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Preparation and Evaluation of Carboxymethyl Cellulose from Recycling Paper Waste

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Abstract

In this study, the rapid production of carboxy methyl cellulose (CMC) from recycling waste paper with an average working hours including waste collection and product preparation up to 50 hours and An optimum temperature between 80-90 ° C using heater as an energy source. The obtained CMC was characterized via FTIR, XRD, techniques to ascertain its structure using two different technique heating with reflex. FTIR spectrum showed characteristic peaks of carboxy methyl cellulose groups and its salt at the wavenumbers of (C=O), (C-O), (C-O-C), and OH at different wavelength. Higher efficiency obtained from heating with reflex technique.

Keywords: Office Waste Paper, Carboxymethylcellulose, Cellulose, Monochloroacetic Acid, Agricultural Waste, Conventional Method.

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Introduction

Office Waste Paper (OWP) serves as a significant source of cellulose due to its high cellulose content and minimal amounts of ink (Joshi et al., 2015). Each year, millions of tons of OWP are generated globally (Wongvitvichot et al., 2021; Asl et al., 2017), with most of it being reused or recycled. A promising solution to the OWP challenge is converting cellulose into high-value materials such as carboxymethyl cellulose (CMC), which holds critical applications in the food and pharmaceutical industries (He et al., 2021; Yang et al., 2023). CMC, a cellulose derivative, is environmentally friendly due to its non-toxic, biodegradable nature and versatile applications, including use as a thickener, water binder, film-former, suspension aid, wound dressing material, scaffold, and drilling fluid (He et al., 2009; Rahman et al., 2021). The production of CMC typically involves two key reaction steps: alkalization and etherification. In the alkalization step, cellulose is activated using NaOH, followed by the etherification process where cellulose reacts at its hydroxyl groups with monochloroacetic acid (MCA) to form CMC, as illustrated in Eq. (1) and (2). These steps collectively constitute the carboxymethylation reaction. However, a side reaction between excess NaOH and MCA can also occur, as depicted in Eq. (3) (Yusoff et al., 2021; Joshi et al., 2017; Marie, 2019).

$$C_5H_6O_{10} \xrightarrow{NaOH} \text{Hydrolyzed cellulose} \quad (1)$$

$$C_5H_6O_{10} + CH_2ClCOOH \xrightarrow{\Delta} CMC + HCl \quad (2)$$

The heat method has also been shown to be an effective method for the synthesis of CMC from agricultural waste, namely wheat straw, barley straw, and rice husk. On the other hand, the conventional method required a rang reaction time between (2-3h) to obtain the best results under the same conditions. both the microwave-assisted method and the conventional method have the disadvantage of being unable to be converted to industrial scale because the reaction time is still long and the energy consumption is high .

As a result, this study investigated the effects of optimum conditions, including temperature and reaction time, on production yield.

Materials and Methods

1. Materials and Instrument

- Cellulose (as pure cellulose) - 10 grams. From recycling.
- Acetic chloride ($CH_2ClCOOH$) - 15 grams (BDH)
- Sodium hydroxide 10% w/volume (NaOH). (BDH)
- Deionized distilled water.
- Ethanol (Scharlau)
- Magnetic heater.
- Balance.
- Round flask, condenser and beakers.

2. Preparation Method

- Used and damaged paper was collected from different places (Iraq-Mosul university).
- The paper was washed and cleaned to remove dirt and other residues stuck on it.
- Prepare a solution of sodium hypochlorate 6% and soak the paper waste for 48 hours with the need to renew the solution every 8 hours with stirring.
- After that, the filtration is done to separate the paper from the solution and with pure water and soak with the same water for another 48 hours and take into account the separation, washing and water exchange every 2 hours.

- After that, a paper paste resulting from the recycling process is produced and squeezed and dried to remove the moisture.
- After drying, take part of the dough and conduct the following treatments on it:

2.1 Dissolving Cellulose

Dissolve 10 grams of cellulose in 100 ml of sodium hydroxide solution at a concentration of 5-10%. Leave the mixture for several hours (2-4 hours) for the cellulose to decompose and become a homogeneous mixture, according to eq.1

$$C_5H_6O_{10} \xrightarrow{NaOH} \text{Hydrolyzed cellulose} \quad (1)$$

After dissolving the cellulose, add 15 g of acetic chloride to the mixture. Stir the mixture well to ensure the reaction[18]

2.2 Reactions

Heat the mixture at a temperature between 60-80 ° C for (0.5,1, 1.5, 2, 2.5, 3) hours. Be sure to monitor the temperature and time to ensure that the product does not disintegrate [19]

$$C_5H_6O_{10} + CH_2ClCOOH \xrightarrow{\Delta} CMC + HCl \quad (2)$$

2.3 Stop the Interactions

After the reaction is finished, cool the mixture and add an amount of water (about 200 ml) to stop the reaction.

2.4 CMC Purification

Filter the mixture to remove any insoluble ingredients. For CMC precipitation, also we can use 50 ml of ethanol.

2.5 Drying the Product

- Dry the product in an oven at a low temperature (about 40-60 ° C) until a dry powder is obtained, the output is CMC, the product will cooling and filter, then neglect the filtrate and take the precipitate, which is supposed to be a carboxy such as cellulose.
- To be sure, we conduct an examination of the product material and compare it with the tests of the original material. The resulting we confirm with FTIR and XRD spectrum [17-19]

Results and Discussion

Fig. 1 XRD Spectrum of Carboxymethylcellulose Samples at Different Reaction Times

- h_6: Carboxy methyl cellulose prepared at 0.5 hours.
- h_5: Carboxy methyl cellulose prepared at 1 hours.
- h_4: Carboxy methyl cellulose prepared at 1.5 hours.
- h_3: Carboxy methyl cellulose prepared at 3 hours.
- h_2: Carboxy methyl cellulose prepared at 2 hours.
- h_1: Carboxy methyl cellulose prepared at 2.5 hours

Through the arithmetic results and the X-ray chart, we find that to prepare this material, the best results were at a temperature of 60-80°C and a time of 2-2.5 hours (h_2, h_1 spectrum), and the reason for this is that a complete interaction between the components occurs within this hours of the reaction, and then if the reaction continues for a longer time, the disintegration of the resulting material occurs. Through the infrared spectrum, we observe peaks at sites (1409,1600,2920,3280cm^{-1}) that represent the active aggregates in and their intensity varies according to the degree of interaction between the aggregates. C-O-C, C=O, C-H, -OH respectively, and this seems obvious in h_1 and h_2 spectrum. In fig. 2 and fig. 3 displays FTIR for Slandered CMC and XRD for Slandered CMC in below.

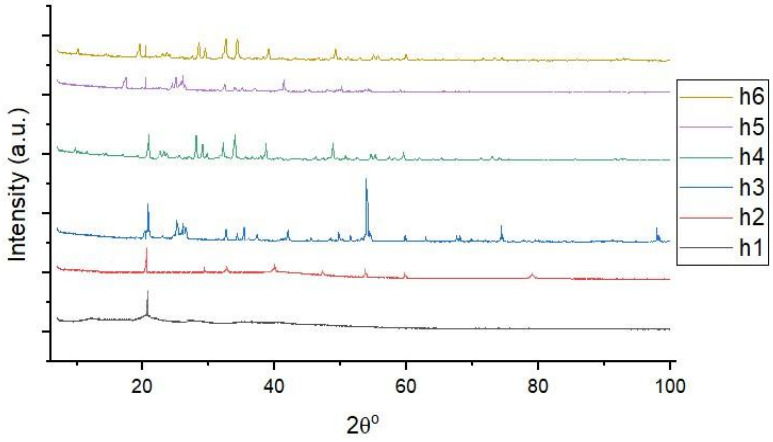


Fig. 1: XRD Spectrum of Carboxymethylcellulose Samples at Different Reaction Times

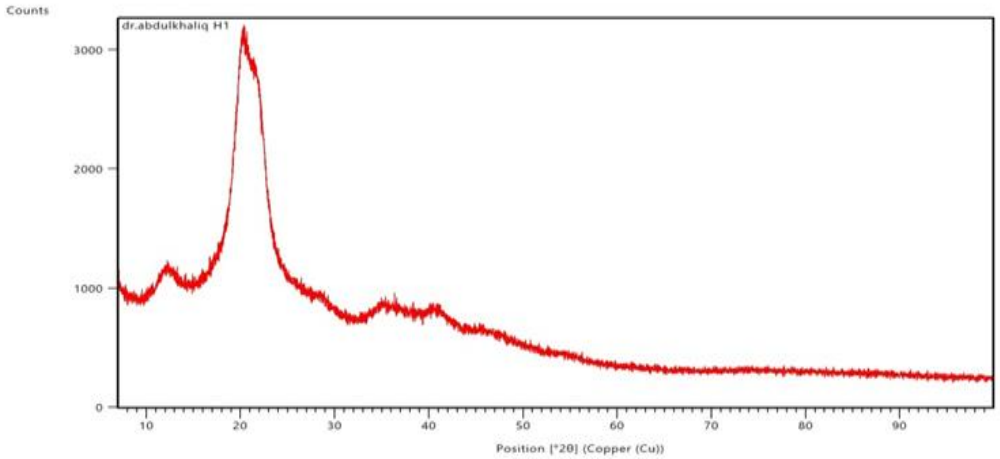


Fig. 2: FTIR for Slandered CMC

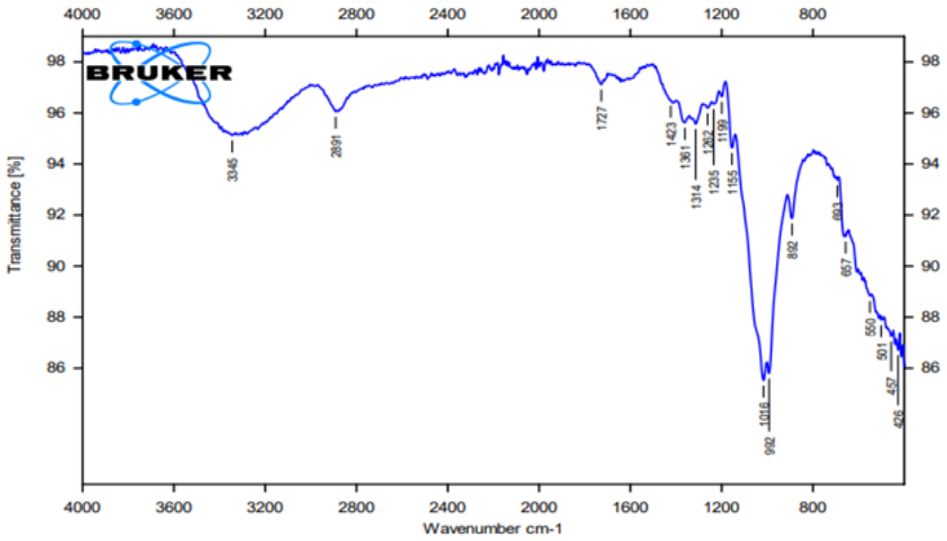


Fig. 3: XRD for Slandered CMC

Table 1: Changes in the Yield of Carboxymethylcellulose with Changes in Temperature and Reaction Times

Reaction time (min)	Temp. ◦C	yield		Reaction time (min)	Temp. ◦C	yield
30	50	30		60	50	31
30	60	33		60	60	39
30	70	37		60	70	42
30	80	41		60	80	44
30	90	41		60	90	44
30	100	40		60	100	43
Reaction time (min)	Temp. ◦C	yield		Reaction time (min)	Temp. ◦C	yield
90	50	33		120	50	34
90	60	40		120	60	45
90	70	49		120	70	52
90	80	52		120	80	66
90	90	51		120	90	64
90	100	48		120	100	62
Reaction time (min)	Temp. ◦C	yield		Reaction time (min)	Temp. ◦C	yield
150	50	37		180	50	39
150	60	56		180	60	51
150	70	63		180	70	59
150	80	74		180	80	68
150	90	73		180	90	64
150	100	68		180	100	57

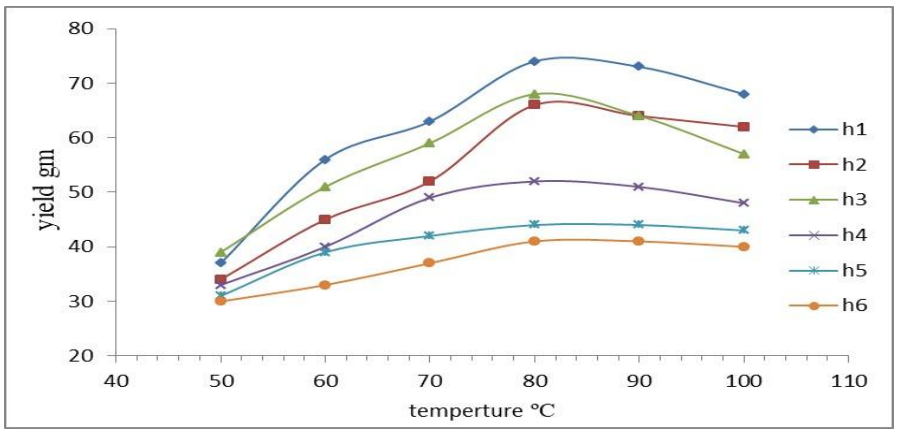


Fig. 4: Yield of Carboxymethylcellulose with Changes in Temperature and Time of Reaction

The tables 1 and Fig. 4 show the change in the production yield of carboxymethylcellulose with the change in temperature and time taken for the reaction. The ideal conditions for temperature and time are that the reaction temperature should be between 80 and 90 °C and the best reaction time is 120-150 minutes, the reason is that the reaction is not complete without these conditions. If the temperature exceeds these parameters, the formed compound will undergo decomposition. Therefore, the above reaction conditions must be adhered for physical and chemical treatments for office paper waste, in addition to that, the work has environmental and economic aspects, as the environment was rid of widespread and polluting paper waste and converted into a material of economic value,

which is one of the important materials at the industrial and food levels. Fig. 5 show the change in the production FTIR of carboxymethylcellulose with the change in temperature and time taken for the reaction.

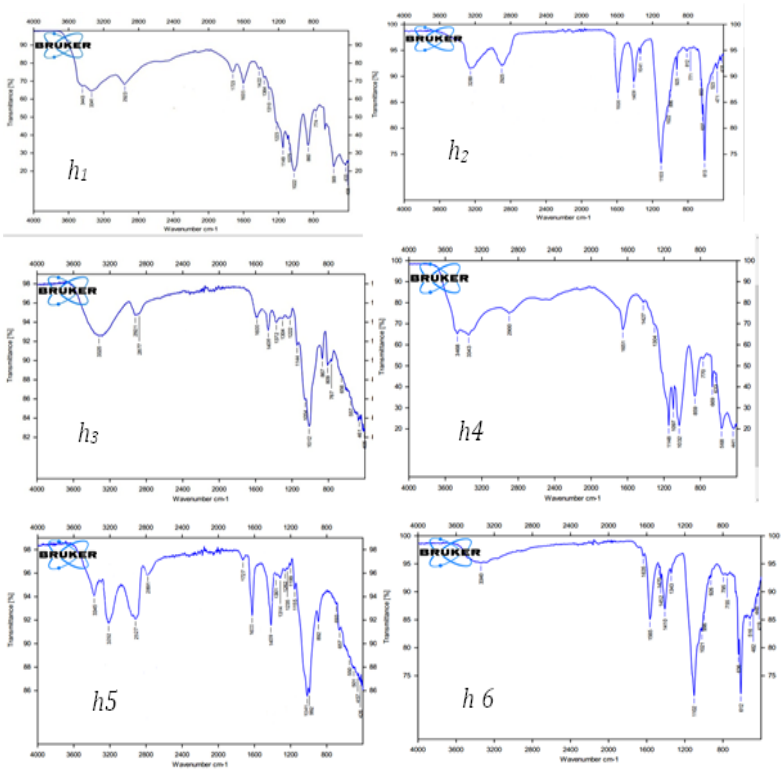


Fig. 5. FTIR of Carboxymethylcellulose with Changes in Temperature and Time of Reaction

Conclusion

The ideal conditions for temperature and time are that the reaction temperature should be between 80 and 90 °C and the best reaction time is 120-150 minutes. By observing the results and interpretations of infrared and X-ray spectra, we clearly found that we obtained the required substance (carboxymethylcellulose) through a series of physical and chemical treatments for office paper waste, in addition to that, the work has environmental and economic aspects, as the environment was rid of widespread and polluting paper waste and converted into a material of economic value, which is one of the important materials at the industrial and food levels.

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