-400 cm^{-1} range to determine the chemical composition of bagasse and the respective extracted cellulose by using FTIR model Nicolet IS-10 FTIR instrument with KBr disc. The peaks as given in the chart indicated that the functional groups were present in the cellulose samples. The resulting spectrum depicts molecular absorption and transmission assigning molecular fingerprints of the sample.

X-Ray Diffraction (XRD) analysis.

The XRD analysis was performed to obtain information about the crystallinity of the produced cellulose by using an X-ray diffractometer to collect (at room temperature), XRD patterns of the extracted cellulose sample. By using a Philips powder diffractometer with Cu K α radiation ($\lambda=0.154\text{nm}$), x-ray diffraction (XRD) patterns of the sample were recorded in the range $2\theta=4-80^\circ$. The instrument was operated at 40Kv and 40mA. The spectra were recorded with a 2θ step of 0.02° at a scanning rate of $2^\circ/\text{min}$.

Results and Discussion.

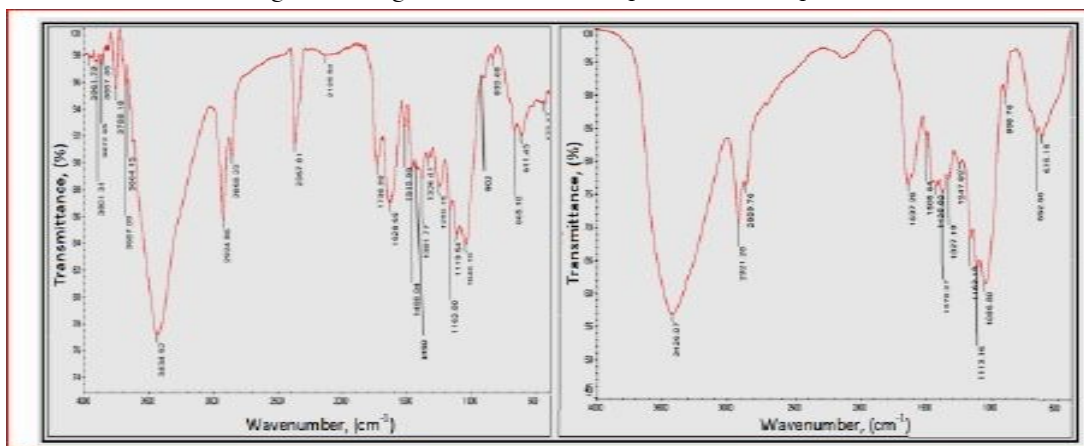
Cellulose extraction and characterization.

Cellulose was extracted from raw sugarcane bagasse by the alkali treatment method of Saad et al, 2022 and Mahmud & Anannya, 2021. Extracted cellulose was found to contain cellulose; $89.55\pm0.63\%$, hemicellulose; $6.14\pm0.81\%$, and lignin; $2.64\pm0.65\%$. Morphological analysis showed that the average diameter of the cellulose was $12.03\mu\text{m}$. The characterization of the dried raw sugarcane bagasse and the extracted cellulose was done by using the Fourier transform infra-red(FTIR) spectroscopy and X-Ray diffraction(XRD) analysis.

Fourier transform infra-red spectroscopy

The qualitative analysis of the sugarcane bagasse fibre (SCB) before and after alkali treatment was determined. Infra-red(IR) spectroscopy was used for the study of interactions between the matter(SCB) and electromagnetic fields in the IR region. The probability of a particular IR frequency being absorbed depends on the actual interaction between the frequency and the molecule. The FTIR spectroscopy was used to identify changes in functional groups and/or

molecular conformation caused by the alkali pre-treatment process. it was observed previously that the three components of the biomass in SCB, cellulose, hemi-cellulose and lignin are most likely to be composed of alkene, esters, aromatics, ketones and alcohols with various oxygen containing functional groups observed such as OH($3400-3200\text{cm}^{-1}$) and C=O($1765-1715\text{cm}^{-1}$). Through the analysis of the functional groups found in the sugarcane bagasse and the extracted cellulose, FTIR spectroscopy was used to demonstrate that lignin and hemicellulose were removed during the cellulose isolation process. The most noticeable difference in the spectra of unmodified SCB and the extracted cellulose fibres were found between 3500 and 600cm^{-1} . According to the FTIR spectrum, there are several peaks in SCB that are not found in the cellulose spectrum. The peaks are approximately 1250cm^{-1} , 1516cm^{-1} and 1736cm^{-1} . These absorptions are attributed to lignin functional groups that are associated with cellulose and hemicellulose prior to the delignification of SCB. The 1250cm^{-1} absorption peak was caused by the C-O stretching vibration of the aryl group in lignin. The C=O stretching vibration of the aromatic ring in lignin is responsible for the peak at 1516cm^{-1} . The peak at 1736cm^{-1} in the bagasse FTIR spectrum is related to the C=O stretching vibration of the carboxylic groups in lignin and hemicellulose. Figure 3 shows two significant absorption bands that must be emphasized, the bands at 1516 and 1250cm^{-1} . In the spectrum of extracted cellulose fibres obtained after the alkaline chemical treatment process, the band at 1516cm^{-1} was absent, and the band at 1250cm^{-1} was also nearly absent indicating that most of the lignin and hemicellulose were removed from sugarcane bagasse after the alkali pre-treatment process.



diffraction examinations was conducted on bagasse fibre and the extracted cellulose to analyse the changes in the crystal form and to establish the impact of alkali (NaOH) treatment on the crystallinity of the final fibres (cellulose extract). Figure 4(a) and (b) shows the XRD profile of untreated bagasse and treated fibre which reveals that cellulose is a material composed of two phases, amorphous and crystalline phases with high purity and crystallinity. The major peaks in the SCB and cellulose diffractograms are at 2 values that correspond to 15.5° and 25.5° of the cellulose 1 polymorphs in natural fibres. Results for crystallinity indicated that SCB had a lower level of crystallinity than the extracted cellulose and an increase in the crystallinity index following the extraction of cellulose from SCB was noticed in similar findings (Candido, Goday & Goncalves, 2017). More so, it was observed that the high amorphous hemicellulose and lignin contents present in the untreated materials are the main causes for their relatively lowest crystallinity index. Results of XRD studies show two distinctive peaks around 2θ value of 15.33° and 22.09° for the bagasse fibre characteristic of typical forms of cellulose 1 structure. Similarly, the extracted cellulose exhibited a distinctive cellulose type 1 pattern with clear peaks at 2θ of 16.35° and 22.47° that were significantly strengthened in comparison with that of the bagasse. Presence of these peaks in

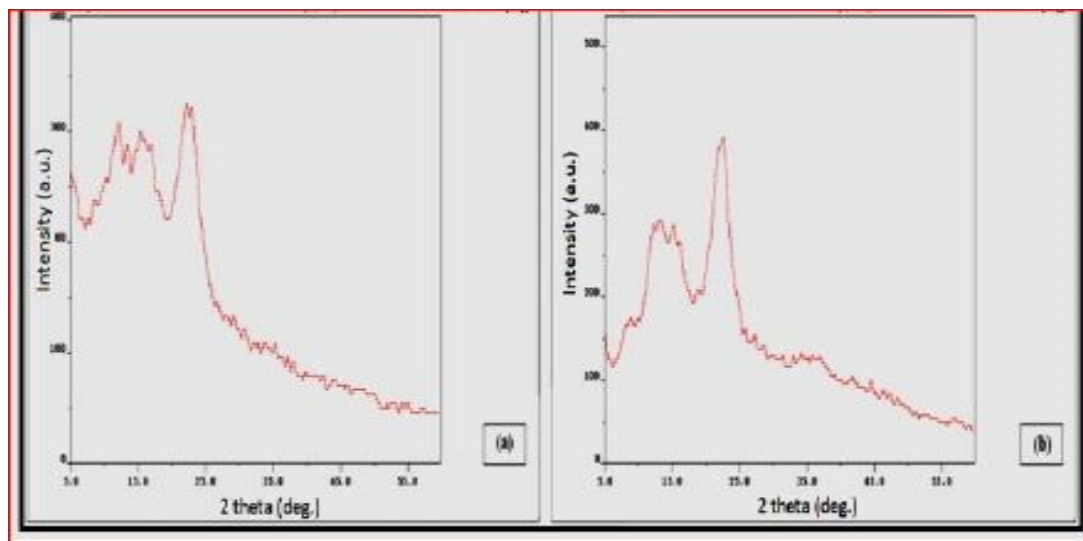


Fig. 6: X-ray diffractograms of Fibre samples: untreated bagasse (a) and extracted cellulose (b). both samples indicates that chemical structure of cellulose was not changed during the chemical treatment. It was also evident that the crystallinity is increased from 31.76% for the raw bagasse fibres to 51.13% for the extracted cellulose as in agreement with the earlier findings of the FTIR analysis. A consistent pattern showed that lignin and hemicellulose content were eliminated during the alkali treatment of the raw bagasse which caused an increase in crystallinity index by 61% in the extracted cellulose (Guilherme et al ,2015; Thomas et al 2015).

Conclusion

Cellulose was successfully extracted from sugarcane bagasse (SCB) by the alkali treatment process and was characterized by Fourier transform infra-red (FTIR) spectroscopy and X-ray diffraction (XRD) analysis. The bagasse fibre and the extracted cellulose's functional groups as well as the crystallinity index were determined. The crystallinity index of the bagasse and the extracted cellulose were 31.76% and 51.13% respectively. The results of FTIR and X-ray diffraction corroborated each other and is supported by findings from other research works. The results of the extractions and the characterizations obtained in this study shows that the natural raw bagasse fibre is a good agricultural waste (biomass) that can be a source of raw material for cellulose extraction and consequently could be valorized in a diverse industrial, textile, and engineering applications including conversion to cellulose acetate (CA) for biodegradable plastic production.

Recommendation

It is highly recommended that other types of agricultural waste (biomass) such as plant stalk/stems, wood chips, saw dusts, rice husks, corn wastes, jute fibre, cotton linters, and flax fibres could be used as feed stocks for cellulose extraction and the optimum yield determined in each case.

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Conflict of Interest

The authors hereby declare no conflict of interest as regards this research work and data supplied.

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