



# Synthesis and characterization of eco-friendly carboxymethyl cellulose based carbon foam using electron beam irradiation

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## ABSTRACT

Carbon foams were prepared by carbonization process without additional stabilization process after manufacturing carboxymethyl cellulose (CMC) composites using a facile electron beam irradiation (EBI) method. A gel fractionation technique was used to study cross-linking degree of infusible structure in the produced CMC composite materials. We observed an increase in cross-linking with increasing CA concentration and EBI doses between 20 kGy and 80 kGy. The CMC composite prepared using 4 wt% CA and 80 kGy EBI represents the highest gel fraction value of ~98%, showing the highest carbon yields and compressive strength due to the increase of cross-linked parts in carbon foam obtained from these CMC composites, which lowers break defects after carbonization. In addition, available surface area was estimated via Brunauer-Emmett-Teller analysis of the carbon foam samples. The carbon foam produced from the CMC composite treated with 4 wt% CA via 80 kGy resulted in highest specific surface area of 372.06 m<sup>2</sup>/g and adsorption pore size of 2.20 nm indicating greater interaction between gas and the carbon atoms.

## 1. Introduction

Carbonaceous materials have good thermal stabilities, chemical stabilities, and non-corrosive properties [1–15]. Carbon foams, one of various carbonaceous materials, have 3-dimensional (3-D) reticular structures of various shapes and sizes. Their high porosity makes them applicable as additives of phase change materials, in sound absorption, physical adsorption, and as catalyst supports. Thus, many researchers have reported on carbon foam-based materials due to their tunable and unique surface functionality and their well-defined macro- and micro-porous 3-D structures [16–25]. Various carbon foam manufacturing processes have been proposed and studied with diverse precursors such as phenolic resin, coal, and pitch obtained from fossil fuel, but utilization of these industrial side products in carbon foam synthesis generated large amounts of toxic gases and shrinkage during carbonization [26–35]. In addition, these processes for manufacturing carbon foam are multi-step, complex, and require large amounts of capital. Therefore, it is highly desirable to develop simple processes for carbon foam manufacturing that use biopolymers as precursors instead of toxic precursors derived from fossil fuel. Generally, biopolymers are biodegradable, biocompatible, and non-toxic. Among them, CMC is a cellulose derivative which has been substituted with sodium carboxylate

groups from the anhydroglucose unit of cellulose, which is one of the most abundant organic polymers obtained from wood or non-wood precursors. Therefore, CMC is classified as a non-toxic natural biopolymer that has the advantages of being biodegradable, biocompatible, and water-soluble. CMC has widely been used as a hydrogel due to its unique porosity [36–41] and still provide eco-friendly, light-weight, and low-cost materials. In addition, electron beam irradiation (EBI) can modify various substances without solvents or additives through chain scission, polymerization and cross-linking [42–44]. Therefore, using the above novel merits of CMC composites and EBI, we prepared eco-friendly carbon foams through facile and simple method via EBI. However, during carbonization over 1000 °C, to survive without decomposing and thus to obtain a high carbon yield, cross-linking degrees in CMC foam composites is critical for commercial applications. In this study, we aimed to prepare 3-dimensional carbon foams from CMC of biopolymer without fossil fuel polymer for various applications. The influences of EBI doses and CA concentrations on the cross-linking of CMC foam composites for the preparation of carbon foams are measured by gel fraction, and the structural, and mechanical properties of the products are characterized by SEM, compressive strength, and BET.

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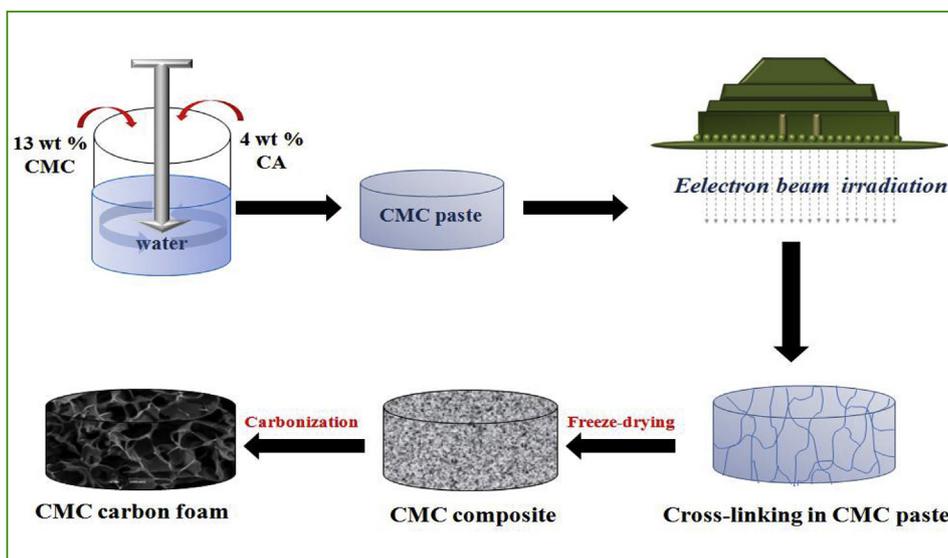


Fig. 1. Schematic diagram of the preparation of CMC carbon foam.

## 2. Experimental

### 2.1. Preparation of CMC composites and their carbon foams

CMC (average MW:  $\approx 250,000$ ) and CA ( $C_6H_8O_7 \cdot H_2O$ ) used in this research were purchased from Sigma-Aldrich Co. As shown in Fig. 1, for preparation of CMC composites, 13 wt% CMC was dissolved in distilled water by stirring at room temperature. 2 wt% and 4 wt% CA were respectively added to the 13 wt% CMC paste. The homogeneous CMC and CA paste mixtures were transferred into plastic molds. Carbonization reaction conditions were determined for optimal cross-linking of CMC and CA, to minimize sample decomposition at high temperature during the carbonization process. The samples were then irradiated at various absorbed doses of 20 kGy, 40 kGy, 60 kGy, 80 kGy, and 100 kGy (an accelerating voltage of 1.14 MeV, a beam current of 7.6 mA, irradiation width of 110 cm, distance between samples and window of 20 cm, dose rate of 6.67 kGy/sec) in air at room temperature. Dosimetry was carried out using cellulose tri-acetate (CTA; ISO/ASTM 51,650 (2013)). All irradiated samples were freeze-dried and CMC foam composites were obtained. The obtained CMC composites were carbonized for 1 h at 1000 °C (heating rate = 2 °C/min) in a tubular furnace under an atmosphere of high-purity nitrogen (99.999%) without stabilization.

### 2.2. Analysis

Gel fraction values were obtained using distilled water, and the soluble fraction was extracted for 24 h at room temperature. The insoluble fraction was completely dried in a vacuum oven at 80 °C. The gel fraction values were calculated according to the following equation;

$$\text{Gel fraction (\%)} = (W_2/W_1) \times 100$$

where  $W_2$  is the weight of the insoluble fraction after extraction with distilled water,  $W_1$  is the known weight of CMC composites before extraction in distilled water. Scanning electron microscopy (SEM) images were obtained using a Jeol JSM 5910 LV microscope to study the morphology of CMC composites and their carbon foams. Compressive strength was measured by an Instron 5050 tester (Instron USA) by method of ASTM standard C365. In the Brunauer-Emmett-Teller (BET) method, the textural properties were observed in terms of nitrogen

adsorption-desorption isotherms specific surface areas, and adsorption-average pore size, obtained using a gas adsorption analyzer (Barrett-Joyner-Halenda model).

## 3. Results and discussion

### 3.1. Gel fraction and carbon yields

Cross-linking reaction between CMC and CA generally happens through esterification by hydroxyl groups of CMC and carboxyl groups of CA during EBI (Fig. 2). The gel fraction exhibit the degree of conversion of the linear structure of precursors to cross-linking structure in composites materials. As shown in Fig. 3, the gel fraction values obtained in CMC composites increased for EBI dose up to 80 kGy, followed by a slight decrease at 100 kGy. This trend is observed for both CA concentrations of 2 wt% and 4 wt%. These results are because excessive radiation over a certain range of EBI dose can occur decomposition by chain scission and then decrease gel fraction. Among CMC composites obtained in this study, CMC composite containing 4 wt% CA via 80 kGy of EBI exhibited the highest gel fraction value of around 97%. Table 1 shows the carbon yields of CMC composites, obtained according to various EBI doses and CA concentration. As exhibited in Table 1, we can see that the carbon yields for the CMC composites mostly increase with increasing EBI dosage up to 80 kGy and CA concentrations from 2 to 4 wt%. The slight decrease in the gel fraction as observed in samples dosed at 100 kGy can be rationalized by the decomposition of the non-cross-linked fraction in CMC composites at 1000 °C during carbonization. Therefore, it is concluded that CMC composites with high gel fraction values directly result in higher carbon yields.

### 3.2. SEM images

Digital photos of Fig. 4 show CMC composites ((a) and (c)) and their carbon foams ((b) and (d)). Carbon foam was prepared through carbonization at 1000 °C without stabilization process. As shown in Fig. 4, cross-linking in composites generally plays an important role in preparing of carbon foam. After carbonization, CMC composite containing 4 wt% CA via 40 kGy of EBI can be seen that most of them burned due to low gel fraction. However, we can see that the carbon foam obtained

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