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# Optimizing the reaction parameters for synthesizing carboxymethyl cellulose from *Hyphaene thebaica* (Doum palm) leaves

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The current investigation focuses on optimizing reaction conditions for preparing carboxymethyl cellulose (CMC) using a new source *Hyphaene thebaica* leaves. The CMC produced under optimal conditions has been subsequently characterized by $^1$H NMR, FTIR and TGA techniques. Various solvent mixtures, including a combination of ethanol and isopropanol have been employed in the study. Additionally, different temperatures, reaction durations, and quantities of monochloroacetic acid (MCAA) have been utilized. The highest degree of substitution (DS) has been found to be 1.16. This result has been achieved by reacting MCAA with cellulose at a molecular ratio of 1:4.5 for 3 hours at 65°C using a solvent combination of (1:1). The produced CMC has an exceptionally unique physical characteristic creamy color and solubility (92%). The presence of a carbonyl group absorption signal in FTIR at 1734 cm$^{-1}$ indicates the production of CMC. In addition, the application of thermogravimetric analysis (TGA) demonstrates that carboxymethyl cellulose exhibits lower thermal stability in comparison to native cellulose.

**Keywords**: *Hyphaene thebaica*, Cellulose, CMC

The trend of utilizing materials that are based on renewable, biodegradable and environmentally friendly resources has found an increasing attention in relatively recent years[1]. Waste biomass lignocellulose is Earth's most abundant biopolymer. This material contains of carbohydrate polymers cellulose, hemicellulose, and lignin as aromatic polymer[2].

Biofuels, bioenergy, and value-added products are made from easily available and well-studied lignocellulosic biomass. Fractionating lignin, cellulose, and hemicellulose may boost process chain value[3].

Lignocellulosic biorefineries represent a series of sequential processing stages that can be executed either by a singular entity or by multiple actors[4]. This conversion technologies, including thermochemical, biochemical, and microorganism's growth, to produce biofuels, chemicals, energy, and value-added products[5]. Plant lignocellulosic polymeric compounds depend on the biomass type. Cellulose, the most prevalent organic material on Earth, has received interest because to its availability from bio mass, plant, animal, and bacterial sources[6].

Cellulose is present in many plant materials, numerous research investigations have been undertaken to explore cellulose derived from various biomass sources, alongside commercially available sources. Prakash *et al*.[7] used a cellulose extraction technique to separate 38% cellulose from a *Mentha arvensis* biomass. After isolating the cellulose, an enzymatic saccharification method was used to turn it into glucose. In their study, Chen *et al*.[8] obtained a nanostructured cellulose yield of 75.4% from the empty fruit bunch of *Elaeis guineensis* using a one-pot oxidative-hydrolysis method. Emmanuel *et al*. extracted and characterized cellulose from different date palm parts using acid-alkaline treatment[9].

Palm plants is a sustainable natural resource and a key component of biomass. Dome palm tree (*Hyphaene thebaica*) is considered one of the oldest tree species, it grows in Mauritania, Senegal, Egypt, Kenya, Tanzania, Yemen, Saudi Arabia, and Sudan in the northern portion of Africa. It is drought-tolerant and occasionally thrives on rocky hillsides[10]. The trees create a significant amount of waste, some of which is composted for arts and crafts and some of which is burnt, polluting the environment[10].

Chemical modification of cellulose using various methods esterification, alkylation, Ionic functionalization and etherification has led to amazing cellulose derivatives with regard to properties and applications[11]. Carboxymethylation of cellulose from diverse biomass resources sugarcane bagasse, sago waste, banana stem, bean hulls and microorganism (*Acetobacter xylinum*) has been thoroughly researched and reported[12–14].

CMC is the most significant ionic cellulose ether and is employed in various areas because it is nontoxic, biodegradable, biocompatible, and water-soluble. Uses in these fields include building materials, cleaning agents, oils, foods, medications, and composites[15]. In the industrial sector, the production of CMC/NaCMC involves several methods, such as the fluidized bed technique, sheet carboxymethylation, rotating drum technique, solvent-less method utilizing a double screw press, and paddle reactor[16].

Commercial production of CMC involves the reaction between cellulose and sodium monochloroacetate in a system consisting of alcohol, water, and sodium hydroxide (NaOH)[17].

Origin of the cellulose source, cellulose purification methods, and carboxymethylation reaction conditions all had a significant role in shaping the characteristics of the resulting CMC products.

Several studies have been conducted to investigate the reaction parameters on CMC properties such as DS, water solubility, viscosity and purity. Wasupon *et al.*[18] produce CMC from office paper waste in alcoholic medium in 30 minutes utilizing an ultrasonic-assisted technique as a source of energy. Saputra *et al.*[19] found that adding NaOH and SCA gradually while swirling at high speeds optimizes the main reaction, reducing by products such sodium glycolate and reagent misuse.

Various researchers have made previous attempts to enhance the DS of CMC through the utilization of diverse methodologies. Regardless of the fact theoretically that the largest possible DS value is 3. Holtzapple *et al.*[20] suggest that for commercially available ones, this figure may rise to 1.4. Insoluble CMC had a DS of less than 0.4, and it was shown that DS increases considerably with both temperature and time, however the effects of time were more pronounced.

Pushpamalar *et al.*[14] employed grinding techniques to extract cellulose from sago waste and optimized CMC with different reaction parameters, the optimized product has DS: 0.821 when subjected to pure isopropyl alcohol, a reaction time of 180 minutes, a sodium monochloroacetate quantity of 6.0 grams, a NaOH of 25%, and a temperature of 45°C.

This study presents the process of purifying (*via* pulping and bleaching method) and characterizing cellulose derived from *Hyphaene thebaica* leaves, which serves as a novel cellulose source. Additionally, the conversion of this cellulose into carboxymethyl cellulose (CMC) is investigated under optimal reaction conditions and characterized.

## Materials and methods

### Sampling and preliminary processing

1.2 kg of dried Doum palm (Hyphaene thebaica) leaves were harvested. Khartoum's National Centre for Research's Medicinal and Aromatic Plants Research Institute verified the sample. After 24–36 hours in tap water, the leaves were sliced into small pieces and grinding to 500 µg. Preserved air-dried material was processed.

### Chemicals

All chemicals were analytical grade obtained from CDH, India except Sodium hypochlorite 5% (Clorox) was purchased from local market.

### Doum palm (Hyphaene thebaica) leaf chemical composition

### Extractible contents

Chemical composition was done using modified method[21]. The Soxhlet extraction device received a 5 g air-dried leaves sample weighed in an extraction thimble. The material was extracted using an isopropanol-xylene solvent for five hours. Then was dried at 60 °C until its weight remained constant after being extracted, and then washed with hot water and ethanol. The percentage of extractable was calculated and each sample underwent the same treatment.

### Lignin content

A flask contained two grams of the extracted material and 20 mL of 70% sulphuric acid. 220 mL of distilled water was added after 2.5 hours of agitation at 25°C.Cooked for two hours, they cooled the concoction down. After 24 hours, the acid was washed out of the lignin by filtering and rinsing it with hot water. Collecting, drying, chilling, and weighing lignin. Repeated drying and weighing reached a uniform weight.

### Holocelluloses content

3 g of air-dried leaf fiber were added in an Erlenmeyer flask containing 200 mL distilled water

then 3 g of sodium chloride was introduced to the mixture followed by 2 mL of anhydrous acetic acid. After an hour in a water bath at 75°C. Twice-hourly acetic acid and sodium chloride additions. An ice bath cooled the flask below 10°C. After filtering and washing with acetone, the holocellulose sample was dried and weighed.

### α-Cellulose content

A volume of 12 mL of a sodium hydroxide solution with a concentration of 18% was introduced into a sample of 2 g of holocellulose. After adding sodium hydroxide solution every five minutes for 30 minutes at 20 °C, a volume of 30 mL of distilled water was introduced and left one hour. The residue of holocellulose was filtered and subsequently transferred to a crucible. It was then subjected to a series of washes using 250 mL of distilled water, followed by 150 mL of sodium hydroxide solution (8%), 20 mL of acetic acid solution (10%) and finally 200 mL water. The -cellulose final product was desiccated and weighed.

### Hemicellulose content

Subtracting the amount of Holocellulose from the amount of **α** -celluloses yields the concentration of hemicellulose in the fiber will determine.

### Purification of cellulose

Modified method[22] was used to purify cellulose. The standard experiment included soaking 350 g of dried leaves in distilled water in a 3L beaker for 30 minutes before decanting. Cooking wet fibers in a 1:10 (w/v) solution of 4% NaOH at 80 degrees Celsius for 4 hours. Supernatant was washed till it became a very light yellow. The filtered slurry was bleached twice for 1 hour at 60°C with a 1.7% (w/v) Clorox solution until it became white. The bleached cellulose was washed with ethanol and dried in an oven at 60°C after being rinsed with distilled water until the iodine test revealed no hypochlorite odor.

### Optimization of carboxymethylation reaction

Different reaction conditions were used for the carboxymethylation process using modified methods[22]; these settings are detailed below.

### Influence of solvent ratio

In a ratio of 1:1, 60 mL of ethanol to isopropanol was added to 1.5 grams of separated cellulose. Thirty minutes were spent slowly adding 15 cc of 40% aqueous NaOH. At RT, stirring took 1 h. Alkalinized cellulose was treated with 3 g of MCAA diluted in alcohol mixture 15 mL. Two hours of refluxing at 55 degrees Celsius followed an hour at RT. Following the neutralization of NaOH with glacial acetic acid, the resultant solution underwent filtration and subsequent multiple washes with ethanol in order to eliminate any residual impurities before being dried at 60°C in an oven, using the identical procedures as in method[22] with a little tweak. For more purification to remove any impurities, dried CMC was dissolved in 50 mL of ethanol 95% 20 mL of 1 M nitric acid and mixed by stirrer for two minutes. After that, we cooked the liquid for 5 minutes while stirring for 15, then let it cool. The solution was centrifuged, and the supernatant was discarded. In order to eliminate acid and salts, the product underwent twice washes using 70 mL of 95% ethanol, followed by a single wash using heated 80% ethanol. In a beaker, the precipitate was heated until the alcohol evaporated and rinsed with methanol. Three hours in a 105°C oven dried the precipitate.

Changing the solvent ratio Isopropanol: Ethanol in two more trials (sample2 = 1:2 and sample 3=2:1) and kept all parameters unchanged (Temperature 55 ºC, MCAA 3g, and time 2h). Each cases DS was calculated, and the best solvent ratio was utilized for the subsequent studies.

### Influence of temperature

The effect of temperature on carboxymethylation occurred at (sample 4 at 55, sample 5 at 65, and sample 6 at 75°C). The solvent ratio was retained at 1:1 based on DS findings from varied solvent ratios. And other parameters were held constant (MCAA 3g and Time 2h).

### Influence of reaction time

Two, three, and four-hour carboxymethylation reactions were performed for sample (7,8 and 9). DS results determined a 1:1 solvent ratio, 65°C as the optimal temperature and the amount of MCAA was kept 3g.

### Influence of MCAA concentration

The carboxymethylation process was done at various MCAA doses (sample 10= 3g, sample 11= 4.5g and sample 12= 6g). Based on the DS all parameters were kept at optimum condition (solvent ratio 1:1, temperature 65°C, and time 3h).

### Determination the degree of substitution (DS)

The ashing process of each product's 0.5 g carboxymethyl cellulose was conducted at a temperature of 700°C for a duration of 20 to 25 minutes. 42 mL of boiling deionized water was used to dissolve the ash sample and subsequently subjected to titration using 0.1 N H2SO4 until a *p*H of 4.4 was reached. In order to remove carbon dioxide, the solution underwent a process of triple boiling prior to each titration. The DS was determined by employing titrated acid (b/mL) and CMC (G/g) in the following equation[23].

$$\mathrm{DS}=\left\{0.162\left(\frac{0.1\mathrm{b}}{\mathrm{G}}\right)\right\} \div \left\{1-0.08\left(\frac{0.01\mathrm{b}}{\mathrm{G}}\right)\right\}$$

### Fourier transform infrared spectroscopy

The Thermo Scientific Nicolet 6700 Fourier Transform Infrared (FTIR) spectrometer, was employed in conjunction with the diamond Smart Orbit Attenuated Total Reflectance (ATR) sampling attachment, to characterizing cellulose and carboxymethyl cellulose. The materials were analyzed with a resolution of 4 $\mathrm{cm}^{-1}$ and spectrum ranged from 4000 to 400 $\mathrm{cm}^{-1}$.

### Nuclear magnetic resonance $^1$H NMR

The 1H NMR tests were carried out at temperatures ranging from –80°C to +130°C. The experiments utilized an Agilent Premium Compact NMR apparatus (DD2 400 MHz, 5 mm) and 300 W Broadband Linear Amplifiers equipped with a 1H/19F, X (15N-31P) and lock PFG probe. The spectra were analyzed using VNMRJ 3.2A. The samples were dissolved using dimethyl sulfoxide.

### Thermogravimetric analysis

The thermal stability of cellulose and carboxymethyl cellulose (CMC) was investigated using the Mettler Toledo® TGA/DSC 1 STARe System. This system enabled the monitoring of heat-induced mass loss from 25 to 1000 °C. In this investigation, a quantity of material weighing 3.146 mg was subjected to heating at a rate of 10 °C/min. The gas Controller GC10 was employed for this purpose.

### Viscosity measurements

For viscosity measurements, 1% CMC aqueous solution with DS 1.16 was created. The studies used Brookfield DV3T. The sample cup received 0.5 mL of CMC solution and cone spindle cp52. Shear rate was 10–200 $\mathrm{S}^{-1}$ and temperature was 25°C.

### Solubility test

A solution containing 1 g of carboxymethyl cellulose (CMC) was prepared by dissolving it in 15 mL of distilled water. The solution was then subjected to agitation for a duration of 6 hours. Subsequently, the solution was transferred to a centrifuge tube and subjected to centrifugation at a speed of 5000 rpm. The resultant solution was decanted and weighed following the process of drying, in order to determine its solubility.

## Results and Discussion

### *Hyphaene thebaica* (Doum palm) leaf chemical composition

A thorough study was undertaken to investigate the chemical constituents present in the leaves of *Hyphaene thebaica*. The findings of this investigation are outlined in Table 1, provided below.

According to the results in a Table 1, hollocellulose accounts for 75% of the fiber. Organic solvent-soluble components make up 9.05% of the fiber. Hyphaene thebaica leaves have a similar chemical composition to ficus leaf fibers (α-cellulose 38.1%, Hemicellulose 30.5%, Lignin, 23.4%)[24], corn stalk (α –cellulose 39%, Hemicellulose 42%, Lignin 7.3%), and napier grass (Holocelluloses 82.4%, α –cellulose 12.4%, Hemicellulose 68.2%, Lignin 10.8%)[25]. One of the most superior types of fibers is sisal (Hemicellulose, 10-14%, α -cellulose, 66-78%, Lignin, 10-14%,)[26] have higher cellulose percentages and lower hemicellulose and lignin percentages, than the present work.

### Carboxymethylation of Cellulose

Optimizing solvent combination, reaction duration, temperature, and cellulose/MCAA mole ratio maximized the carboxymethylation of *Hyphaene thebaica* leaf cellulose. The DS values at various carboxymethylation reaction parameters are illustrated in Fig. 1.

### Influence of solvent ratio on carboxymethylation

It was investigated how the solvent-to-substituent ratio affected the degree of substitution using the

Table 1 — The chemical composition of *Hyphaene thebaica*

| Constituents | Percentage ± SD* % |
|---|---|
| Soluble organic | 9.03±1.33 |
| Liginin | 26.6±2.47 |
| Holocelluloses* | 74.32±0.65 |
| α-cellulose | 35.5±3.83 |
| Hemicellulose | 41.9±3.14 |

*Holocelluloses = α-cellulose + Hemicellulose
*SD: standard deviation

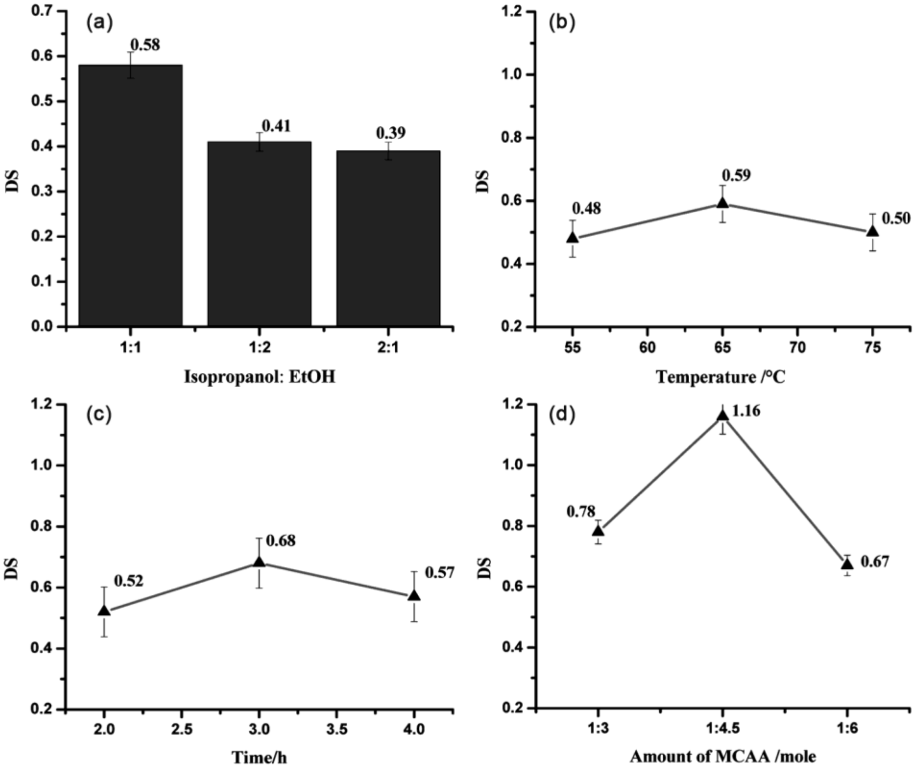

Fig. 1 — Influence of (A) solvent ratio (B) temperature (C) reaction time and (D) amount of MCAA on carboxymethylation reaction.

experimental parameters outlined in Fig. 1(A). These parameters included a reaction temperature of 55°C, a reaction duration of 2 hours, and 1:3 ratio of cellulose and MCAA. Yielded the highest DS value (0.58) when the alcoholic ratio of was 1:1. In carboxymethylation process, the primary function is to augment the solubility and availability of the etherifying substance along the cellulose chain and reaction sites, as a consequence, it is considered to improve CMC while inhibiting glycolate production. Different alcohol mixtures (ethanol and isopropanol) have different degrees of carboxymethylation, which may be seen by comparing the polarity and stereochemistry of those mixtures. The adequacy of the reaction decreases as the polarity of the solvent increases[27]. According to the DS data, it was determined that the optimal choice was a blend of isopropyl alcohol and ethanol. This is consistent with earlier study reports[28,29]. Based on the results above, the solvent mixture of 1:1 was chosen for the subsequent carboxymethylation trials since it produced the greatest DS value.

## Influence of temperature on carboxymethylation

Fig. 1(B) depicts the variation in DS as a function of reaction temperature. The chart clearly shows that as the temperature rises, the DS initially climbs before falling from 0.48 to 0.59 and 0.50 at 55°C, 65°C, and 75°C, respectively. The initial appreciation of the DS's value can be ascribed to the inclination towards carboxymethylation production *via* the glycolate process. However, the predilection for glycolate production, which was well demonstrated during experimental work, is what causes the drop in DS with additional temperature increase. Similar results were observed as well[30].

Based on the results, the temperature of 65 °C produced the greatest DS value, which was adopted in the subsequent carboxymethylation studies.

## Influence of reaction time on carboxymethylation

Fig. 1(C) illustrates the relationship between DS and reaction time at the optimal temperature and optimal solvent ratio. According to the data presented an increase in DS was observed as reaction time increased up until the three-hour, after which it subsequently decreased. The observed reduction in degree of substitution as reaction time rises might be related to glycolate production rather than carboxymethylation. The deceleration of the reaction rate can be attributed to the gel formation phenomenon, which hinders the penetration of the etherifying agent into the cellulose substrate. Additionally, the increase in viscosity of the medium poses challenges to the stirring process. These observations align with the findings previously documented in the literature[31].

### Influence of MCAA on carboxymethylation

The MCAA dose was changed to see how it affected the DS value. The alcohol solvent ratio was 1:1, the reaction temperature was 65°C, and the reaction duration was 3 hours. Fig. 1(D) depicts the relationship between DS and MCAA concentration. As seen in the graph, the overall tendency is a rise with rising MCAA levels, followed by a quick fall with increasing MCAA quantities. The presence of MCAA in close proximity to cellulose molecules may be responsible for the initial rise, which drives the creation of carboxymethylated compounds. The decrease in DS value that has been observed may be explained by the formation of glycolate due to an excessive presence of MCAA in close proximity to cellulose molecules. This explanation aligns with prior research findings[29].

### Physical properties of CMC

*Hyphaene thebaica* leaf cellulose was transformed to CMC through monochloroacetic acid based alkaline carboxymethylation. Table 2 show the conversion %, color and solubility, and in Fig. 2 the viscosity of CMC with DS 1.16.

Using Brookfield viscometer, the rheological properties of 1% CMC solution were measured at RT and the shear rate range was 10~200 $s^{-1}$. Fig. 2 plots viscosity as a function of shear rate diverse flow behaviors is detected. The viscosity decreases when the shear rate increases similar results were obtained[21], these exhibit shear-thinning behaviors of CMC solution.

### FTIR spectroscopic analysis

FTIR spectrum of isolated cellulose and prepared CMC was depicted Fig. 3. The vibrational frequency associated with the stretching of the hydroxyl (-OH) functional group, along with the occurrence of intramolecular and intermolecular hydrogen bonding, obviously generate a wide absorption band observed at a wavenumber of 3430 $cm^{-1}$. C-H and $CH_2$ stretching vibrations cause the band at 2898 $cm^{-1}$ similar tendency of cellulose reported[32]. According to reported data[33], the bands at 1431-1323 $cm^{-1}$ are caused by $-CH_2$ and CH bending vibrations, whereas the bands observed at wavenumbers 1060 $cm^{-1}$ and 1166 $cm^{-1}$ can be attributed to the vibrations of C-O and C-O-C groups, respectively. Finally, out-of-plane C-OH bending causes the band at 602 $cm^{-1}$.[14]

As shown in Fig. 3, the CMC product displayed totally typical absorption bands, with the development of a well-defined band at 1734 $cm^{-1}$ made by stretching vibration of the carbonyl group (C=O) of the carboxymethyl substituent being the only distinctive characteristic[33]. The hydroxyl (OH) group's peak at 3483 $cm^{-1}$ is brighter than cellulose's. The hydroxyl-carbonyl interaction increases intensity. Othman *et al*. found similar results[34]. Furthermore, the

Table 2 — Physical properties of prepared CMC

| Parameter | CMC /DS 1.16 |
|---|---|
| Conversion (%) | 66% |
| Color | Creamy |
| Solubility at 25°C | 92% |
| Viscosity (1% w/v, 25°C, shear rate 20 $S^{-1}$) | 26 (CP) |

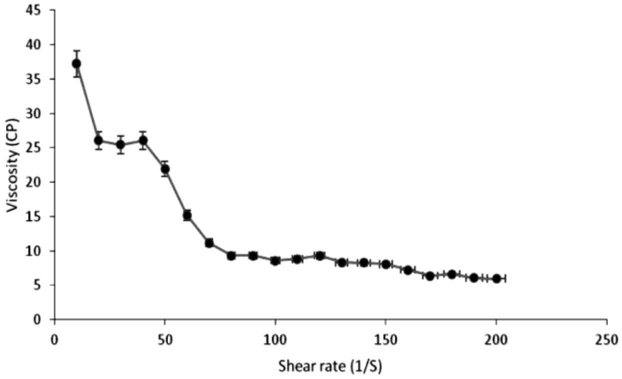

Fig. 2 — Viscosity as a function of the shear rate for 1% CMC solution.

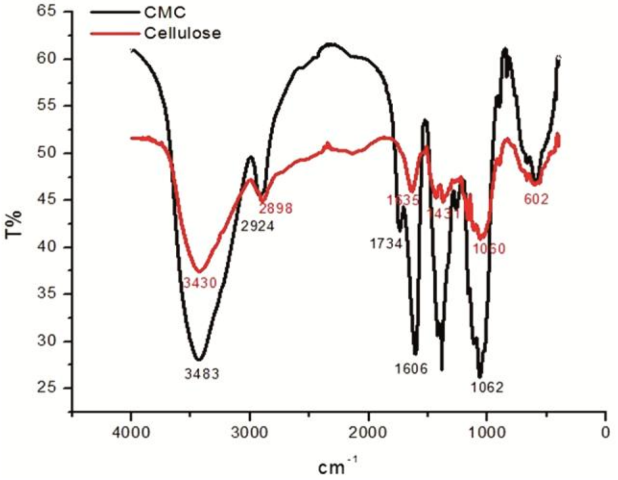

Fig. 3 — The FT-IR spectra of isolated cellulose and optimized CMC.

area between 1200 cm$^{-1}$ and 1000 cm$^{-1}$ becomes stronger and more distinct, which might be attributable to C-O-C ether stretching vibrations induced by the carboxymethyl substituent.[14]

### Nuclear magnetic resonance analysis

The findings of highly resolved $^1$H NMR spectra of cellulose and CMC spectrum of the samples exhibits all the distinctive attributes that have been documented Bisht *et al.*[35] Fig. 4 observed the proton positions of the glucose unit in cellulose are depicted with chemical shifts of 4.65, 3.74, 3.75, 3.76, 3.77, and 3.04 ppm assigned to H1, H2, H3, H5, H4, and H6, respectively. Furthermore, the protons of hydroxyl group in anhydrous glucose units(AGU) signals were shown at 5.40, 4.49, and 4.29 ppm[35]. DMSO and $H_2O$ are responsible for the significant peaks at 2.5 and 3.1-3.4 similar reported with Kono *et al.*[36] The observed disparities in chemical shifts ascribed to the heterogeneous electronic surroundings encompassing the carbon atoms in AGU, which emanate from their interactions with atoms that either donate or withdraw electrons, such as carbon or oxygen atoms.

Fig. 5 displays the $^1$H NMR spectra of CMC. The spectral analysis reveals that the solvent DMSO exhibits peaks within the range of 2.4-2.6 ppm, the maximum concentration of water is observed at 3.355 ppm[36]. Although the H NMR spectra was not recorded with great clarity, the signals acquired in the ranges of 3.4-3.6 and 4.6 are the close ranges of CMC corresponding to AGU and acetate group as previously assigned by Kono *et al.*[37] experienced a reduction in intensity as a result of the strong absorption peaks exhibited by DMSO and water. As a result, these peaks were observed to manifest either as shoulder peaks or to be entirely absent in specific cases.

### Thermogravimetric analysis

In order to evaluate the thermal stability of the purified cellulose and the synthesized carboxymethyl cellulose (CMC) with a degree of substitution (DS) of 1.16, Thermogravimetric and differential thermal analysis (TG/DTG/DTA) were conducted. As depicted in Fig. 6, the TG/DTG and DTA of cellulose

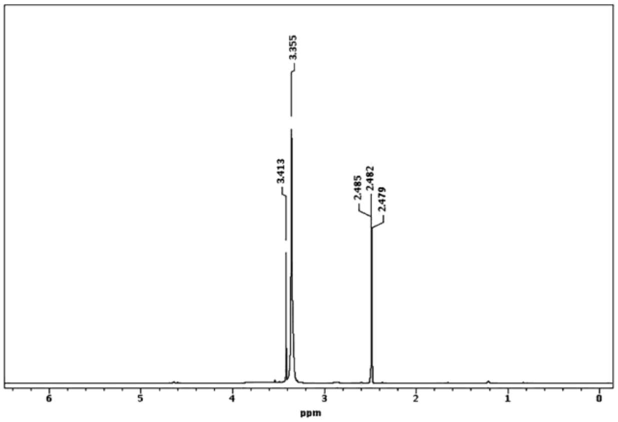

Fig. 5 — $^1$H NMR of synthesized CMC.

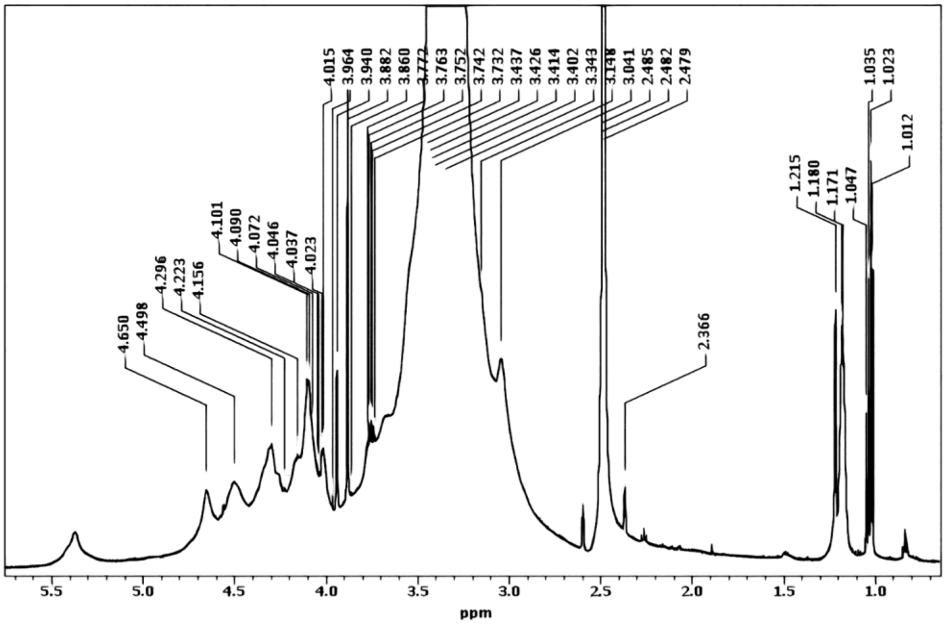

Fig. 4 — $^1$H NMR of isolated cellulose.

curves exhibit two distinct stages of mass reduction. The initial occurrence transpired at temperatures below 100°C, potentially attributable to the phenomenon of moisture evaporation. Currently, the observed mass reduction stands at 6%. On the other hand, the initiation of the second phase of degradation begins at approximately 200°C and reaches its maximum at 340°C, whereas the termination occurs at approximately 400°C. During the pinnacle of the stage, there is an approximate loss of 52% in mass.

The second instance of cellulose degradation can potentially be ascribed to the cleavage of glycosidic bonds, as well as the pyrolysis. The application of temperatures ranging from 400°C to 1000°C to the sample resulted in a decrease in mass of approximately 91%. The degradation of cellulose at elevated temperatures can be attributed to the additional breakdown of cellulose molecules and the formation of char, the above mention results consistent with El-Sayed *et al*., Nada *et al*.[24,25]

The thermal stability of carboxymethyl cellulose (CMC) was evaluated. According to the data presented in Fig. 7 which is similar finding with Rani *et al*.[38] It can be observed that there is no statistically significant reduction in sample weight until reaching a temperature of 200°C. Visible degradation initiates at temperatures exceeding 200°C specifically around 220°C and persists until approximately 330°C, with the temperature at which degradation is most pronounced being 280°C. The observed reduction in

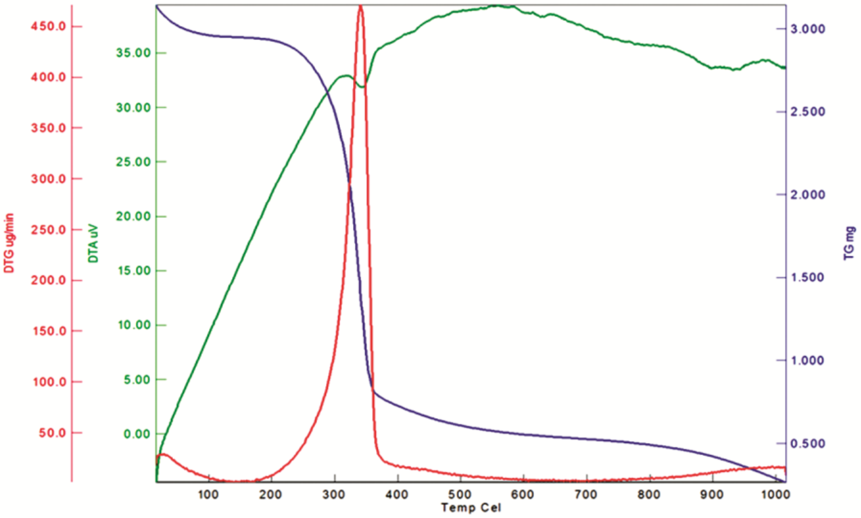

Fig. 6 — TG/DTG and DTA curves of pure isolated cellulose.

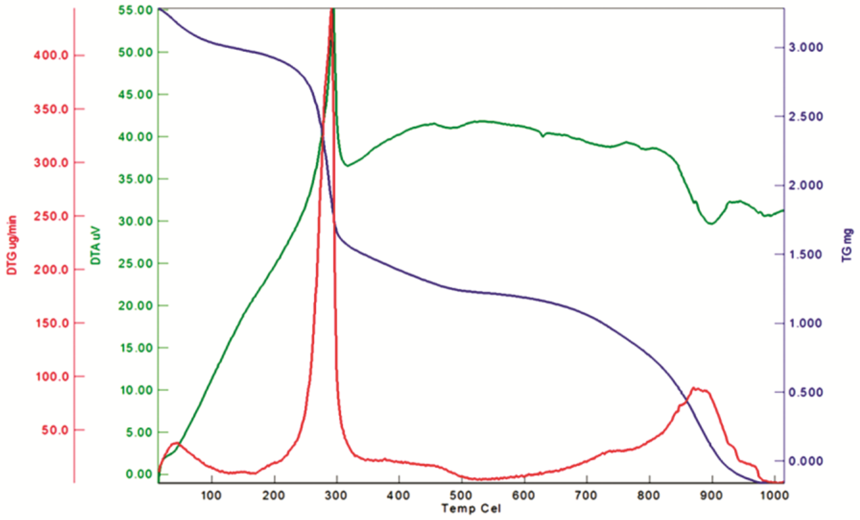

Fig. 7 — TG/DTG and DTA curves of pure synthesized CMC.

mass was approximately 32%. The samples exhibit a gradual degradation above 400°C, with the rate of degradation increasing significantly as the temperature approaches approximately 650°C. Subsequently, the degradation reaches its peak at around 880°C. At this juncture, the specimen had undergone complete disintegration, resulting in an almost negligible residual mass.

## Conclusion

As a novel source of cellulose, Doum palm (*Hyphaene thebaica*) leaves were utilized in this research project to produce carboxymethyl cellulose (CMC) with best degree of substitution (1.16) using just mild processing conditions. The solvent ratio used as equal parts of ethanol (ETOH) and isopropanol. The reaction was conducted at a temperature of 65°C for a duration of 3 hours. The quantity of monochloroacetic acid (MCAA) used in relation to cellulose was in a ratio of 1:4.5. Those parameters were found to produce the best results from the carboxymethylation process. It was discovered that the CMC that was created had great rheological characteristics in addition to having a strong thermal stability and a high degree of solubility in water.

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