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CHEMICAL TREATMENT OF SUGARCANE BAGASSE FOR THE PRODUCTION OF CELLULOSIC FIBERS

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Abstract: The extraction of cellulosic fibers from sugarcane bagasse and the characterization of these fibers are carried out in this study. Sugarcane bagasse was treated with an alkaline solution in order to get cellulosic fibers. Sugarcane bagasse were analyzed by X-ray diffraction, Fourier transformed infra-red, Scanning electronic microscopic, Transmission electronic microscopic and differential thermal analysis/thermogravimetric analysis in nitrogen gas. Influence of the temperature and the mass of the fiber are studied, the temperature has an effect on the color of the fiber and the concentration of sodium hydroxide used, and more the soaking time increases the fiber mass decreases. It is found that the extraction yield of the fibers decreases as soaking time increases, the highest extraction yield is obtained at a temperature of 60°C with a concentration of sodium hydroxide of 0.1mol/L and the moisture content is 31.1%. The results obtained show that the process of extracting the fiber and controlling certain parameters such as temperature, concentration of sodium hydroxide and soaking time have an effect on the quality of the fiber obtained.
Keywords: Extraction; Sugarcane bagasse; cellulosic fibers; soaking time

INTRODUCTION

Waste from sugarcane is a real problem in the sugar industry in its management and valorization, yet the applications of sugarcane bagasse is no longer to be demonstrated in the paper industries by the production of paper pulp and energy fields by the production of bio fuel [1].

Natural cellulose fibers are often extracted from lignocellulosic substances using biochemical and chemical methods. Sometimes traditional methods are used to extract natural cellulose fiber using microorganisms but they have limitations [2,3]. Although atmospheric retting provides better quality fibers, it requires relatively longer duration and it is difficult to control the fiber quality.

Chemical extraction is recognized as effective in view of the convincing results obtained by the separation of lignin from cellulose. Alkaline treatment with sodium hydroxide has shown its effectiveness and several studies have been reported [4,5], delignification methods with sodium hypochlorite are frequently used

[6]. The cellulose from sugarcane bagasse can be easily obtained by acid hydrolysis followed by an alkaline pulping process. During the hydrolysis, the reagents acts on the lignin, breaking the macromolecule into units of low molecular weight, which are soluble in the liquor [7]. However this reaction is not strong enough to eliminate all lignin and hemicelluloses residues; therefore an additional process can be used. The bleaching treatment can be made with sodium chloride, which can result in cellulose degradation [8], and different properties and characteristics of cellulose arise. Chemical concentration, temperature and duration of treatment are the main factors determining the quality of chemically extracted fibers [9].

Cellulose is a main component of plant cell walls, including wood, cotton, jute, hemp, cereal straw, etc. It is constantly being renewed by photosynthesis. Its presence in plant tissues makes them compounds of vital importance and opens up new perspectives for research. It's a macromolecular component, made by the linking of smaller molecules. The links in the cellulose

chain are a type of sugar: ß-D-glucose, anhydroglucose units linked at the one and four carbon atoms by b-glycosidic bonds [10, 11]. The sugar units are linked when water is eliminated by combining the -OH group and H highlighted in gray. Linking just two of these sugars produces a disaccharide called cellobiose [12].

Nowadays, the promotion of local materials is an important focus in the field of materials sciences. Thus, from sugarcane bagasse, materials with new properties can be made, in textiles and in industries, in view of the high percentage of cellulose in their composition [13].

The aim of this work is to use chemical treatment to produce cellulosic fibers from sugarcane bagasse while evaluating the parameters that can influence this extraction such that the concentration of sodium hydroxide, the temperature. The moisture content have been determined.

MATERIALS AND METHODS

» **Chemicals reagents and Sample**

Sodium hydroxide, Hydrogen peroxide, amino-phosphonic acid, and persulfate were purcha*-sed from Himedia Laboratories Pvt.Ltd, India. Samples of sugarcane bagasse were filled by the Cameroon Sugar Company to Nkoteng (Cameroon, central Africa).

» **Chemical treatment of sample – Alkaline extraction and bleaching of Sugarcane Fibers**

5g of sugarcane bagasse are immersed in a beaker of 500mL containing 300mL of sodium hydroxide (0.1mol/L) and 40mL of hydrogen peroxide (30%), together with a stabilizer-chelating 30mL of aminophosphonic acid (0.1mol/L). After 2 h of rest, 20mL of 0.1mol/L of per sulfate (0.1mol/L) is added to the mixture and the whole is brought to rest for 4 h. Then the sugarcane bagasse is filtered and washed several times with distilled water to neutralize the pH of fibers, were conducted to eliminate the excess of sodium hydroxide in the fibers. After all alkaline extraction, fibers were oven-dried at 60°C for 24 hours, then conditioned at standard laboratory conditions. The untreated sugarcane bagasse containing 44.5% of cellulose, 30.15% of hemicelluloses 20.5% of lignin, 3.5% of ash and 1.2% of wax on a dry weight basis [14].

» **Determination of moisture content of the bagasse fibers**

It involves placing in the oven for 24 hours the mass of loaded sample or 1 g of crude bagasse for the case of our study, and to let it dry at a temperature of 105°C. After 24 h, the sample is cooled in a desiccator and weighed using the scale to determine the exact mass (dry mass). This operation is repeated under the same conditions before confirming the results obtained and making the determination of the moisture content according to the following formula described in the literature [15].

$$\text{Moisture content (\%)} = \frac{m - ms}{m} \times 100 \qquad (1)$$

Or (m) initial mass of bagasse, (ms) mass of the dry bagasse obtained.

» **Characterization of fibers from sugar cane bagasse**

The structural (physical) analysis of the samples was evaluated by X-ray diffraction (XRD) using a using a Panalytical, Netherlands (Model: PW3040/60 X'pert PRO) equipped with a Cu anode (kα radiation, λ = 1.54056 Å) and using a voltage of 40 KV and a current of 30 mA. IR spectra were scanned using KBr pellets in the region 4000 to 400 cm^{-1} with a resolution of 0.125 cm^{-1}, on a spectrometer Brucker Optik GmbH, Germany (model Tensor 27) equipped with the Opus TM software which provides an intuitive interface and facilitates the analysis of scans. Differential thermal analysis (DTA) and thermo-gravimetric analysis (TGA) were recorded using a SDT Q600 V8.3 Build 101 simultaneous DSC-TGA instrument. Approximately 1.2530 mg of fibers material was placed on the microbalance of the STA analyzer, which was purged with nitrogen gas.

The measurements were recorded from room temperature to 1100°C under nitrogen flow (100 mL.min^{-1}) with a heating rate of 20°C/min. Data analysis was performed using Universal V4.7A TA software package. The SEM micrographs of the sugar cane bagasse were taken in Hitachi (Japan) S-3000H electron microscope with an accelerating voltage of 15kV. Transmission electron micrographs of the functionalized clays are taken in FEI, The Netherlands (Model:Tecnai 20) transmission -electron microscope with an accelerating voltage of 200 keV. Ultrathin sections of bulk specimens (~100 nm thickness) are obtained at -85°C using an ultra-microtome fitted with a diamond knife.

RESULTS AND DISCUSSION

» **Structural Properties of Extracted Fiber from Sugar Cane Bagasse**

Figure 1 shows the results of x-ray diffraction of different samples which exhibit peaks at the same positions 2θ = 16.79° and 22.23° implying that the sugarcane bagasse used is composed typical cellulose-I structure [16].

The XRD patterns were obtained over the angular range 2θ = 10–50º. The Scherrer equation was used to calculate the average crystallite size (D) [17,18] of cellulose I structure in respect of (200) plane:

$$D = \frac{K\lambda}{\beta_{1/2} Cos\theta} \qquad (5)$$

The degree of crystallinity (X$_c$) was calculated using following equation [18].

$$X_c = \left\{ \frac{Kc}{\beta_{1/2}} \right\} \qquad (6)$$

where K is the correction factor and usually taken to be 0.91, λ is the radiation wavelength, θ where is the diffraction angle in degrees (002) and (110), $\beta_{1/2}$ is the

full width at half maximum (FWHM) of the (200) X-ray reflection (in degree), Kc is a constant set at 0.24 [19]. We also see the peaks (110) and (200) increase with temperature which justifies the fact that the temperature influence on the crystallinity of cellulose. Crystallite size of sugarcane bagasse was calculated as 2.33nm. The crystallinity index of the sugarcane bagasse was calculated as 35.6%.
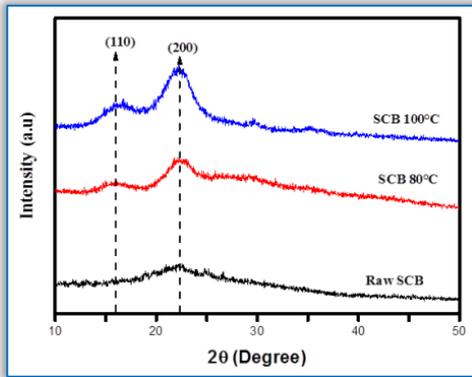

Figure 1: XRD patterns of Raw SCB and Treated SCB at different temperature.

Fig.2. shows the spectrum of treated. The band at 3695.75 3334.2cm^{-1} is depicting the stretching of hydroxyl group in treated bagasse. The absorption band at 2921.75-2354.20 cm^{-1} is attributed to C-H stretching, the absorbance at 1434.47, 1375.17, 1249.35 and 1041.41 cm^{-1} corresponds to the aromatic skeleton vibration, ring breathing in the C-O stretching in lignin [20]. The bands at 1375.17cm^{-1} are attributed to absorption by C-H and C-O stretching in acetyl group in hemicellulose respectively. The strong band at 1041.41 cm^{-1} in pretreated sugarcane bagasse is assign to C-O stretching in cellulose, hemicellulose and lignin or C-O-C stretching in cellulose and hemicelluloses. The band at 971.38 cm^{-1} is due to glucosidic linkage [21]. The spectral band observed in the region 1731.42–1635.05 cm^{-1} are due to the O–H bending due to adsorbed water [22] The region of FTIR spectrum between 682.03 to 473.08 cm^{-1} was not considered this because the vibration area of Si-O-Si from traces of sand grains fixed on sugarcane bagasse.
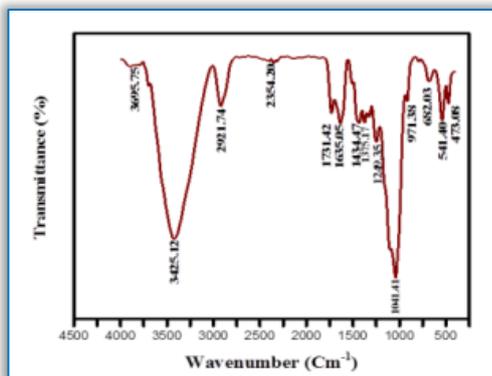

Figure 2: FTIR Spectrum of Treated SCB

Figure 3 shows the FTIR spectrum of cane bagasse after extraction of cellulose with sodium hydroxide, the samples were heat treated at different temperatures. These spectra present the same peaks as the previous one but the difference lies in the areas of 3695.75 3334.2cm^{-1}, and 1731.42-1635.05 cm^{-1} assigned to the peaks of OH. The intensities of these peaks decrease with increase in temperature which means the phenomenon of dehydration that occurs.
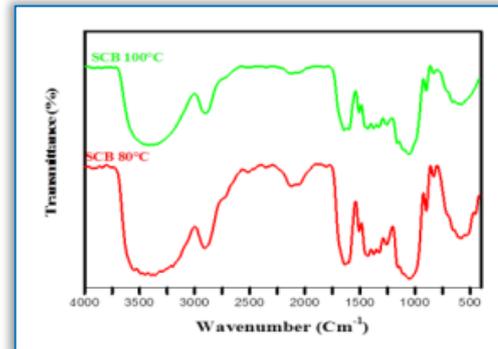

Figure 3: FTIR Spectrum of heated SCB at different Temperature

» **Thermal analysis**

TGA Curves of the fibers exhibited three degradation stages. The first stage of weight loss occurred at 188.62°C, which corresponds to dehydration in the sample. The second stage of weight loss for presented thermal degradation peak at 367.30°C and the third stage at 529.65°C corresponds to the thermal degradation of cellulose (See figure 4).
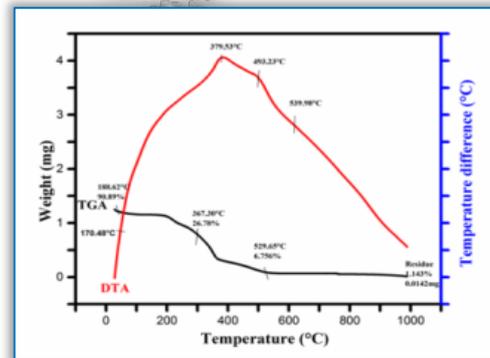

Figure 4: TGA and DTA Curves of Treated SCB

The DTA curves show the endothermic peak 170.48°C, which is attributed to the removal of moisture when the sample was heated. Two exothermal peaks appear at 379.53°C and 493.23°C, respectively, and they are attributed to charring, while a large endothermic peak at 379.53°C is related to the cellulose fraction. This behavior is related to the full decomposition of cellulose that might be attributed to quick devolatilization reactions, leading to very little solid residue (0.0142 mg).

» **Morphology and Elemental composition Analysis**

Figure 5 shows the SEM images of untreated and treated bagasse under different magnifications. There was no

apparent difference in the morphology of untreated and treated sugarcane bagasse when they were both examined under lower magnification. However, a relatively more porous structure can be observed for treated sugarcane bagasse when higher magnification was applied. Treated bagasse has a more irregular and corrugated surface with thinner fibers compared to the untreated sugarcane bagasse as illustrated in Figure 5(a) and (b).
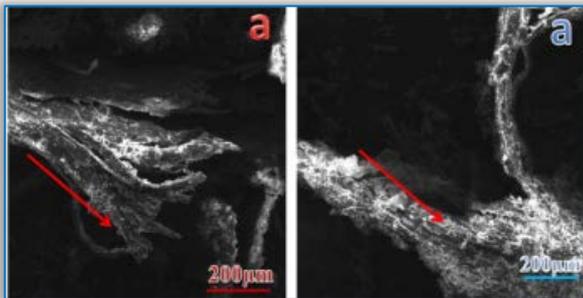

Figure 5: SEM of a) Untreated SCB, b) Treated SCB.

» **Transmission Electronic Microscopic imaging of Sugar cane Bagasse morphology**

The fig.6 shows the sugarcane bagasse treated with sodium hydroxide. It has the porous structure of the bagasse with very small dimensions fibers observable disparately.
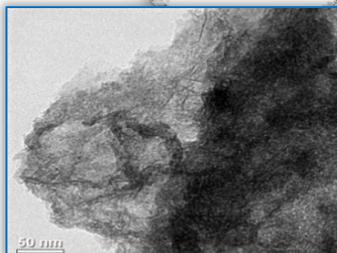

Figure 6: TEM of Treated SCB.

EXTRACTION PARAMETERS

» **Influence of Temperature and soaking time**

At issue in this part of the fibers extracted by the use of products chemicals in particular sodium hydroxide according to a number of parameters (temperature, soaking time, etc.). Kinetic extractions fibers in order to obtain higher yields have led to the following results (Figure 7, 8 and 9).
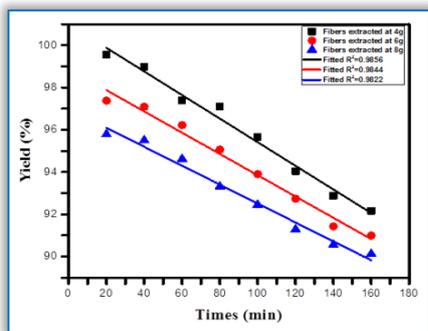

Figure 7: Kinetic of Extraction of SCB Fibers at different mass (at 80°C).
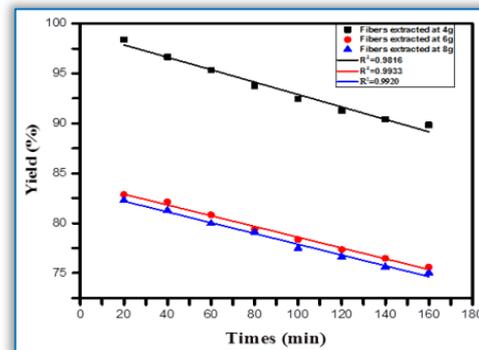

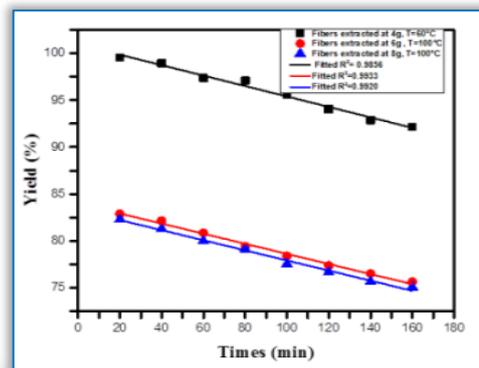Figure 8: Kinetic of Extraction of SCB Fibers at different mass (at 100°C).

Figure 9: Kinetic of Extraction of SCB Fibers at different mass (at 80°C and 100°C).

It is found that the extraction yield of the fibers decreases as preparation time increases. This yield loss is also due to the increasing of concentration and an increase in temperature. The difference between yields between 60 and 100°C is due to the very high temperature rise and the corrosive nature of the sodium hydroxide.

At 100°C, it is also observed that the extraction yield does not vary significantly, due to the fact that the temperature of the bath is constant and that only the concentration of sodium hydroxide. The mass of fibers decreases progressively during each preparation when the soaking time increases

» **Influence of Concentration of sodium hydroxide and Temperature**

By maintaining the temperature constant and varying the quantity of sodium hydroxide, this affects the quality of the fiber, the appearance of which changes and appears bushy when the concentration of hydroxide sodium is low, but when this concentration increases, the appearance of the fiber is like whiskers and somewhat supple which shows the action of sodium hydroxide is remarkable (Figure 10 and Figure 11).

At 80°C, 0.2mol/L of sodium hydroxide, we find that the resulting fibers are finer and distinguished easily, the fineness of the fibers is due to the corrosive nature of the sodium hydroxide, the temperature rise and the time of cooking (figure 12).

Figure 10: Fibers extracted at 60°C for 0.1mol/L of sodium hydroxide.

Figure 11: Fibers extracted at 60°C for 0.2mol/L of sodium hydroxide

Figure 12: Fibers extracted at 80 ° C for 0.2mol/L of sodium hydroxide.

At 100°C, we find that the resulting fibers change shape and become pasty, and the yield drops considerably. This is due to the increase in the concentration of the extraction bath, the elevation of the temperature and cooking time.

Figure 13: Fibers extracted at 100 ° C for 0.3mol/L of sodium hydroxide.

» **Moisture content of the fiber**

Using the formula above, the calculation of the content moisture is 31.1%.

CONCLUSION

Fiber bundles were chemically extracted from raw bagasse of sugarcane. The alkaline extraction was the best and most efficient way to remove lignin since the solution was more concentrated. The kinetics of extraction of sugarcane fibers chemically extracted at different concentration of sodium hydroxide showed that the mass of the sample extracted fibers decreases with time, and with the rise in bath temperature.

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