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Chemical composition and structural characterization of treated sugarcane bagasse with alkaline solution

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Abstract

Sugarcane bagasse is a natural raw material of agricultural origin, these biomass materials have a lot of potential future applications when converted into useful and promising products. *Sugarcane bagasse* was treated with *potassium hydroxide* (0.3N) in order to create more active sites on the fiber after treatment. *Sugarcane bagasse* and its sequence on the new formed fiber and the results of this treatment was characterized and identified by several techniques. The following FTIR, SEM, AFM analysis and Van Soest measurement for fiber identification were used for *sugarcane bagasse* characterization before and after treatment with *potassium hydroxide* and the results will be shown later. Van Soest measurement showed a decrease of *cellulose* and *hemicelluloses* before and after treatment with an amount 17.97% and 27.08%, respectively . FTIR spectrum showed that the most absorption characterized bands were shifted from 3392.17 cm^{-1} to 3388.32 cm^{-1} after treatment with *potassium hydroxide*. SEM showed an increase in internal surface area of the lignocellulose particles and weakening of the structural after treatment with KOH solution leading to a swelling of the biomass and confirming the increasing of the internal surface area of the lignocelluloses particles after KOH treatment. AFM showed a decreasing in the mean radius and average surface roughness values after *Sugarcane bagasse*, SB-KOH treatment and increase in the surface area by 8.5327nm. *Sugarcane bagasse* treated with alkaline solution is promising for future applications in the industry in comparison with untreated *sugarcane bagasse*.

1. Introduction

Industrial human activities have increased in recent years such as mining, traffic and materials chemicals causing an increase in the harmful effects resulting from those activities which have reached large levels of environmental pollution harmful to human live and the ecosystem and water [1]. The importance of finding environmentally friendly solutions for the recycling of waste resulting from industrial activities especially organic are of large interest. The *Sugarcane bagasse* is considered to be one of the harmful waste to the environment without any treatment because this biomass contain *cellulose*, polyposis, *lignin*, *hemicelluloses*, small amounts of extractives and mineral substances. Sugarcane, a tropical crop, requires special environment, such as, an optimum high temperature, availability of sunshine and higher rainfall (1250–2500 mm) for its growth [2]. These materials have been used as a main source for the production of sugar, food and drinks [3, 4].

Sugars are bound together in a long chain called polysaccharides and form a structural part of plant cell walls [5] . According to previous studies, alkaline treatment has been shown to dissolve *hemicelluloses, lignin and cellulose* swelling [3, 6]. Based on previous research and experiences cellulosic, fibers have been identified and explained having a crystalline structure containing sugar molecules by a cellulose sheath in the form of cover that protects cellulose from harmful bacteria. Figure 1 shows the structure of cellulose micro fibrils. All previous studies report the importance of *Sugarcane bagasse* and the interesting of extending the research into the composition showing the importance of low cost and wide spread raw naturals with common use by humans as a source of fuel and a fodder for animals [7,8].
This paper reports a comprehensive chemical composition and structural characterization of treated *sugarcane bagasse* with alkaline solution emphasizing the importance of sugarcane bagasse and its future potential usage.

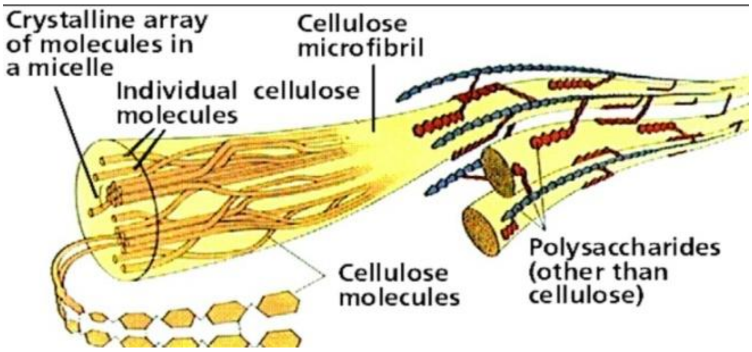


Figure 1: structure of cellulose micro fibrils. (This image was copyright Dennis Kunkel at www.Dennis Kunkel.com, used with permission).

2. Material and Methods

2.1. Sample preparation

The starting materials were comprised of KOH (0.3N), KBr, and Ethanol. All other chemicals and reagents used were of the highest commercially available purity purchased from Merck and used without as received.
Sugarcane bagasse samples were collected from local juice seller shops at AL-Mohafaza Square area in Damascus City. The collected bagasse was placed into plastic bags and manually sliced into small pieces with an average size of 3 cm then, thoroughly washed several times with tap water to remove dust, ligneous, *cellulose* and trapped impurities with similar procedures followed by Salih [9]. After that, it was then soaked in distilled water for 48 h and then dried for 24 h at 60°C and sieved to 680 µm. The two separate samples were: *sugarcane bagasse* SB-N (*sugarcane bagasse* without any alkaline treatment) and SB-KOH (sugarcane bagasse treated with KOH solution).
The dried SB-N was treated with (0.3N KOH) solution. With a ratio of 1:3 and placed in closed container and heated in a furnace for 24h at 105°C The dried material was washed with distilled water until the amendment. The biomass was further dried in an oven at 105°C for 24 h. The obtained biomass after treatment, SB-KOH was stored in polyethylene bottle before further use.

2.2. Van Soest method for fiber Determination

This method was performed based on AOAC (Association of Official Analytical Chemists scheme) and it consists of three stages using VELP Scientificsa, FIWE, Raw fiber extractor device in order to complete assigning the fiber determination properties , the analyzes were carried out in the ACSAD (Arab center for the studies of arid lands and dry zones) [10,11].

2.3. Fourier transforms infrared spectroscopy (FTIR) analysis

The properties of the reaction products were characterized by Fourier Transform Infrared spectrophotometer, FTIR., Using Jasco FT4200 type A, Serial C077661018 taken as KBr disc for the samples (SB-N,SB-KOH) in the range of 4000-400 cm^{-1} with a resolution of 4 cm^{-1}.

2.4. Characterization of sugarcane bagasse (SEM)

The SEM measurements of the powdered samples were carried out as follows: The samples were applied directly on a double-sided adhesive carbon tape. Then, an air gun was used to blow away almost all the fine power, hence only the suitable size of the powder was left, which could be subjected to analysis. Tescan Vega II XMU Scanning Electron Microscope (SEM) system was utilized to gain information about the surface morphology of the samples. Additionally, an EDAX TEAM™ energy dispersive X-ray (EDX) system, which is associated with the SEM technique, was also utilized for obtaining the elemental composition of the samples. The whole system is fully automated to enable simple control of the sample holder as well as straight forward data acquisition. An accelerating voltage of 20 kV and an electron beam current of about 90 μ Awas were applied. The beam was scanned over different grains of the samples, at various magnification modes, to gain general understanding of both the morphology and composition.

2.5. Characterization of sugarcane bagasse (AFM)

To allow Atomic Force Macroscopic (AFM) measurements of the powdered samples, the samples were dissolved with ethanol (0.05gr per 5 ml), the prepared solution is dispersed using ultrasonic machine , and then drops the prepared solution placed on a glass slide, then dried at 80°C for 15min , then measured using Atomic Force Macroscopic (AFM), the objective of the analysis towards the topographic study of the surface , the roughness of the surface , the size of the granules (half-grain diameter) and the mean distribution .

3. Results and discussion

Van Soest method for fiber Determination:

The results of the analysis were shown in Table1 which exhibiting that the *cellulose* content in not treated *sugarcane bagasse* SB-N and reached 49.64% and the treated content SB-KOH was about 31.67%, and the *hemicellulos* value decrease from 34.34% to 6.52%, while the percentage of lignin and Ash increased almost doubled. The decreased in the *cellulose* and *hemicellulos* in the alkaline treatment allowed a new active sites formation and increasing the surface area of *sugarcane bagasse* SB-KOH. These results are consistent with previous reported study [12].

Table1: The values from the Van Soest method for fiber Determination.

Proportions in dry matter%					
	Dry matter ratio	**lignin**	***cellulose***	***hemicellulose***	**Ash**
***sugarcane bagasse* untreated**	94.7	6.39	49.64	34.34	1.45
***sugarcane bagasse* treatment with KOH**	96.46	11.24	31.67	6.52	2.42

Fourier transforms infrared spectroscopy (FTIR) analysis:

Figurer 2 shows the FTIR spectrum of (SB-N) without any alkaline treatment and Figure 3 show the spectra of *Sugarcane bagasse* after treatment (SB-KOH), The IR spectrum of untreated *Sugarcane bagasse* (SB-N) shows various absorption bands indicating the complex structure of Sugarcane residues and the multiplicity of functional groups, and the number of characteristic absorption bands in the spectrum were appeared at 3392 cm^{-1} and assigned to stretching vibration of (-OH) group [13] in the glucose component unit for the cellulose molecule. Another characteristic absorption band was observed at 2927 cm^{-1} which is assigned to the elastic vibration of the alpha (C-H) bond [13]. The absorption band at 1628 cm^{-1} is due to (C=O) carbonyl group stretching [14]. The absorption band at 1429 cm^{-1} is due to the elastic vibration of the (C-O-C) bending formation band of the ester groups in the *cellulose*[15], in addition to the absorption band at 1060 cm^{1-} which is due to the elastic vibration of the (C-OH) groups [15]. By comparing the *cellulose* spectrum before and after the treatment it is noted that most of the absorption bands were shifted to lower wave numbers 3.85cm^{-1}. These shifts are consistent with (Nadiah Ameram) finding due to the effect of treating *cellulose* with KOH [16].

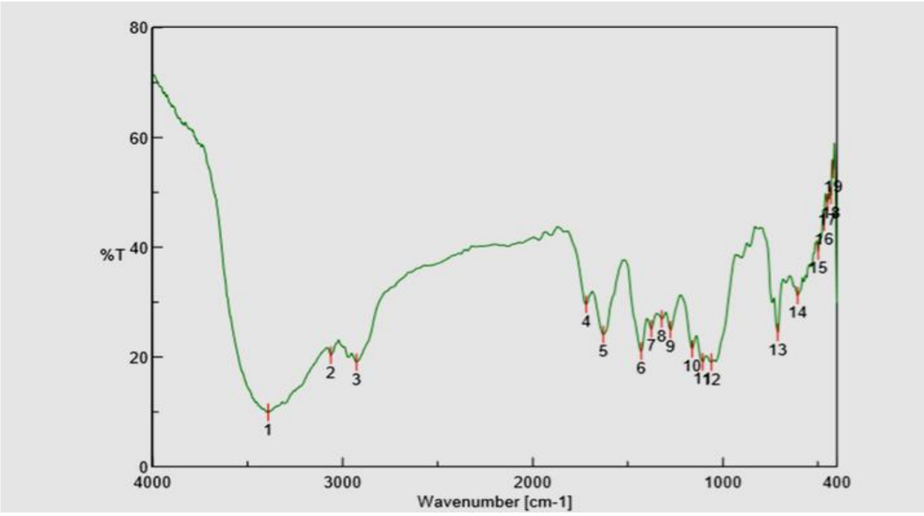


Figure 2: FTIR spectrum of Untreated *Sugarcane Bagasse* (SB-N).

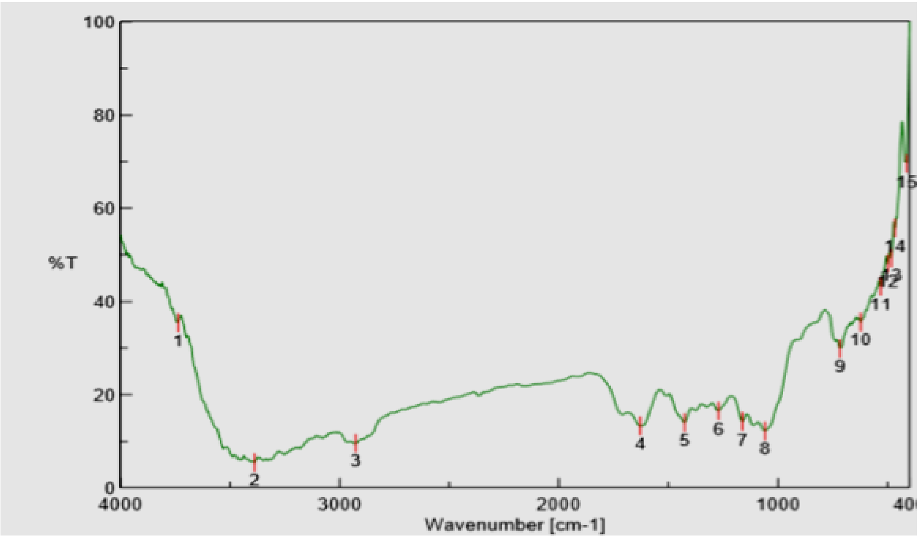
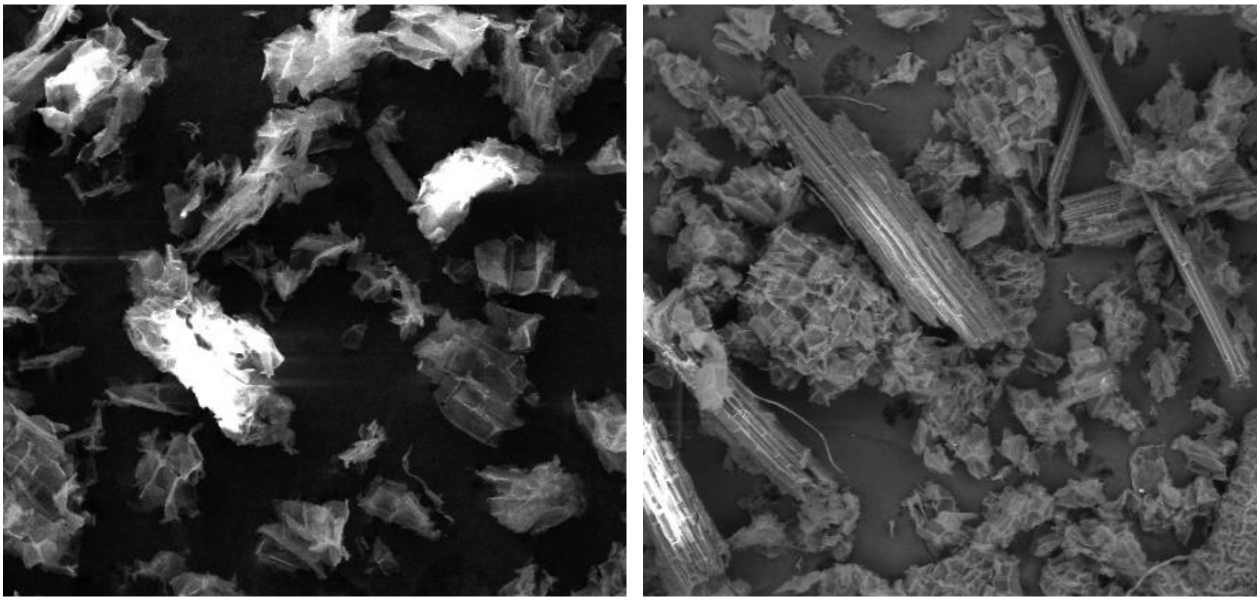


Figure 3: FTIR spectrum of Treated *Sugarcane Bagasse* (SB-KOH) before adsorption.

Morphological analysis of sugarcane bagasse (SEM) :

The morphology of SB-KOH fibers was investigated using SEM to determine the change of fiber surface and morphology during extraction processing and the results are presented Figure 4. Alkaline treatment also has a remarkable effect on the bagasse morphology, especially on the fiber bundles. The images obtained from fiber surfaces of the samples at 0.3N concentration of KOH are remarkable. After being treated with KOH, the bagasse bundles were started to dismantle and the fibers were detached from the others. Alkaline pretreatment by adding KOH solution could cause a swelling of the biomass, which would result in the increasing of the internal surface area of the lingo *cellulose* particles, as well as the weakening of the structural integrity of the lingo *cellulose* and breaking off the bond linkages between *lignin* and the other carbohydrates (*cellulose* and *hemicellulose*), causing in greater accessibility and digestibility of the *cellulose* fraction, and thus, it could be depolymerized into fermentable sugars [17,18].



(a) (b)

Figure 4: Morphology of the SB-KOH by SEM analysis with magnification (a-70x,b-50x)

Characterization of sugarcane bagasse (AFM) :

After drying the glass slides at 80°C for 15min ,surface morphology was measured by the Atomic Force Macroscopic (AFM)and using a special image processing program (Nano surface assay scan), we calculated both the average surface roughness (Sq), the diameter of the grains formed , and the mean value of the surface roughness coefficient (Sa) , Figure 5 shows images (2D-3D AFM) measuring (3µmx2µm) *Sugarcane bagasse* (SB-N,SB-KOH), Table 2 shows the values from the analysis. From Table 2, we notice a decrease in the average surface roughness of 8.5327nm in treated *Sugarcane bagasse* SB-KOH in comparison with SB-N, this indicates an increasing surface area, and the mean value of the surface roughness coefficient in the SB-KOH with nano scale dimensions assuring the positive factor in increasing surface area [19].

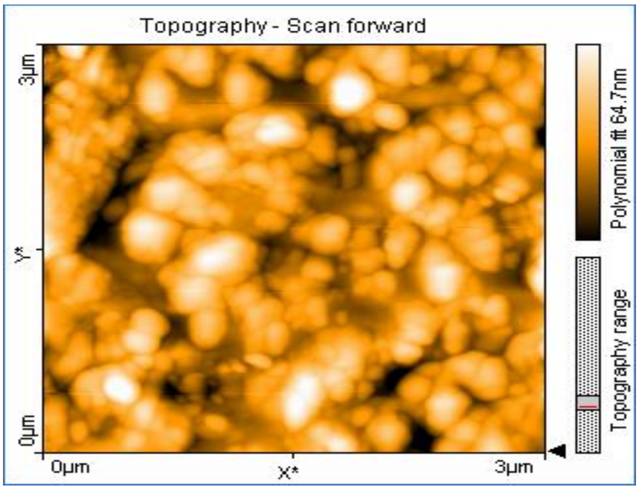


Figure 5: Image 2DAFM measuring 3µmx2µm SB-**N**

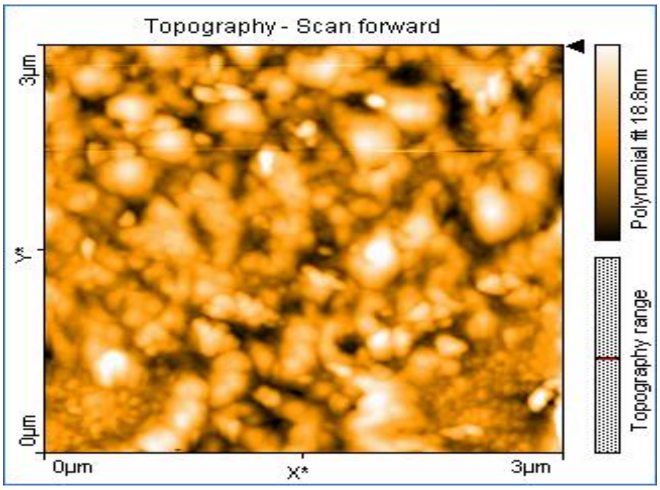


Figure 5 : image 2DAFM measuring 3µmx2µm SB-KOH

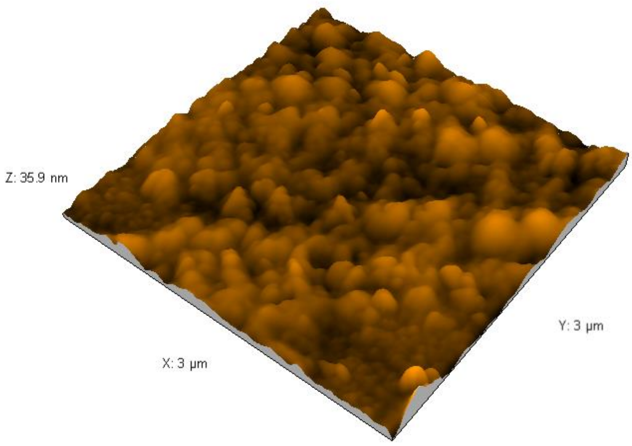


Figure 5: image 3DAFM measuring 3µmx^{2}µm SB- N

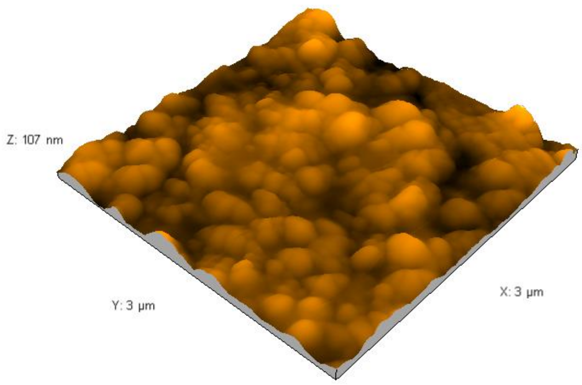
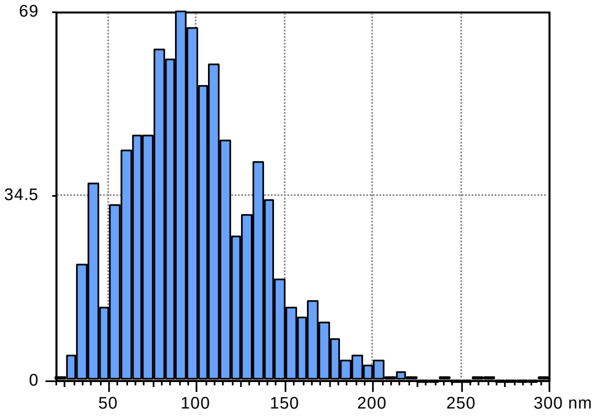


Figure 5: image 3DAFM measuring 3µmx2µm SB-KOH

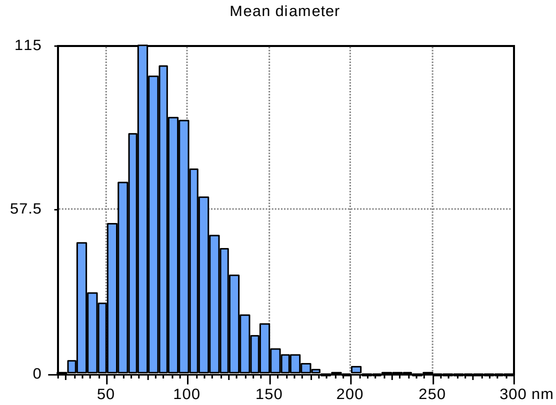
Table 2: The values from the AFM analysis.

	Sq	**Sa**
SB-N	12.075 nm	9.5273 nm
SB-KOH	3.5423 nm	2788.1 pm

Figure 6 shows the distribution curves in terms of the radii of granules of *Sugarcane bagasse*, and it has been noticed the displacement of 25nm shift in the distribution curves of alkaline treated sample, SB-KOH. The smaller mean radius of the granules, the higher the ratio of the surface to the volume, the surface area becomes larger and the curing process becomes better are observed. Similar results were recently obtained showing that the study of composites reinforced with sugarcane bagasse fibers treated with NaOH showed that alkali treatment results in increased composite stiffness and strength compared to materials prepared with the untreated fibers. A maximum is achieved in these properties at around 5 wt% NaOH content of the treating solution [20].



Sa = 9.5273 nm ; Sq = 12.075 nm



Sa = 2788.1 pm ; Sq = 3.5423 nm

Figure 6: the distribution curves in terms of the radii SB-N **Figure 6:** the distribution curves in terms of the radiiSB-KOH

Conclusion

Based on the findings of the study, the following conclusions were drawn. The results of *Van Soest method* showed a decrease in the percentage of cellulose by 17.97% and decrease in the percentage of *hemicellulose* by 27.08% in alkaline treated samples compared with the untreated .FTIR showed that comparing the *cellulose* spectrum before and after the treatment it is noted that most of the absorption bands were shifted with respect to lower 3.85 cm^{-1} wavenumbers. These shifts demonstrated the effect of cellulose treatment with KOH.

SEM showed an increase in internal surface area of the lignocellulose particles and weakening of the structural and alkaline pretreatment by adding KOH solution and that could cause a swelling of the biomass, which would result in the increase of the internal surface area of the lingo *cellulose* particles. AFM showed a decreased in the mean radius and average surface roughness values of the treated *Sugarcane bagasse* SB-KOH and this indicates an increase in the surface area of the sample.

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