

Synthesis and Characterisation of Carboxymethyl Cellulose Based Bentonite Polymer Blend



Shalini Palani, Ranjitha Jambulingam, Anand Mohanam, Gokul Raghavendra Srinivasan

Abstract: Polymer blend of carboxymethyl cellulose (CMC) and bentonite clay were prepared in the presence and absence of the cross linking agent glutaraldehyde. The synthesized compounds were characterized using FT-IR, XRD, TGA and DSC analysis. The percentage of yield obtained is higher for the polymer blend prepared with glutaraldehyde as cross linking agent. The result of FTIR studies showed the various peaks corresponding to the functional groups present in the two polymer, which clearly indicates the presence of strong cross linking between the polymer and cross linking agent. Similarly, thermal gravimetric analysis revealed that enhanced thermal stability for the polymer blends along with glutaraldehyde. X-ray diffraction studies prove that the polymer blends have higher crystallinity when blending with glutaraldehyde.

Keywords : Polymer blend of carboxymethyl cellulose (CMC) and bentonite clay, X-ray, FTIR Spectral studies..

I. INTRODUCTION

Naturally isolated carbohydrate polymers have unique physical and bio-chemical properties advantageous for bio-engineering applications. Cellulose derivatives such as carboxymethylcellulose (CMC) and hydroxypropylcellulose are biocompatible [1] and have been applied in drug delivery formulations [2-5]. A large variety of CMC derivatives was prepared by modification of the hydroxyl groups of CMC using acyl chlorides, anhydrides, isocyanates as well as sulphation, phosphorylation, and silylation agents. CMC is commercially available in large quantities and known to act as water binder, thickener, suspending agent, emulsion stabilizer and anti-redeposition agent in detergents [6&7]. CMC is used as medicine, food, paper making, printing & dyeing, and

protective colloid thickener and in dispersion in aqueous solvents. Sodium salts of carboxymethyl cellulose form the basis for the production of cellophane. Recently, a new series of polymeric surfactants based on CMC grafted with macro monomers was reported to show excellent surface activity due to their unique structure [8-10]. Similarly, further attempts has been to substitute the environmentally unfriendly and toxic classical esterification processes, the earlier trans-esterification reactions with fatty acid methyl esters were reinvestigated and used also under solvent-free conditions [11]. The polymer blend used is the most convenient and the fastest, as well as the cheapest materials. The present research paper describes about the bentonite clay blended with CMC at various ratios (1:1, 1:2, and 1:3) and the synthesized product was subsequently analyzed as to their compatibility.

II. MATERIALS AND METHODOLOGY

Carboxymethyl cellulose and Glutaraldehyde were purchased from SD-fine-chemicals, India.

A. Polymer Blend preparation

Preparation of Carboxymethyl cellulose and Bentonite clay

About 0.5g of Carboxymethyl cellulose was dissolved in 25mL of double distilled water. Simultaneously, about 0.5g of Bentonite clay was dissolved in 25mL of double distilled water. These two solutions were mixed in the ratio of 1:1, 1:2 and 2:1. These solutions were stirred in a magnetic stirrer for about 30 minutes. The stirred solution were poured into petri dishes and dried.

Preparation of Carboxymethyl cellulose and Bentonite clay with Glutaraldehyde:

About 0.5 g of Carboxymethyl cellulose was dissolved in 25mL of double distilled water. Simultaneously, about 0.5g of Bentonite clay was dissolved in 25mL of double distilled water. These two solutions were mixed in the ratio of 1:1, 1:2 and 2:1. To this 3mL of Glutaraldehyde was added as cross linking agent. These solutions were stirred in a magnetic stirrer for about 30 minutes. The stirred solution were poured into petri dishes and dried.

B. FTIR Characterization

FTIR Measurements were performed on these polymer blending samples using AVATAR spectrophotometer using KBr pellets.

Manuscript published on January 30, 2020.

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The FTIR spectra were obtained in the wave number range from 4000 to 450 cm⁻¹.

C. Differential scanning calorimetric (DSC) Analysis

DSC of the samples was measured using a DSC Q10v9.0 Build 275 Instrument. The temperature range was varied from room temperature 30°C-350°C with the heating rate of 10°C/min

D. Thermogravimetric analysis (TGA)

The polymer blended samples was prepared under different conditions and tested in a SDT Q600v80 Build 275 Instrument. The temperature range was varied from room temperature to 850°C with the heating rate of 20°C/min.

E. X-ray diffraction (XRD) Analysis

The x-ray diffraction patterns of the samples were tested using x-ray scattering 'SHIMADZU-6000' Diffractometer using thin filter Cu (K α) radiation source (LAMBDA = 0.154nm). The scattering angle (2 θ) was from 10 to 60° at a scan rate of 5°/min.

III. RESULTS AND DISCUSSIONS

The synthesized polymer blends were characterised using FT-IR spectral analysis. The IR spectra of the Carboxymethyl cellulose and Bentonite blend with Glutaraldehyde and without Glutaraldehyde as shown in the figure-1. Table 1 consolidates the FTIR spectral data of pure pure carboxymethyl cellulose with CMC/bentonite blend (1:1) with Glutaraldehyde.

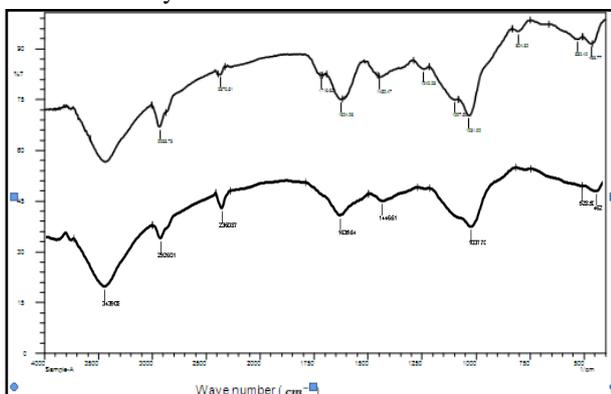


Figure-1: FTIR spectra of the synthesized polymer blends

Table 1: FTIR spectral data of pure pure carboxymethyl cellulose with CMC/bentonite blend (1:1) with Glutaraldehyde

Pure carboxymethyl cellulose	
Wavenumber (cm ⁻¹)	Bond Activity
3435	O-H stretching
2924	CH stretching of CH ₂ and CH ₃ groups
2376	COO ⁻ and O-H bending
1636	C=O stretching
1464	C-H bending
CMC/bentonite blend (1:1) with Glutaraldehyde	
Wavenumber (cm ⁻¹)	Bond Activity
3435.22	O-H stretching
2918.3	CH stretching of CH ₂ and CH ₃

	groups
2376.3	COO ⁻ and O-H bending
1604.77	C=O stretching
1425.4	C-H bending

The spectrum of pure carboxymethyl cellulose shows a broad band at 3435 cm⁻¹ which is due to the O-H stretching. The band at 2376 cm⁻¹ is assigned for the combination band from COO⁻ and O-H bending. The band at frequencies 1330, 1045, 590 and 503 cm⁻¹ is due to the presence of -C-H bending, C-O, C-C, C-O-H bending and C-C bending. Similarly, in the case of pure bentonite, the peak at 3624 cm⁻¹ indicates the Si-OH, Al-OH stretching and bending band. The band at 3439 cm⁻¹ and 2368 cm⁻¹ O-H and Si-H stretching. When comparing the FTIR spectral results of CMC/bentonite blend (1:1) with Glutaraldehyde to pure carboxymethyl cellulose, the most prominent peaks of CMC are shifted from the wave numbers 3435.22, 2918.3, 2376.3, 2144.84, 1604.77 and 1425.4 cm⁻¹. Based on the FTIR spectral data, the CMC was properly mixed with cross linking agent.

Likewise, XRD of carboxymethyl cellulose/bentonite with and without glutaraldehyde is shown in the figure-2. Table 2 tabulates the diffraction data of the carboxymethyl cellulose/bentonite with glutaraldehyde. The carboxymethyl cellulose exhibits single broad peaks at 2 θ =19° which is exactly matching with the X-ray pattern of pure carboxymethyl cellulose. Carboxymethyl cellulose/bentonite blend prepared with and without cross linking agent shows almost three distinct peaks at around 12°, 20° and 25° exactly matching with JCPAD XRD pattern.

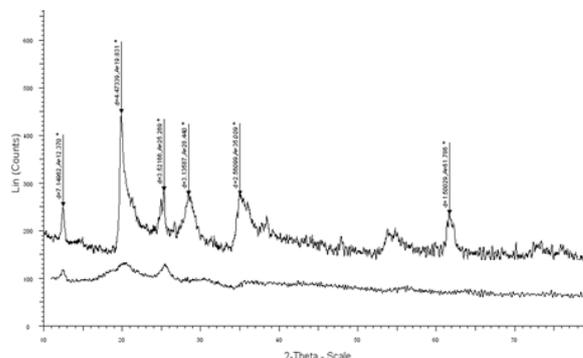


Figure-2: XRD pattern of the synthesized polymer blends

Table 2 tabulates the diffraction data of the carboxymethyl cellulose/bentonite with glutaraldehyde

d-spacing (Å)	2 θ (°C)
7.14962	12.370
4.47339	19.831
3.52166	25.269
3.13587	28.440
2.56099	35.009
1.50029	61.786

TGA studies of carboxymethyl cellulose /bentonite (1:1, 1:2, 2:1) polymer blend prepared with and without glutaraldehyde as shown in the figure-3. It shows eight different decomposition temperatures.

Maximum decomposition occurs at around from 350° C, Maximum weight loss occur at 150-400°C. On comparing the TGA results of the blends (Carboxymethyl cellulose/Bentonite) prepared at various ratio (1:1, 1:2, 2:1) with and without cross linking agent glutaraldehyde, it was observed that the blend of Carboxymethyl cellulose and Bentonite prepared in the presence of cross linking agent glutaraldehyde was found to be thermally most stable when compared to Carboxymethyl cellulose/Bentonite blend in the absence of glutaraldehyde.

This was confirmed from the amount of blend remained as residue at end of the experiment and the various decomposition temperatures.

DSC curves of pure carboxymethyl cellulose, Bentonite and carboxymethyl cellulose/bentonite blend 1:1 with and without glutaraldehyde. Broader endothermic peaks are observed at various temperatures indicating the crystallization of the blended polymers as well as evaporation of water in the sample. The glass transition temperature of pure carboxymethyl cellulose is 185.65 °C, Whereas the glass temperature of carboxymethyl cellulose/bentonite blend (1:1) with glutaraldehyde blend is 213.32 °C and the glass temperature of carboxymethyl cellulose (1:1) blend without glutaraldehyde is 186.4 °C On comparing the DSC curves of carboxymethyl cellulose it was found that the endothermic peaks and the glass temperature of carboxymethyl cellulose/bentonite blends with and without cross linking agent are shifted to higher values. It confirms that the polymer has higher thermal stability than the original carboxymethyl cellulose.

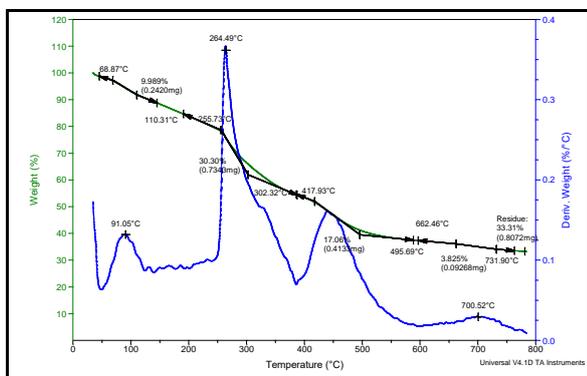


Figure 3: TGA Thermogram of Carboxymethyl/Bentonite blend (1:1) with Glutaraldehyde

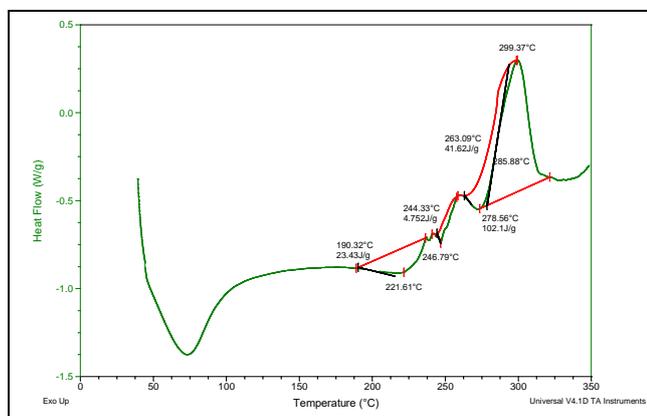


Figure 4: TGA Thermogram of Carboxymethyl/Bentonite blend (1:1) with Glutaraldehyde

IV. CONCLUSIONS

Carboxymethyl cellulose and bentonite blend were prepared by a simple solution-blending method followed by a cross linking procedure. All samples were characterized by FTIR studies thus proving the component polymers participate in the blend preparation. X ray diffraction studies prove that the polymer blends have higher crystallinity when blending with cross linking agent. The TGA and DSC studies proves that the Carboxymethyl cellulose/Bentonite blend prepared in the presence of cross linking agent glutaraldehyde was found to be thermally more stable when compared to Carboxymethyl cellulose/Bentonite blend in the absence of glutaraldehyde,. The thermal stability of the polymers increases due to blending and cross linking. The shift of glass transition temperature from pure Carboxymethyl cellulose (Tg-186.65°C) and pure Bentonite (Tg-209°C) to Carboxymethyl cellulose blend (1:1) with Glutaraldehyde (Tg-213°C) and same blend without Glutaraldehyde (Tg-186.4°C) was a good measures of miscibility in the amorphous phase.

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Ms. Shalini Palani graduated her master degree in Chemistry from St.Peter's University, Chennai. Her areas of interest are bioenergy and waste to energy conversion using biotechnology. She has published many papers in international Journals and presented papers in international conferences.



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