

# Selective extraction and modification of cellulose from sugar cane bagasse (*Saccharum officinarum*)

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Received: 30-09-2021

Accepted: 25-05-2022

Published online: 01-09-2022

**Citation:** Rincon-Fuentes L, Moreno-Bastidas L, Medina-Vargas O. Selective extraction and modification of cellulose from sugar cane bagasse (*Saccharum officinarum*), *Universitas Scientiarum*, 27(3): 254–272, 2022. doi: 10.11144/Javeriana.SC273.seam

**Funding:** n.a.

**Electronic supplementary material:** n.a.

## Abstract

This paper's aim was the synthesis of a flocculant useful in the production of *panela* as an alternative to replace polyacrylamide and improve food safety in Boyacá, a Colombian district. The cellulose was extracted by using a treatment with potassium hydroxide and followed by acid hydrolysis. From the extracted cellulose, cationic dialdehyde cellulose (CDAC) was synthesized and both cellulose and CDAC were characterized using infrared spectroscopy, FTIR, thermogravimetric analysis (TGA), differential thermal analysis (DSC) Scanning Electron Microscopy (SEM) and RAMAN. The cellulose extracted by hydrolysis from sugarcane bagasse with bleaching, gave yield of 50 % and crystallinity index of 77 %. Its modification to aldehyde is 95 % reliable at a temperature of 65 °C and 2 h; with higher temperature and time the performance is affected. The dialdehyde process allows a modification to be carried out and to be more easily attacked at carbons 2 and 3, leaving cationized cellulose for later use as a flocculant in the *panela* production process as a traditional solution of flocculants and polyacrylamide. Extraction, modification, and tests for the clarification of the *panela* juice were carried out in triplicate.

**Keywords:** sugarcane; cellulose; flocculant; polyacrylamide.

## 1. Introduction

Colombia is one of the countries with the highest production of sugar cane (*Saccharum Officinarum*) used to produce *panela* in Latin America and the world [1]. It is the second largest producer of *panela* in the world and the agro-industrial production of *panela* is the second one, after coffee production [2]. The production of *panela* in Colombia is traditional and artisanal, with little use of technology. There are 27 districts in which sugar cane is grown, with the largest production concentrated in Santander, Cundinamarca, Boyacá, Valle del Cauca, and Antioquia. In Colombia, *panela* is primarily consumed in either a pulverized form or in solid blocks.

In Latin America, *panela* is produced under different names in different regions. It is known as *Panela* in Colombia, Guatemala, Panamá, Ecuador, and Bolivia; *Chancaca* in Perú and Chile; *Rapadura* in Brazil and Argentina; *Rapadura* in the Dominican Republic and Ecuador; *Papelón* in Venezuela; *Piloncillo* in Mexico; *Tapa Dulce* in Costa Rica; *Atado de Dulce* in Nicaragua; and *Empalizado* in Bolivia. Sugar cane is typically cultivated in areas with temperate or tropical climates, where it is grown for approximately 9 to 24 months to achieve the maximum amount of sucrose to produce *panela*.



One important agricultural residue from sugarcane is its bagasse. Nowadays, bagasse has been considered as a suitable cellulose source. All the methodologies to obtain cellulose are based on the separation of the fibers. There are two main processes: the alkaline method and the sulfite method [3]. The acid hydrolysis of cellulosic compounds may generate a 48 % yield [4].

Most of the hemicellulose is hydrolyzed during pretreatment and the lignin is released or may even break down. One of the most efficient pretreatments is the so-called steam explosion, in which the lignocellulosic material is subjected to high temperatures (180 °C to 240 °C) with pressurized steam (0.7 MPa to 4.9 MPa) for short times (10 minutes) [5].

The annual production of sugar cane bagasse in the world is approximately 100 million tons. In Colombia, more than 6.5 tons/year of sugar cane bagasse are produced. Due to its fibrous nature, bagasse is widely used as fuel in sugar mill stoves in papermaking, and as a source of thermal or mechanical energy. It is primarily used in other industrial chemical products [2]. Its role as a raw material used in important industries such as paper, clothing, and medicine among others is noticeable.

Bagasse is chemically composed of cellulose, lignin, hemicellulose, and ash among other compounds. It contains between 40 % and 44 % cellulose, an abundant, renewable, biodegradable, and environmentally biocompatible polymer. The fibrils are made from polymerized linear chains of cellulose arranged in a semi-crystalline structure (40 % to 70 %) depending on the origin and isolation method from cellulose [3]. More than 10 billion tons of cellulose worldwide are of vegetable origin. Cellulose is a rigid and linear homopolymer polysaccharide consisting of D-anhydro glucopyranose (AGU) units. They are linked together by  $\beta(1 \rightarrow 4)$  glycosidic bonds at the C<sub>1</sub> and C<sub>4</sub> carbon bonds of adjacent glucose moieties. Solid state AGU units rotate 180° with respect to each other and have three hydroxyl groups (–OH) at the C<sub>2</sub>, C<sub>3</sub>, and C<sub>6</sub> positions. The  $\beta(1 \rightarrow 4)$  glycosidic bonds are oriented in a planar structure where the number of repeating units per string,  $n$ , depends on the cellulose source (wood or cotton). There are different types of crystalline cellulosic polymorphisms; and there, the –OH groups are responsible for the hydrophilicity of intramolecular hydrogen bonds, symmetric stretching of the primary alcohol, and the  $\beta(1 \rightarrow 4)$  glycosidic bonds of cellulose. In this study, cellulose I and II are studied, which are found in nature as two allomorphs called I $\alpha$  with a triclinic structure and I $\beta$  with a monoclinic structure [4].

The main difference between the polymorphic structures I $\alpha$  and I $\beta$  is the relative displacement to the cellulose sheets along the plane of the lattice (110) in the triclinic structure and the lattice plane (200). In the monoclinic structure, cellulose II is in the thermodynamically stable crystalline form, which can also be obtained from cellulose I by regenerative processes.

Novel routes and effortless, low-cost, and green process similar to this paper have been recently published [6]. Different studies have been carried out on the transformation of cellulose as a flocculant in drinking water and wastewater. One of the modification processes for cellulose relies on locating groups with positive charges (cationic cellulose). Due to its low toxicity and good reactivity, cationic cellulose produces an esterification reaction. The transformation of cationized cellulose shows a significant improvement in the ability to remove suspended solids [5]. The objective of this study seeks to describe the synthesis of a cationic cellulose that acts as a flocculant resulting from sugar cane bagasse biomass. This substitute for traditional flocculants such as polyacrylamide in the production of *panela* is necessary to find a solution for improving food safety in Boyacá, Colombia.

## 2. Materials and Methods

### 2.1. Extraction and procurement of cellulose

The sugarcane bagasse was obtained from the biomass residues generated by the milling process to produce *panela* at the *Buena Vista* sugar cane mill in the municipality of Santana in Boyacá. All the chemical reagents used were from commercial sources and of analytical grade. Acetic acid (from Sigma-Aldrich Co), sulfuric acid, potassium hydroxide (KOH) (from Merck laboratories), were used as purchased.

Cellulose extraction was performed using the method proposed by [4] with modifications, mainly in the separation of the cellulose fibers. First, the cane bagasse was carefully washed and dried at 70 °C in an oven. The bagasse was then ground to an approximate size of 10 microns. The modification of the cellulose was done according to the methodology suggested by [7]. The oxidation of carbons C1 and C2 was done using sodium periodate (NaIO<sub>4</sub>) and catalyzed with LiCl. The obtained product was dialdehyde cellulose (DAC). The reaction was performed in dark amber glass flasks to avoid photo-induced decomposition of the periodate. It was stirred for 2 hours at a temperature of 65 °C. At the end of the reaction time, ethylene glycol with an excess of 2 % was added to stop the oxidation reaction and the resulting product was washed with distilled water.

The degree of oxidation from the oxime reaction was determined from the content of aldehyde groups per gram of cellulose [7]. This process utilized hydroxylamine to obtain an acidic pH while stirring for 24 hours. This step was completed with a subsequent potentiometric titration using 0.1 M NaOH until a pH of 3.5 was achieved according to Equation 1 shown below.

$$\text{Aldehyde} = \frac{V(\text{NaOH}) \times N(\text{NaOH})}{W}. \quad (1)$$

Where:  $V(\text{NaOH})$ : Volume (ml) of NaOH.  $N(\text{NaOH})$ : Normality of NaOH, and  $W$ : dry weight (g) of DAC.

From the amount of aldehyde groups in the oxidized cellulose, the cellulose dialdehyde content was modified by increasing the degree of substitution. This procedure was accomplished by adding deionized water and 2-hydrazinyl-2-oxoethyl trimethyl titanium chloride and (Girard's reagent, GT) in a molar ratio of GT/aldehyde of 1 : 7.8 at a pH of 4.5. The pH was adjusted with diluted HCl for a period of 4 hours at 20 °C while stirring. The cationic cellulose was washed with isopropanol and subsequently with distilled water. The cationic product was then dried in the oven at 60 °C and stored in a desiccator [8] [9].

It is worth mentioning that the study on extracting cellulose from raw garlic fibers in Colombia [10] also used an autoclave in addition to a mild chemical treatment. The chemical treatments included alkaline extraction, bleaching, and acid hydrolysis, but with a moderate concentration of chemicals. The extracted cellulose microfibrils were characterized by infrared spectroscopy, Fourier transform infrared spectroscopy (FTIR), thermo gravimetric analysis (TGA), X-ray powder diffractometry (XRD), and scanning electron microscopy (SEM). The FTIR spectra, as well as the results from XRD, TGA, and SEM showed changes in the peaks corresponding to hemicelluloses and lignin, showing its removal from the fiber surface, due to the former treatments. This study helped to illustrate the potential of garlic by-products to produce cellulose derivatives and its potential use for the preparation of other useful compounds.

Similarly, [11] developed a simple method to estimate the fuel ratio (Fixed carbon)/(volatile matter). Molecular weight (MW) from pyrolysis was carried out using a rotary kiln (20 t MW/day). The proportion of coal fuel was estimated by thermal analysis using a mixture of Polyvinyl chloride/CE to represent biomass and polyethylene, polypropene, and polystyrene; and to represent plastics, considering the elemental composition of the molecular weight. The proportion of fuel depended on the amount of biomass and plastics in MW as well as on the pyrolysis temperature which was like the theoretical estimate.

A study on Mucilages and cellulosic derivatives as clarifiers for the improvement of non-centrifuged sugar (NCS or *panela*) production was performed by [12]. The study found that non-centrifuged cane sugar (NCS) is the second most important Colombian agricultural production in Colombia. Unfortunately, the sugarcane industry faces some challenges caused by the controversial nutritional and safety factors of NCS.

Although the Colombian NCS producers use natural mucilage as clarifiers, the uncontrolled application of these ingredients has caused an extinction risk for the mucilage plants from which those mucilages are extracted. Other producers use acrylamide as a clarifier; however, the health consequences have caused concerns among consumers and food safety authorities. The intention of this study was to find a sustainable, natural, non-toxic, and economical clarifier for Colombian NCS producers.

## 2.2. Analysis using FTIR Spectroscopy

For the FTIR analysis, a Perkin Elmer 1600 spectrometer was used with a working range from  $4000\text{ cm}^{-1}$  to  $500\text{ cm}^{-1}$ . The spectra were taken in transmission mode, using dry KBr to prepare the sample in a transparent pellet form. Cellulose samples from sugar cane bagasse and cationic cellulose were analyzed. The percentage of lignin extracted from the cane bagasse was 50 %. It considered the fact that it was subjected to an alkaline treatment for 24 hours, which guaranteed the removal of lignin from the structure of the sugarcane bagasse.

## 2.3. Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DSC)

The TGA and DSC analyses of the samples were carried out at the Institute for Research in Materials Science and Technology (INCITEMA), located at the UPTC in Tunja, Boyacá. The thermograms of the samples were acquired on a TGA Q600 thermogravimetric analyzer which utilized 90  $\mu\text{l}$  alumina crucibles that contained approximately 13 mg of the sample. A non-oxidizing nitrogen atmosphere was used for all tests with a flow rate of 40 ml/min while a 20 ml/min flow rate of argon was used as a purge gas. The temperature range varied from  $20\text{ }^{\circ}\text{C}$  to  $600\text{ }^{\circ}\text{C}$  with a heating rate of  $10\text{ }^{\circ}\text{C}/\text{min}$ . This step was performed to create a more accurate evaluation of the temperature at which the greatest weight loss occurs. Curves of weight and temperature (thermogravimetric derivative curves, DSC) were evaluated with the approximate duration of the measurements, i.e., 70 minutes. There were no intermediate peaks which are usually associated with the rapid degradation of hemicellulose.

## 2.4. X-ray diffraction analysis (XRD)

To determine the crystallinity of the samples and evaluate the purity and morphology of the extracted cellulose, an analysis of the sample was carried out in a PAN analytical X'pert PRO-MPD diffractor. It was equipped with an ultrafast X'Celerator detector in a Bragg-Brentano arrangement using a Cu K $\alpha$  radiation source ( $\lambda = 1.54186 \text{ \AA}$ ) between 5 and 90° with steps of 0.02° and a voltage of 40 kV.

To calculate the crystallinity index (% CI), the Segal method was used using Equation (2) [13]:

$$\% \text{ CI} = \frac{I_{100} - I_{\text{am}}}{I_{200}} \times 100. \quad (2)$$

Where  $I_{\text{am}}$  is the intensity of the diffraction in the amorphous material.

## 2.5. Scanning Electron Microscopy (SEM) Analysis

The SEM analysis was also performed at INCITEMA, where micrographs were obtained on a CARL ZEISS EVO MA 10 microscope. The high vacuum mode and a voltage between 5 kV and 15 kV were used. The samples were subjected to gold plating using PVD prior to their analysis by SEM.

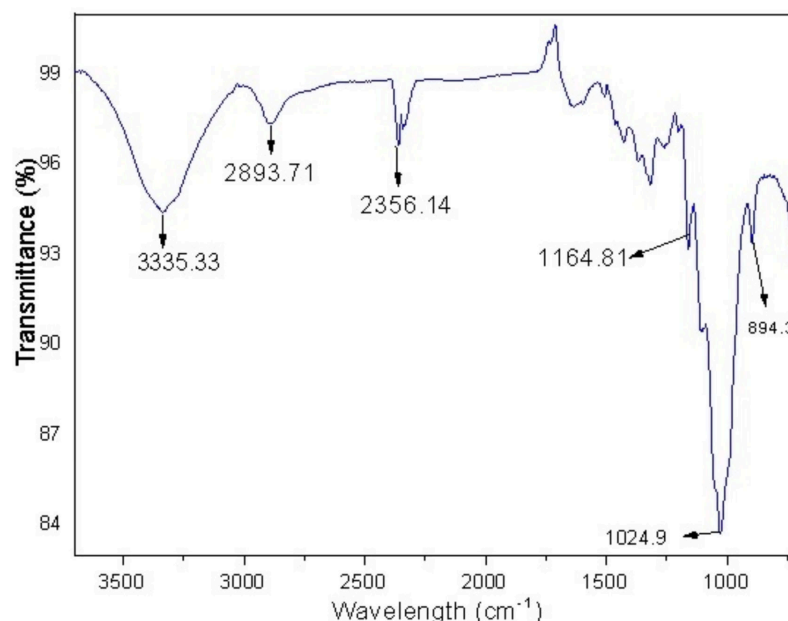
# 3. Results and Discussion

## 3.1. FTIR Spectroscopy Analysis

In the infrared spectrum as seen in **Figure 1** and **Table 1**, observed bands at 3322.32 cm<sup>-1</sup>, 1053.92 cm<sup>-1</sup>, and 893 cm<sup>-1</sup> are associated with the -OH groups. These are responsible for the hydrophilicity of the intramolecular hydrogen bonds, symmetric stretching of the primary alcohol, and  $\beta(1 \rightarrow 4)$  glycosidic bonds from cellulose, respectively. The band at 2893.71 cm<sup>-1</sup> is associated with stretching in the CH, CH<sub>2</sub>, and CH<sub>3</sub> molecules, indicating the complete conversion of cellulose type I to cellulose type II after alkaline hydrolysis.

In the spectrum, the characteristic bands for lignin found at 1730 cm<sup>-1</sup> and 1595 cm<sup>-1</sup>, which correspond to the acetyl groups and vibrations of aromatic rings, respectively, do not appear. This demonstrates the elimination of lignin by the proposed procedure [14]. The percentage of lignin extracted from the cane bagasse was approximately 50 %, after having considered that it was subjected to an alkaline treatment for 24 hours, guaranteeing the removal of lignin from the structure of the cane bagasse. When applying an alkaline pre-treatment, the removal of the lignin present in the material was possible to prepare it for later use.

**Figure 2** shows the FTIR spectrum of CDAC cationized cellulose. In **Table 2** presents the bands associated with cationized cellulose with the band at 1459 cm<sup>-1</sup> corresponding to the methyl groups, the band at 1420 cm<sup>-1</sup> corresponding to the C-N bond of the quaternary ammonium group of the modified cellulose, and the band at 1504.03 cm<sup>-1</sup> corresponding to the bending of the CH<sub>2</sub> groups. The N-N bond is seen in the band at 923.75 cm<sup>-1</sup>, which corroborates the modification of cellulose in the methodology proposed by [15] [16] and agrees with the results included in [1, 17].



**Figure 1.** Infrared spectrum of sugar cane bagasse cellulose.

### 3.2. Thermal properties of CDAC

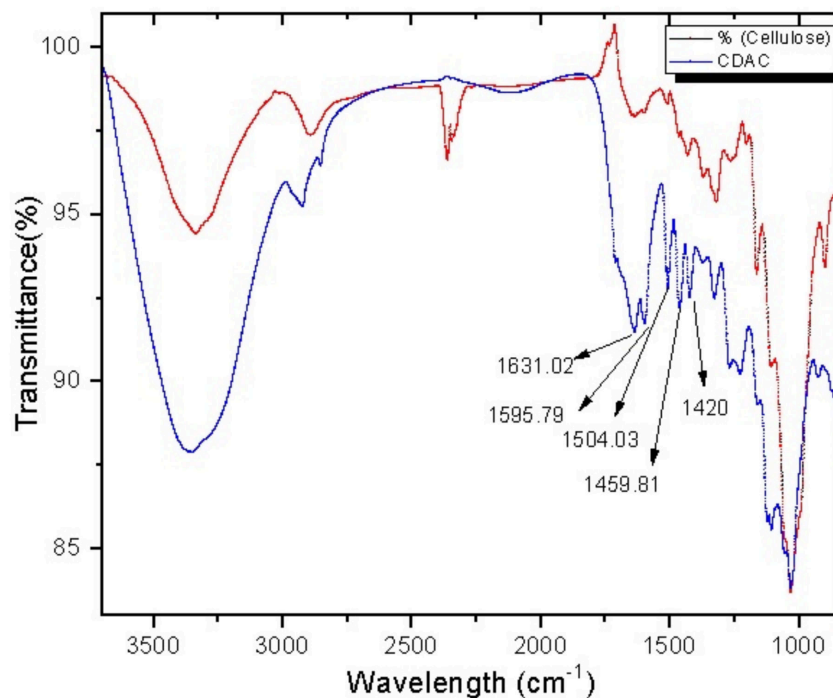
Figure 2 presents the variations of CDAC in response to heat treatment. Initially, a loss in mass is recorded, followed by an equilibrium, then an even greater decrease in mass until the final equilibrium is reached after 600 °C.

A loss in mass of 2.122 mg (19 % of decomposed CDAC) was seen, corresponding to a structural change in the compound because of calcination. Additionally, an endotherm corresponding to cellulose is observed ranging from 299 °C to 44 °C, with a loss of mass of 0.68 mg or 6 % with respect to the residual cellulose. This result is like the one reported by [24].

The thermal stability behavior of the extracted cellulose was studied, in which an absence of lignin and hemicelluloses peaks was observed. In contrast, the characteristic peak for cellulose is observed as described by [13].

**Table 1.** Absorption bands for the spectrum of cellulose in sugar.

Wavelength	Assignment of absorption bands for bagasse cellulose
3335.33	—OH stretching
2893.71	CH, CH <sub>2</sub> , and CH <sub>3</sub> stretching
2356.14	H—O—H flexing of water adsorbed
1426.60	Flexing of CH <sub>2</sub>
1361.03	C—H deformation
1257.38	C—O stretching of ether bond
1024.90	C—O—C asymmetric bridge stretching
894.30	C-O symmetrical stretching of a primary alcohol



**Figure 2.** Infrared spectrum for caationized cellulose.

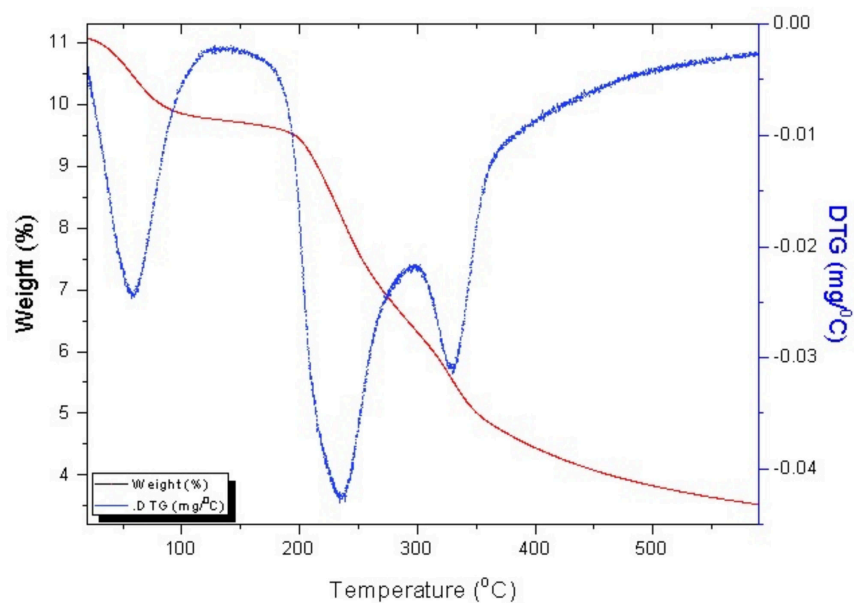
In **Figure 3**, the results of the TGA and DTA speed analyses of loss in mass expressed in (%/min) at 52.20 °C (First peak in Figure 3) are shown. These peaks can be attributed to water loss. After 100 °C, a second loss of mass occurs between 290 °C and 380 °C with a maximum loss rate (%/min) occurring at 358.85 °C. This loss of mass is associated with cellulose which was 6.6444 mg, corresponding to 80 % of the initial mass (8.3230 mg) and 2 % of ash corresponding to 0.2226 mg.

The figure does not contain intermediate peaks which are usually associated with the rapid degradation of hemicellulose. This typically occurs at temperatures lower than that of cellulose (220 °C to 315 °C), due to its amorphous nature as stated by [13]. As a result, the extraction of cellulose was deemed to be adequate, since no traces of hemicellulose are evident. There was also an 81.42 % loss in mass between 220 °C and 340 °C, which would be associated with cellulose pyrolysis [18, 19].

**Table 2.** Assignment of the main.

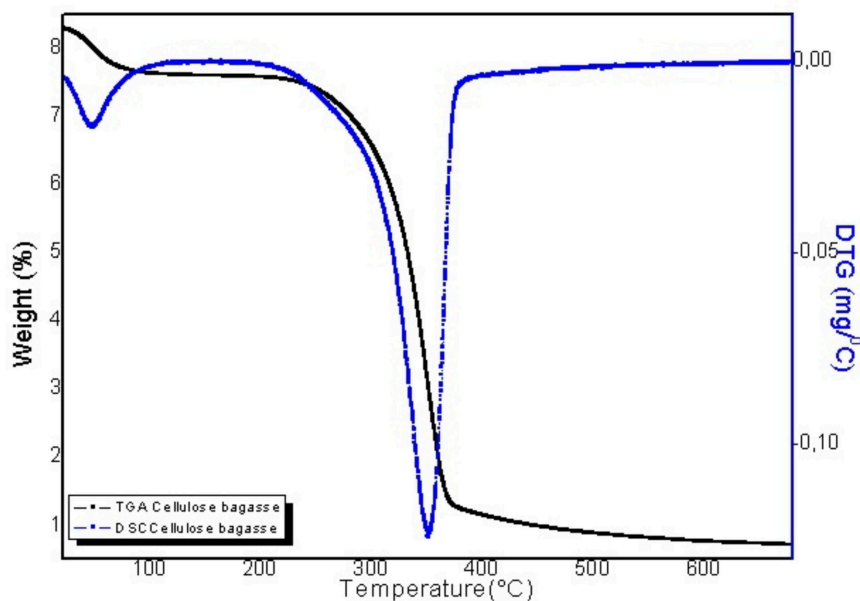
Wavelength	Assignment of absorption bands for CDAC
3322.00	—OH stretching
1631.00	Carbolnyl groups and nitrogen bonds of the GT
1595.00	C—N—C
1504.00	Flexing of CH <sub>2</sub> bond of CDAC
1459.00	Methylene and methyl groups folding
1420.00	C—N quaternary ammonium group
923.75	N—N bond of GT





**Figure 3.** Thermal properties of CDAC.

Table 2 summarizes the loss of mass for each of the stages of the thermo gravimetric analysis of the sample. The sample shows a 6.6444 mg loss of mass associated with cellulose corresponding to 80 % of the initial mass (8.3230 mg) and a 2 % loss of ash corresponding to 0.2226 mg. On the other hand, the % humidity and relative humidity of the extracted cellulose in relation to the loss of mass of the sugar cane bagasse resulted in  $T_{\text{initial}} = 202^{\circ}\text{C}$ ,  $T_{\text{final}} = 352.3^{\circ}\text{C}$ ,  $T_{\text{loss}} = 91.07\%$ , and  $T_{\text{residual}} = 7.60\%$ .



**Figure 4.** DCS and TGA of cellulose and cane bagasse.



### 3.3. X-ray diffraction (XRD) and X-ray diffraction analysis (XRD)

The crystallinity of the cellulose extracted from the sugar cane bagasse was analyzed using XRD and the diffractogram is presented in **Figure 4**. The characteristic crystalline phases of the cellulose obtained using alkaline methods are shown according to the methodology proposed by [12].

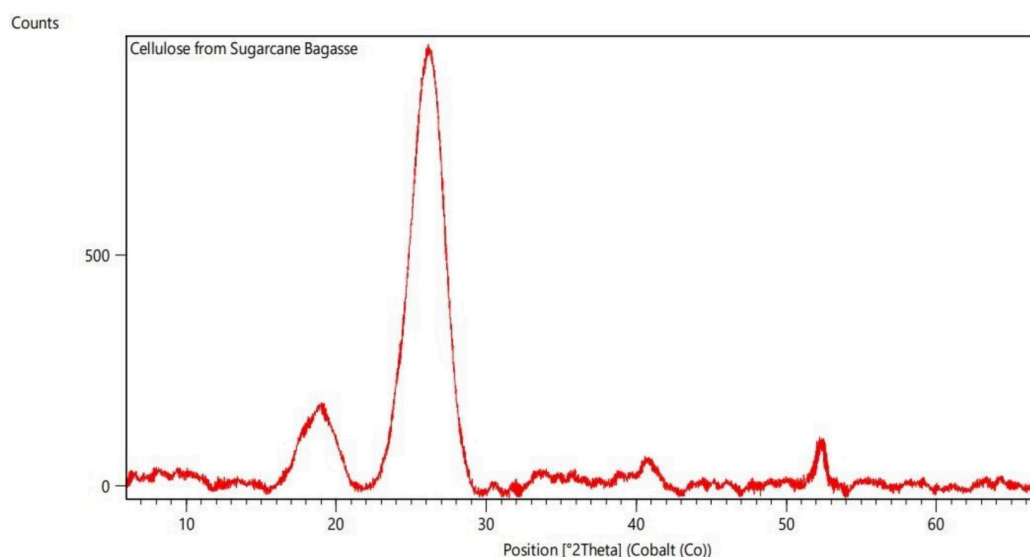
As described in [20], two well-defined peaks are evident. The peak of maximum intensity was found around  $2\theta = 26.2^\circ$  corresponding to the plane (200) of the crystalline phase of cellulose. Another peak at  $2\theta = (19.3 \pm 0.7)^\circ$  corresponding to the plane (110) are characteristic of cellulose as stated in [20, 14]. These two peaks are associated with the amorphous phase of the natural polymer due to the low ordering degree in the carbohydrate chains.

The crystallinity index was measured using the height of the maximum peak  $I_{200}$ ,  $2\theta = 26.5^\circ$ , and the minimum peak  $I_{am}$ ,  $2\theta = 18.6^\circ$ , which represents the crystalline and amorphous nature of cellulose. The crystallinity percentage of the cellulose obtained was 77.2 %, which was like that reported by [21].

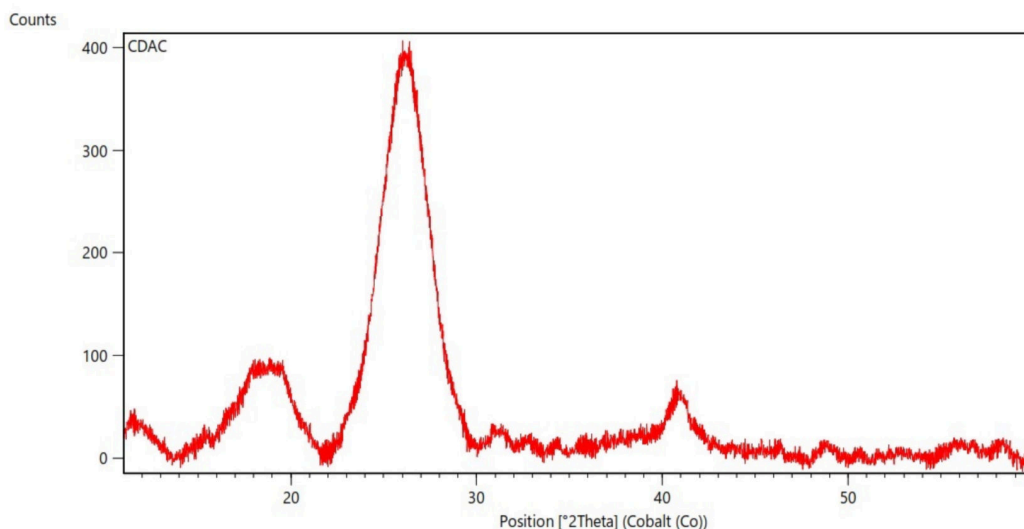
Cationized cellulose has two well-defined peaks; the highest intensity peak at  $2\theta = 26.2^\circ$  corresponding to the cellulose plane (200) which defines the reduction of the crystalline phase of cationic cellulose; and a lower intensity peak at  $2\theta = 18.6^\circ$  corresponding to the (110) plane, which shows the decrease in crystallinity of cellulose when it is cationically modified with a crystallinity index of 75 %. As seen in **Figure 5**, its crystallinity is reduced by 2 % which agrees with the results described by [22].

### 3.4. Scanning Electron Microscopy (SEM) Analysis

The morphology of cationic cellulose shows an alteration with respect to the morphology of unmodified cellulose caused by the cationization reaction. **Figure 7** corresponds to the unmodified cellulose in which disaggregated fibers with a smooth surface and an approximate diameter of  $(4.5 \pm 1.0) \mu\text{m}$  can be seen.



**Figure 5.** XRD of cellulose extracted from sugarcane bagasse.

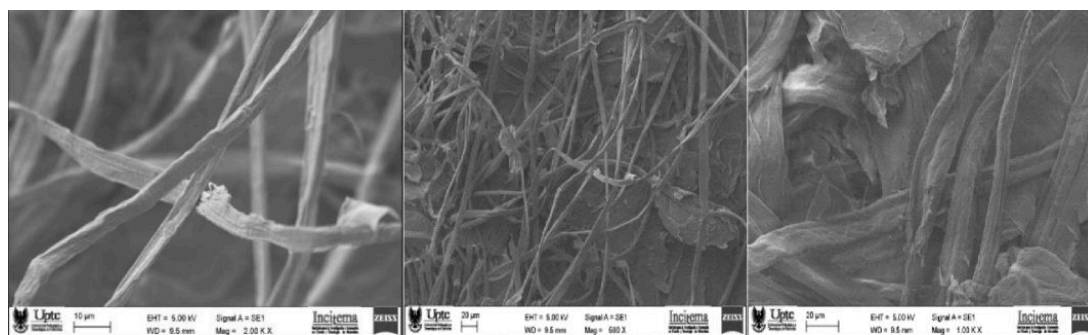


**Figure 6.** XRD of cationized cellulose.

In **Figure 8**, the cationic cellulose has a rough and irregular morphology, and the size of the fibers is reduced from  $4.5\ \mu\text{m}$  to  $1.94\ \mu\text{m}$ . This results in an increase in the surface area of the cellulose, which is consistent with the results published by [23]. The modifications in the surface of the fibers after the cationization process are derived from the changes not only at the charge level in the structure of the original cellulose, but also from the presence of the functional groups of greater volume and mass. These originated from the substitution of  $-\text{OH}$  due to the reaction with Grignard's reagent.

From the micrographs of the extracted sugar cane bagasse cellulose showed in Figure 7, a fibrous structure is observed with the presence of exfoliated layers that correspond to the primary walls of the sugar cane bagasse without chemical treatment [14]. Once the lignin was removed by oxidation reactions and the subsequent hydrolysis of the hemicelluloses, the cellulose microfibrils that were separated from each other are observed.

Figure 8 highlights the rough and irregular morphology of the fibers. It also demonstrates a decrease in size of the fibers from  $4.5\ \mu\text{m}$  to  $1.94\ \mu\text{m}$ . This results in an increase in the surface area of the cellulose which is consistent with the results published by [23]. The modifications in



**Figure 7.** CEM micrographs of sugarcane bagasse cellulose.

the surface of the fibers after the cationization process are probably derived from the changes not only at the charge level in the structure of the original cellulose, but also from the presence of the functional groups of greater volume and mass. These originated from the substitution of  $-OH$  due to the reaction with Grignard's reagent.

A greater detail of the morphology of the cellulose microfibrils is presented in the micrograph seen in Figure 8. Here, the separation between them can be seen which comprises a network of long and tangled cellulosic filaments. It can also be seen from these micrographs that the diameter is  $(4.5 \pm 1.0) \mu m$  and that they are oriented and appear on the surface, which indicates that it was a favorable extraction [20].

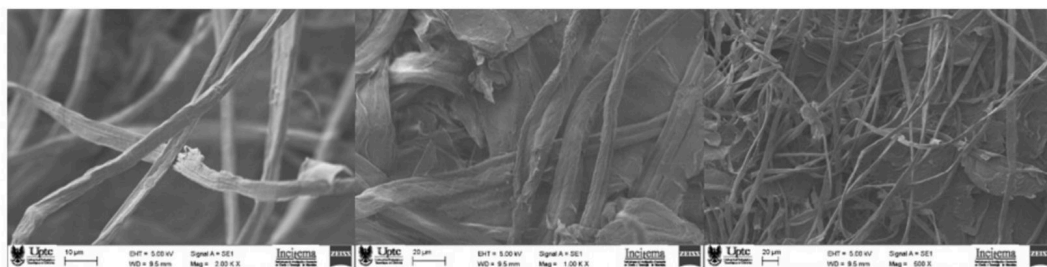
### 3.5. Comparison of the flocculating activity of CDAC with conventional clarifiers

To demonstrate the flocculating activity of the cellulosic derivatives obtained in this study, an attempt was made to simulate the process performed at a conventional mill. The time and temperatures at which the most used flocculants are added and whether vegetable mucilage or polyacrylamide was used was also simulated. A more complete study of these activities is reported in the study by [3].

For the first clarification stage in the production of *panela*, the reference value for transmittance percentage was 33.3 % with the addition of 1.25 ml of the mucilage solution extracted from Balso, (*Heliocarpus Americanus* L.). In the second clarification stage, the transmittance percentage was 45.3 %, after adding 1.25 ml of flocculant.

By adding 1.25 ml of the cationized cellulose solution at a concentration of  $1360 \text{ mg l}^{-1}$ , a transmittance percentage of 32.5 % was achieved. In the second clarification stage, another 1.25 ml of the solution of the product synthesized in this study was added and a transmittance percentage of 47.2 % was obtained. A transmittance percentage of 30.6 % was achieved with the addition of 1.25 ml of the food grade cationic polyacrylamide solution ASROFLOC SA, with a concentration of  $1000 \text{ mg l}^{-1}$ ; a transmittance of 43.1 % was observed with an additional volume of 1.25 ml.

In **Figure 9**, the three clarification processes of the sugar cane juices are compared. There was no difference between the use of the mucilage and cationized cellulose solutions produced in this study, as in the first stage as well as for the second clarification stage. As a result, it can be inferred that the cationized cellulose proposed in this study can be a promising replacement for the mucilage extracted from Balso (*Heliocarpus Americanus* L.). The polyacrylamide solution presented a slightly lower value in the clarification of sugar cane juices than the above-mentioned solutions.

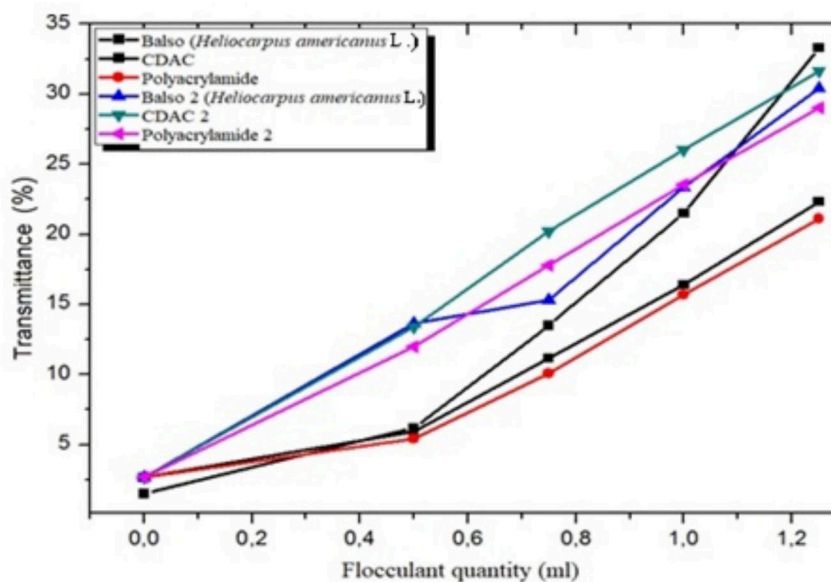


**Figure 8.** CEM micrographs of cationic cellulose.

**Table 3.** Transmittance values, at 760 nm, applying different doses of CDAC in comparison with extracts of Balso and polyacrylamide. FC = Flocculant quantity.

Wavelength $\lambda = 760$ nm			
First dosage			
FC (mL)	Balso ( <i>Heliocarpus Americanus</i> L.)	CDAC	Polyacrylamide
0.00	1.5	1.5	1.5
0.50	6.2	6.7	5.9
0.75	13.5	14.9	13.2
1.00	21.5	23.2	22.1
1.25	33.3	32.5	30.6
Second dosage			
FC (mL)	Balso ( <i>Heliocarpus Americanus</i> L.)	CDAC	Polyacrylamide
0.00	1.5	1.5	1.5
0.50	18.9	18.5	16.2
0.75	21.5	29.2	25.4
1.00	34.1	38.4	34.4
1.25	45.3	47.2	43.1

**Figure 10** presents the images of the sugar cane juice clarified with the three flocculating solutions used in this study. It is important to highlight that the floc formed from the CDAC and Balso (*Heliocarpus Americanus* L.) solution underwent a flotation process unlike the floc formed with

**Figure 9.** Clarifying comparison of flocculant CDAC, polyacrylamide, and Balso *Heliocarpus americanus* L., on sugarcane juices.

the polyacrylamide solution which settled. This is important in the technological process of producing *panela* whose equipment is designed for collecting floating flocs. This is advantageous for proposing cationic cellulose as a substitute for the B also mucilage that is used in the industry.

#### 4. Conclusions

- The synthesized cationic cellulose showed characteristic bands in the FT-IR spectrum such as the band at  $1420\text{ cm}^{-1}$  of the C–N bond of the quaternary ammonium group, and the band at  $1595\text{ cm}^{-1}$  belonging to the imine bond, characteristic of the C–N–C groups that allowed to corroborate the efficiency of the reaction in this work.
- The transmittance percentage values for the first stage (32.5 %) and for the second stage (47.2 %) in the sugarcane juices used in the production of *panela* were like those obtained with the Balso mucilage (*Heliocarpus americanus* L. and cationic polyacrylamide.
- The bagasse from sugar cane, usually considered a byproduct from the production of *panela*, if subjected to adequate extraction and bleaching processes, provide cellulose for its use as a substitute of polyacrylamide and vegetable extracts such as that from Balso (*Heliocarpus americanus* L.).

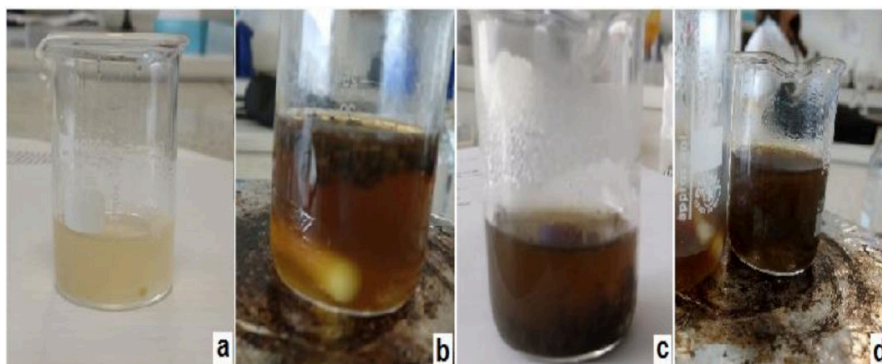
#### 5. Acknowledgements

Authors express gratitude:

To the director of research studies. DIN-UPTC, for managing the resources necessary to perform this study.

To the INCITEMA group of the UPTC, for their collaboration in the preparation and characterization of the samples.

To Engineer Karol Sarasty Zambrano, from Fedepanela for accompanying us to Trapiche Buena Vista in the municipality of Santana, Boyacá, and to Mr. Rodolfo Peña for allowing us to take a sample of the sugarcane juice from his factory.



**Figure 10.** (a) Synthesized CDAC Bio flocculant solution. (b) Sugarcane juice clarified with CDAC. (c) Sugar cane juice clarified with polyacrylamide. (d) Sugar cane juice clarified with mucilage solution extracted from Balso (*Heliocarpus Americanus* L.)

## 6. Conflict of Interest

The authors certify that they have no affiliations with or involvement in any organization or entity with any financial interest (such as honoraria; educational grants; participation in speakers' membership, employment, consultancies, stock ownership, or other equity interest; and expert testimony or patent arrangements), or non (such as personal or professional relationships, affiliations, knowledge or beliefs) in the subject matter or materials discussed in this manuscript. Potential conflicts of interest related to individual authors' commitments. Potential conflicts of interest related to commitments of editors, journal staff, or reviewers.

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### Extracción selectiva y modificación de la celulosa del bagazo de caña de azúcar (*Saccharum officinarum*)

**Resumen:** El objetivo de este trabajo fue la síntesis de un floculante útil en la producción de panela como una alternativa para remplazar la poliacrilamida en la producción de panela y mejorar la seguridad alimentaria en Boyacá, un distrito colombiano. La celulosa fue extraída mediante un tratamiento con hidróxido de potasio seguido de hidrólisis ácida. A partir de la celulosa extraída se sintetizó celulosa dialdehídica catiónica (CDAC) y tanto la celulosa como la CDAC se caracterizaron mediante espectroscopia infrarroja, FTIR, análisis termogravimétrico (TGA), análisis térmico diferencial (DSC), microscopía electrónica de barrido (SEM) y RAMAN. La celulosa extraída por hidrólisis del bagazo de la caña de azúcar con blanqueo produjo un rendimiento del 50 % y un índice de cristalinidad del 77 %. Su modificación a aldehído es fiable en un 95 % a una temperatura de 65 °C y 2 h; con temperaturas y tiempos mayores, el rendimiento se ve afectado. El proceso de dialdehído permite llevar a cabo una modificación y ser atacada más fácilmente en los carbonos 2 y 3, dejando celulosa cationizada para su posterior uso como floculante en el proceso de producción de panela como una solución frente a floculantes tradicionales y poliacrilamida. Tanto la extracción como la modificación y las pruebas de clarificación del jugo de panela se realizaron por triplicado.

**Palabras clave:** caña de azúcar; celulosa; floculante; poliacrilamida.

### Extração seletiva e modificação de celulose do bagaço de cana de açúcar (*Saccharum officinarum*)

**Resumo:** O intuito deste estudo foi sintetizar um floculante que fosse útil na produção de rapadura como alternativa ao uso de poliacrilamida para melhorar a segurança alimentar em Boyacá, um distrito colombiano. A celulose foi extraída mediante um tratamento com hidróxido de potássio, seguido por hidrólise ácida. A partir da celulose extraída foi sintetizada celulose dialdeídica catiônica (CDAC) e tanto a celulose como a CDAC foram caracterizadas usando espectroscopia de infravermelho, FTIR, análise termogravimétrica (TGA), análise térmica diferencial (DSC), microscopia eletrônica de varredura (SEM) e RAMAN. A celulose extraída do bagaço de cana de açúcar por hidrólise com branqueamento teve um rendimento de 50 % e um índice de cristalinidade de 77 % e sua modificação ao aldeído é 95 % confiável a uma temperatura de 65 °C e 2 h, maiores temperaturas e tempos afetam o rendimento. O processo de dialdeído permite realizar uma modificação para atacar com maior facilidade os carbonos 2 e 3, deixando celulose cationizada que pode ser usada como floculante na produção de rapadura, oferecendo uma alternativa aos floculantes tradicionais e à poliacrilamida. A extração, modificação e provas de clarificação do suco de rapadura foram realizados em triplicado.

**Palavras-chave:** cana de açúcar; celulose; floculante; poliacrilamida.

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