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Characterization of carboxymethyl cellulose derivatized from melon seed shells α-cellulose

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Abstract

This study focuses on converting melon seed shells, which are typically discarded as waste, into carboxymethyl cellulose (CMC), a valuable cellulose derivative with a wide range of industrial applications. By repurposing agricultural waste, specifically melon seed shells, this process adds value to an otherwise unused material. The dried and ground melon seed shells were initially soaked in hot water and subsequently treated with a 20% w/v sodium hydroxide (NaOH) solution at 90–100°C for 2 hours. The isolated cellulose fiber was transformed into carboxymethyl cellulose through alkalization and etherification processes. The physicochemical, micromeritic and pre-formulation characterization of the derivatized melon Peel carboxymethyl cellulose (MP-CMC) was carried out. The MP-CMC gave a yield of 48% and a degree of substitution of 1.732 (57.7 % degree of substitution). The micromeritic analysis indicated that MP-CMC demonstrated outstanding flow characteristics, with densities (bulk and tapped) of 0.3907 g/mL and 0.4238 g/mL, respectively, along with an angle of repose measuring 27.46°. Other analyses such as FTIR spectroscopy, X-ray diffraction and SEM imaging were employed to assess the structural, morphological and crystalline properties of MP-CMC. It can be concluded that MP-CMC if studied further could possibly be a potential pharmaceutical excipient

Keywords: Melon Seed shells; Agriculture waste; Carboxymethylcellulose; Pharmaceutical Excipient; Characterization

1. Introduction

The increasing global emphasis on sustainable and environmentally friendly practices has driven researchers to explore innovative avenues for utilising agricultural by-products to create value-added materials[3].Cellulose, a prominent component of plant cell walls, possesses inherent biopolymeric properties that make it an attractive candidate for modification and utilisation[5]. Carboxymethylation is one way cellulose can be modified. It is a chemical process that introduces carboxymethyl groups onto the cellulose backbone, enhancing its solubility, swelling capacity, and reactivity, thus rendering it suitable for an array of applications [19].

Carboxymethyl cellulose (CMC), commonly referred to as cellulose gum, is a modified form of cellulose where carboxymethyl groups ($-CH_2-COOH$) are introduced by substituting some hydroxyl groups present in the glucopyranose units of the cellulose backbone. It is commonly utilized in its sodium salt form, referred to as sodium carboxymethyl cellulose. The characteristics of CMC are influenced by factors such as the degree of substitution within the cellulose structure, the length of the cellulose polymer chain, and the distribution pattern of carboxymethyl groups. Carboxymethyl cellulose has a wide range of applications, such as component in various non-food products, including K-Y Jelly, toothpaste and laxatives, a lubricant in non-volatile eye drops (artificial tears), a viscosity modifier and water retention agent, thickener, and emulsion stabiliser, an adhesive or fixative in conservation-restoration processes, etc. [21].

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Studies have shown that CMC can be derived from different sources, including the pseudo stem of Cavendish banana, sago waste, palm kernel cake, cotton ginning industry waste, pulp of sugar beet, sugarcane straw, waste paper, and corn stalks [16]. The production and analysis of carboxymethyl cellulose (CMC) derived from melon seed shells present a promising approach. Melon seed shells, often regarded as agricultural waste, present an untapped resource with potential applications in various industries.

Cucumeropsis mannii, an indigenous melon species, thrives in tropical Africa, specifically in regions west of the East African Rift [20]. The plant serves as a valuable source of food and oil. *Cucumeropsis mannii* seeds are visually characterised by their large size and white appearance, sometimes displaying a brownish hue. This study delves into the conversion of melon seed shell cellulose into CMC, exploring its transformation from an underutilised residue into a versatile biopolymer.

Cellulose has been extracted from several agricultural wastes and converted into microcrystalline cellulose or carboxymethyl cellulose but information on the extraction of cellulose from melon peel and conversion into carboxymethyl cellulose is scanty. The purpose of the study therefore is to extract cellulose from melon seed shells and subsequently modify it through carboxymethylation to produce CMC. By characterising the synthesised CMC, its physical, chemical, and functional properties will be assessed, paving the way for potential applications across industries.

2. Material and methods

The materials utilized in this study included sodium hydroxide, isopropanol, monochloroacetic acid, methanol, 90% glacial acetic acid, 70% ethanol, and melon seed shells, which were sourced from a local market in Warri, Delta State, Nigeria.

2.1. Isolation cellulose from Melon seed shell

Cellulose extraction from the melon seed shell was performed following a modified version of the method previously described by Ibikunle et al. [12]**.** A 200 g sample of melon seed shell was weighed and immersed in hot water for 1 hour, followed by filtration. The retained residue was subsequently treated with a 20% w/v sodium chloride solution at a liquid-to-material ratio of 10:1 (v:w) and heated at 90–100°C for 2 hours.

The obtained cellulose slurry was subjected to filtration and multiple washes with distilled water. Once cooled, the cellulose fibers were further rinsed with distilled water until a neutral pH was attained. The purified cellulose was then dried in an oven at 60°C for 8 hours before being weighed using an analytical balance. The percentage yield of the isolated alpha-cellulose was calculated using the following equation:

$$\%\, Yield = \frac{Mass\ of\ cellulose\ (g)}{Mass\ of\ melon\ seed\ shell\ sample\ (g)}\ x\ 100\ ----Eqn\ 1$$

2.2. Derivatization of Melon peel Carboxymethyl cellulose (MP-CMC)

The transformation of isolated cellulose from melon shell into carboxymethyl cellulose (CMC) involved a two-step process: alkalization followed by etherification, conducted under heterogeneous conditions. During the alkalization pretreatment, 50 g of cellulose was placed in a 1000 mL beaker, followed by the addition of 500 mL of isopropanol. The mixture was allowed to stand for 5 minutes before 400 mL (120 g) of aqueous sodium hydroxide was gradually added while stirring continuously on a magnetic stirrer for 1 hour. This reaction between the hydroxyl (-OH) groups of cellulose and sodium hydroxide is referred to as the mercerization process.

Following alkali treatment, the etherification process was initiated by adding 80 g of monochloroacetic acid (MCA) at 70°C, and the reaction mixture was stirred for 1 hour. Longer time of reaction will increase degradation of polymer and will reduce the DS value as well [7].The slurry was then soaked in methanol for 4 hours, neutralised with 90% acetic acid until it reach pH 6-8.The slurry was filtered and then purified by washing with 70% ethanol to remove undesired by-product to get pure CMC. Then, the pure MP-CMC was filtered and dried at 60°C in an oven for 24 h [12].

2.3. Micrometric properties of Melon Seed shell Carboxymethylcellulose (MP-CMC)

2.3.1. Determination of Flow Rate

The flow rate was determined according to (Uzondu *et al.*[24]with little adjustments.

$$\text{Flow rate (g/secs)} = \frac{w}{t} \quad ——————— \quad Eqn\ 2$$

Where W= weight in grams of MP-CMC t= time in seconds

2.3.2. Angle of repose

This was determined following standard USP (2010) method [23]

$$Angle\ of\ repose = \frac{tan^{-1}(2h)}{d} ——————— \quad Eqn\ 3$$

Where; h = height of the heap of powder and d= diameter of the base of the heap of powder.

2.3.3. Bulk and Tap Densities

A 25 g portion of the powder sample was placed in a clean, dry 50 mL measuring cylinder, and the initial volume (V_0) occupied by the sample before tapping was recorded. The cylinder was tapped 100 times, and the volume after tapping (V_{100}) was recorded. Bulk and tap densities were then determined by calculating the ratio of the sample's weight to its respective bulk and tapped volumes (V_0 and V_{100}) Using these density values, Carr's index and Hausner ratio were also calculated based on the obtained bulk and tapped density values.

$$\text{Bulk Density} = \frac{Weight\ of\ Powder\ Sample\ (g)}{Bulk\ Volume\ of\ Powder\ Sample\ (ml)}. \quad ——————— \quad Eqn\ 4$$

$$\text{Tapped Density} = \frac{Weight\ of\ Powder\ Sample\ (g)}{Tapped\ volume\ of\ Powder\ sample\ (ml)} ——————— \quad Eqn\ 5$$

$$\text{Carr's Index} = 100\ X\ \frac{Vo-Vf}{Vo} ——————— \quad Eqn\ 6$$

$$\text{Hausner ratio} = \frac{Vo}{Vf} ——————— \quad Eqn\ 7$$

Where V_o = Unsettled apparent volume and V_f = Final Tapped volume

2.3.4. True density

The true density (Dt) of CMC powder were determined by the liquid displacement method using xylene and pycnometer as described by Ohwoavworhua and Adelakun[17] and Ejikeme[8]

$$Dt = \frac{\left(\frac{W2}{V}\right) * W3}{W3-W4+W2+W} ——————— \text{Eqn 8}$$

Where, V=Volume of the pycnometer (ml), W=Weight of the empty pycnometer(g).

W1=Weight of filled pycnometer (g) , W2=Different between W1 and W

W3=weight of Dry CMC, W4=Weight of the fluid + weight of Dry CMC

2.4. Physicochemical Properties of Melon Seed shell CMC

2.4.1. Percentage yield

The cellulose yield extracted from the melon seed shell, as well as the yield of the synthesized CMC, was determined based on dry weight measurements.

$$\%\ Yield = \frac{Mass\ of\ cellulose\ (g)}{Mass\ of\ melon\ seed\ shell\ sample\ (g)}\ x\ 100\ ----\qquad From\ Eqn\ 1$$

$$(\%) Yield\ of\ CMC\ = \frac{Weight\ of\ dried\ CMC\ (g)}{Dry\ weight\ of\ cellulose\ (g)} x\ 100\% \text{ ——————————Eqn 9}$$

2.4.2. Swelling index

The dry MP-CMC powder was placed into a 10 mL measuring cylinder and tapped until it reached a settled volume of 3 mL. Water was then added to bring the total volume up to the 10 mL mark. The mixture was left undisturbed for 24 hours to allow the powder to swell. After this period, the volume occupied by the swollen wet powder was recorded.

$$Swelling\ index = \frac{Vf-Vi}{Vi} * 100. \qquad \text{———————— Eqn 10}$$

Where *Vf* represents the final volume, and *Vi* denotes the initial volume.

2.4.3. Test for cellulose

Schulze's solution (Chlor-Zinc iodide) is used to stain cellulose. To prepare the solution, 20 g of anhydrous zinc chloride was dissolved in 8.5 mL of water and allowed to cool. Separately, 1 g of potassium iodide and 0.5 mL of iodine were dissolved in 29 mL of water. This iodine solution was then added dropwise to the zinc chloride solution until a persistent iodide precipitate formed upon agitation. Finally, 1 g of cellulose was treated with the prepared solution, and the resulting color change was observed.

2.4.4. Solubility Test

The solubility of carboxymethyl cellulose (CMC) derived from the melon seed shell was assessed by dissolving it in both water and ethanol.

2.4.5. Loss on drying of CMC

A clean, dry crucible was weighed, and its initial mass was recorded as (M1). A 1 g sample of MP-CMC powder (M) was then placed into the crucible, and the new total weight (M2) was measured. The crucible containing the CMC sample was heated in an oven at 105°C for one hour. During the heating process, the crucible and its contents were periodically weighed until a constant final weight (M3) was achieved. This procedure was performed in duplicate for the MP-CMC powder derived from the melon seed shell.

$$Moisture\ content = \frac{M2-M3}{M} * 100 \qquad \text{———————— Eqn 11}$$

Where M2 is the weight of the crucible and sample before drying, M3 is the weight of the crucible and sample after drying and M is the weight of the sample

2.4.6. pH Determination

A 1 g sample of the Melon seed shell powder was dispersed in 100 mL of water and the pH of the 1% dispersion of MP-CMC was determined. [18]

2.4.7. Total Ash value determination

The determination of total ash values of the Melon Seed shell CMC was carried out as described by Kadam *et al*., [13] A 3 g quantity of MP-CMC powder was transferred into a crucible and incinerated over the burner. The sample was incinerated in a muffle furnace for six hours at a temperature range of 600–650°C. The resulting ash was white, carbon-free, and subsequently cooled before being weighed using an ashless filter paper.

2.4.8. Degree of substitution of Melon Peel CMC (MP-CMC)

The degree of substitution (DS) was determined using potentiometric back titration [22]. To begin, 4 g of dry melon seed shell carboxymethyl cellulose (CMC) was stirred in 75 ml of 95% ethanol for 5 minutes. To convert CMC into its acid form (H-CMC), 5 ml of 2M nitric acid was added under high temperature. The mixture was then allowed to cool while stirring for an additional 10 minutes, leading to the formation of solid and liquid phases. The liquid phase was removed, and the solid phase was washed five times with 20 ml of 80% ethanol at 60°C. The

resulting precipitate was then rinsed with a small quantity of anhydrous methanol and filtered. Drying was carried out at 100°C for 3 hours, followed by cooling in a desiccator for 30 minutes.

Next, approximately 0.5 g of the dried acid CMC was weighed into a 250 ml Erlenmeyer flask, where it was mixed with 100 ml of distilled water and stirred. A volume of 25 ml of 0.3 M NaOH was then added, and the solution was heated to a boil for 15 minutes. Once the product dissolved completely, the mixture was titrated with 0.3 M HCl. Phenolphthalein was used as an indicator, with the endpoint observed as a color change from dark pink to colorless.

The DS was calculated according to the equations:

$$Degree\ of\ substitution\ = \frac{0.162 * A}{1-(0.058 * A)}. \qquad \text{———————— Eqn 12}$$

$$A = \frac{BC - DE}{F}$$

Where;

- A = milli-equivalents of consumed acid per gram of specimen
- B = volume of Sodium hydroxide added
- C = concentration in normality of sodium hydroxide added
- D = volume of consumed chloric acid
- E = concentration in normality of chloric acid used
- F = specimen grams used

The molecular weight of the anhydrous glucose unit is 162 while 58 is the net increment in the anhydrous glucose unit for every substituted carboxymethyl group [7].

2.4.9. Fourier Transform infrared spectroscopy (FT-IR)

The FT- IR spectroscopic method was used for the characterization of Cellulose and CMC samples.[2] The FT- IR were taken from 4000 -650 cm^{-1}

2.4.10. X-ray Diffraction (XRD) analysis

The crystallinity of melon seed shell CMC was analyzed using X-ray diffraction (XRD) [25]. Diffraction patterns were recorded within a 2-theta range of 10° to 70°, utilizing a CuKα radiation source with a generator supply of 0 kV and 0 mA..

3. Results and discussion

3.1. Percentage yield

The yield of the extracted CMC in this research work compared favourably with the work of Hadiza *et al.*,[10] (78.4%) on CMC from Peanut shell and (77.1%) on Corn stalk.

The cellulose yield was determined to be 52.5%, while the CMC yield derived from alpha cellulose was recorded at 48%

3.2. Physicochemical Properties of Melon seed shell CMC

Table 1 Physicochemical Properties of MP-CMC

Parameters	**Results**
Percentage Yield (%)	48
pH	6.63

Swelling index(%) in 24 hours	36.9
Loss on drying (%)	10
Total ash (%)	40
Solubility	Soluble in hot water and ethanol

The yield of Melon shell Carboxymethyl Cellulose obtained is a good one (48%) considering issues of inconsistent or low yields of desired compounds associated with extractions from natural sources.

The swelling Index of the Melon shell CMC at 24 hours is 36.9%, showing poor hydration and water intake capacities.

The pH analysis indicates that the sample is slightly acidic and likely non-irritating to the mucous membrane, suggesting good biocompatibility. The observed pH level may be attributed to impurities, possibly resulting from residual unreacted MCA..

The result for loss on drying was 10%, this indicates that the CMC obtained is hygroscopic in nature and there is need to store it in an airtight container. [9].

The total Ash value is high, this indicates the presence of a high amount of impurities. This could be as a result of the site of collection of the melon seed shell and the presence of some unreacted starting materials as further investigation will show

3.3. Identification Test for Cellulose

The identification test carried out on Melon Seed shell alpha cellulose showed a slight violet blue colour to confirm the presence of cellulose in the sample.

3.4. Micrometric Properties

Table 2 Micrometric Properties of MP-CMC

Parameters	**Results**
Bulk Density	0.3907g/ml
Tapped Density	0.4238g/ml
Hausner ratio	1.0856
Compressibility Index	7.877%
Angle of Repose	27.46^{0}
True Density	1.645g/ml

Generally, the higher the bulk and tap densities, the better the tendency of the material to flow and to rearrange under compression[6]. The average bulk density obtained for Melon seed shell CMC was recorded as 0.3907g/ml while the average tapped density was recorded as 0.4238g/ml.

The findings on flow rate, angle of repose, and other derived parameters, including Hausner's ratio and Carr's index, are summarized in Table 2. Based on these results, the CMC derived from the melon seed shell exhibited excellent flow properties, as it passed freely through the funnel during the flow rate assessment, with an angle of repose measured at 27.46°.

The angle of repose is commonly used to describe the flow properties of solid materials and is closely associated with inter-particle resistance or the opposition to movement between particles.

3.5. Degree of Substitution (DS) Determination

The degree of substitution (DS) of carboxymethyl cellulose derived from the melon seed shell was assessed using potentiometric titration. The physicochemical properties of CMC are influenced by its degree of substitution. The DS of

standard commercial CMC is between 0.5 - 1.5 [14]. When the DS is below 0.4, the CMC is swellable but insoluble, while above this value, then the CMC is fully soluble with its hydro affinity increasing with increasing DS [4]. In this study, the degree of substitution obtained for MP-CMC is 1.732 which indicates a high degree of substitution. Hence they are not swellable but are soluble in water.

3.6. Fourier Transform infrared spectroscopy

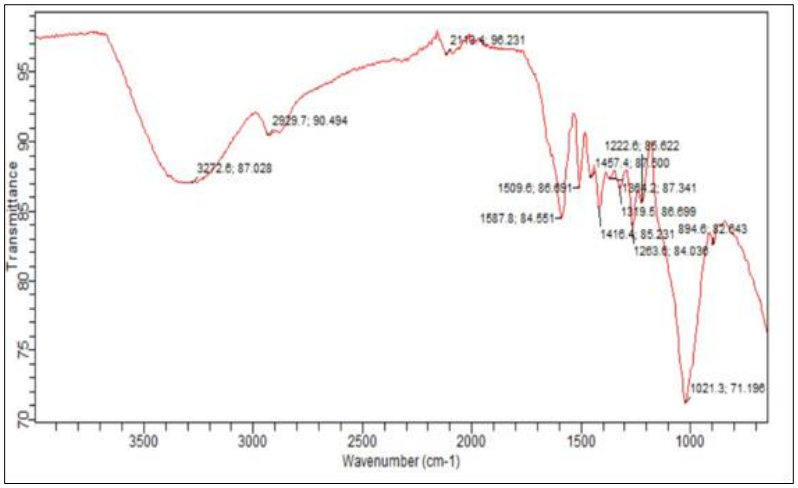

Figure 1 FTIR spectrum of synthesised MP-CMC

A broad absorption band detected at 3272.6 cm^{-1} is associated with the stretching of the -OH group, along with intra- and intermolecular hydrogen bonding. The absorption band at 2929.7 cm^{-1} corresponds to C-H stretching vibrations. The appearance of a distinct and intense absorption band at 1587.8 cm^{-1} confirms the presence of the COO group, indicating successful carboxymethylation. The intensity of this band suggests a high degree of substitution. Additionally, the peak at 1416.4 cm^{-1} in the CMC sample is linked to the C-O bond, with its intensity reflecting the extent of carboxymethyl group substitution. The band at 1457.4 cm^{-1} is attributed to $-CH_2$ scissoring and -OH bonding. Additionally, the band at 894.6 cm^{-1} is associated with asymmetric bridge stretching (C-O-C) in cellulose, as well as the β-1,4-glycosidic bonds linking glucose units within the cellulose structure.

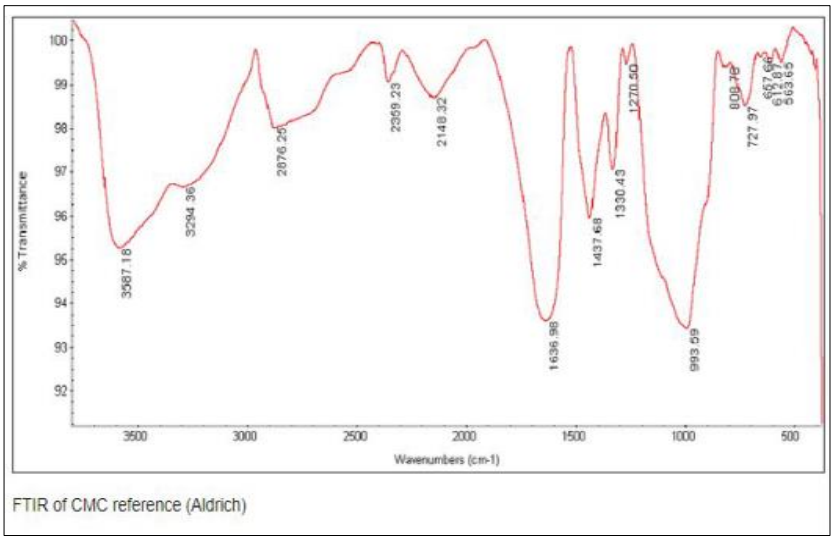

Figure 2 FTIR of CMC reference (Aldrich)

The peaks of the pure CMC are not really the same as the synthesised MP-CMC.

The synthesised MP-CMC is not purified and could be responsible for the changes. The process conditions may also be responsible for the differences in peaks.

3.7. Scanning electron microscopy Analysis of Melon Shell CMC

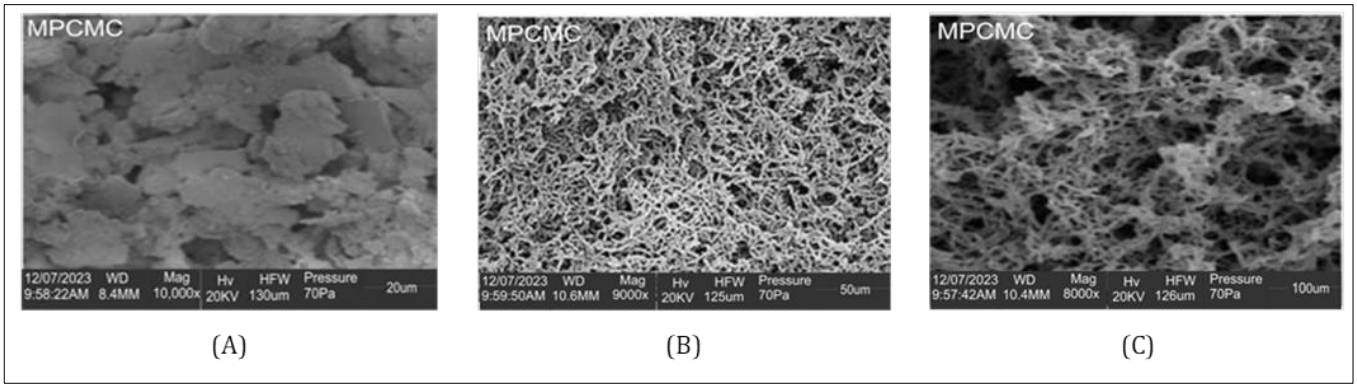

Figure 3 SEM images of MP-CMC at 10,000x, 9000x and 8000x

The images show a progressively detailed structure across the three magnifications, revealing the porous and fibrous nature of the MP-CMC.

At magnification 10,000x the surface appears denser with larger agglomerates, indicating a rough and compact structure.

At magnifications 8000x and 9000x, a highly porous network is visible, suggesting a microporous structure that could facilitate the absorption or entrapment of molecules. This porosity may contribute to improved water retention and swelling capacity, which is a desirable property in various biomedical and industrial applications.[11].

The images show no visible cracks or significant structural defects, suggesting that the MP-CMC maintains structural integrity.

The presence of interconnected pores implies potential for use in applications requiring controlled release or filtration.[1]

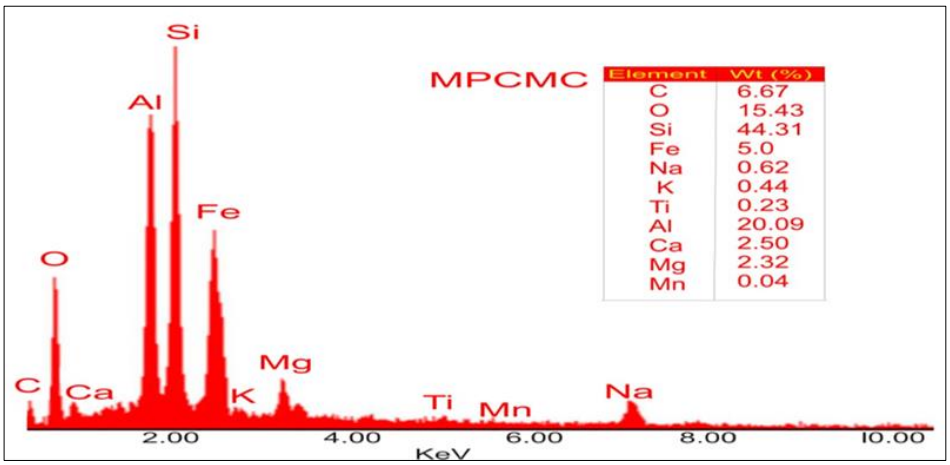

Figure 4 SEM- EDX elemental analysis

The SEM-EDX analysis provides insight into the elemental composition of MP-CMC, displaying the elements present along with their respective percentage weights. The X-axis, labeled in kilo electron volts (KeV), represents the energy levels of X-rays and gamma rays, while the Y-axis indicates the count. As previously indicated by the ash value assessment, this analysis verifies the presence of some impurities in the sample.

3.8. X-ray Diffractograms of Melon seed shell CMC

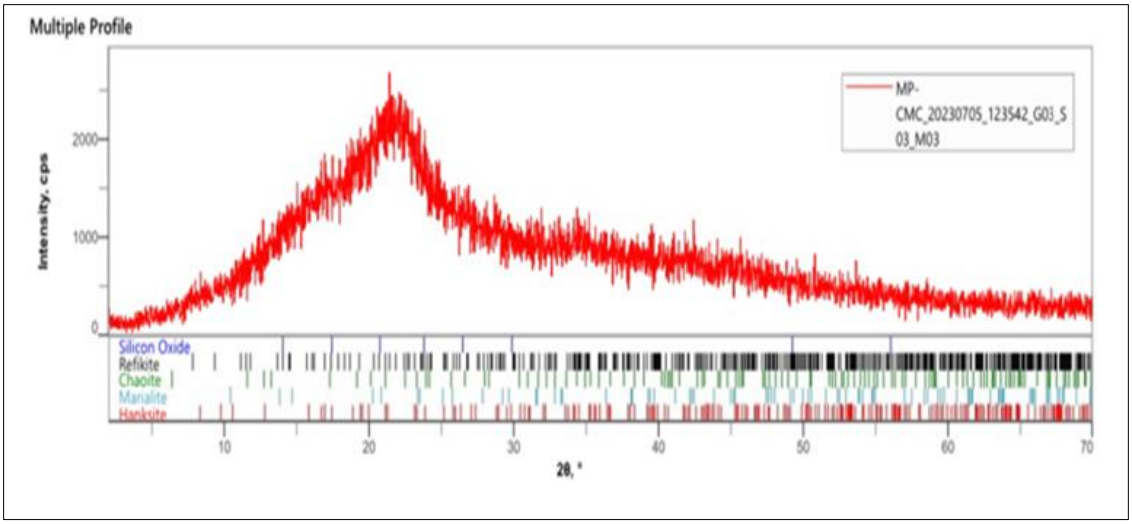

Figure 5 XRD pattern of Melon shell CMC (MP-CMC)

The X-ray diffraction (XRD) analysis of melon shell CMC revealed a broad peak, as shown in the XRD pattern above, indicating its amorphous nature. This characteristic may result from the disruption or cleavage of hydrogen bonds due to carboxymethyl substitution at the hydroxyl groups of cellulose [15]. The findings further validate that the sample with a degree of substitution of 1.732 exhibits an amorphous structure.

4. Conclusion

The results of this study provide significant insights into the physicochemical characteristics of CMC synthesized from the melon shell (*Cucumeropsis mannii*). The extracted melon shell CMC demonstrated moderate cellulose and CMC yields, with percentage values of 52.5% and 48%, respectively, indicating its potential as a sustainable source for industrial-grade cellulose production. Its high degree of substitution further enhances its applicability in various industrial processes. However, further research is necessary to evaluate the suitability of the modified MP-CMC as a pharmaceutical excipient.

Compliance with ethical standards

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Disclosure of conflict of interest

All authors declare they have no conflicting interest.

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