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The Sustainable Production of Carboxymethyl Cellulose from Waste Cotton: A Comprehensive Chemical Analysis

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ABSTRACT

At first, hemicellulose and lignin were eliminated in order to recover cellulose from discarded cotton. Following the extraction of cellulose, monochloroacetic acid and 30% NaOH were used for etherification and alkalization in an isopropanol-ethanol (70:30) medium to generate carboxymethyl cellulose. The yield of produced cellulose and carboxymethyl cellulose was calculated, and they by Fourier-transform infrared spectroscopy, X-ray diffraction, scanning electron microscopy, and thermogravimetric analysis. The yield of cellulose was 97.66%, and carboxymethyl cellulose was 171.05%. The degree of substitution was ascertained using the back titration method. The degree of substitution of CMC was 1.659. It indicated that the synthesised carboxymethyl cellulose was soluble in water. The successful etherification of cellulose became apparent by the occurrence of a new, strong band at 1600 cm^{-1} (COO^{-}). The amorphous character of carboxymethyl cellulose was confirmed by the loss of several crystalline peaks in cellulose and the development of peaks around 9.4°. The estimated crystallinity index of carboxymethyl cellulose was 30%, a significantly lower figure than the 55.3% crystallinity index for cellulose. Solubility was triggered by lower crystallinity index values. The carboxymethyl cellulose formation was reaffirmed by the rod-like structure. It showed TGA breakdown thermograms at temperatures between 205°C and 401°C. Antimicrobial activity results showed that CMC was not sensitive towards the microbes. But it was found to be susceptible to *Aspergillusniger*. In this work, a higher DS and high yield been attained.

INTRODUCTION

One of the most important crops for humankind is cotton. Even though its main purpose is not to produce food, it is among the ten most often planted crops worldwide. Apart from valuing its fibres, cultures discovered ways to use the entire plant for a range of applications, from medicines and reproductive control to textile[1]. As worldwide reliance on cotton escalates, so does the amount of waste generated. Cotton waste has been either burned or land filled, which culminates in numerous ecological risks [2]. Cotton's strength as a functional material is related to the rigidity of a cellulose chain and its crystalline structure, which facilitates intra-molecular and intermolecular hydrogen bonding [3]. Cotton contains more than 90% cellulose [4].

Cellulose can be used in clothing, biofuels, explosives, and so on.

Cellulose is the primary structural component of plants, responsible for structural support, strength, and stability in plant cell walls. It is a naturally occurring linear polymer made up of anhydrous glucose units. It contains repeated glycosidic linkages at locations C-1 and C-4. It contains two secondary OH groups and one primary OH group at C-2, C-3, and C-6, allowing for easy conversion into various derivatives [5]. Cellulose is recalcitrant in most solvents, including water. The substantial hydrogen bonding prevalent in cellulose's crystalline areas is often attributed to its insolubility [6]. Because of this, cellulose has limited applications. To overcome its limitation, cellulose is modified into various cellulose derivatives whose reactive hydroxyl groups are chemically altered by various reactions such as esterification and etherification, resulting in derivatives such as cellulose esters (e.g., cellulose acetate), cellulose ethers (e.g., carboxymethyl cellulose, methyl cellulose, ethyl cellulose, cellulose sulphate, and cellulose nitrate, etc). Cellulose is converted into carboxymethyl cellulose (CMC) through the carboxymethylation process [7].

CMC is an anionic, water-soluble derivative of cellulose, a linear polysaccharide containing anhydroglucose units. ß-1, 4-glycosidic linkages connect the repeating units. CMC differs from cellulose by the CH_2COOH group. The CH_2COOH group gets substituted in the place of hydrogen atoms of some hydroxyl groups in the cellulose structure [8]. It is a water-based, innate, and biodegradable substance for the carboxymethyl group. When CMC is discharged into the water, no negative impacts on aquatic life have been reported. Its non-toxicity makes it suitable for use as a food additive [9]. CMC quality diverges by application type, encompassing technical, semi-purified, and purified. Purified CMC is a free-flowing powder with a white to creamy yellow colour. It has no taste or odour [10]. The applications for CMC-based blended materials are numerous due to their simple, low-cost synthesis process, abundance of raw materials, characteristic surface properties, mechanical strength, different formability, hydrophilicity, viscosity, rheological properties, and hundreds of other contrasting aspects [11]. CMC is used in numerous fields, including the medical field, cosmetic industries, food packaging applications, textiles, oil drilling, and paper manufacturing industries. It is widely used in wound healing, biosensors, implants, and bone cement [12]. So, in this current study, carboxymethyl cellulose with high yield and high degree of substitution was synthesized from waste cotton (WC). To produce CMC from WC, two phases were required. 1) Conversion of WC into cellulose. 2) Conversion of cellulose to CMC: This stage requires three steps: alkalization, carboxymethylation, and neutralisation [13-15]. The synthesized CMC was characterized using distinct techniques to study its properties.

MATERIALS AND METHODOLOGY

WC was gathered from small-scale handloom industry in Kanyakumari district. Sodium hydroxide (NaOH), monochloroacetic acid ($ClCH_2COOH$), isopropanol, ethanol, and hydrogen peroxide (H_2O_2), glacial acetic acid were the analytical grade chemicals used in this investigation and were procured from Molychem, Mumbai.

Conversion of WC into Cellulose

The cellulosic components in WC should be garnered so that CMC production doesn't get interrupted. WC was cleaned up, dried, and boiled with a 30% NaOH solution for 10 minutes until it turned brown. WC entails not only cellulose but also hemi-cellulose and lignin. These two illicit substances needed to be removed so that only cellulose could be retrieved. H_2O_2 was used for bleaching the cellulose, and it turned from brown to white. The cellulose residue was filtered, rinsed completely with water, dried, and the process of turning them into a derivative compound began.

Conversion of cellulose to CMC

Cellulose from WC was incorporated with isopropanol and ethanol (70:30) mixture in a beaker and stirred for 45 minutes at room temperature using a magnetic stirrer. 30% NaOH was added dropwise while stirring. After 45 minutes, the temperature was raised to 55° C. Monochloroacetic acid was added, and the amount added was half the weight of cellulose. The mixture was stirred for 90 minutes. To remove undesirable salts, the residue was neutralised with glacial acetic acid, filtered, and washed with 70% ethanol. The upper phase was discarded. The extracted CMC was oven-dried at 55 °C. Figure shows the representation of the preparation of CMC.

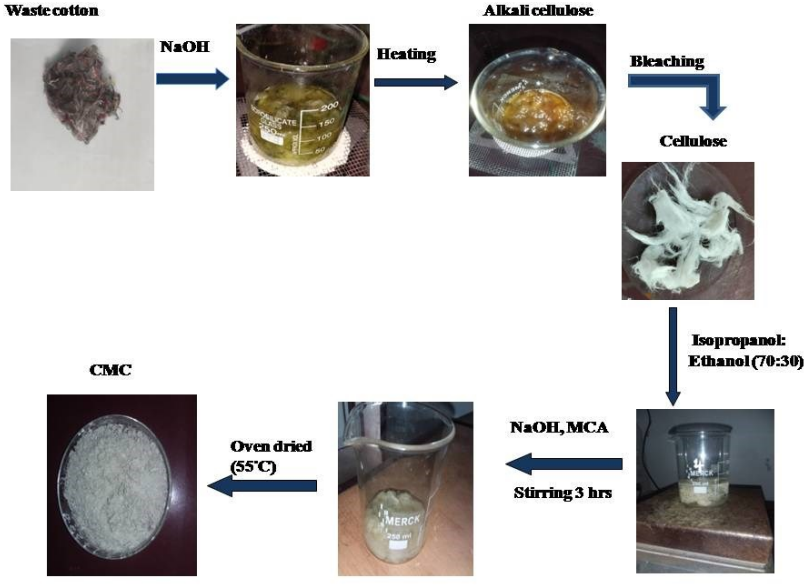

Figure 1 Schematic representation of the preparation of CMC

Cellulose and CMC Yield

The yield of cellulose and CMC was determined on a dry-weight basis. The total yield of cellulose and CMC was calculated using equations 1 and 2 [16].

$$Celluloseyield\ (\%) = \frac{\text{Weight of cellulose(g)}}{\text{weight of waste cotton(g)}} \times 100 \text{ ---- (1)}$$

$$CMCyield\ (\%) = \frac{Weight\ of\ CMC(g)}{weight\ of\ cellulose(g)} \times 100 \text{ ------ (2)}$$

Degree of substitution (DS)

2 g of CMC was dispersed in 10 mL of acetone and swirled for 10 minutes. 6 ml of 6N HCl was added to the dispersion and stirred continuously for 20 minutes. Filtered precipitates were washed with a 70:30 ethanol: water solution. The precipitate was filtered and dried at 50 °C. 20 ml of 0.2 N NaOH was used to dissolve 0.5 g of dried CMC. 50 ml of distilled water was added. The solution was then added to a 100-mL standard flask. It was filled with distilled water to the mark. A 25 ml solution was taken in a conical flask and titrated against 0.05 N HCl using phenolphthalein as an indicator. The disappearance of a pale, persistent pink colour (back titration) indicates the end point [17].

$$A = \frac{BC-DE}{F} \text{ ---------- (3)}$$

$$DS = \frac{0.162 \times A}{1-(0.058 \times A)} \text{ ----- (4)}$$

Where A = Milli-equivalent of consumed HCl per gram of sample, B = Vol. of NaOH, C = Normality of NaOH, D = Volume of HCl, E = Normality of used HCl, F = Weight of used CMC in gram,162 = Molecular weight of anhydrous glucose unit, 58 = Net increment in mass of anhydrous glucose unit for each substituted CMC group.

Characterization

Fourier-Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy, or Fourier transform infrared spectroscopy, encompasses the vibration of molecules. Each functional group has its own vibrational energy, which can be used to identify a molecule by combining all of the functional groups. Elmer-2000 was used to gauge the functional groups of cellulose and CMC between 4000 cm^{-1} and 400 cm^{-1}, at a resolution of 4 cm^{-1}.

X-ray Diffraction Spectroscopy (XRD)

XRD analysis was carried out to determine the crystalline and amorphous nature of cellulose and CMC. Powder X-ray measurement was performed on a Rigaku 1V diffractometer with Cu kα radiation at 40 and 40 mA in the range of 2θ = 5- 80°and the scanning rate is 10°/min.

Scanning Electron Microscopy (SEM)

A scanning electron microscope (SEM) was used to examine the surface morphology of the CMC sample. A focused stream of electrons is projected and scanned across a sample's surface using a scanning electron microscope (SEM), which uses detectors to gather the various signals that are produced. It was carried out using a ZEISS instrument.

Thermogravimetric analysis and Differential Thermogravimetry (TGA-DTG)

Thermogravimetric analysis (TGA) is widely used to identify specific properties of materials that experience mass loss or gain owing to decomposition, oxidation, or volatile loss (such as moisture). Differential thermogravimetry, or DTG, is a TGA derivative that gauges weight change rate in relation to temperature. It is useful for figuring out the temperature at which a substance experiences a certain thermal event, such as combustion or breakdown.

Antimicrobial activity

Antimicrobial activities of CMC evaluated using the disc diffusion method. For the antimicrobial analysis, *E. coli* and *Aspergillusniger* were the test microorganisms to study the inhibitory effects of CMC. For antimicrobial testing, a standardised inoculum should be utilised. Muller Hinton Agar Medium dissolved in 1000 millilitres of distilled water to create the medium. For 15 minutes, the dissolved medium (pH 7.3) was autoclaved. After cooling and thoroughly mixing the autoclaved solution, Petri plates containing a culture of pathogenic bacteria were swabbed. CMC was poured onto the disc, placed on top of Mueller Hinton medium, and the plates were incubated at 37° °C for 24 hours. After the incubation period, the zone of inhibition was measured using a transparent ruler. The zone of inhibition was expressed in millimeters. If there were no zone of inhibition, the sample seemed to be inactive against bacteria or fungi.

RESULTS AND DISCUSSION

The organoleptic properties and solubility of CMC were observed. The carboxymethyl cellulose derived from discarded cotton was white in hue, odor-free and easily soluble in water

Cellulose and CMC yield

The yield of cellulose and CMC was calculated. The yield of obtained cellulose was 97.66%, and CMC was 171.05%. The key goal for cellulose-derived CMC synthesis was to activate the hydroxyl groups present in cellulose, as exemplified by their ability to function in an alkaline environment. Monochloroacetic acid reacted with alkali cellulose to change it into CMC. The high yield of CMC over cellulose was because of the inclusion of monochloroacetic acid during the carboxymethylation process. Carboxymethyl groups were substituted for OH groups in cellulose, resulting in increased mass. Consequently, the amount of monochloroacetic acid added was not taken into account while calculating the yield percentage [18,19].

Degree of substitution (DS)

Cellulose is modified to CMC to increase its applications. Since DS is the primary metric for identifying the characteristics of the product, an accurate estimate of the DS of CMC is crucial. To ascertain the DS of CMC, back titration was employed. As DS increased, so did polymer solubility. When the DS ≤0.4, the polymers become soluble in dilute alkali. When DS ≥ 0.4, it becomes water-soluble. The determined DS of the CMC was 1.659, which was found to be water soluble. Several publications pointed out that DS is determined by parameters such as 1) alkali dosage, 2) etherifying agent, and 3) temperature. Alkali dosages of up to 30% are ideal for CMC synthesis. Beyond or below that value, it begins to react with by-products, resulting in inefficient synthesis of CMC. Excessive levels of etherifying agent (MCA) increase the exiguity of mercerized cellulose alkoxide, lowering DS. Temperatures up to 55°C promote better carboxymethylation; temperatures above that point cause cellulose breakdown and low DS levels

Characterization

Fourier-Transform Infrared Spectroscopy (FTIR)

the FTIR spectra of waste cotton, cellulose, and CMC. The broad OH, CH, and CH_2 groups induce absorption bands that appear at 3436 cm^{-1}, 2923 cm^{-1}, and 1459 cm^{-1} in waste cotton and show absorption bands at 3502 cm^{-1}, 2891 $cm^{-1,}$ and 1420 cm^{-1} for cellulose. In reference to the spectra of waste cotton and cellulose, it turned out that the absorption bands at 1506 cm^{-1} not visible in the latter. This may ensure that the lignin is being removed, since the absorption bands in lignin exhibit frequencies of 1506 cm^{-} The peaks between 950 and 850 cm^{-1} were prompted by glycosidic connections. C-O-C pyronose groups can be seen by the distinctive peaks at 1112.99 cm^{-1} and 1124 cm^{-1}.

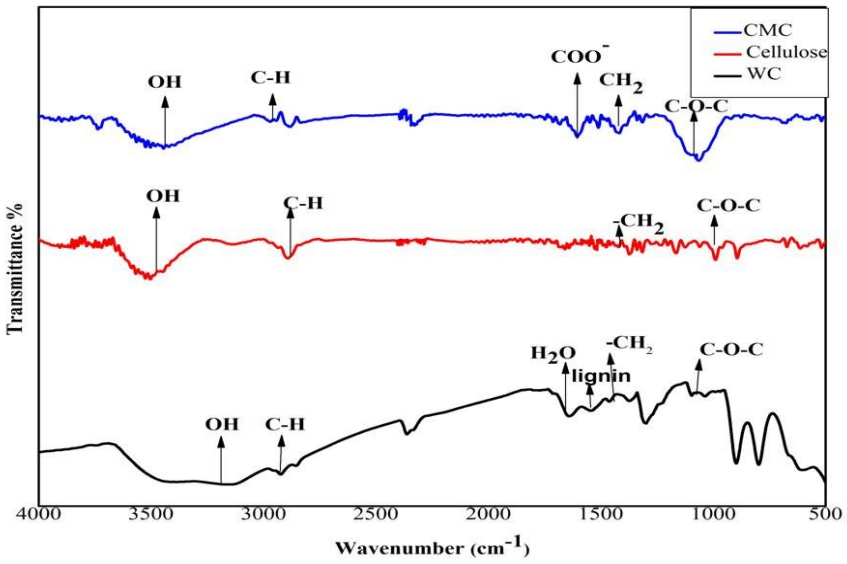

Figure 2 FTIR spectra of WC, cellulose, and CMC

The observed peaks ensured that carboxymethyl groups had been substituted for cellulose. Akin functional groups were seen in CMC and cellulose. It exhibited absorption bands at 3444.63 cm^{-1}, C-H vibrations at 2881 $cm^{-1}$was due to the OH group, $-CH_2$ scissoring at 1420 cm^{-1}, and an ether group ($-CH-O-CH_2-$) at 1000-1200 cm^{-1}. A broad OH band can be seen in CMC compared to cellulose. It was pertinent to observe that the strong absorption band that appeared at 1600 cm^{-1} verified the existence of the COO group. In CMC, there was an increase in the methyl group ($-CH_2$), carbonyl group (C=O), and ether group (-O). In the meantime, monochloroacetic acid caused the carboxymethyl groups to be substituted, resulting in a reduction in the hydroxyl group (OH) in CMC, in contrast to cellulose. These findings validated the production of CMC and the carboxymethylation process [23].

X-ray Diffraction Spectroscopy (XRD)

XRD has been used to determine the crystallinity. Figure 3 XRD peaks of cellulose and CMC. The crystallinity in cellulose was shown by sharp, narrow peaks at diffraction angles 16.4°, 21.19°, 37.74°, and 44.07°, and the background noise line indicated the amorphous region[24,25]. The peaks at 9.4°, 20.1°, 31.3°, and 44.7° showed the crystallinity of CMC [26, 27]. The estimated crystallinity index of cellulose was found to be 55.3%. While the XRD curve of carboxymethyl cellulose showed a clear difference when compared with cellulose. Some crystalline peaks had and some other sharp peaks in cellulose clearly diminished in CMC. Significant changes in peak height, width, and peak shifts were seen. While 30% NaOH was employed, the chains of cellulose molecules stretched and induced pressure on nearby crystallites, which caused the structure to unravel or cleavage of hydrogen bonds, as well as the crystalline structure This caused the phase transition from crystalline to amorphous. In contrast to cellulose, carboxymethyl cellulose was found to have a drastically lower speculated crystallinity index of 30%. As a consequence, CMC's lower index fosters higher solubility. This pointed out that CMC was formed and that it was amorphous.

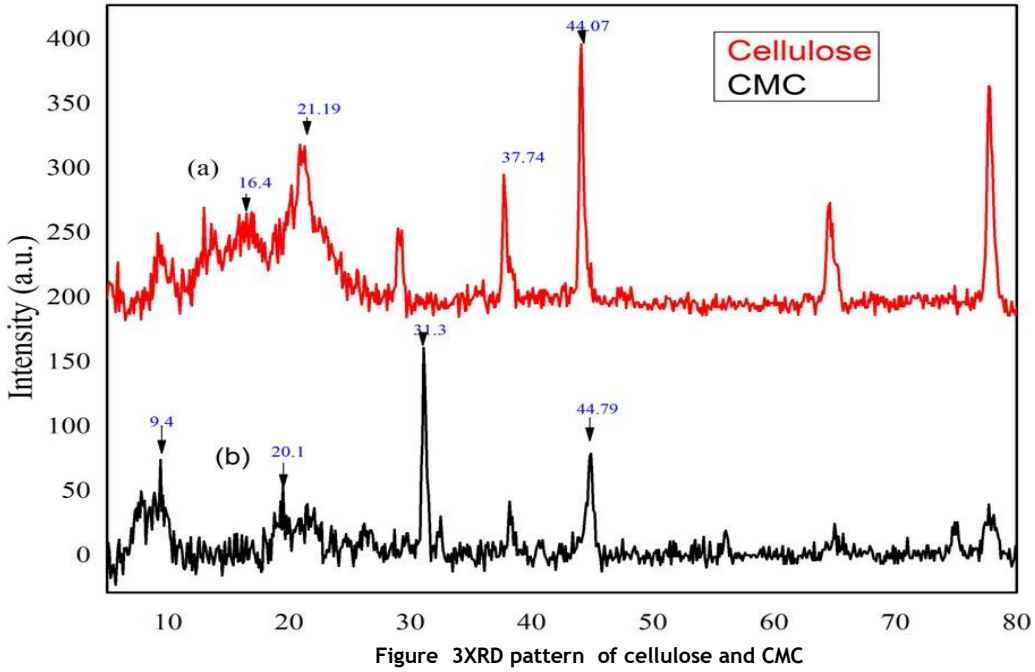

Figure 3XRD pattern of cellulose and CMC

Scanning Electron Microscopy (SEM)

A scanning electron microscope was used to analyse the surface's morphology. Figure 4 (a, b, c, and d) the surface morphology of cellulose and CMC. The cellulose surface obtained from discarded cotton (figures 4a and 4 b) displayed a lengthy, smooth surface together with a regular, dense, and fibrous structure. However, surface homogeneity declined for CMC molecules. Following carboxymethylation, the surface structure of CMC became weakened and deviated from its intended form. The NaOH partially disrupted the structure of the cellulose by penetrating its amorphous regions. These results correlated with those of other CMC molecule images that had been revealed [26, 28]. The CMC surface's fibrous structure was arranged in a woven network. The etherifying agents had more access to cellulose molecules during CMC production, which makes the cellulose structure, and lose its crystallinity.

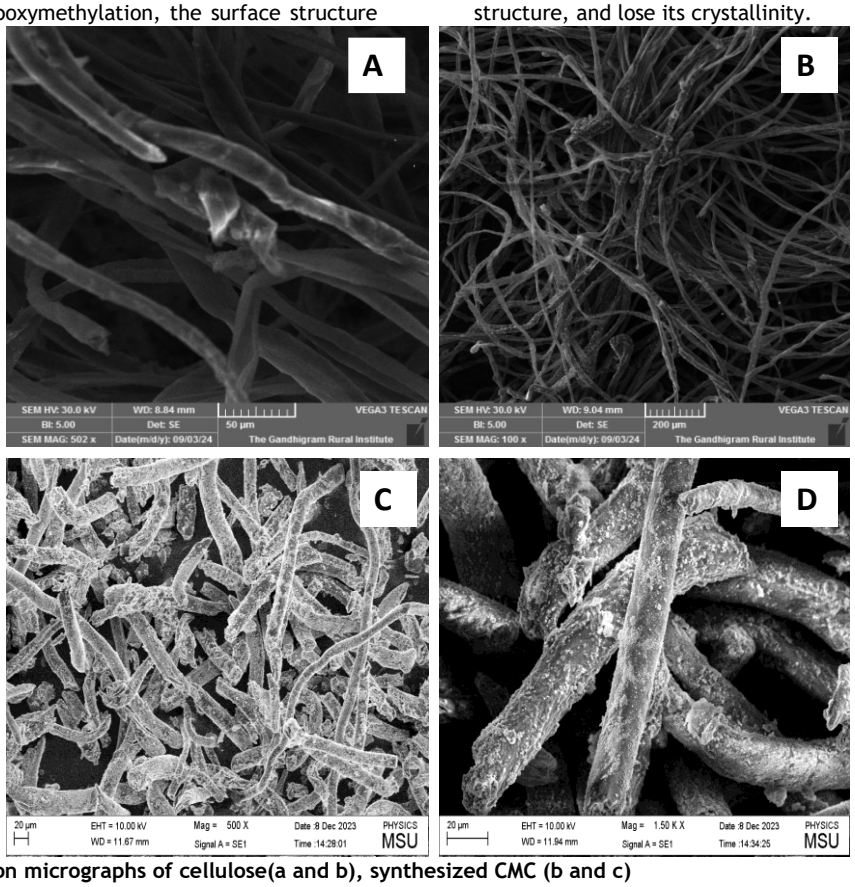

Figure 4 Scanning electron micrographs of cellulose(a and b), synthesized CMC (b and c)

Thermogravimetric analysis and Differential Thermogravimetry (TGA-DTG)

Figure 5 (a) the TGA of cellulose. The thermal breakdown of cellulose was examined between 36 and 500 ◦C. For cellulose, the TGA curves showed two different weight-decreasing stages during thermal degradation. Weight loss at the early and slow pyrolysis stages of the curve had been correlated to water vaporisation, and volatilisation happened with a weight loss of 5 % between 36-123◦C, which can be ascribed to reduced hydrogen bonding and intermolecular interactions in that temperature range. The second weight was recorded between 123-362◦C experiencing weight loss of roughly 35% respectively. DTG curve showed the maximum decomposition at 321°C.

For CMC, five transition temperatures at 32, 129, 205,401, and 725°C were shown by the TGA curve. During CMC decomposition with a weight loss of 12.44%, the volatile molecules were eliminated between 32 to 100 °C. Diverse simultaneous processes, including dehydration, depolymerisation, and disintegration, contributed to the second weight loss at 129°C (mass loss of 8%). Conversely to the third phase, there was a little decrease in weight in the second stage. With a weight loss of approximately 35.70%, the main degradation stage, also known as the pyrolytic stage, began roughly at 205°C and terminated at 401°C. Almost 56% CMC weight loss was observed at temperatures lower than 401°C. The heat breakdown of the carbohydrate polymers was the cause of the third weight loss. There were several stages involved in the degradation of CMC. Based on the DTG thermogram, decomposition of CMC showed endothermic curves. At 233 - 398°C, the endothermic characteristics were noted as a result of CMC breakdown, and it showed the maximum decomposition at 300°C.

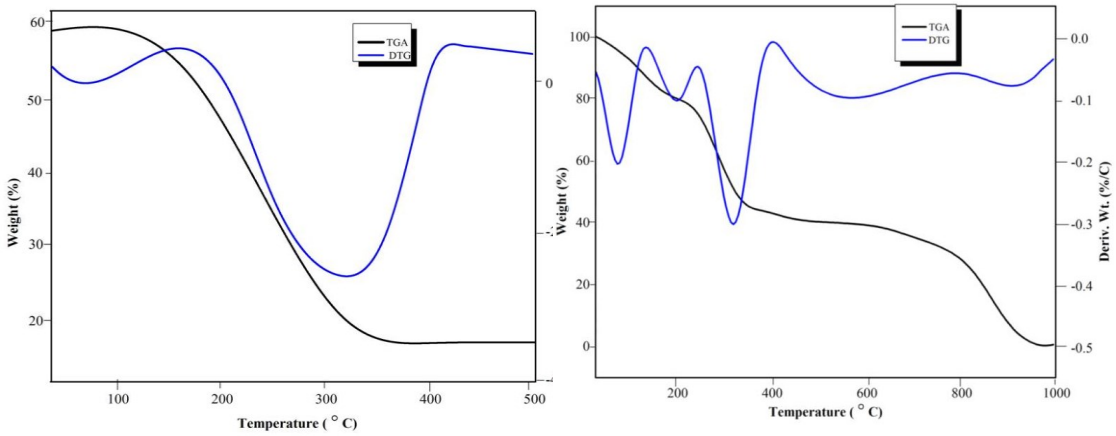

Figure 5 (a) TGA and DTA curves of cellulose

The decarboxylation of carboxylic groups in CMC and the release of CO_2 were the triggers of the weight loss of the CMC sample. Due to their thermal instability, the broken molecules were subjected to further breakdown after they were created. The process continued until the ends of the polymer chains, resulting in aromatised units and ultimately a cross-linked carbon skeleton. The molecular structure and bonding energy of CMC were altered during the conversion from cellulose, leading to the distinct thermal behaviour of CMC. However, compared to cellulose (36°C), the primary weight loss of CMC (32°C) was pushed to a lower temperature, which may support CMC's reduced thermal stability. This may be due to the negatively charged carboxymethyl groups in CMC and due to inter- and intra-molecular hydrogen bonds between cellulose fibres. During the CMC preparation process, mercerisation with NaOH enhanced the amorphous structure of CMC

Antimicrobial activity

CMC's antibacterial and antifungal properties were evaluated against the gram-negative bacterium *Escherichia coli* and the fungus species *Aspergillusniger*. CMC showed little to no effectiveness against and Figure 6 the zone of inhibition around the well. The zone of inhibition values for *E. coli* and *Aspergillusniger* were 10 and 9 mm, respectively. Because the carboxymethyl cellulose had a very low zone of inhibition value, it was determined that it had little effect in inhibiting the growth of both bacteria and fungi. Thus, bacterial or fungal growth was possible.

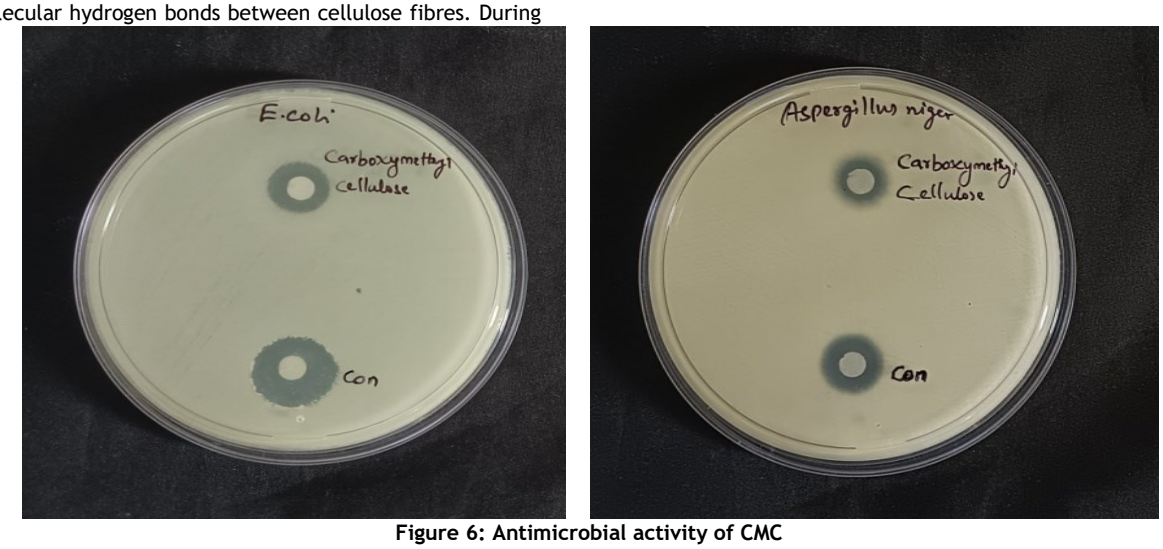

Figure 6: Antimicrobial activity of CMC

CONCLUSION

The following conclusions were drawn from the outcomes of our observations and experiments. WC serves as a reliable supply of cellulose for the production of CMC. A high yield of CMC with a high degree of substitution was obtained by infusing 30% NaOH, 1:1 (Cellulose: MCA), and 70:30 (isopropanol: ethanol) in the process. Characterizations such as FTIR, XRD, SEM, and TGA-DTG revealed the effective synthesis of CMC. Antimicrobial activity results showed low activity against.

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