

Characterization and Physicochemical Analysis of Carboxymethyl Cellulose Extracted From Sugarcane Baggasse: A Greener Approach to Waste Management

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Abstract

In Nigeria, Sugarcane Bagasse (one of the by-products of sugarcane processing) is typically seen as an agricultural waste with few commercial utilization and the accumulation of this sugarcane bagasse is considered as an environmental concern as it causes environmental pollution in areas where it is disposed. The conversion of this waste to wealth was studied by way of revealing its remarkable potential for use as a grant of cellulose (an unconventional source) for the manufacturing of cellulose derivatives (derivatized herbal polymers) such as Carboxymethyl Cellulose (CMC) which is quite referred to for its good sized industrial applications. 39.2% of cellulose was taken out from sugarcane bagasse via alkali remedy and nitric acid hydrolyzed process. The cellulose was then remodelled to carboxymethyl cellulose (CMC) thru mercerization with varying sodium hydroxide (NaOH) concentrations (10%, 20% and 30% respectively) and therefore etherified with monochloroacetic acid at unique working reaction instances (1h, 1.5h and 2h respectively). The cellulose samples were designated as 10% CMC₁, 20% CMC_{1.5} and 30% CMC₂ respectively. The derivatized samples of cellulose had been characterised for their bodily evaluation and chemical compositions. Parameters studied have been hydrated density, water and oil retention capacities, hemicellulose content, lignin content, moisture and ash contents. Fourier Transform Infrared Spectroscopic assessment was carried out on all samples of derivatized cellulose for their micro-structural studies. The absolute degree of substitution (DS_{abs}) of all derivatized cellulose samples was studied and increased up to 20% (w/v) of the derivatized cellulose prepared at 1.5h reaction time. This increment was attributed to greater carboxymethyl organizations substituted by the usage of the hydroxyl organizations of the cellulose polymers which in flip transformed the crystalline vicinity in cellulose to an amorphous place with increasing NaOH concentrations thereby improving the attainability of CMC to immobilize water in a system. A decrease in DS of CMC synthesized at 30% (w/v) NaOH concentration was due to interaction of part of monochloroacetic acid (MCA) molecule with NaOH and this resulted into low yield of the derivatized CMC produced at 30% (w/v) NaOH concentration.

Keywords: Sugarcane Bagasse, Cellulose, Carboxyl Methyl Cellulose (CMC), Natural Polymer, Synthesis, Characterization

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I. Introduction

Sugarcane (*Saccharum officinarum*) is a C4 plant and it is in a position to convert up to 1% of incident photo voltaic electricity into biomass. It is one of the most giant industrial cash plants that is commonly grown in over 200 nations during the world in tropical and subtropical climates the place moist and dry seasons alternate. The pinnacle producers of sugarcane in order of production, on a world groundwork are Brazil, India, China, Thailand, Pakistan and Mexico. Australia, South Africa, Cuba, Peru, Sudan, Egypt amongst others have little brilliant influence as producers of sugarcane on a world degree manufacturing (Alvaris, 2008; Kajima, 2012). In the year 2011, Brazil produced 625 million tonnes of sugarcane accompanied by India and China (Chandel et al., 2012; FAO, 2015). In Nigeria, Sugarcane is usually grown generally in most northern states of the country such as Adamawa, Niger, Sokoto, Taraba, Kano, Katsina, Jigawa, as correct as in Kwara state, Kogi State and Ogun State (papalanto) and over 15 million a lot of sugarcane was once produced in the year 2013 (Agboire et al., 2002). Basically, sucrose extraction is the germane cause at the back of sugarcane plantation across the globe for the synthesis of sugar (Agboire et al., 2002). Apart from sugar extraction from sugarcane,

the tender specie of sugarcane is often chewed by numerous Nigerians for its sweetness and nutrient content. Tender sugarcane can also be domestically processed into “Mazarkwaila” and “Elewa” used singly or mixed in foods (Agboire et al., 2002).

After the pressing process of sugarcane for juice extraction, a solid fibrous residue otherwise known as sugarcane bagasse is often generated in huge amount as a by-product. Sugarcane bagasse is often considered as an agro-industrial waste whose accumulation creates environmental pollution as they form heaps in areas where they are disposed. However, in an effort to solve this complicated waste problem by turning it to wealth and conserve the environment, advances in green technology for sustainability and carbon neutrality has geared towards economic utilization of renewable biomass often considered as agricultural wastes in the manufacture of value added products that will be of immense use to the entire humanity across the globe (Lois-Correa, 2012; Loh et al., 2013; De Souza et al., 2013; Bizzo et al., 2014; Lombardi et al., 2015; Dotaniya et al., 2016; Sampaio et al., 2019; Nunes et al., 2020; Quiroga et al., 2020).

Renewable biomass are lignocellulosic substances which are identified to be the most good sized residue on earth comprising of cellulose which is a linear polysaccharide (high molecular weight biopolymer) made up with the useful resource of β -1, 4-D-glucose units, hemicellulose which is a heterogeneous polysaccharide having differentiated quantities of hexose and pentose sugars and lignin which is an aromatic macromolecule that gives an immoderate complexity and recalcitrance to the lignocellulosic form (Singh and Khatri, 2012). Sugarcane bagasse is an intricate material that belongs to the type of renewable biomass that are lignocellulosic in nature comprising of cellulose, hemicellulose and the complex substance known as lignin that is mostly composed of fragrant phenolic compounds which gives stiffness to the sugarcane fiber. The relative extent of these factors in the bagasse depend on the breed of the sugarcane, its age and the size of the stems (Mandal and Chakrabarty, 2011). Millions of tons of sugarcane bagasse are generated on yearly basis globally and the primary constituent of the bagasse being the affluent cellulosic fraction can acquire up to (23-53%) in the complete fiber composition thereby making it viable for use as a source of cellulose for the manufacturing of large scale derivatized cellulosic substances that are used in a wide array of industrial fields (Varshney and Naithani, 2011; Singh and Singh, 2012). The complicated shape of sugarcane bagasse exists in such a way that, the cellulose fiber is embedded in a composite shape comprising brilliant non-cellulosic cementing materials. Hence, isolation of the pure cellulose from this complicated form has indeed come to be the purpose for sizeable look up work through many researchers. Native cellulose is a renewable biopolymer that has attracted a lot of interest globally for being an uncooked material for the manufacturing of many industrial products. It is estimated to be 1.5×10^{11} tons being generated globally on yearly basis in a pure way (Cao et al., 2009). Untreated cellulose in its pure form is a high molecular weight polymer that is linear, crystalline, biodegradable, non-toxic and insoluble in conventional natural solvents and water due to the presence of various strong inter and intra-molecular hydrogen bonding in its structure which must be broken if dissolution of cellulose is to be achieved for its good sized applicability (Klemm et al., 2005; Olayemi et al., 2020).

This research work focused on the removal of cellulose from sugarcane bagasse via pre-treatment followed by carboxymethylation of the extracted cellulose at different reaction times and different alkali concentrations. The effect of alkali concentration and working reaction time on the quantity of carboxymethyl cellulose (CMC) generated were investigated by performing indistinguishable reactions using three different alkali concentrations and three working reaction times. The aim of this work was to investigate the feasibility of increasing the economic value of the cellulosic agricultural waste and solve the problem of environmental waste pollution.

In the synthesis of carboxymethyl cellulose, extracted native cellulose is often reacted with the solution of sodium hydroxide to activate the native cellulose. When this activated alkali cellulose is reacted with monochloroacetic acid (MCA) or its salt (Na-MCA), a series of carboxymethylation reactions occur resulting in the evolution of carboxymethyl cellulose as the product of interest and Glycolic acid as the by-product (Stigsson et al., 2001;). During carboxymethylation reaction, each hydrogen atom in the unprocessed cellulose hydroxyl group is substituted with carboxymethyl group, which is responsible for the polymer's solubility in water. Carboxymethyl cellulose (CMC) is a linear, long chain, water soluble, anionic, odorless, tasteless, free-flowing, biodegradable, viscous, non-toxic, non-allergenic cellulose ether that has found use in industrial settings of medicine, pharmaceutical, petroleum, paper, detergent, cosmetics, textiles, food and confectionary (Varshney and Naithani, 2011; Singh and Singh, 2012; Lawal et al., 2018; Ogunneye et al., 2019).

II. Materials And Procedure

2.1 MATERIALS / CHEMICALS

Sugarcane stalks were purchased from a local market popularly known as “Shasha,” in Ibadan, Oyo state. Chemicals used in this research were analytically graded and these include Sodium hydroxide, Monochloroacetic acid (MCA), Sulfuric Acid, Hydrochloric Acid and Glacial Acetic Acid which were supplied by Sigma-Aldrich. Absolute Methanol was supplied by Chem Tec Free Zone. Isopropanol was supplied by shell

chemicals. 95% ethanol was supplied by Ideal SMA. Nitric Acid was supplied by J.H.D. Benzene was supplied by B.D.H. Potassium Hydroxide was supplied by Lobachemie.

2.2 METHODOLOGY

2.2.1 ESTIMATION OF SUGARCANE BAGASSE CHEMICAL COMPOSITIONS

2.2.2 ESTIMATION OF MOISTURE CONTENT (ASTM, 2005: D 2216).

10 g of ground bagasse was weighed accurately in a dry Petri dish and dried at 105°C for three hours. The weighed bagasse was allowed to cool in a desiccator, reweighed and the new weight was written down. The drying-cooling-weighing manner was ingeminated till a constant weight was attained. The value of the moisture was estimated using the equation below:

$$\text{Moisture content (\%)} = \frac{W_1 - W_2}{W_1} \times 100$$

Where

W_1 = Weight of the moist bagasse sample.

W_2 = Weight of the dry bagasse sample.

2.2.3 ESTIMATION OF ASH CONTENT (ASTM, 2005: D 2866).

About 3g of powdered bagasse was put in an accurately weighed porcelain crucible (A g). Both the crucible and the bagasse were collectively weighed and written down as(B g). The weighed bagasse was ashed in an electric muffle furnace, at 625 ±25°C for 6 h, cooled in the desiccator for 15 minutes and the crucible holding the ash was weighed (C g). The presence of unburnt particles was confirmed with the usage of a platinum wire in order to ensure complete ashing. The ash content material was calculated using the equation below:

$$\text{Ash content (\%)} = \frac{C - A}{B - A} \times 100$$

Where

A = Weight of empty crucible (gram)

B = Weight of crucible and sample (gram)

C = Weight of crucible and ash (gram)

2.2.4 ESTIMATION OF LIGNIN CONTENT

Lignin, expressed as Klason lignin used to be estimated directly in accordance to the method of the Institute of paper chemistry, Appleton, Wisconsin 428, 1951 as follows:

Ground bagasse was first removed with ethanol – benzene mixture (1:1) for 8 h and dried at room temperature. One gram of the extracted sample was once put in a one hundred ml beaker and then treated with 20ml of 72% sulfuric acid, which was delivered to the weighed bit of cellulose drop-wise with uniform mixing using a small glass rod. After total disintegration, the reaction was once allowed to stand and the beaker was covered with a watch glass and left over night at room temperature. It was once then transferred quantitatively to a 1 litre round bottom flask, diluted to 3% sulfuric acid, and boiled for 4 hours underneath reflux. The lignin was filtered on a pre-weighed filter paper and washed with warm distilled water until neutrality. The lignin was once then dried at 105°C for 6 h and gravimetrically estimated in accordance to the following equation:

$$\text{Lignin content (\%)} = \frac{\text{weight of lignin}}{\text{weight of pattern}} \times 100.$$

2.2.5 ESTIMATION OF HEMICELLULOSES.

Hemicellulose content was once determined with the usage of the technique described through Abdel-Halim (2014). Accurately weighed ground bagasse material (X) was extracted with 10% KOH using a material to liquor ratio of 1:20 for 10 h at 50°C. At the end of the extraction process, the device used to be allowed to cool and filtered. The filtrate was once made acidic through use of glacial acetic acid until pH 6 was attained. The filtrate was once then combined with a solution of two volumes of ethanol. Precipitate recovery was achieved via filtration and freeze-drying and then weighed (Y). The hemicelluloses content material (%) was evaluated with the usage of the equation below:

$$\text{Hemicellulose content material (\%)} = \frac{Y}{X} \times 100.$$

2.3 EXTRACTION OF CELLULOSE FROM SUGARCANE BAGASSE (SCB).

The technique employed for cellulose isolation is outlined in fig.1. Firstly, freshly accrued sugarcane stalks have been reduced into portions with a knife and then beaten manually (chewed) leaving the fibrous residues behind which is known as bagasse containing minute quantity of sugar residues. These sugar residues were totally washed off from the bagasse using water after which the bagasse was then dried. The sugar residue was removed in order to decrease any microbial attack in the course of sun-drying to 8% moisture. The dry bagasse was once collected, cut into small pieces and then grinded into first-class particles (powdered form) with the use of a milling machine. 25 g of sugarcane bagasse powder was subjected to cooking (95 °C) with 750 ml of 0.5 M

NaOH for two hours at uniform mixing. The dark slurry acquired was filtered and washed with 1 L of distilled water and then dried. The dried cellulose was once refluxed with two successive parts of mixture containing 20 percent (v/v) of nitric acid in ethanol. The combination was then filtered and washed with cold distilled water until the filtrate did not turn to pink when phenolphthalein and a drop of 0.5 M NaOH was drop-wisely added to it (Filho, Rosana, Vieira, Meireles, Daniel, Hernane, Ribeiro and Messaddeq, 2007). The residue was dried in an oven at 60 °C overnight to consistent mass. Lastly, the dried cellulose was mashed into powder and stored in the polyethylene bag for the cellulose modification in the subsequent process.

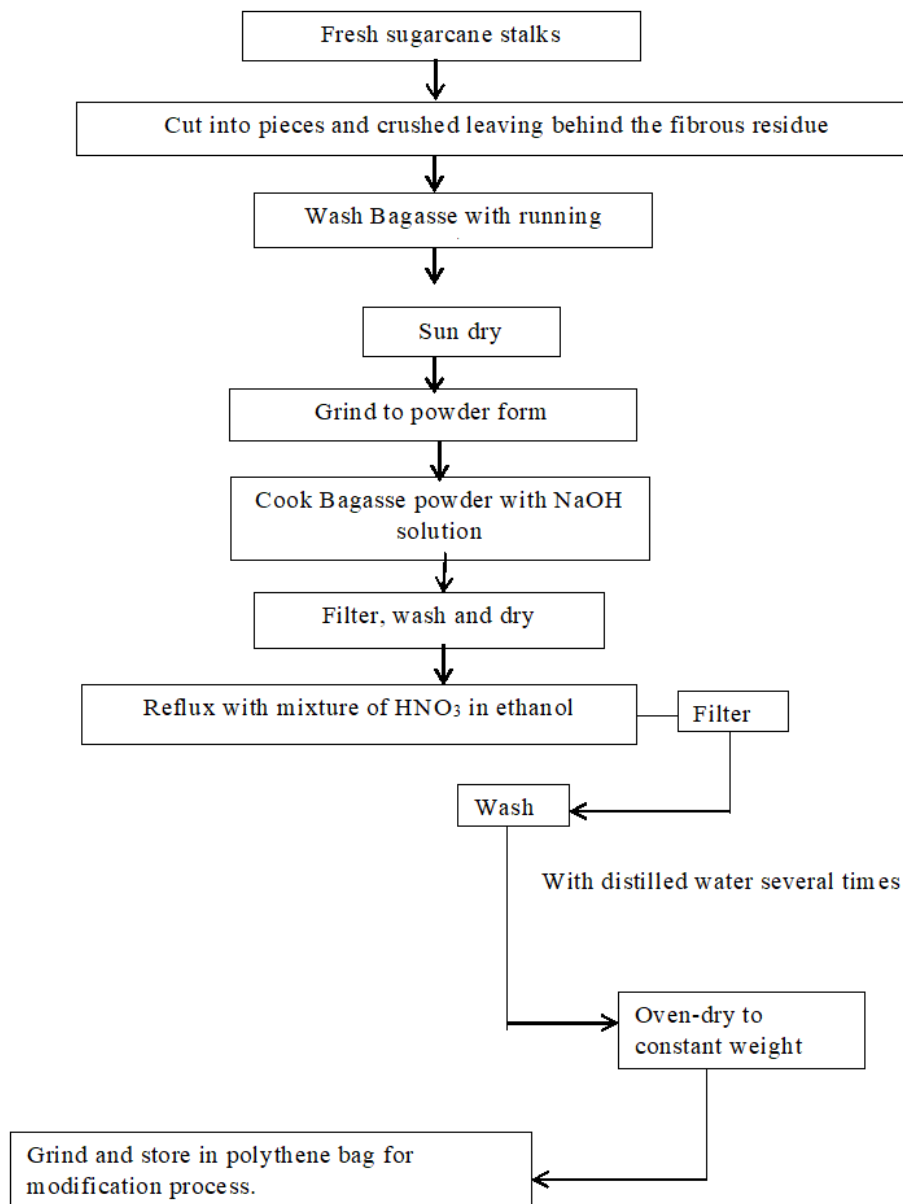


Fig. 1: Flow diagram for the isolation of cellulose from sugarcane bagasse

2.3.1 ESTIMATION OF THE CELLULOSE CONTENT

The cellulose content in percentage was quantified by finding the sum of hemicelluloses, lignin, moisture and ash and then discounted from 100. Alternatively, the yield of cellulose extracted from the SCB was quantified based on the dry weight basis. The yield of cellulose, expressed in a percentage, was quantified based on the amount of sugarcane bagasse (SCB) using the following equation:

$$\text{Yield of cellulose (\%)} = \frac{\text{Weight of cellulose obtained (g)}}{\text{Weight of ground bagasse used (g)}} \times 100\%$$

2.4 PREPARATION OF CARBOXYMETHYL CELLULOSE FROM SUGARCANE BAGASSE CELLULOSE.

Three samples of carboxymethyl cellulose from sugarcane bagasse had been synthesized according to the system described by using Bhattacharya, Singhal and Kulkarni (1995). First, accurately weighed 5 g of cellulose powder from SCB was suspended in one hundred and fifty ml of isopropanol and stirred mechanically at room temperature. Then, 15 ml each of (10%, 20% and 30% w/v) NaOH was added to the mixture in a drop wise manner and mixed for an hour to achieve alkaline cellulose. 6 g of MCA was incorporated into the mixture with non-stop stirring to achieve etherification reaction which in this case was carboxymethylation reaction, and the reaction time varied from 1 h to 2 h. The mixture was once covered with aluminium foil and positioned in an oven with warm air at 60 °C for 3.5 hours. Then the suspension was soaked in hundred cm³ volume of methanol overnight. The next day, the suspension was neutralized with 90% acetic acid to pH 7, and then filtered using a sintered funnel. The product of interest was washed three times by soaking in 50 ml of ethanol for 10 minutes to take away undesirable by-products, and then it was washed once more with one hundred ml of absolute methanol for the ultimate time. The ensuing CMC from SCB was once filtered and dried in an oven at 60 °C to a constant mass and stored in a dry place.

By purification, approximately 5g each of earlier synthesized CMCs with different concentrations of NaOH was once dissolved in a hundred ml of distilled water at a temperature of 80°C with consistent stirring for 10 minutes. Each of the weighed product of interest was then centrifuged for 1 minute at 4000 rpm. The dissolved CMC was re-precipitated in 100 ml of acetone. The extracted CMC was once filtered and dried in an oven at 60 °C until a steady mass was got and saved in a desiccator for further analysis. The carboxymethylcellulose samples had been denoted as CMC_{1,0}, CMC_{1,5} and CMC_{2,0} in accordance to the working reaction time varied between 1-2 hours all through the chemical modification process.

2.4.1 ESTIMATION OF THE CARBOXYMETHYL CELLULOSE CONTENT.

The yield of CMC synthesized was measured based on the dry weight basis. The yield was mathematically assessed by dividing the net dry weight of CMC with 5g of dry cellulose according to the equation stated below:
Yield of CMC (%) = $\frac{\text{Weight of dried CMC (g)}}{\text{dry weight of cellulose (g)}} \times 100$.

2.5 ESTIMATION OF THE PHYSICAL (FUNCTIONAL) PROPERTIES OF NATIVE AND CARBOXYMETHYL CELLULOSE ISOLATED FROM SCB.

2.5.1 ANALYSIS OF THE NATIVE CELLULOSE AND CARBOXYMETHYL CELLULOSE FROM SCB FOR HYDRATED DENSITY.

The method adopted by Prakhongpan, Nitithamyong and Luanpituksa, (2002) was used to determine the hydrated density. A calibrated 10 ml graduate cylinder was filled with a known amount of distilled water (3mL), and a known weight of each sample (1g) was added carefully to prevent the sample from sticking to the cylinder's wall. The volume of the water before adding the sample was subtracted from the volume of water after adding the sample and the difference was put in record as mL of water displaced. Results were expressed as grams of the sample per mL of water displaced. The hydrated density was calculated using the following equation:

$$\text{Hydrated density (g/mL)} = \frac{\text{grams of sample (g)}}{\text{mL of water displaced.}}$$

2.5.2 ANALYSIS OF THE NATIVE CELLULOSE AND CARBOXYMETHYL CELLULOSE FROM SCB FOR WATER AND OIL RETENTION CAPACITIES.

The method described by Riantong, Worasit, Teeraporn and Chiraporn, (2014) was employed to analyze oil and water retention capacities. For the water retention capacity (WRC), 2 g of sample was mixed using a glass rod with 30 mL of distilled water in a 50 mL centrifuge tube. The slurry was allowed to stand for 10 min, and then centrifuged at 2000 × g for 15 min. After centrifugation, the supernatant was drained and the wet sample precipitate was weighed. The result was expressed as gram of water per gram of the sample. For the oil retention capacity (ORC), the adopted methodology was similar to the one described for the WRC except that vegetable oil was used in place of distilled or deionized water. The WRC and ORC were determined using the equation below:

$$\begin{aligned} \text{WRC (g water / g dried sample)} &= \frac{\text{gram of water}}{\text{gram of the sample}} \\ \text{ORC (g oil / g dried sample)} &= \frac{\text{gram of oil}}{\text{gram of the sample}} \end{aligned}$$

2.6 CHARACTERIZATION OF CARBOXYMETHYL CELLULOSE

2.6.1 DETERMINATION OF THE ABSOLUTE DEGREE OF SUBSTITUTION (DS_{abs}) OF CARBOXYMETHYL CELLULOSE

The method employed for the determination of the degree of substitution was titrimetry using the standard method (ASTM, 2005; D1439). In this case, 4 g of the dry powder of sodium carboxymethyl cellulose (Na-CMC) was dispersed in 95 % ethanol (75mL) for 5 minutes. Then, 5 mL of 2 M nitric acid was incorporated, and the dispersion stirred to convert Na-CMC to its acid or its protonated form, H-CMC and the

solution was subjected to boiling using hot plate. Afterwards, the solution was removed from the hot plate and further stirred for 10 min.

The mixture was then separated into two parts, which was a solid and liquid phase. The liquid phase was removed, and the solid phase was washed with 20 mL of 80 % ethanol at 60°C for 5 times. Then, the precipitate was washed with a small quantity of anhydrous methanol and filtered. Lastly, the precipitate was dried at 100°C for 3 h and cooled in a desiccator over silica gel for half an hour. 0.5 g of dry H-CMC was weighed in 250 mL Erlenmeyer flask and 100 mL of distilled water was added and stirred. Then, 0.3M NaOH (25mL) was added and heated to boil for 15 min. After the dissolution of the product, the mixture was titrated with 0.3 M HCl. Phenolphthalein was added to observe the color change from dark pink to colorless. The titration was repeated twice, and the average volume of HCl used was recorded. The DS was determined as follows (Elomaa, et al., 2004).

$$\text{Degree of substitution (DS}_{\text{abs}}) = 162 \times \% \text{CM} / [5900 - (58 \times \% \text{CM})]$$

Where %CM is regarded as carboxymethyl content and it is mathematically expressed as:

$$\% \text{CM (carboxymethyl content)} = [(V_0 - V_n) M \times 0.059 \times 100] / m$$

Where

V_0 = volume of hydrochloric acid used to titrate blank (mL).

V_n = volume of hydrochloric acid used to titrate samples of modified cellulose (mL).

M = molar concentration of hydrochloric acid used which in this case was 0.3M.

m = weight of modified cellulose taken (g).

162g/mol is denoted as the molar mass of anhydroglucose unit.

58 g/mol is designated as the molar mass of carboxymethyl residue (-CH₂COOH).

2.6.2 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FT-IR ANALYSIS)

The functional groups of the cellulose and Carboxymethyl cellulose synthesized at various reaction times and sodium hydroxide concentrations were determined using Fourier Transform Infrared Spectrophotometer. The samples were dehumidified in an oven at 60°C. About 0.2 mg of each sample and 2 mg of potassium bromide were mixed and ground finely before the mixture was compressed to form a transparent pellet. The infrared spectra of these samples were measured in the transmission of a wavelength number between 4000 and 400 cm⁻¹

III. Results And Discussion

3.1 PROXIMATE ANALYSIS

TABLE 1: Chemical compositions of sugarcane bagasse.

Determination	Values (%)
Cellulose content	39.20
Hemicellulose content	28.00
Lignin content	21.13
Ash content	01.67
Moisture content	10.00

Table 1 indicates the chemical composition of sugar cane bagasse used for the synthesis of carboxymethylcellulose. The cellulose content obtained as a result of alkaline processing of sugar cane bagasse and subsequent hydrolysis with nitric acid was 39.2%, and it was white or cream in color. It has been cautioned that the output may additionally be due to the process used in the extraction process. The end result of cellulose yield showed a similar yield for stem fibers such as rice, wheat and barley, which consist of cellulose of about 28-48%, 29-51% and 31-45%, respectively, and by means of 26-43% in bamboo cane fibers (Han and Rowell, 1996). It has been conceived that the determined colour of the product shows that the delignification procedure (bleaching process) was not at some stage employed in the synthesis of cellulose. The important purpose of any bleaching treatment, irrespective of the nature of the bleaching agent, is to attain white cellulose fibers. This whiteness is accomplished via the elimination of the colouring agent that exists in the natural composition of cellulose fibrewith the aid of the use of a bleaching agent. Depending on the nature of the bleaching agent, it both oxidizes and restores the colouring substance, thereby destroying it to simpler compounds, which is therefore dissolved and washed out, resulting in lifelong whiteness. The decreased native cellulose content material yielded in this study suggests the existence of low molecular weight oligosaccharides, which might also affect the yield and first-rate of the derivative.

The values determined for hemicellulose, ash, moisture and lignin in the course of this findings are in Table 1. The hemicellulose content was higher than 25%, as formerly suggested in the research conducted by Abdel-Halim (2014). The higher content shows that the method used to determine it is not in a position to do away with most of the hemicellulose from the milled sugarcane bagasse. It has been pronounced that the

persistence of hemicellulose means that it is likely chemically crosslinked with cellulose and/or lignin. Crosslinking between hemicellulose, lignin and cellulose forms a secure three-d structure (Huber, 2006). As for the value of the lignin content material, a lower value than the 23.9% lignin suggested by Abdel-Halim (2014) was obtained. The got value is an indication that the process used in its determination allowed the elimination of most of the lignin, which is hydrophobic in nature. The moisture and ash content material had been greater and lower, respectively, in contrast with the result gottenby Abdel-Halim (2014) in similar atmospheric conditions.

3.2 PERCENTAGE YIELD OF CARBOXYMETHYL CELLULOSE SAMPLES

TABLE 2: The percentage yield of CMC synthesized with various NaOH concentrations.

Sample	Mass of cellulose	Mass of CMC	Yield	
		(g)	(g)	(%)
10% NaOH – CMC _{1,0}		5.00	5.50	110.00
20% NaOH – CMC _{1,5}		5.00	6.81	136.20
30% NaOH – CMC _{2,0}		5.00	6.63	132.60

10% NaOH-CMC_{1,0} = Carboxymethyl sugarcane bagasse cellulose prepared with 10% (w/v) of NaOH at 2h working reaction time.

20% NaOH-CMC_{1,5} = Carboxymethyl sugarcane bagasse cellulose prepared with 20% (w/v) of NaOH for 1.5 h working reaction time.

30% NaOH-CMC_{2,0} = Carboxymethyl sugarcane bagasse cellulose prepared with 30% (w/v) of NaOH for 2 h working reaction time.

Table 2 offers the yield of CMC synthesized with a variety of NaOH concentrations (10 – 30%) within the working reaction time studied (1-2 h). The yield of CMC prepared at non-identical reaction times (1-2 h) accelerated with increasing NaOH concentrations. This is due to the fact when native starch reacted with chloroacetic acid in an alkaline environment, the hydroxyl groups of the cellulose molecule were chemically changed into an alkoxide form (Cellulose-O-) which then again reacted with more chloro-acetic acid giving rise to a Cellulose –O-CH₂ –COO- form. Therefore, the rise in percentage yield is attributable to the presence of carboxymethyl moiety with their higher mass. In addition, it was determined that the yield of all samples of CMC also increased with increasing reaction time.

3.3 HYDRATED DENSITIES OF ISOLATED CELLULOSE AND CMCs FROM SCB

The hydrated densities of the isolated cellulose and prepared samples of CMCs are presented in Table 3. It was suggested that the value obtained for unmodified cellulose was an indication that all samples of CMCs have higher values of hydrated densities due to no incorporation of sodium hydroxide into the matrices of the native cellulose. The absence of alkali incorporation into the structure of cellulose lowered its surface area which in turn made the larger pores in the structure of the cellulose to be occupied coupled with a low degree of swelling capacity. Furthermore, lack of alkali incorporation also inhibited the smaller network of the pores responsible for water retainment in the cellulose structure to become widened.

TABLE 3: Hydrated densities of isolated cellulose and CMCs from SCB

Samples	Hydrated density (g/mL)
Native cellulose	2.50 g/mL
10% CMC _{1,0}	2.86 g/mL
20% CMC _{1,5}	3.57 g/mL
30% CMC _{2,0}	5.00 g/mL

3.4 WATER AND OIL RETENTION CAPACITIES OF ISOLATED CELLULOSE AND CMCs FROM SCB.

The water retention capacity of cellulose depends on the number of free hydroxyl groups not linked with each other (Fengel and Wegener, 2011a). The WRC and ORC of both native and all samples of carboxymethyl cellulose were determined and are presented in Table 4. As observed from the table, water retention values of all prepared samples of CMCs increased after carboxymethylation and were concluded to have higher values than their native counterpart. This is probably due to the increased alkaline concentration of the CMCs known to increase the surface area of the cellulose structure, swelling capacity of the cellulose and the diffusing capacity of the hydrophilic modifying reagent used, and this in turn increased the tendency of the cellulose fibre to absorb water as more hydroxyl groups of the cellulose become more accessible for water. Normally, fibre has the propensity to soak up water and this is attributed to the large number of hydroxyl groups its structure which have the capacity to form a hydrogen bond with water. According to spiller (2001), estimating the water retention capacity (WRC) in the fibre's structure indicated that the fibre can absorb large

quantities of water due to its soluble and insoluble nature towards water. Ang (1991) also found that the WRC was noticed to increase with an increasing fibre length. The characteristics of fibre imbibing and swelling in water are important not only in food application, but also in the proper functioning of human gastrointestinal tract (Riantong, Worasit, Kongbangkerdand and Sodshit, 2014).

Particle size of cellulosic materials also influences water retention capacity. The strong linkage between cellulose, hemicellulose and lignin forms a tight structure and this gives rise to large particle size that limits water retention capacity (Xu, Reddy and Yang, 2009). On the contrary, Riantong et al., (2014) reported that large particle size increases water retention capacity. Similarly, Riantong et al., (2014) reported that smaller particle size increases ORC as opposed the research conducted by Sansawat (2008) and Prakhonpan et al., (2002). Hence, water absorption capacity of cellulose is determined by the number of free hydroxyl moieties not connected with each other (Fengel and Wegener, 2011a).

The oil retention capacity (ORC) of both native and all samples of CMCs also increased with increasing sodium hydroxide concentration as observed from the table. Native cellulose had the least value of ORC while the CMC prepared with 30% NaOH concentration for 2 h working reaction time had the highest value of ORC. The high value noticed was proposed to be due to the introduction of carboxymethyl moieties which disrupts the inter and intra molecular hydrogen bonds in the cellulosic structure and this in turn limits the electrostatic repulsion among the cellulose molecules, thereby facilitating the absorbance of oil by the cellulose structure.

The ability of the WRC and ORC of cellulose could be divergent based on the nature of the raw materials (Chen et al., 1988). Table 5 presents the comparison between the WRC and ORC property of cellulose from SCB and other sources of cellulose. It was reported that cellulose with high WRC and ORC is appropriate, befitting and satisfactory for products that require expansion in volume and optimization in texture such as bakery products (Chen et al., 1988).

TABLE 4: Water and Oil capacities of isolated cellulose and CMCs from SCB

Samples	WRC (g H ₂ O/g dried sample)	ORC(goil/ g dried sample)
Native cellulose	3.10	1.50
10% NaOH-CMC _{1,0}	3.33	1.60
20% NaOH-CMC _{1,5}	3.58	2.07
30% NaOH-CMC _{2,0}	4.10	2.17

TABLE 5: WRC and ORC values of cellulose from different sources.

Cellulose sources	WRC (g / g)	ORC (g / g)
Pineapple core cellulose	9.92	2.15
Orange residue dietary fibre	13.36	2.01
Banana peel cellulose	2.91	0.08

WAC = Water retention capacity

ORC = Oil retention capacity

10% NaOH-CMC_{1,0} = Carboxymethyl sugarcane bagasse cellulose prepared with 10% (w/v) of NaOH at 2h working reaction time.

20% NaOH-CMC_{1,5} = Carboxymethyl sugarcane bagasse cellulose prepared with 20% (w/v) of NaOH for 1.5 h working reaction time.

30% NaOH-CMC_{2,0} = Carboxymethyl sugarcane bagasse cellulose prepared with 30% (w/v) of NaOH for 2 h working reaction time.

3.5 DEGREE OF SUBSTITUTION OF CARBOXYMETHYL CELLULOSE

The DSabs for CMC synthesized with a range of NaOH concentrations varied between 10 – 30% at distinct reaction instances (1-2 h) is in Table 6. Values acquired from this work ranged between 0.45 – 0.59. In this study, increment in NaOH concentration resulted in an increasing DS up to 20% NaOH concentration. This indicates that NaOH acts as a catalyst to actuate cellulose molecules. Reuben and Conner (1983) synthesized CMC by means of alkalization of cellulose accompanied by etherification reaction using monochloroacetic acid (MCA) and the DSabs was between 0.4 – 1.3. Besides, preceding reports through Waring and Parsons (2001) indicated that a low value of absolute degree of substitution below 0.4 means that the derivatized cellulose displayed swellability but not soluble. Above this value, CMC is soluble as its hydro-affinity will increase with increasing substitutional degree. Comparison between the DSabs value of carboxymethyl cellulose synthesized from SCB cellulose and other sources of cellulose showed that the latter's values were much non-identical and this was attributed to different experimental conditions and chemicals used as presented in Table 7.

TABLE 6: DSabs Of CMC synthesized with various NaOH concentrations at different reaction times.

Sample	Absolute DS
10% NaOH – CMC _{1,0}	0.4529
20% NaOH – CMC _{1,5}	0.5905
30% NaOH – CMC _{2,0}	0.5203

TABLE 7: DSabs values of CMC from different cellulose sources.

Cellulose sources	DSabs	Reference
Cotton fibre	0.15 – 0.70	Heydarzadeh, et al. (2009)
Sago waste	0.33 – 0.82	Pushpamalar, et al. (2006)
Water hyacinth	0.24 – 0.73	Barai, et al. (1997)
Sugar beet pulp	0.11 – 0.67	Togrul and Arslan (2003)
Banana pseudo stems	0.24 – 0.75	A dinugraha, et al. (2005)

It appears that there is a direct relationship between percentage yield and absolute substitutional degree (DSabs) of all samples of CMC synthesized as represented in Table 8 and fig. 2. The greater the DS, the greater the yield of CMC prepared at each working reaction times and NaOH concentrations. A rise in DS was due to greater carboxymethyl moieties substituted for the hydroxyl groups of the cellulose polymers thereby enhancing the potential of CMC to immobilize water in a system. Therefore, a rise in DS thus increased the quantity of all samples of CMC. However, a decrease in DS of CMC organized at an excessive NaOH concentration of 30% (w/v) lowered the yield of CMC due to the fact that some accessible sites of the MCA molecule tend to react with NaOH.

TABLE 8: Relationship between absolute degree of substitution and yield of all samples of CMCs

Sample	Yield of CMC (%)	DSabs
10% NaOH – CMC _{1,0}	110.00	0.4529
20% NaOH – CMC _{1,5}	136.20	0.5905
30% NaOH – CMC _{2,0}	132.60	0.5203

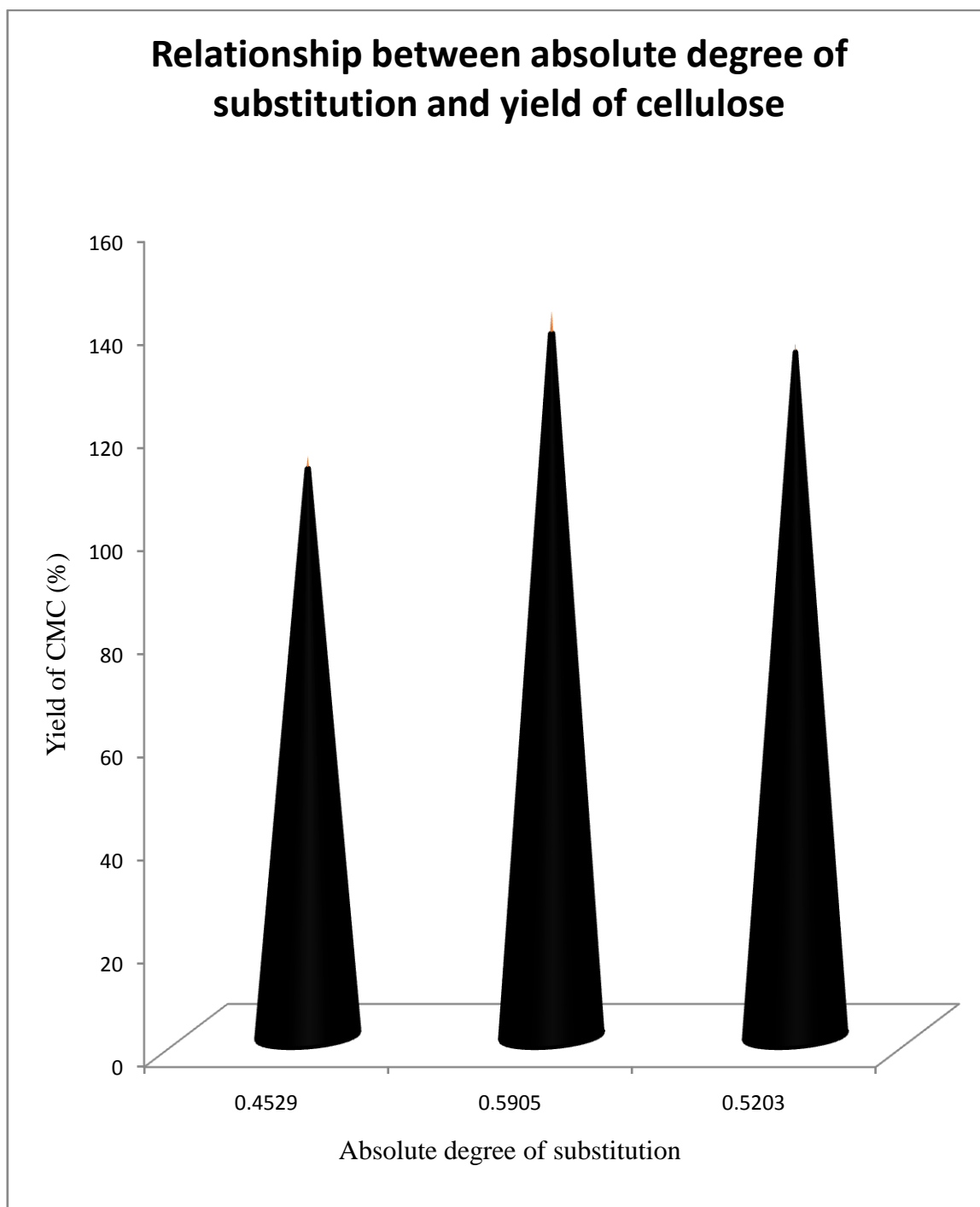


FIG. 2: Relationship between absolute degree of substitution and yield of all samples of prepared CMCs.

3.6 INFLUENCE OF REACTION PARAMETERS ON THE DEGREE OF SUBSTITUTION.

3.6.1 EFFECT OF NaOH CONCENTRATIONS

The effect of rising NaOH concentration in response to the degree of substitution is presented in fig. 3. As observed, the DSabs of all samples of CMCs heightened with NaOH concentration and attained a highest DSabs of 0.59 at a NaOH concentration of 20 % (w/v). This observation was due to marked change in appearance of the crystalline environment of the cellulose which was chemically modified into amorphous form of cellulose and thus, atoms at C2, C3 and C6 of anhydroglucose unit (AGU) should without difficulty be accessed by MCA. During the carboxymethylation process, the sodium hydroxide (NaOH) created an alkaline surroundings for the reaction and additionally facilitated the swelling of the cellulose with a widened surface area. It additionally supported diffusion and penetration of the etherifying agent to the bulk of the cellulose granular structure with the goal of permitting the etherification technique to structure cellulose alkoxide.

However, when the NaOH concentration reached 30%, the degree of substitution (DS) of CMC_{2.0} reduced due to degradation of cellulose chains by the alkaline hydrolysis. Rise in the NaOH concentration additionally improved the inactivation of monochloroacetic acid which led to the formation of sodium glycolate as a spinoff in the synthesis of carboxymethyl cellulose. This commentary is in accordance with previous reports on carboxymethyl cellulose made from sugar beet pulp (Togrul and Arslan, 2003), banana pseudo stem (Adinugraha, Marseno and Haryadi, 2005), and sago waste (Pushpamalar, Langford and Lim, 2006). Barai, Singhal and Kulkarni (1997) reported that at high NaOH concentration, glycolate formation appeared to be preferential, accordingly lowering the degree of substitution and CMC content material as part of monochloroacetic acid (MCA) molecule tends to react with NaOH.

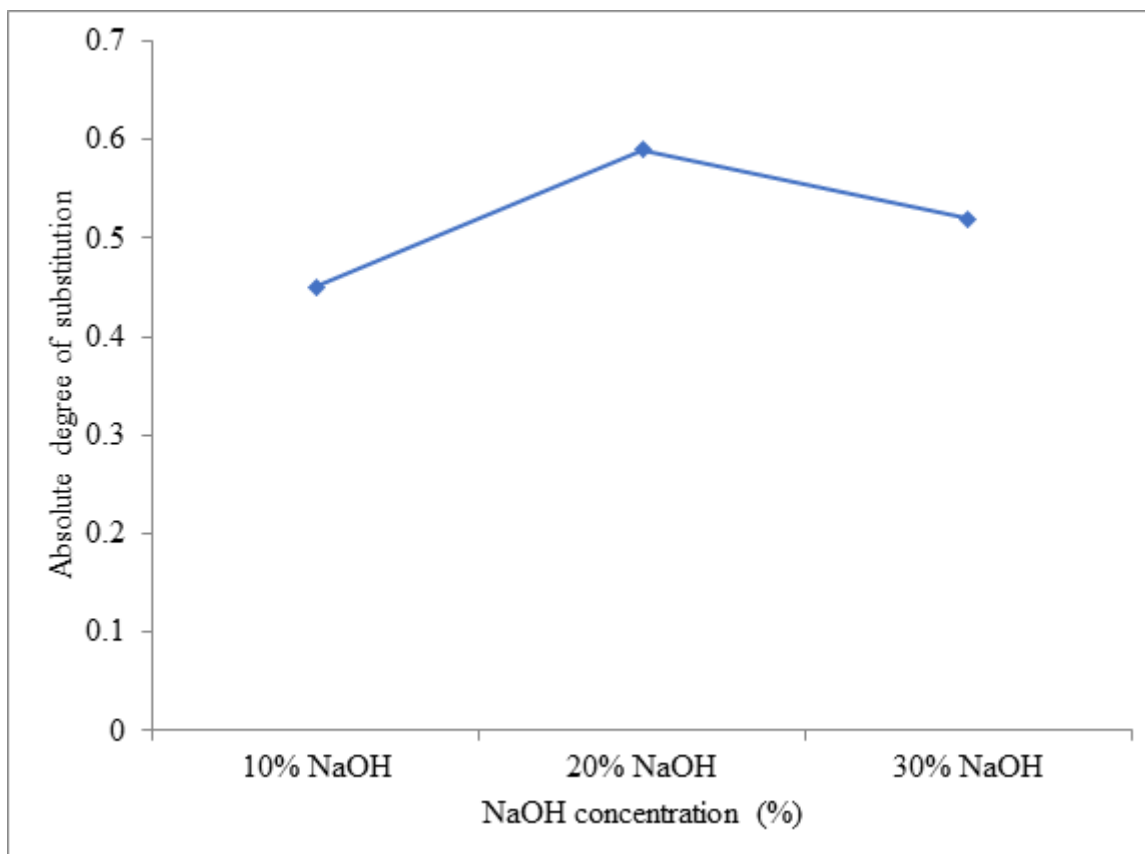


FIG.3: The effect of sodium hydroxide concentration on the DSabs for the carboxymethylation of sugarcane bagasse cellulose.

3.6.2 EFFECT OF REACTION DURATION

The impact of reaction time on the absolute substitutional degree is presented in fig. 4. Tremendous enhancement in the substitutional degree upon increasing the response duration up to 1.5 h was once observed. The rise in the DSabs is directly proportional to the reaction duration time and this in turn enhanced duration of contact of the etherifying reagent and the cellulose molecules. Prolonging the carboxymethylation duration beyond this 1.5 h was discovered to be accompanied by a decrease in the degree of substitution of the prepared carboxymethyl cellulose. This behavior counseled that a very low concentration of MCA was unused in the reactionmedium and consequently, a decline in the degree of substitution emerged after 1.5 h working response time.

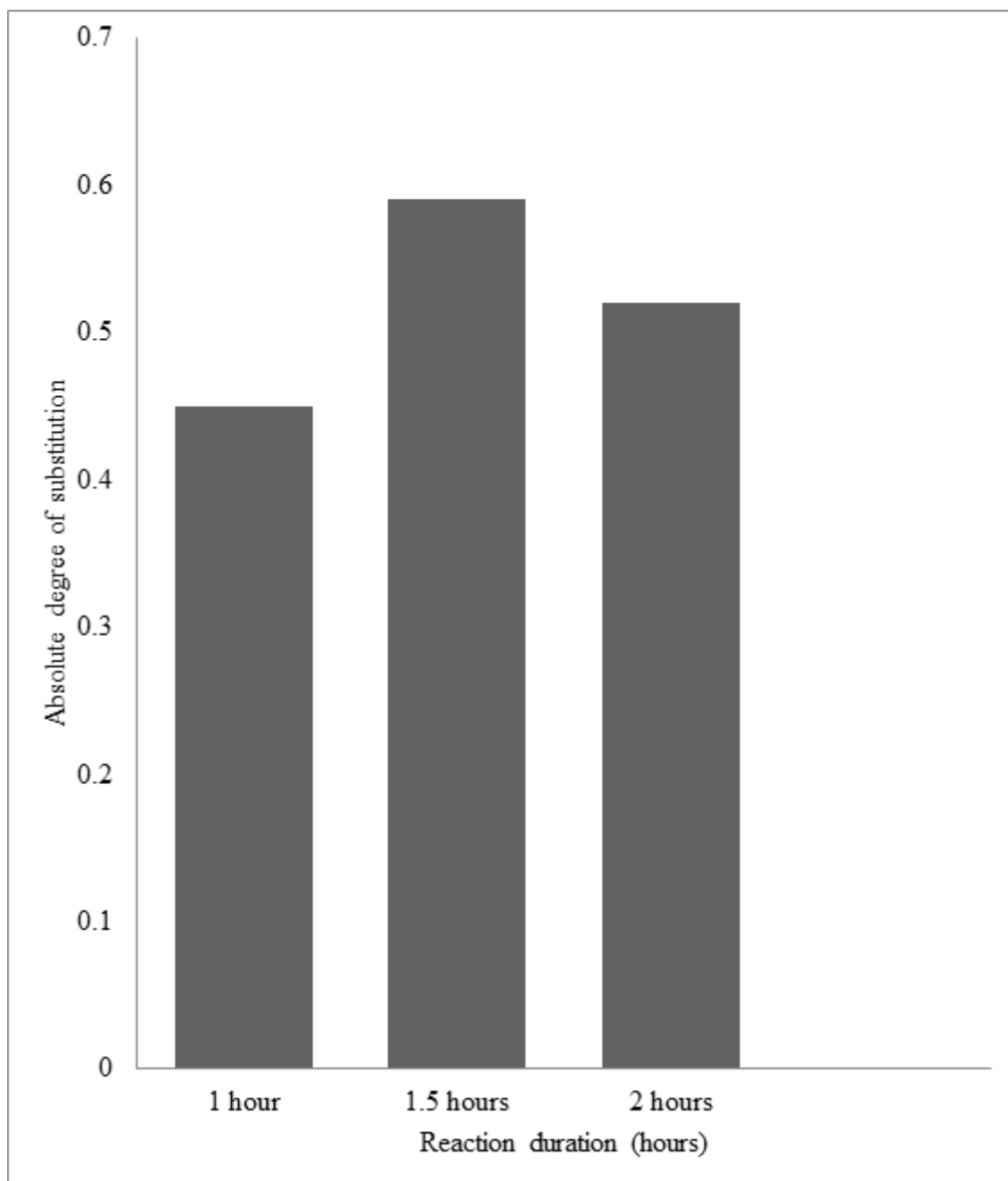


FIG. 4: The effect of varying reaction duration on the DSabs for the carboxymethylation of sugarcane bagasse cellulose prepared at different NaOH concentrations.

3.7 SPECTROSCOPIC CHARACTERIZATION BY FT-IR

Fourier transform-infrared spectroscopy was used to verify the structure of both native and all samples of CMCs subjected to chemical modifications with the use of varieties of chemical treatments. Fig. 5 and Fig. 6 exhibit the FT-IR spectra of sugarcane bagasse and the chemically extracted cellulose respectively. The top established at 1729 cm^{-1} in the spectrum of grounded sugarcane bagasse is assigned to the C=O stretching vibration of the acetyl and uronic ester moiety from pectin, hemicellulose or the carboxylic group of ferulic and p-coumaric acids of lignin. Similarly, the absorbance at 1635 cm^{-1} and faintly at 1509 cm^{-1} correspond to aromatic C=C in the plane symmetrical stretching vibration of the dominant ring existing in lignin. The emergence of the transmittance at 1252 cm^{-1} in sugarcane bagasse (SCB) spectrum is the C-O out of plane stretching vibration of the aryl-alkyl ether moiety in lignin. All these pin-pointed peaks disappeared in the spectrum of native cellulose after alkali and nitric acid treatment respectively due to the full-size removal of hemicelluloses and lignin materials. The presence of 1640 cm^{-1} height in the spectrum of native cellulose is the OH bending of the absorbed water while the band at 3444 cm^{-1} is ascribed to OH-stretching vibration that elucidates the formation of hydrogen bonds in the shape of the cellulose. The bands at 2911 cm^{-1} , 1441 cm^{-1} ,

1373 cm^{-1} , 1044 cm^{-1} and 897 cm^{-1} in fig. 6 were assigned to C-H stretching vibration, CH_2 bending vibration, C-H bending vibration, C-O vibration and C-O-C stretching at β -(1-4)-glycosidic linkages respectively. Furthermore, the FT-IR spectra of all samples of carboxymethyl cellulose synthesized with different alkaline concentrations and varied working reaction times as indicated in fig. 7, 8 and fig. 9 respectively confirmed the emblematic absorptions of the cellulose spine and the presence of the carboxyl, C=O groups at 1609–1640 cm^{-1} . Two peaks at the wavenumber about 1600 – 1640 cm^{-1} and 1400 – 1450 cm^{-1} were determined and these two absorption peaks indicated that carboxymethylation took place in all the three samples of derivatized cellulose prepared. However, increased intensity of C=O band was observed after carboxymethylation as extra carboxylic reagents were introduced. The peak discovered at 1379 cm^{-1} in the spectrum of 10% NaOH-CMC_{1.0} is attributed to C-H asymmetric stretching vibration. The bands around 1424-1441 cm^{-1} are assigned to CH_2 scissoring vibration in all samples of derivatized cellulose. The absorption peaks around 1327-1330 cm^{-1} in all samples of CMCs have been assigned to C-H bending vibration.

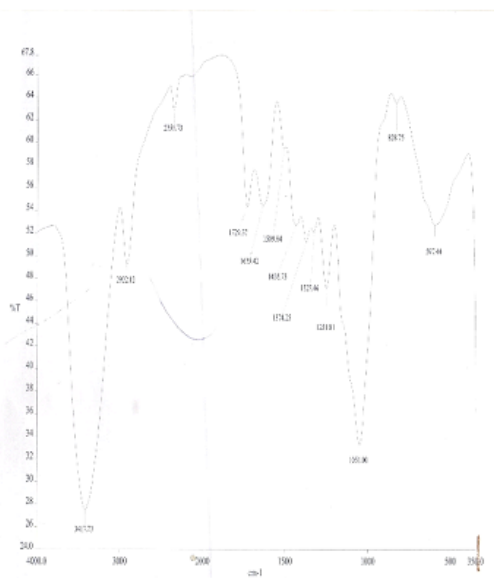


Fig. 5: Spectra of Sugarcane Bagasse

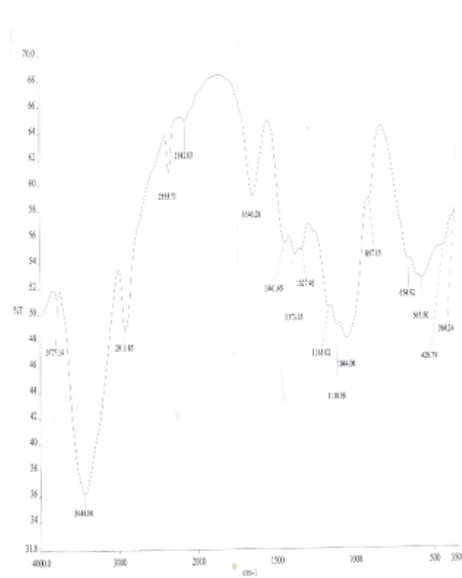


Fig. 6: Spectra of Native Cellulose

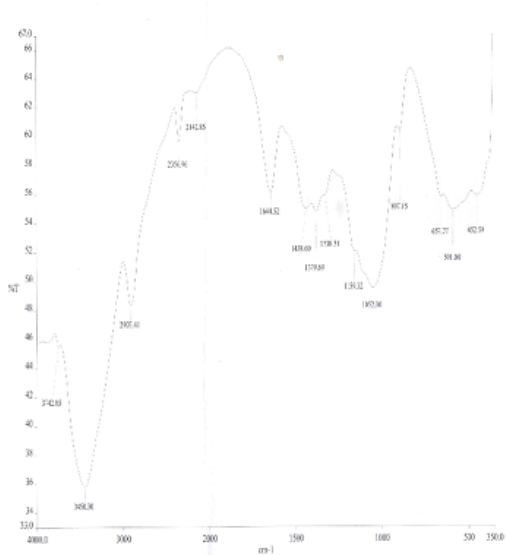


Fig. 7: Spectra of 10% CMC

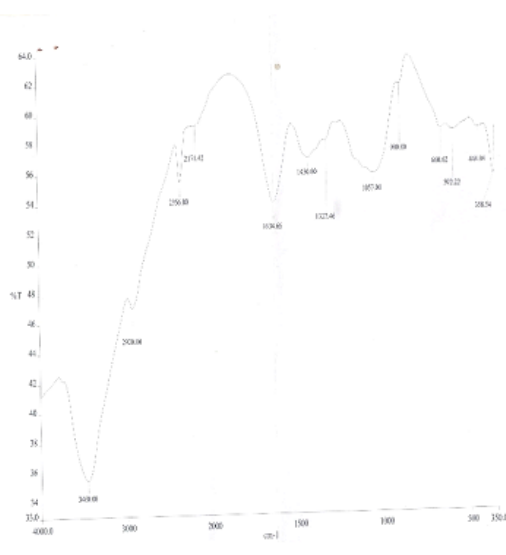


Fig. 8: Spectra of 20% CMC

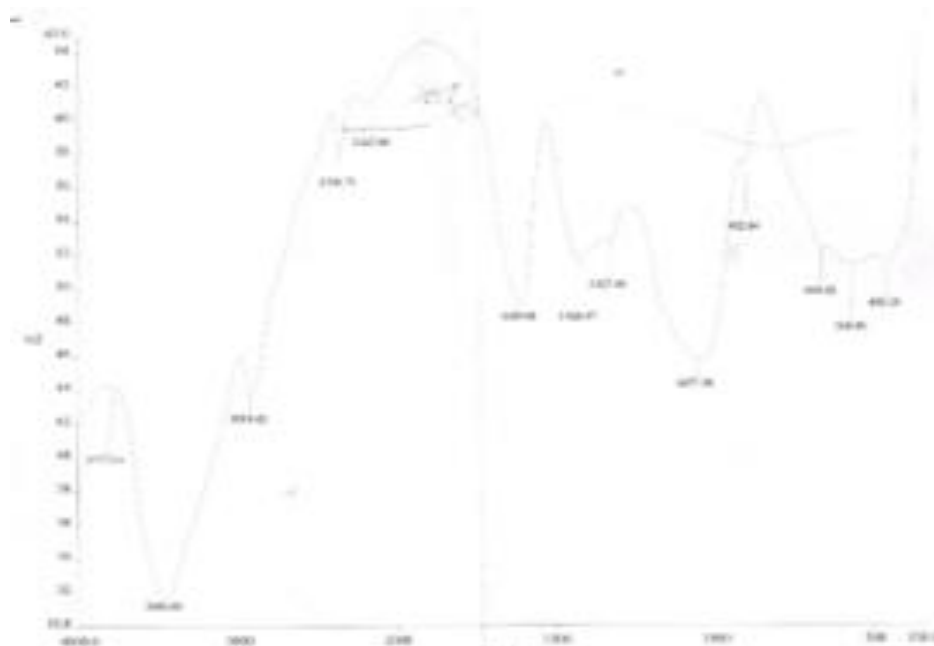


Fig. 9: Spectra of 30% CMC

IV. Conclusion And Recommendation

In conclusion, results recorded in this study have indicated that all derivatized samples of cellulose via carboxymethylation extracted from sugarcane bagasse have good functional properties that can be utilized in different fields of applications and this will in turn position the non-conventional cellulose for good market globally. However, it is recommended that carboxymethyl cellulose prepared with 20% (w/v) NaOH concentrations for 1.5 hours of reaction time could be a potential additive in bakery products due to its little or no resistance to water and oil retainment in its structure. Though, much work needs to be conducted in this area because little or no researches have been carried out with regards to comparing the water and oil retention capacities of CMCs prepared at different alkaline concentrations and different reaction times.

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