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EXTRACTION, PREPARATION AND CHARACTERIZATION OF CARBOXYMETHYL CELLULOSE FROM CORN STALK AND ITS APPLICATION ON *ESCHERICHIA COLI*

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ABSTRACT

Corn Stalk (CS) was treated with alkali, acid hydrolyzed, and bleached, in order to extract native cellulose (NC). By employing chloroacetic acid to etherify the NC, carboxymethyl cellulose was produced. The changed cellulose's chemical composition, degree of substitution, proximate analysis, structural crystallinity, and thermal analysis were all reported. Modified cellulose had lower levels of fat and ash, according to proximate analysis. The crystallinity index of CMC was lower than that of the NC. The surface disruption during carboxylmethylation was confirmed by Scanning Electron Microscopy (SEM), which showed rough spongy-like mass for modified cellulose and irregular rod-like morphology for native cellulose, respectively. The EDX analysis confirmed that cellulose was extracted from CS. FTIR and XRD results confirmed that the extracted cellulose had been modified to carboxymethyl cellulose. Improved stability following chemical alteration of the NC was demonstrated by thermal analysis. The native cellulose and carboxymethyl cellulose exhibited little or no response against *Escherichia coli.*

KEYWORDS: Carboxymethyl cellulose, Corn stalk, Modification, Native celluse, and *Escherichia coli.*

1.0 INTRODUCTION

The majority of plant cell walls are composed of cellulose, making it the most prevalent biopolymer found in nature.[1] According to Holtzapple.[2] It is a homopolymer of anhydroglucose, with the glucose residues joined in a ß-1,4 manner. Cellulose is the component that gives plant cell walls their mechanical strength. The structural characteristics of cellulose are attributed to its ability to maintain a semi-crystalline state of aggregation, which is uncommon for a polysaccharide, even in an aqueous environment.[3] The structural characteristics of cellulose are attributed to its ability to maintain a semi-crystalline state of aggregation, which is uncommon for a polysaccharide, even in an aqueous environment.[3]

Cellulose has long had a significant place among the many raw materials that nature has made available to mankind for use in industry. Since the beginning of time, it has been utilized by humans as a building material or as a flexible starting material for chemical reactions that produce synthetic cellulose-based threads and biofilms, as well as a range of stable cellulose derivatives used in a variety of household and industrial applications.[4] Even before the polymeric nature of cellulose was known and understood, it was utilized for a variety of metabolic transformations. Due to their enhanced mechanical, thermal, and reinforcing qualities, low density, and non-toxic nature, cellulose derivatives find extensive use in the food, paper, adhesive, cosmetics, pharmaceutical, textile, and detergent industries.[5-7] A significant cellulose derivative, carboxymethyl cellulose (CMC) finds extensive use in the food sector, as well as in detergents, cosmetics, medicines, textiles, paper, adhesives, and ceramics.[8] CMC is a water-soluble cellulose derivative that contains β-D-glucopyranose-2-

(carboxymethyl) -monosodium salt and β-D-glucopyranose, which are joined by β-l,4-glycosidic linkages. Additionally, it functions as an emulsifier, thickener, and viscosity modifier.[9]

Globally, the production of waste from agricultural goods has been a significant problem. Efforts to reduce waste production and increase the efficiency of raw material use have been prompted by the need for sustainable development.[10,11] Approximately 200 million tonnes of agricultural waste are produced worldwide each year, according to research. These wastes are plentiful, inexpensive, and renewable resources that can be used to make cellulose. 34–40 % of this agricultural waste is composed of cellulose, which may be separated from it.[12]

The primary components of a corn stalk are the leaves and stems, which are made up of the cortex and pith. One of the traits of corn stalks is the abundance of pith in the stem. Of the entire weight of the stalks, 40%, 35%, and 15% are made up of the leaf, cortex, and pith, respectively. Both the juvenile and mature stages of corn stalks are nutritious. Crude protein content in mature corn stalks is highest in the leaves, next in the tassels and stalks, and lowest in the bracts.[13] The highest amount of crude fiber is found in the bark, the lowest is found in leaves. The tassels have the least amount of crude fat, whereas the leaves have the most. From highest to lowest, the nutritional value of the various sections is arranged as follows: leaves, tassels, stalks, and bracts.

Escherichia coli is a Gram negative prokaryotic, rod-shaped bacterium that is commonly found in the lower intestine of warm-blooded animals. *E. coli* causes severe infectious diseases associated with high rates of mortality and morbidity.[14] Several pathogenic *E. coli* are characterized by different genes associated with their virulence. Many enteric infections caused by *E. coli* are transmitted by inter-human contacts such as those caused by Entero-invasive *E. coli* (EIEC), Enteropathogenic *E. coli* (EPEC) and Enteroaggregative *E. coli* (EAggEC).[15,16] Shiga toxin-producing *E. coli* (STEC) or Enterotoxigenic *E. coli* (ETEC) are primarily transmitted to humans through the consumption of contaminated food and water.[16-18]

Many agricultural wastes with enormous economic potential are currently considered a nuisance in most parts of Nigeria because their underlying benefits are not well understood. Given the current state of the economy and the prevalence of malnutrition, the separation, modification, and use of cellulose-based products from these underutilized sources would not only provide an extra source of income but also reduce pollution in the environment and encourage healthy living. Thus, separating, altering, and characterizing the cellulose from corn stalks using: Fourier Transform Infrared Spectrophotometer (FTIR), Scanning Electron Microscope and Energy Dispersive X-ray analyser (SEM-EDX), Thermogravimetric analyser (TGA), and X-ray diffractometer (XRD), were the primary goal of this investigation. *Escherichia coli* was then used to assess the antimicrobial qualities of the native cellulose (NC) and the carboxymethyl cellulose (CMC).

2. MATERIALS AND METHODS

2.1. Materials

Dried Corn Stalk (CS) was collected from Lambo Lasunwon area of Ikorodu, Lagos State, Nigeria. Every chemical used was of analytical quality.

2.2. Extraction of native cellulose from Corn stalk

1,000 g of the powdered fibers were macerated with 15 % Nitric acid and 1.2 % Sodium sulphite, delignified at 95 °C for 180 mins, washed and filtered many times with distilled water until the pH was 7, and the residue was then cooled. Following this, the residue was treated with 6 % Sodium hydroxide and 1.2 % Sodium sulphite at 60 °C for 120 mins. Next, 12 % sodium hydroxide was added, and the temperature was raised to 80 °C for 60 mins. Finally, it was filtered and washed several times until the pH was 7. It was later bleached with Sodium hypochlorite at 60 °C in a hot water bath for 15 mins until white pure native cellulose (NC) was obtained. The resulting native cellulose was washed thoroughly with distilled water until the filtrate became neutral (pH = 7). Finally, the residue was air-dried and the percentage yield of the native cellulose was determined.

2.2.1. Percentage Yield

The percentage yield of the native cellulose was determined by comparing the dried weight of the isolated native cellulose with the weight of the starting material and calculated:

$$Percentage\ yield = \frac{weight\ of\ cellulose}{weight\ of\ sample} \times 100$$

Equation 1

2.3. Determination of Hemicellulose

150 mL of Sodium Hydroxide (NaOH) solution (0.5 mol/L) was added to 2 g of CS with extractives free. The temperature 80 °C was controlled by using a hot plate for 3.5 hours. After that, the sample was washed with deionized water until it was free from Na^+. The Na^+ was detected by using pH paper and the reading should be closed to 7. The sample was dried in an oven at 105 – 110 °C until constant weight was obtained.[19]

2.4. Determination of Lignin

30 mL of 98 % Sulphuric acid was added to 2 g of CS with extractives free. The sample was left at ambient temperature for 24 h before boiled at temperature 100 °C controlled by using a hot plate for 1 h. The mixture was filtered and the solid residue was washed by using deionized water until sulfate ion was undetectable. Detection of sulfate ion was done via titration process with 10 % of Barium chloride solution. The sample was dried in an oven at 105 – 110 °C until constant weight

was obtained. The final weight of residue is recorded as lignin content.[19]

2.5. Preparation of Carboxymethyl Cellulose

Preparation of CMC samples was carried out using a method described by Lawal et al.[20] NaOH (10 g) was dissolved in 50 ml of distilled water and placed on a magnetic stirrer at 250 rpm until the NaOH was dissolved. Isopropanol (100 ml) was added to the mixture after which native cellulose (10 g) was added to the mixture and was stirred for 1 h magnetically. After 1 h, chloroacetic acid (15 g) was added and the mixture was stirred continuously for 2 h. The pH was adjusted to 10 and the temperature was varied and adjusted to 40 °C, 50 °C, and 60 °C for the respective samples. The mixture was dispersed in acetone (100 mL) for some hours, then filtered and washed with methanol until the filtrate tested negative to silver nitrate i.e it will not give white precipitate and it was then air-dried.

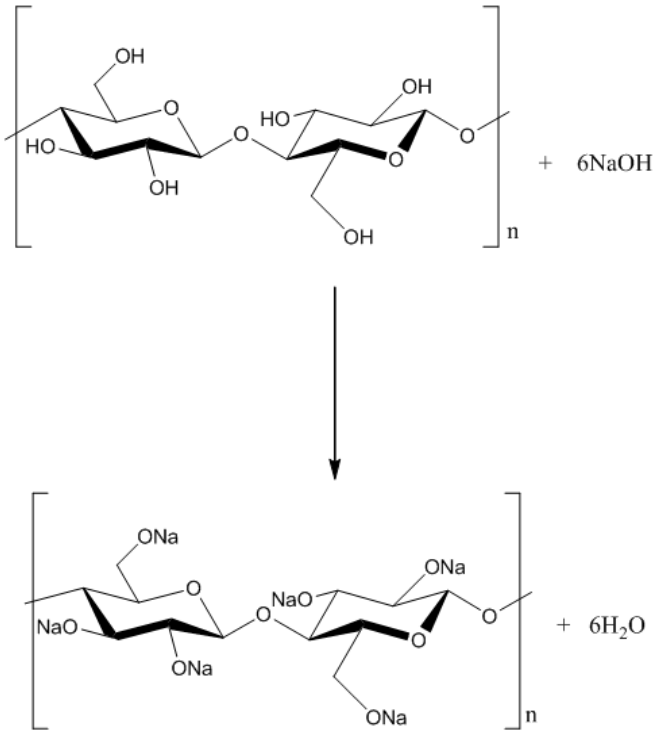


Figure 1: Reaction Scheme 1 for the Preparation of β-D glucopyranose-2-carboxylmethyl monosodium salt.

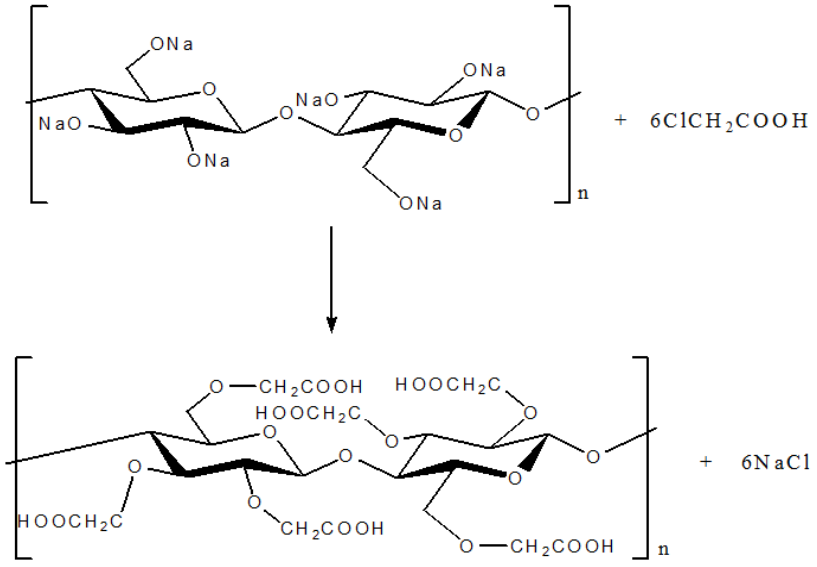


Figure 2: Reaction Scheme 2 for the Preparation of carboxymethyl cellulose.

2.5.1. Determination of Degree of Substitution (DS) of Carboxymethyl Cellulose

The DS was determined using the titrimetric method as described by Lawal et al.[21] 2 g sample of CMC was dissolved in 1 % aqueous NaCl solution (100 ml) and titrated with 1 M NaOH solution using phenolphthalein indicator until a colourless solution was detected. The DS was determined as follows;

$$DS = \frac{nNaOH \times M_o}{M_c - nNaOH \times M_r} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (Equation\ ii)$$

$$M_c = \frac{M_p - (M_p \times F)}{100} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (Equation\ iii)$$

M_o =	Molar mass of the anhydroglucose unit = 162g/mol
M_r=	Molar mass of carboxymethyl residue = 58g/mol
$_n$NaOH =	Quantity of sodium hydroxide used [mol]
M_p=	Weight of modified cellulose taken [g]
M_c =	Corrected weight of modified cellulose [g]
F =	Moisture content

2.6. Determination of Proximate and structural analysis of NC and CMC

Proximate analysis (moisture content, total ash, crude fat) was carried out in triplicate on NC and CMC samples using standard AOAC methods[22] without modification.

2.7. FTIR Spectroscopy

FTIR spectra were recorded in transmission mode on an Agilent Technologies Cary 630 FTIR using potassium bromide pellets (1.5 mg sample in 200 mg KBr) that were dried under vacuum (80 ^{0}C, 72 h) prior to the measurements. 64 scans were collected in the range of 400–4000 cm^{-1}.

2.8. Scanning Electron Microscopy and Energy Dispersive X-ray (SEM-EDX),

Gemini 1 Scanning Electron Microscopy was used to investigate the surface morphology studies of the NC and CMC. Samples were vacuum-coated with Au. Additionally, their elemental compositions were examined by Energy Dispersive X- ray analysis at a working distance of 14.5–15.5 mm at 20 kV.

2.9. Thermal Analysis

Thermogravimetric analysis (TGA) is a process in which a material is decomposed by heat, which causes bonds within a substance to be broken. TGA plays an important role in determining the thermal stability of the substance. Thermogravimatic analysis (TGA) of the samples was carried out using TGA 4000 by Perkin Elmer Netherlands. The NC and CMC samples were analyzed over a continuous increase in temperature from 30 °C to 900 °C at a heating rate of 20 °C min^{-1}.

2.10. X-Ray Diffractometry

The X-ray diffraction patterns of the powdered samples of the NC and CMC were determined using Genius IF by Xenemetrix Ltd Israel with Cu Kα (1.5418 Å) radiation at an angular incidence of 10 – 70 ° at 40 kV and 40 mA. The crystallinity index (CrI) of cellulose was determined according to Eq. (iv):

$$CrI = \frac{I_{200} - I_{AM}}{I_{200}} \times 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (Equation\ iv)$$

I_{200} = Intensity of the cellulose crystalline peak

I_{AM} = Intensity of the cellulose amorphous peak after subtraction of the background signal

2.11. Test organism used for the study

To study the antimicrobial activity of native cellulose and carboxymethyl cellulose against *Escherichia coli* a Gram-negative bacterium was used for the study. The microorganism was maintained at 4°C on a nutrient agar slants in the Department of Chemical Sciences, Lagos State University of Science and Technology and fresh subculture was made before use.

2.12. Inoculum preparation

A loopful of isolated colonies of the organism was inoculated into 4 mL of peptone water in a test tube and incubated at 37°C for 4 h. These bacterial suspensions was then adjusted with peptone water to obtain turbidity visually comparable to that of 0.5 McFarland standards using standard procedure described by Momoh et al.[14] 0.5 mL of 1.75% (w/v) $BaCl_2$. $2H_2O$ and 99.5 mL of 1% (v/v) H_2SO_4 were combined to create the 0.5 McFarland standard. The turbidity of the solution was approximately 1 x 10^8 colony forming units per milliliter (CFU/mL) (Momoh et al.[14]

2.13. Determination of diameter of zone of inhibition using agar well diffusion method

Antimicrobial activities of the native cellulose and carboxymethyl cellulose were tested performed against the *Escherichia coli*. The native cellulose and carboxymethyl cellulose were tested for antibacterial activity using the agar well-diffusion method as described by Momoh et al.[23] Eighteen hours of broth culture of the microorganism was suspended into a sterile nutrient broth. The broth was standardized by adding 9 percent normal saline to it and comparing their

turbidity to McFarland standard which is about 1 x 10^8 colony forming units per mL. Petri dish plates were prepared by loading 25 mL of an autoclaved nutrient agar on sterile plates and left to solidify. A total of 100 µL of a standardized culture adjusted to 0.5 McFarland of the organism was added into the different agar plates. After that, three wells were punched out of each plate's surface using a sterile cork borer. 100 µL of 200 mg/nL each of the native cellulose and carboxymethyl cellulose were poured onto each of the three wells on all plates and allowed to diffuse at room temperature for 2 h. The Petri dish plates were incubated in the laboratory at 37ºC for 18-24 h for antibacterial study. The diameters of the inhibition zone in mm were measured. The susceptibility of the *E. coli* to the native cellulose and carboxymethyl cellulose were assayed using modified standard procedure described by Momoh et al.[23] The experiment was repeated thrice and the average values were recorded. The inhibitory responses were divided into five categories: weak response (+), zone diameter between 10-15 mm; little or no response (zero), zone diameter <10 mm; moderate response (++), zone diameter between 16-20 mm; strong response (+++), zone diameter between 21-30 mm and potent response (++++) zone diameter greater than 30 mm. The negative control was a solvent blank. For positive control, Azithromycine antibiotic was employed.

2.14 Statistical Treatment

3. RESULT AND DISCUSSION

3.1. Structural and Proximate Analysis

The findings of the percentage yield of cellulose, lignin, and hemicellulose were shown in Table 1. The yield of NC from corn stalk (CS) was 45.89 % based on the total weight of the dried sample. The percentages of hemicellulose and lignin were 38.22 % and 14.50 %, respectively. This outcome surpasses the corn cob yield shown in other studies.[24] The high hemicellulose concentration suggests that CS is a rich source of hemicellulose, which could make it good for use as a food packaging material, binder, or stabilizer.[25] The results of the proximate analysis of NC and CMC were shown in Table 2. The increased hydrophilicity of CMC relative to its original form may have contributed to the observed increase in NC's moisture content (from 0.28 % to 0.56 %) following alteration. The observed decrease in the percentage of ash content of CMC (0.23 %) compared to NC (0.38 %) may have been caused by the erosion of the cellulose's mineral content during modification, while the degradative effect of carboxymethylation processes may have contributed to the NC's crude fat dropping from 6.48 % to 2.75 % upon etherification.[25]

Table 1: Percentage yield of cellulose, Lignin and Hemicellulose.

Sample	(%)		
	Cellulose	Lignin	Hemicellulose
	45.89	14.50	38.22

Table 2: Moisture content, Ash Content and Crude fat of native cellulose and carboxymethyl cellulose.

	Moisture Content (%)		Ash Content (%)		Crude fat (%)	
Sample	NC	CMC	NC	CMC	NC	CMC
	0.28 ±0.01	0.56 ±0.03	0.38 ±0.01	0.23 ±0.02	6.48 ±0.33	2.75 ±0.31

3.2. Degree of Substitution

The results of the degree of carboxymethyl cellulose substitution at different temperatures are displayed in Table 3. The degree of substitution of the CMC increases with temperature, peaking at 60 °C. It makes sense to work at temperatures lower than the cellulose's gelatinization temperature to avoid poor product recovery and to reduce the amount of contact between the cellulose and the etherifying reagent. This result is in line with published observations of etherified starch.[20] The results of the degree of CMC substitution at different etherifying agent masses are displayed in Table 4. The degree of substitution of the CMC increases with the amount of the etherifying agent. Because more hydrogen is substituted in the cellulose's hydroxyl group, the degree of substitution rises with the concentration of chloroacetic acid. This result validates previous studies conducted by Lawal et al.[20] The degree of CMC replacement at different times is shown in Table 5. The degree of replacement increases with time, reaching a maximum of 0.62. The degree of substitution increases as the etherifying agent's contact time with the cellulose increases. This finding aligns with previous studies carried out by Lawal et al.[20]

Table 3: Degree of substitution of CMC at different Temperatures.

	Temperature (oC)		
30	40	50	60
0.29 ±0.05	0.36 ±0.03	0.40 ±0.01	0.49 ±0.01

Table 4: Degree of substitution of Carboxymethyl cellulose at different volumes.

	Mass (g)		
20	25	30	35
0.52 ±0.02	0.59 ±0.02	0.62 ±0.04	0.67 ±0.02

Table 5: Degree of substitution of Carboxymethyl cellulose at different times.

	Time (h)		
2	3	4	5
0.69 ±0.01	0.71 ±0.04	0.74 ±0.05	0.87 ±0.01

3.3. Scanning Electron Microscopy (SEM)

Fig. 3 displays the surface morphology of the native cellulose fiber that was extracted from CS. The surface has a somewhat rod-like texture that was curled and fluffy. The fiber surface has a small flaw that might have been brought on by the extraction process's usage of powerful chemicals and high temperature with void spaces. The morphology of carboxymethylated cellulose (Figure 4) exhibited less void spaces and more dispersed fiber, rough surface with compacted fibers that are spiral, which may have been a result of the effect of the etherifying agent (chloroacetic acid) at an increased temperature and time.[26,27]

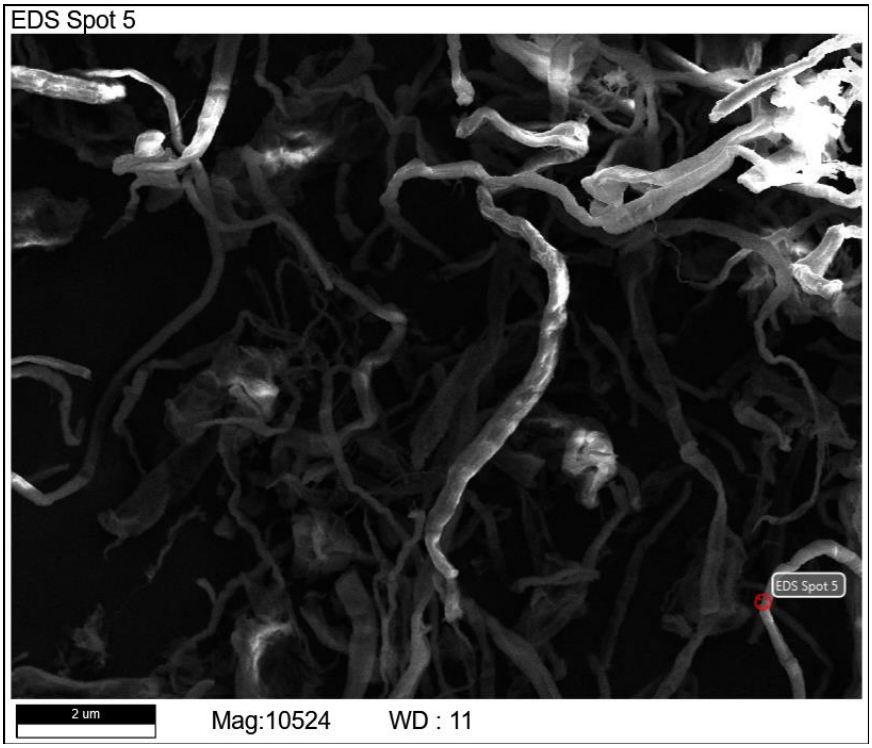
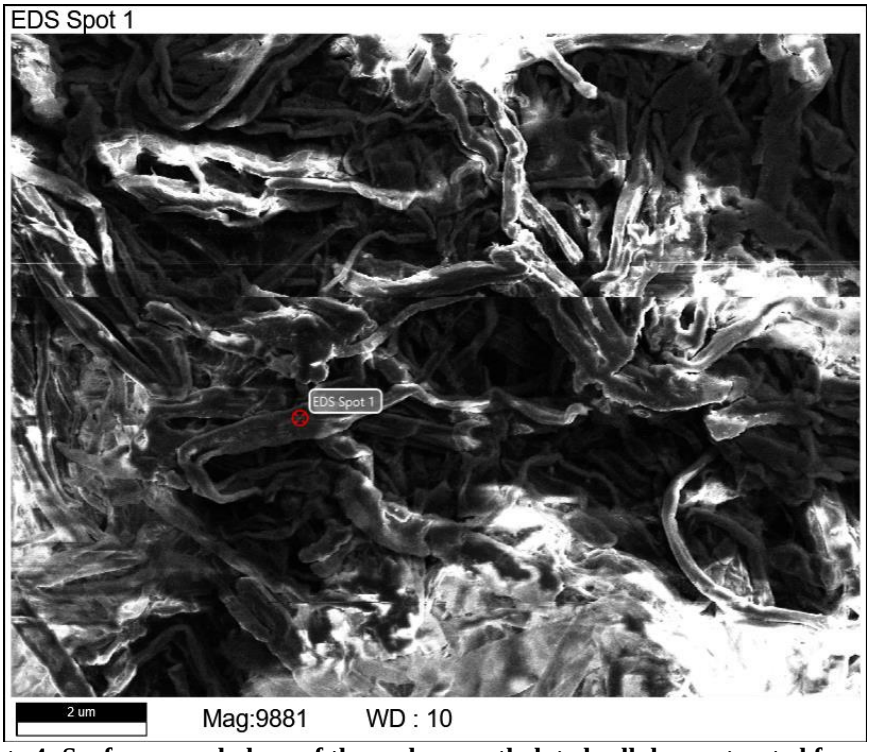


Figure 3: Surface morphology of the native cellulose extracted from CS.



Figutr 4: Surface morphology of the carboxymethylated cellulose extracted from CS.

3.4 Energy-dispersive X-ray spectroscopy (EDX) analysis

The elemental composition of the NC and CMC from CS was confirmed by energy-dispersive X-ray spectroscopy (EDX) analysis. According to Figure 5 EDS data, the NC primarily included the cellulose-specific peaks C (70.59 %) and O (29.41 %). The same distinctive peaks of C (61.16 %) and O (36.60 %) that were generated from the sodium hydroxide and monochloroacetic acid during etherification were also seen in the CMC (Figure 6), with lesser amounts of Na (2.09 %) and Cl (0.15 %). This demonstrated that the extraction procedure and CMC generation were successful.[28,29]

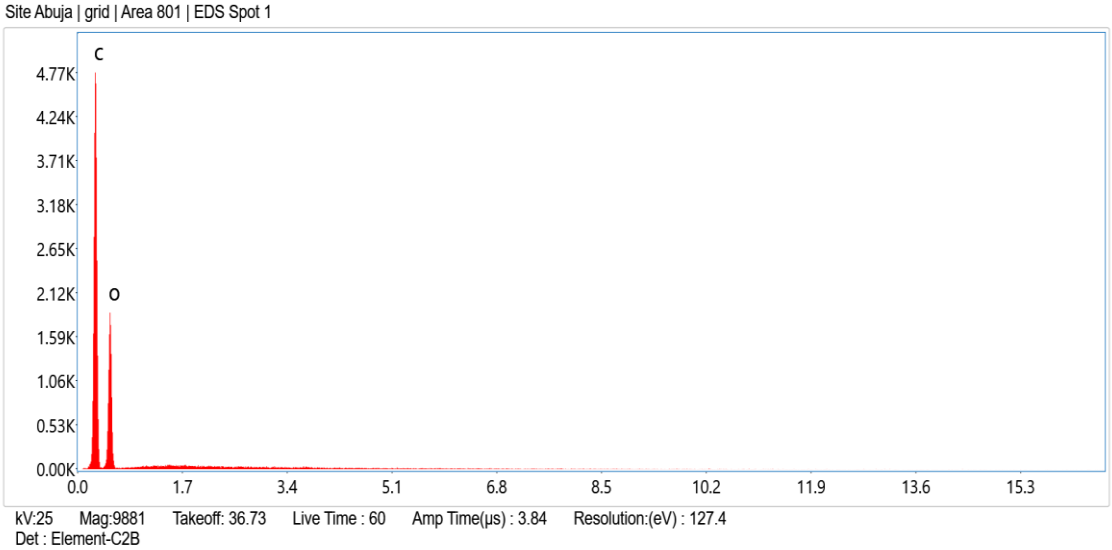


Figure 5: EDX spectrum of native cellulose.

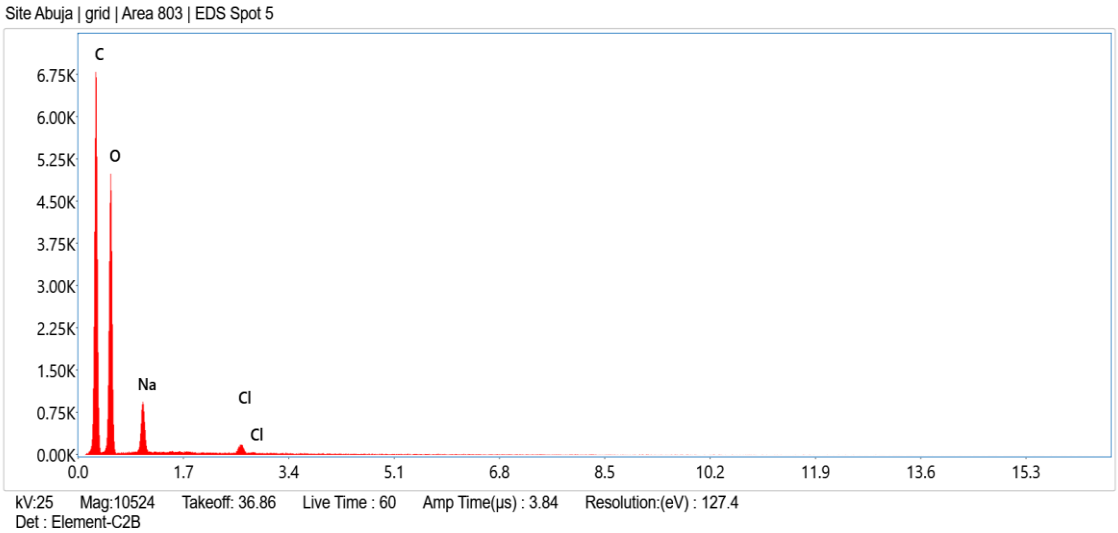


Figure 6: EDX spectrum of carboxymethylated cellulose.

3.5. FT-IR Spectroscopy

In order to verify whether the NC was successfully extracted from CS and converted to CMC, the FTIR spectra of the NC and CMC that were obtained from CS were analyzed to discover the kind of functional groups that were present in them. Figure 7. displays the NC and CMC FTIR spectra from CS, confirming a typical cellulose profile in the range of 1500 to 650 cm^{-1}. Specifically, the vibration and elongation of the hydroxyl-O-H bonds are responsible for the presence of three distinctive peaks between 1150 and 950 cm^{-1}. Furthermore, the peak features of glucosidic units can be seen around the 950–850 cm^{-1} range. The (-O-H) bending is indicated by the peak at 1307 cm^{-1}. In another study, a characteristic band at 1217 cm^{-1} was ascribed to structural carbohydrates.[30] The absorbed water's bending mode is represented by the band at 1638 cm^{-1}.[31-33] Additionally, the spectrum shows that there is a stretching vibration near the peak with a wave number of 2,892 cm^{-1}, which turns out to be (-C-H). Lastly, the stretching frequency of the (-OH) group and the intramolecular and intermolecular hydrogen bonding inside cellulose macromolecules are responsible for the absorption band at 3339 cm^{-1}.[31,32] A study shows that at 3323 cm^{-1}, the stretching vibrations of aromatic O–H groups for carbohydrates was detected.[34] The produced

CMC's FT-IR spectrum analysis reveals bands at 3332 cm^{-1} indicate OH-stretching,[35] 2897 cm^{-1} (C-H), and 1156 cm^{-1} (C-O-C). The -CH_2 scissoring and bending vibrations are attributed to the band at around 1421 cm^{-1}. The creation of carboxylic acid salt is confirmed by the appearance of the carboxylate anion COO^- absorption band at 1584 cm^{-1}. The frequencies of carboxyl groups as salts are approximately 1600-1640 cm^{-1}.[36] The structural assignments previously published[33,36,37] were likewise in agreement with the spectroscopic FTIR data of the produced CMC. The synthesis of carboxymethyl cellulose was suggested by the decrease in the peak at 3200–3500 cm^{-1} and the emergence of the peak centered around 1584 cm^{-1}.[25,38]

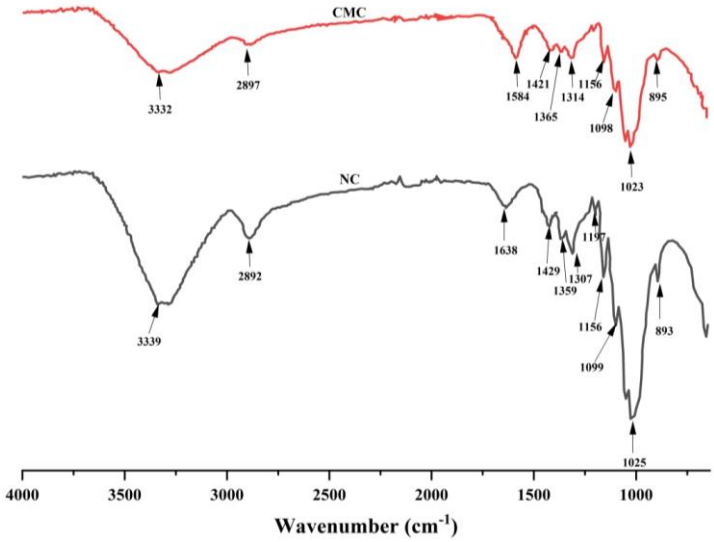


Figure 7: FTIR spectra of NC and CMC from CS.

3.6. X-Ray Diffractometry

Figure 8 displays the XRD diffractograms of NC and CMC that were acquired from CS. Cellulose was characterized by peaks at 2θ = 16°, 22°, and 35°.[31,32,39] The cellulose crystalline proportion produces the peak with the maximum intensity, whereas the amorphous portion produces the background noise line (Moussa *et al.*, 2019).[33] Similar to those reported by Golbaghi et al.[40], the strength of the peak at 2θ = 22° decreased upon modification for CMC. For NC and CMC, the estimated crystallinity index (Crl) was 87.79 and 72.65%, respectively. There are notable variations in peak widths, shifts, and heights. The NC samples have distinct crystalline peaks, but the CMC samples show a decrease in peak intensity. Due to the breaking of weak hydrogen bonds in the crystalline portion of cellulose when treated with sodium hydroxide, the CMC exhibited a lower degree of crystallinity than the NC, indicating that NC was converted to CMC. The outcome is in line with research by Lawal et al.[20] and Xin et al.[41]

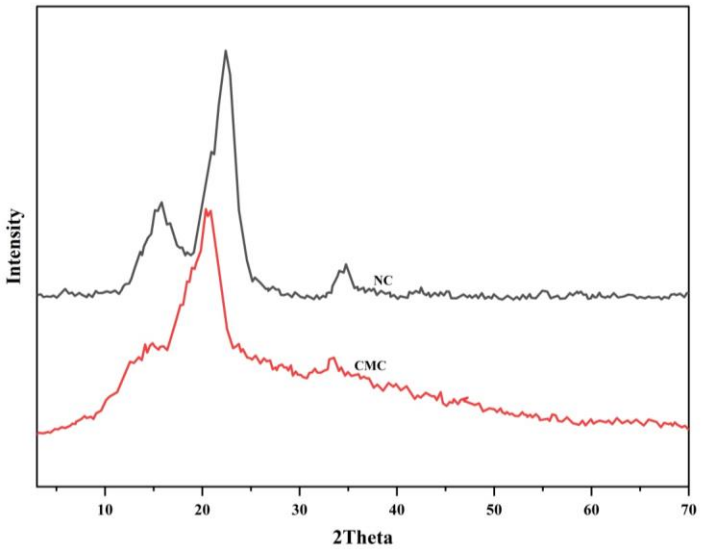


Fig. 8: XRD diffractogram of NC and CMC from CS.

3.7. Thermal Analysis

Figures 9 and 10 show the results of thermogravimetric analysis (TGA) and differential thermogravimetric analysis (DTG) for NC and CMC from CS. TGA and DTG can be used to assess the degradation and thermal stability of cellulose and its derivatives. In three regimes, the TGA curves displayed two separate weight-decreasing phases. Between 50 and 155 °C, moisture evaporation caused the initial weight loss; NC and CMC fibers lost 4.50 % and 7.90 % of their weight, respectively. With maximal breakdown temperatures of around 370.52 °C and 379.54 °C, respectively, the second weight drop was seen for NC and CMC fibers between 280 and 410 °C, leading to weight losses of approximately 70.50 % and 31.00 %. In the second step, weight loss was brought on by the thermal breakdown of carbohydrate polymers.[42] Since CMC lost less weight than NC during the second stage of thermal breakdown, it is clear that CMC has greater thermal stability than cellulose fibers. This is in line with earlier research on modified starch and cellulose[20,43] and the work of Sodeinde et al., 2020[25] on CMC from Delonixregia pods. At roughly 370.52 °C and 379.54 °C, respectively, the DTG thermograms for NC and CMC displayed strong endothermic peaks. This demonstrates a deterioration process that produces volatile compounds.[44]

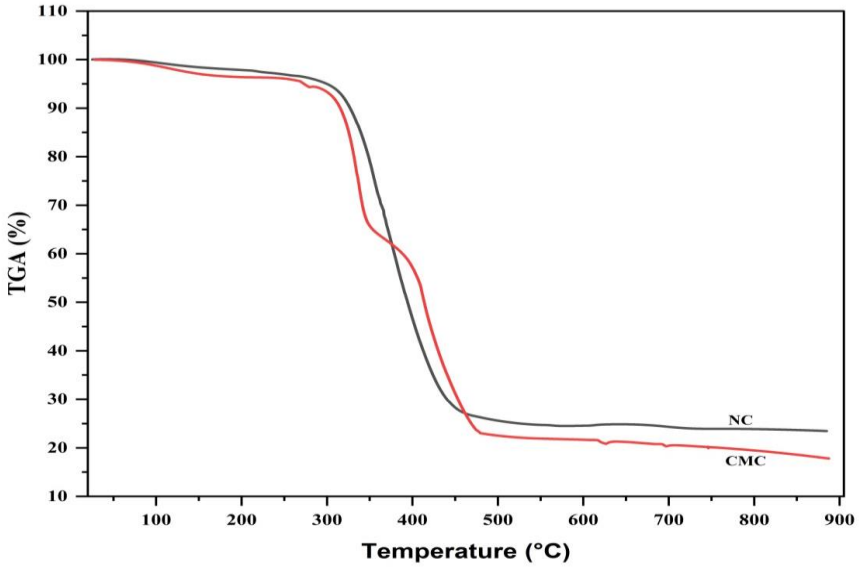


Figure 9: TGA, curves of the NC and CMC from CS.

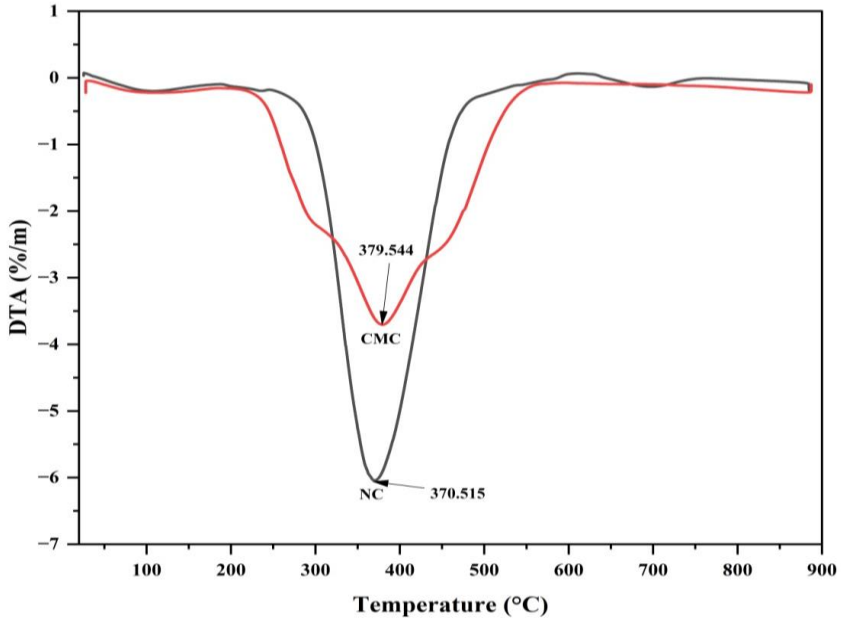


Figure 10: DTG, curves of the NC and CMC from CS.

3.8 Microbial study

Figure 11a. Zone of inhibition at 50 mg/mL for native cellulose against *E. coli.*

Figure 11b. Zone of inhibition at 50 mg/mL for native cellulose against *E. coli.*

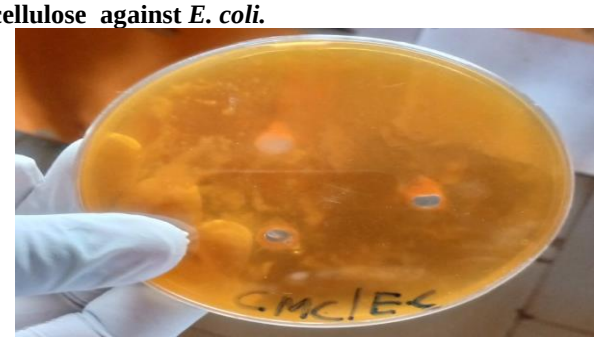


Figure 11c. Zone of inhibition at 50 mg/mL for carboxymethyl cellulose against *E. coli.*

Figure 11d: Zone of inhibition at 20 mg/mL of azithromycin solution against *E. coli.*

Table 6: Zone of inhibition for native cellulose, carboxymethyl cellulose and Azithromycin solution against *Escherichia coli.*

Test organism	Zone of inhibition for native cellulose at 50 mg/mL (mm)	Zone of inhibition for carboxymethyl cellulose at 50 mg/mL (mm)	Zone of inhibition for Azithromycin solution at 20 mg/mL (mm)
Escherichia coli	5.13 ± 0.32^{c}	7.66 ± 0.23^{b}	24.66 ± 0.97^{a}

Data represent means ± SD. a=highest, b= medium, c=lowest. Those alphabets that have different letters are statistically significant ($P<0.05$).

The native cellulose and carboxymethyl cellulose exhibited little or no response against *E .coli*. However, the carboxymethyl cellulose gave a better result than the native cellulose. In a research work, it was observed that pure CMC–BC film (without nanoparticles) showed no inhibition zones, confirming that CMC alone is not antimicrobial as shown in our study. Their study also shows that when ZnO and Ag nanoparticles are embedded or loaded on the CMC, it enhanced both the zone of inhibition size and biofilm prevention, showing a synergistic antimicrobial effect.[45] In another study, it was observed that carboxymethyl cellulose/acrylic acid (CMC/AAc) hydrogel reinforced with ZnO nanoparticles confer antimicrobial activity while pure CMC/AAc polymer is inert.[46] In our study, the azithromycin solution used exhibited strong antibacterial activity against *E. coli*. In a study carried out by Momoh et al.[14] it was observed that at 12.50 mg/mL, azithromycin exhibited strong response against *E. coli* and moderate response against *S. aureus* while at higher concentration of 25 mg/mL, the drug exhibited potent response against *E. coli* with zone diameter greater than 30 mm and strong response against *S. aureus* with zone diameter less than 30 mm.[14] In another study, it was observed that azithromycin solution at 20 mg/mL, showed moderate response to both *S. aureus* and *E. coli* and at 5 mg/mL the drug exhibited little or no response which is in line with previous study.[47] Momoh and Olaleye[15] study shows that azithromycin solution at 25 mg/ml showed potent response against *E. coli* with zone of inhibition of 37.34±0.65 and strong response with zone of inhibition of 23.37±0.48 at concentration of 12.50 mg/mL. At a lower concentration of 5.00 mg/mL used in that study, the antibiotic exhibited weak response against *E. coli* with zone of inhibition of 15.37 ± 0.33. The study supports the results of azithromycin solution obtained in this research work.

4. CONCLUSION

It was possible to successfully extract and characterize cellulose from CS. Through an etherification procedure, the isolated cellulose was modified into CMC. Temperature, etherifying agent amount, and duration all increase the degree of substitution of the modified

cellulose. The modification method was validated by FTIR, SEM-EDX, XRD, and TGA/DTG. The native cellulose and carboxymethyl cellulose exhibited little or no response against *Escherichia coli.*

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