

## OPTIMIZED SYNTHESIS OF CARBOXYMETHYL CELLULOSE FROM MICROCRYSTALLINE CELLULOSE AND NANOCELLULOSE VIA SUSPENSION METHOD

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### Abstract

Carboxymethyl cellulose (CMC) is a versatile polysaccharide derivative with widespread applications in various industries, including food, pharmaceuticals, and cosmetics. In this study, we present a novel synthesis method for CMC utilizing a suspension approach based on microcrystalline cellulose (MCC) and nanocellulose (NC). The suspension method offers several advantages, including simplified processing, reduced reaction times, and improved product uniformity compared to traditional methods.

The synthesis process involves the conversion of MCC and NC into CMC through carboxymethylation reactions mediated by sodium hydroxide and monochloroacetic acid. We investigate the influence of reaction parameters, such as reaction time, temperature, and reactant concentrations, on the degree of substitution (DS). Characterization techniques, including Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD), are employed to analyze the chemical structure and crystalline properties of the synthesized CMC. Our results demonstrate that the suspension method facilitates efficient carboxymethylation of MCC and NC, yielding CMC suspensions with tunable DS values and tailored rheological behaviors. Moreover, the incorporation of NC enhances the mechanical properties and stability of the CMC suspensions, offering potential advantages for applications requiring enhanced performance characteristics.

Overall, this study provides valuable insights into the synthesis of CMC via the suspension method using MCC and NC as precursors, highlighting its potential for the development of functional cellulose-based materials with tailored properties for various industrial applications.

**Keywords:** nanocellulose, microcrystalline cellulose, carboxymethylcellulose, degree of polymerization, degree of substitution, monochloroacetic acid.

### Introduction

Cellulose, as the most abundant biopolymer on Earth, has attracted significant attention in various fields due to its renewable nature, biodegradability, and versatile properties [1-2]

Among its derivatives, carboxymethyl cellulose (CMC) stands out as a widely utilized polymer with applications ranging from pharmaceuticals and food additives to personal care products and industrial processes. [3-4]. Carboxymethyl cellulose, a water-soluble derivative of cellulose, has garnered significant attention in various industrial applications due to its unique

# OPTIMIZED SYNTHESIS OF CARBOXYMETHYL CELLULOSE FROM MICROCRYSTALLINE CELLULOSE AND NANOCELLULOSE VIA SUSPENSION METHOD

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properties, including high viscosity, biocompatibility, and film-forming ability [5]. The synthesis of CMC from cellulose sources presents a crucial avenue for expanding its applicability and enhancing sustainability in diverse industries. The synthesis of CMC involves the carboxymethylation of cellulose, a process that modifies the hydroxyl groups of cellulose chains with carboxymethyl ( $-\text{CH}_2\text{COOH}$ ) moieties, imparting water solubility and enhancing its functionality [6]. The synthesis of CMC typically involves the reaction of cellulose with sodium chloroacetate in the presence of an alkaline catalyst, followed by subsequent purification steps to remove by-products and residual reagents. This carboxymethylation process results in the introduction of carboxymethyl groups onto the cellulose backbone, leading to the formation of CMC with tailored properties suited for specific applications.

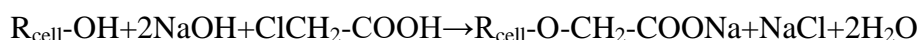
In recent years, there has been growing interest in developing efficient and environmentally friendly methods for the synthesis of CMC. Among these methods, the suspension method, leveraging microcrystalline cellulose (MCC) and nanocellulose as starting materials, has emerged as a promising approach. The suspension method offers several advantages, including facile scalability, reduced energy consumption, and the ability to tailor CMC properties through controlled reaction conditions.

Microcrystalline cellulose, derived from cellulose pulp or cotton, possesses a well-defined crystalline structure and high surface area, making it an ideal precursor for CMC synthesis [7]. Nanocellulose, comprising nanofibrils or nanoparticles with dimensions on the nanometer scale, further enhances the reactivity and functionality of the resulting CMC due to its increased surface area and accessibility of hydroxyl groups [8].

There are three ways to carry out the carboxymethylation reaction of cellulose and its derivatives: suspension (in the liquid phase or in a heterogeneous solvent environment), solid phase (without a solvent) and homogeneous (in solution). The production of CMC involves two reaction stages: mercerization and esterification by the slag method [9-11].

To obtain Na-CMC from modified cellulose forms using the suspension method, cellulose is treated with a solution of sodium hydroxide in alcohol, and monochloroacetic acid is used as an alkylating reagent [12]. CMC preparations with high solubility can be obtained by the suspension method with lower consumption of O-alkylating reagent [13], ethanol [14], propanol-2 [15-16], propanol-1, tertiary butyl alcohol, benzene, acetone, alcohols, a mixture can be used [17] and other organic solvents [18]. The esterification reaction of NC and MCC was carried out in ethanol using the well-known suspension technology [19-20].

The main step in the carboxymethylation reaction is the formation of alkali cellulose, which changes the crystalline structure of cellulose and increases the penetration of chemicals through fiber swelling [21-23].



The resulting cellulose ester has the ability to dissolve in water and form viscous solutions due to the replacement of hydrogen in the hydroxyl groups of cellulose with one group -

CH<sub>2</sub>COONa. The solubility of CMC in water depends on the degree of alkylation and DP. Drugs with the same DS and DP values can differ significantly in solubility due to the chemical heterogeneity of cellulose.

The scope of application of CMC is influenced by the molecular weight and DS of the product. Unlike other cellulose ethers, CMC is an ionic polymer and in aqueous solutions exhibits the properties of a polyelectrolyte, which determines its areas of application. In industry, Na-CMC is usually obtained in the range of DS 0.4-1.4 and DP 300-3000 [24]. CMC samples with a degree of substitution above 0.9 are called polyanionic cellulose. Conventional CMC is soluble in water if its degree of substitution exceeds 0.5 [25].

The physicochemical properties of CMC are mainly determined by the degree of substitution (DS), dispersity and degree of polymerization. DS represents the number of carboxymethyl groups per molecular unit of anhydroglucose units. Basically, all hydroxyl groups (OH-2, OH-3 and OH-6) in the anhydroglucose unit can be substituted, and the maximum degree of substitution (DS) is 3 [26]. Purified CMC is a white powdery substance without taste or odor [27-28].

In this article, we present a comprehensive study on the synthesis of carboxymethyl cellulose via the suspension method utilizing microcrystalline cellulose and nanocellulose as starting materials. We explore the influence of reaction parameters such as reaction time, temperature, and concentration of reagents on the yield, degree of substitution of the synthesized CMC. Furthermore, we investigate the structural characteristics of the obtained CMC products through analytical techniques such as Fourier-transform infrared spectroscopy (FTIR), and X-ray diffraction (XRD).

Through systematic experimentation and analysis, we aim to elucidate the key factors governing the synthesis of CMC via the suspension method and provide insights into optimizing reaction conditions for obtaining CMC with tailored properties suitable for specific applications.

### **Materials and methods**

NC-1, NC-2 and MCC were used as raw materials for synthesis of CMC. NC-1 was prepared by the hydrolysis of cotton cellulose in aqueous solution of H<sub>2</sub>SO<sub>4</sub>: acid concentration 61%, temperature 40°C, modulus 1:10, reaction duration 90 minutes.

NC-2 was prepared by the hydrolysis of cotton cellulose in a 66% aqueous solution of H<sub>2</sub>SO<sub>4</sub>: acid concentration 66%, temperature 25-30°C, modulus 1:10, reaction duration 60 minutes [29].

MCC was extracted by the hydrolysis of cotton cellulose in aqueous solution of HNO<sub>3</sub>: acid concentration 4%, temperature 100-110°C, module 1:10, duration 60 minutes [30].

To synthesize Na-CMC, 10 g of NC (or MCC) are added to 75 ml of ethyl alcohol and stirred for 15 minutes. Then variable volumes of NaOH solution are added in this suspension. Alkaline treatment was carried out for 120 min at 40°C for NC-1; for 120 min at 25°C for NC-2; for 120 min at 40°C for MCC. After, a solution of monochloroacetic acid (MCAA) dissolved in 50 ml of ethyl alcohol was added with stirring, and the solution was kept at 25°C for 15 minutes. After this, the reaction temperature was increased to 70°C for NC-1, 55°C for NC-2, 70°C for MCC and the esterification reaction was carried out for 120 minutes. The product is

# OPTIMIZED SYNTHESIS OF CARBOXYMETHYL CELLULOSE FROM MICROCRYSTALLINE CELLULOSE AND NANOCELLULOSE VIA SUSPENSION METHOD

---

filtered and washed with 70% aqueous solution of ethanol. Na-CMC was dried at a temperature of 60–70°C to a certain humidity.

Infrared (IR) spectroscopic studies were carried out on an Inventio-S IR Fure spectrometer (Bruker, Germany) in the wavelength range from 500 cm<sup>-1</sup> to 4000 cm<sup>-1</sup>.

XRD studies were carried out using an XRD Miniflex 600 (Rigaku, Japan) with monochromatic CuK $\alpha$  radiation isolated by a nickel filter with a wavelength of 1.5418 Å at 40 kV and the current strength of 15 mA. The spectrum was recorded in the interval  $2\theta = 5^\circ\text{--}40^\circ$ . The data processing of experimental diffraction patterns, peak deconvolution, describing the peaks used by Miller indices, peak shape, and the basis for the amorphous contribution were conducted using the software “SmartLab Studio II” and data base PDF-2 (2020 Powder diffraction file, ICDD).

To determine the DS, 0.7–1.5 g of Na-CMC mass is taken and dissolved in 100 ml of distilled water. After the complete dissolution of CMC, add 20 ml of 94% ethyl alcohol. Then, add 0.5 mol/l sulfuric acid solution dropwise to the solution until the pH reaches 2.2–2.4. Next, add 25 ml of 0.1 mol/l copper sulfate solution to the resulting solution and slowly add a 5% ammonia solution until the pH reaches 4–4.1. The solution should be filtered through two layers of filter paper. Wash the filtered Cu-CMC three times with 100 ml of 70% ethyl alcohol solution and two times with 50 ml of 94% ethyl alcohol solution. Finally, dry the resulting Cu-CMC at a temperature of 105°C for 2 hours until a constant weight is achieved.

The mass of the dried salt is determined, and then it is dissolved in 100 mL of distilled water. 10 mL of a 5% ammonia solution is added to the solution and stirred until completely dissolved. Slowly, a 6 mol/L acetic acid solution is added drop by drop until the green colour turns blue. After the colour changes, add 5 mL of acetic acid solution. Then, 15 g of KI salt is added to the solution and placed in a dark place for 10 minutes. The solution is titrated with 0.1 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution. When the colour of the solution becomes completely white, the volume of titrant solution consumed is measured and calculated using the following formula.

$$\gamma = \frac{162 \cdot X_1}{31,77 - 0,888 \cdot X_1} \cdot 100$$

Here, 162- the molecular weight of the cellulose unit, g,

31.77- equivalent molar mass of copper reacting with carboxyl group, g

The mass fraction of copper in Cu-CMC is found using the following formula.

$$X_1 = \frac{V \cdot 0.006357}{M \cdot 100}$$

V- volume of sodium thiosulfate consumed for titration, ml

m - mass of Cu-CMC, g

0.006357- mass of copper contained in 1 ml of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

### Results and discussion

To synthesize Na-CMC using the suspension method, cellulose is treated with a solution of sodium hydroxide in alcohol, and monochloroacetic acid is used as an alkylating reagent. By the suspension method, Na-CMC is synthesized with a high DS, DP, and solubility are synthesized.

The gradual destruction of various levels of the morphological structure and supramolecular organization of natural cellulose during its transformation into MCC and further into NC has a significant impact on certain chemical transformations. On the one hand, the substantial increase in the surface area of cellulose during its transformation to MCC and NC can have a decisive influence. On the other hand, in the processes of cellulose destruction, primarily hydrolytic, an area with an amorphous and mesomorphic structure is removed, which undergoes chemical transformations.

**Table 1**

**The characteristics of CMC samples**

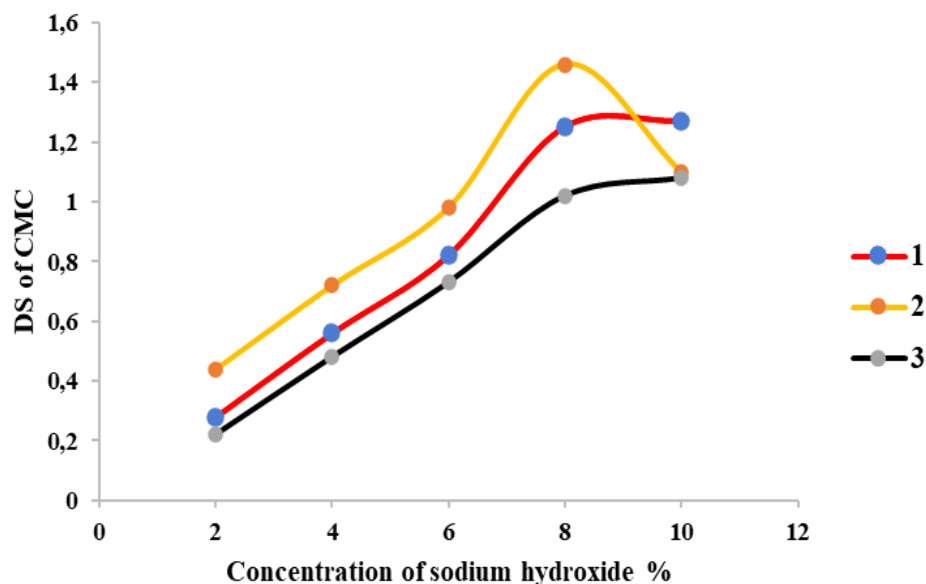
№	Sample	DP	Na-CMC		
			DP	DS	solubility, %
1	NC-1	178	120	1.25	100
2	NC-2	108	78	1.46	100
3	MCC	367	275	0,92	100

The reactivity of cellulose and its derivatives in heterogeneous processes is determined, first of all, by the content and nature of functional groups, as well as their surface area. From this point of view, cellulose can be considered as a polymeric alcohol, the elementary units of the macromolecule which contain three hydroxyl groups. It is the individual properties of the hydroxyl groups that, as a result of their chemical transformations, allow the synthesis of cellulose ethers and esters.

As can be seen from Table 1, the high reactivity of nanocellulose compared to microcrystalline is observed during carboxymethylation reactions. Comparative studies have shown that, under the same carboxymethylation conditions, the quality of the resulting carboxymethylated cellulose samples has a linear dependence on the nature of the feedstock. The degree of substitution increases with the transition from micro to nanosized cellulose particles. Shortened cellulosic particles have increased surface area, which increases the chance of contact between reactants and cellulose particles, thus increase the reaction rate, and ultimately increases the DS value.

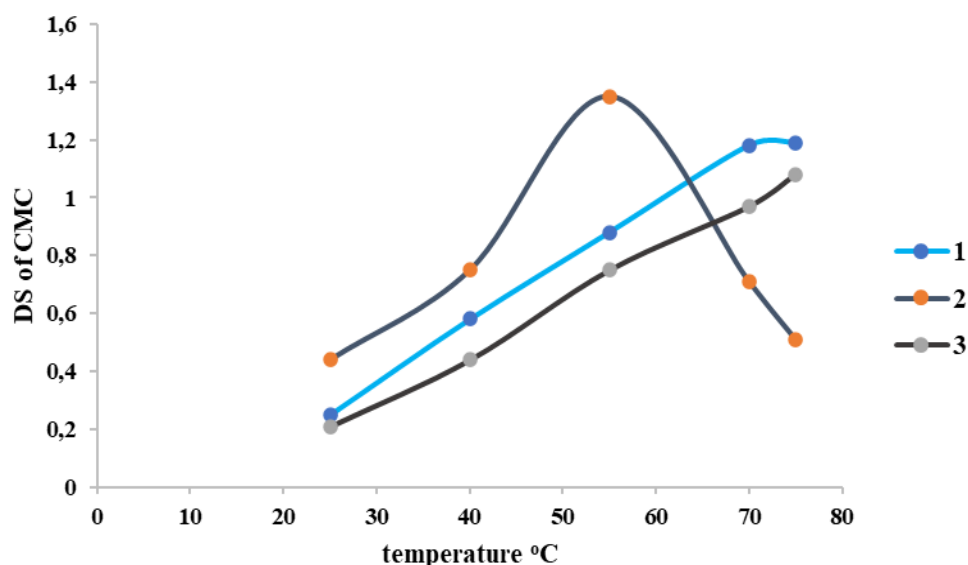
The influence of the carboxymethylation reaction conditions on NC-1, NC-2, and MCC, as well as their impact on the physicochemical parameters of the resulting Na-CMC samples, was studied. The optimal conditions for obtaining water-soluble Na-CMC samples from NC-1, NC-2 and MCC in ethanol were also determined.

# OPTIMIZED SYNTHESIS OF CARBOXYMETHYL CELLULOSE FROM MICROCRYSTALLINE CELLULOSE AND NANOCCELLULOSE VIA SUSPENSION METHOD



**Figure 1.** Dependence of the DS of Na-CMC on sodium hydroxide concentration. Na-CMC obtained from NC-2 (1), NC-1 (1), and MCC(3). Alkaline treatment time: 30 min, Etherification time: 60-90 min.

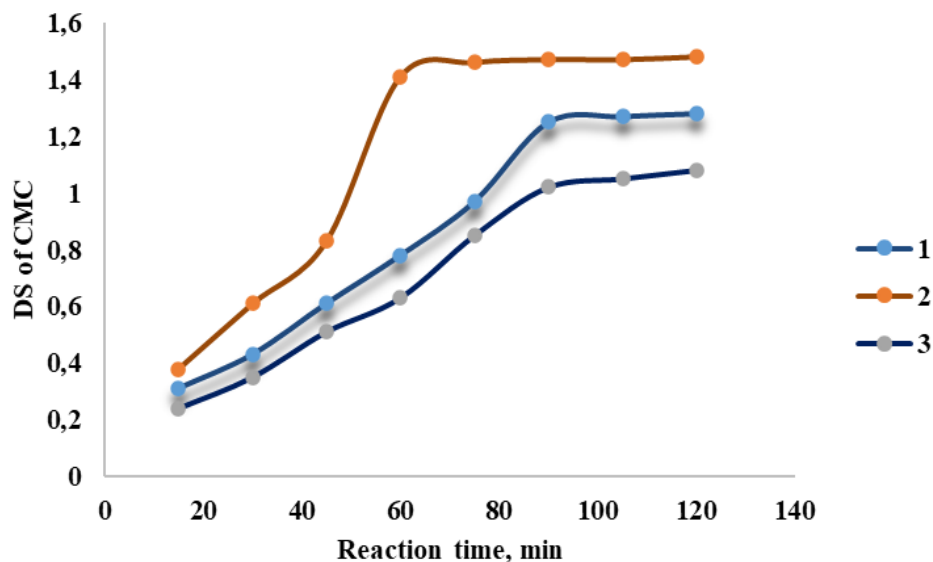
An increase in the DS of Na-CMC was observed as the concentration of sodium hydroxide increased. However, if the alkali concentration exceeds 8%, the DS of Na-CMC decreases. This decrease indicates an increase in the rate of hydrolysis and side reactions of MCAA.



**Figure 2.** Dependence of reaction temperature on the degree of substitution of Na-CMC. (Na-CMC obtained from 1-NC-2, 2-NC-1, 3-MCC)

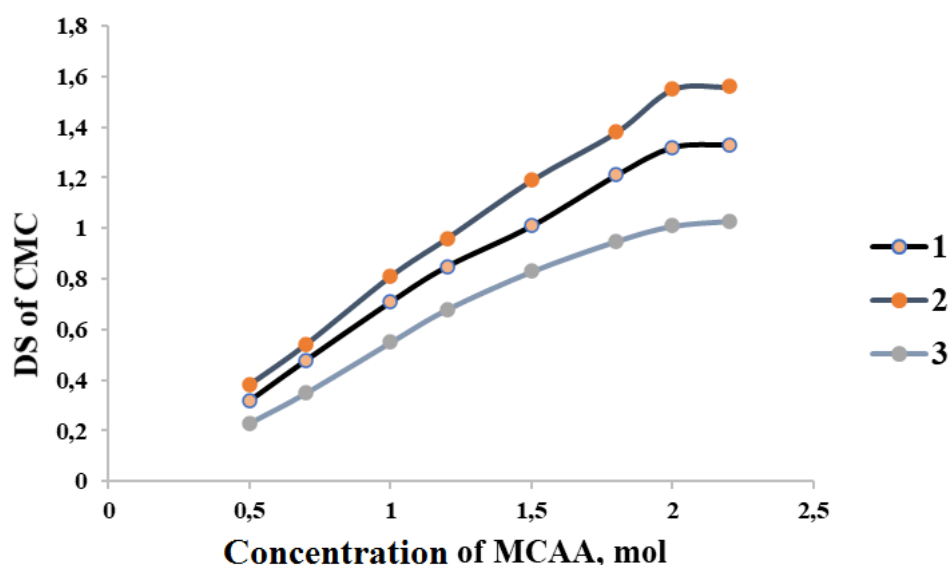
When the carboxymethylation reaction is conducted at a temperature of 70°C, the degree of substitution (DS) of Na-CMC obtained from NC-1 and MCC becomes an important fac-

tor. It is clear that the exchange rate remains consistent when introducing reagents during the intermediate reaction. However, it has been observed that the yield of Na-CMC decreases when the carboxymethylation reaction is performed at temperatures exceeding 55°C in NC-2. This decrease in yield is attributed to a depolymerization process triggered by the increase in temperature, as NC-2 is in an amorphous state.



**Figure 3.** Dependence of the DS of Na-CMC on the duration of the carboxymethylation reaction: Na-CMC obtained from NC-2 (1), NC-1 (2), MCC(3)

The study focused on examining the impact of time on the DS of Na-CMC obtained using the suspension method. In the beginning, the reaction rate was high, and the degree of substitution of Na-CMC reached its highest value for NC-2 after 60 minutes, while for NC-1 and MCC, it took 90 minutes to reach the maximum value.



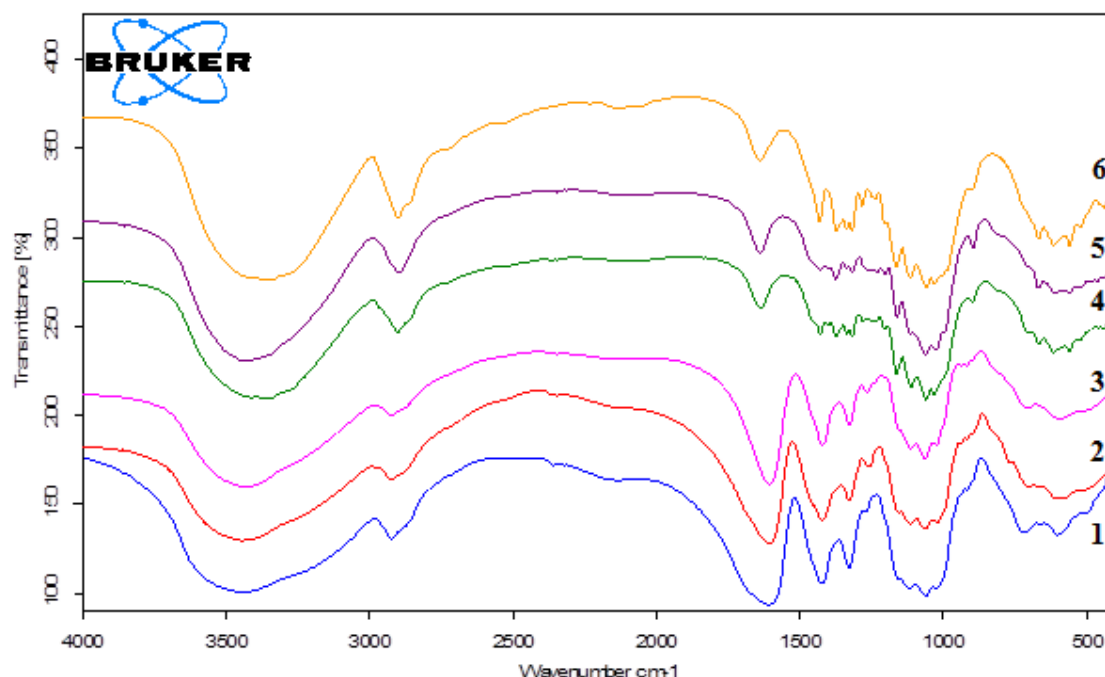
**Figure 4.** Dependence of DS on MCAA concentration: Na-CMC obtained from NC-2 (1), NC-1 (2), MCC (3)



# OPTIMIZED SYNTHESIS OF CARBOXYMETHYL CELLULOSE FROM MICROCRYSTALLINE CELLULOSE AND NANOCELLULOSE VIA SUSPENSION METHOD

In Figure 4, we can observe that DS has a high value when the MCAA concentration is 2 moles.

The FTIR spectrum of Na-CMC obtained from NC-1, NC-2, and MCC displayed absorption regions in the stretching and deformation vibrations that are characteristic of cellulose structure. Furthermore, changes in intensity were observed in several peaks (Fig. 5).

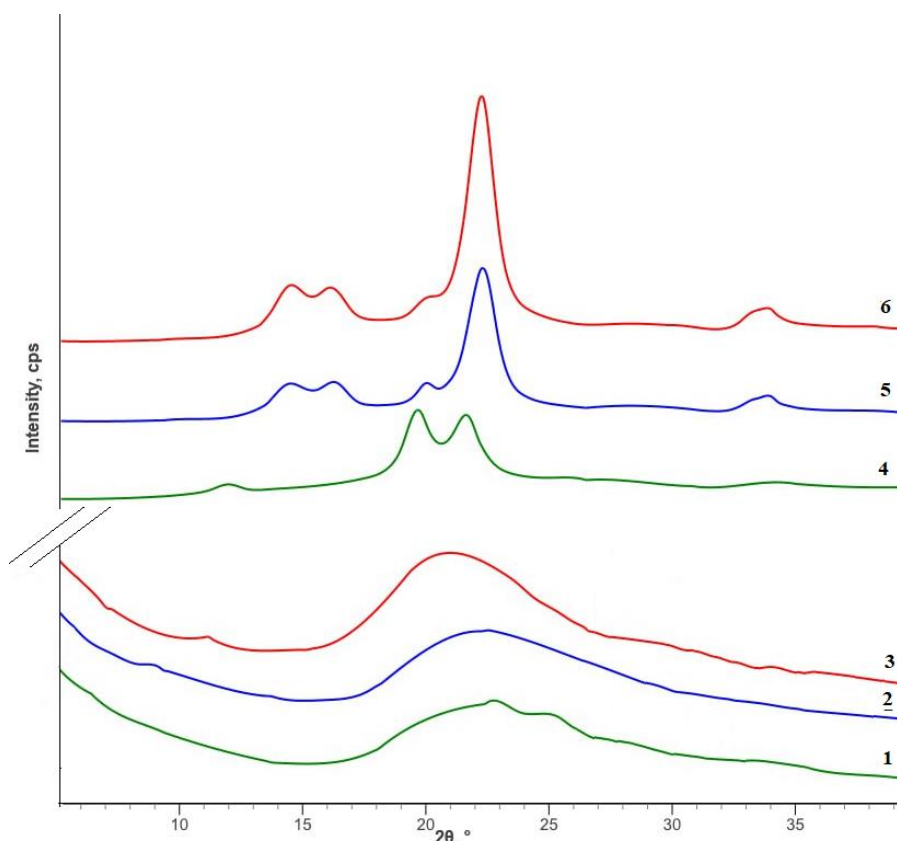


**Figure 5.** FTIR spectra of samples: Na-CMC obtained from MCC (1), NC-1 (2), NC-2 (3), and neat MCC (4), NC-1 (5), NC-2 (6).

The absorption regions ranging from  $1605$  to  $1420\text{ cm}^{-1}$  correspond to two carboxyl and methyl groups of Na-CMC [31]. There are additional absorption bands in the range of  $2150$ – $2370\text{ cm}^{-1}$ , which may be attributed to the presence of by-products from Na-CMC reactions or an overlap of the Na-CMC and water bands.

A wide absorption band in the range of  $3420$ – $2925\text{ cm}^{-1}$  is characteristic of vibrations of the hydroxyl (OH) and methylene (C-H) asymmetric branches [32]. Strong vibrations at  $1605\text{ cm}^{-1}$  confirm the presence of carbonyl groups (C=O). The absorption bands at  $1420\text{ cm}^{-1}$  and  $1325\text{ cm}^{-1}$  belong to  $(-\text{CH}_2)$  and free hydroxyl groups (OH), respectively. Absorption (CO) in the region of  $1050$ – $1095\text{ cm}^{-1}$  indicates stretching vibrations of the bands. As the DS of Na-CMC increases, the intensity of this band decreases, indicating that the hydroxyl groups form in-plane bending vibrations.





**Figure 6.** X-ray diffraction patterns of Na-CMC obtained from NC-2 (1), MCC (2), NC-1 (3) and neat NC-2 (4), MCC (5), NC-1 (6).

The X-ray diffraction patterns reveal differences in the structure of MCC, NC-1, and NC-2. These structures are characterized by the arrangement of elementary units within a crystal cell and the presence of areas exhibiting coherent scattering of X-ray radiation. Upon examining the X-ray diffraction analysis, it is clear that both MCC and NC-1 exhibit crystalline reflections at  $2\theta = 14.9^\circ$ ,  $16.4^\circ$ ,  $20.7^\circ$ ,  $22.75^\circ$ , and  $34^\circ$ . These reflections arise from the diffraction of X-rays from various planes, each with an interplanar distance of  $d = 5.93 \text{ \AA}$ ,  $5.39 \text{ \AA}$ ,  $4.28 \text{ \AA}$ ,  $3.9 \text{ \AA}$ , and  $2.58 \text{ \AA}$  respectively (Fig. 6). In the case of the NC-2 sample, a transition from the crystalline structure of cellulose I to cellulose II is observed (Fig. 6). Furthermore, carboxymethylation leads to amorphization in all samples, as evidenced by a broad amorphous halo in the diffraction patterns with a peak at  $2\theta = 21^\circ$ .

### Conclusions

In this study, sodium carboxymethyl cellulose (Na-CMC) samples with high degrees of substitution (DS) were successfully synthesized from NC-1, NC-2, and microcrystalline cellulose (MCC), using the suspension method. The physicochemical properties of the synthesized Na-CMC samples were comprehensively characterized, and the factors influencing their production were systematically investigated.

Through the optimization of synthesis parameters including sodium hydroxide concentration, monochloroacetic acid (MCAA) concentration, reaction temperature, and reaction time, the optimal conditions for Na-CMC synthesis from NC-1, NC-2, and MCC were determined. A

# OPTIMIZED SYNTHESIS OF CARBOXYMETHYL CELLULOSE FROM MICROCRYSTALLINE CELLULOSE AND NANOCELLULOSE VIA SUSPENSION METHOD

sodium hydroxide concentration of 8%, MCAA concentration of 2 mol, reaction temperature of 55°C, and reaction time of 60-90 minutes were found to yield Na-CMC samples with desirable DS values.

The obtained Na-CMC samples exhibited DS values of 1.25, 1.46, and 0.92 for NC-1, NC-2, and MCC, respectively, indicating successful carboxymethylation of cellulose chains. These DS values reflect the extent of carboxymethylation and are indicative of the functionalization degree of Na-CMC, which plays a crucial role in determining its physicochemical properties and applications.

Overall, the optimized synthesis conditions and characterized properties of Na-CMC derived from NC-1, NC-2, and MCC provide valuable insights for the production of tailored cellulose derivatives with specific functionalities. The established methodology offers a systematic approach for synthesizing Na-CMC with controlled DS values, paving the way for its utilization in various industrial applications including food, pharmaceuticals, textiles, and cosmetics.

Furthermore, the findings of this study contribute to the understanding of the synthesis parameters affecting the production of Na-CMC and provide a basis for further research in optimizing synthesis protocols and exploring novel cellulose-based materials with enhanced properties and functionalities.

In conclusion, the successful synthesis and characterization of Na-CMC samples under optimized conditions underscore the potential of cellulose derivatives as versatile materials for diverse applications, with implications for advancing sustainable and innovative solutions in materials science and industrial manufacturing.

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# **OPTIMIZED SYNTHESIS OF CARBOXYMETHYL CELLULOSE FROM MICROCRYSTALLINE CELLULOSE AND NANOCELLULOSE VIA SUSPENSION METHOD**

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