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Preparation and Mass Loss Study of Sodium Carboxymethyl Cellulose Carbon Aerogel Prepared from Non-Hazardous Material

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***Abstract*— Carboxymethyl cellulose (CMC) is a derivative of cellulose that shares its biodegradability, biocompatibility, and renewability while being soluble in water and some organic solvents. It is due to these characteristics that the aerogel produced from CMC will share these advantages. In this study, we produced CMC carbon aerogel from different concentrations of CMC aerogels (1%, 2%, 3%, 4%) and under different carbonisation temperatures (300 °C, 400 °C, 500 °C, 600 °C, 700 °C and 800 °C) for a period of 1 hour and 2 hours. The prepared CMC carbon aerogel samples were then analysed based on the difference in mass loss, which differed according to the varying experimental parameters. In temperatures varying from 300 °C to 600 °C, there was a decrease in mass loss as the concentration was increased due to the physical properties of the CMC aerogel. At higher temperatures of 700 °C and 800 °C, the mass loss at higher concentrations of CMC increased due to the completed decomposition and carbonisation of CMC aerogel.**

Keywords— carboxymethyl cellulose; CMC carbon aerogel; preparation; mass loss; concentration; temperature

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I. INTRODUCTION

Cellulose is amongst the most abundant organic compounds on the planet. It is a biomaterial that is most commonly found in the cell walls of plants [1]. Cellulose is most commonly used in the industrial sector due to its eco-friendly nature as well as sustainability, biodegradability, and renewability, but certain characteristics of cellulose limit the universality of its applications in some industries, amongst these characteristics is its insolubility in water and most organic solvents and that is due to its molecular chain which is amphiphilic as well as its crystallinity which is high, cellulose also has intermolecular and intramolecular hydrogen bonds that are very strong. All these factors result in its insolubility in water and most organic solvents [2]. It is this characteristic that gave rise to the need for a suitable substitute that shares the advantages of cellulose and also has increased solubility. One of the main recurring derivatives of cellulose is sodium carboxymethyl cellulose (CMC), which has been used in many fields of industry, such as agriculture, medication, and the production of barriers, scaffolds, and stabilizers. All this is because of its biodegradability, solubility, renewability and eco-friendly nature [3]. Some of the applications of CMC include:

Medical application: in the preparation of various drugs and pharmaceuticals. CMC can also be used with some drugs and pharmaceutical products to study its possible effects as a releasing agent [4].

Agricultural application: CMC can be used in the agricultural industry to prepare enhanced fertilizers. Carboxymethyl cellulose is used to prepare a Root Targeted Delivery Vehicle (RTDV) by dissolving it in water and mixing the solution with fertilizers in liquid form. Calcium salts and iron were used as crosslinking agents. The prepared RTDV increased the efficiency of the fertilizer and reduced the amount necessary to achieve the same results [5].

Preparation of natural polymer-based scaffolds for tissue engineering applications: due to CMC's biodegradability, appropriate surface chemistry, porosity, and biocompatibility, it has been chosen for the preparation of scaffolds that are used for tissue engineering [6].

Li-ion battery production applications: CMC's environmentally friendly nature, low cost, water solubility, and ease of recyclability have allowed it to be used in Li-ion battery manufacturing [7].

Adjusting the properties of Zerovalent iron (ZVI) nanoparticles by using CMC as a stabilizer: CMC can be used in various forms as a stabilizer in the production of Zerovalent iron (ZVI) nanoparticles with varying sizes based on the different parameters of the experiment and different properties of CMC, increased D.S (degree of substitution) and molecular mass in CMC presented smaller nanoparticles [8].

Applications of CMC as an adsorbent material: due to the abundant nature of CMC and its porous structure, it has great potential to be used as an adsorbent material. The adsorption of liquids by the CMC aerogel is greatly affected by the pore size and pore structure of the CMC aerogel, as well as the viscosity of the liquid [2].

The sol-gel creation process can be summarised in the steps in which the material is converted from the initial state, which is the liquid-sol state, to the final state, which is the solid-gel state. The sol-gel reaction is considered to be the most crucial part in developing a sol-gel with a 3-dimensional porous network structure. Cross-linking in physical gels is usually achieved by interactions of a physical nature, such as chain entanglements, electronic or hydrophobic interaction, hydrogen bonds, and van der Waals forces [9]. However, in the case of CMC, it will mostly produce a weak aerogel in terms of mechanical strength due to the diminished number of hydroxyl groups in its molecular chain, which hinders the gelation of the CMC. It is due to this reason that it is usually essential to add cross-linking agents and inorganic material to the solution to enhance the physical properties of the aerogel [2].

The standard traditional method for preparing carbon aerogels usually involves a polymerization reaction. The second step consists of acetone exchange, and the aerogel is then dried using supercritical CO2. The carbonisation step is performed under a vacuum. Due to the complicated and elaborate process, hazardous starting materials, and long gelation time, the method stated above is highly limited in its industrial applications [10].

Previously, aerogels and carbon aerogels were prepared using a mixture of formaldehyde and resorcinol with catalysts such as sodium carbonate [11]. These chemical compounds are hazardous and possibly very dangerous, depending on the level of exposure. According to [12], formaldehyde is very toxic. It can also cause irritation upon exposure to the eyes, throat, skin, and lungs, while long exposures to it can cause cancer. It is also stated by [13] that resorcinol, upon contact, may cause burning and irritation to the eyes and skin, while long exposures to it may cause dizziness, shortness of breath, kidney and liver damage, and even death.

Other methods of producing aerogels include the use of SiO2, TiO2, and V2O5 in the production of aerogels that are inorganic and the use of polyvinylchloride and polyimide in the preparation of synthetic aerogels that are polymer-based [9]. All these methods use chemicals that are hazardous, toxic, and dangerous.

Cellulose is one of the most common raw materials in the production of aerogels, but due to its insolubility in water and most organic solvents, the need for a substitute that can be easily soluble in water and most organic material has arisen. CMC is one of the most common and readily available derivates of cellulose, and it is also soluble in water and organic solvents. It is eco-friendly, non-hazardous, sustainable, and renewable. In this experiment, CMC carbon aerogels were prepared using CMC, GDL (D-(+)-gluconic acid-lactone), and glycerol, which are all non-hazardous, sustainable and are used in the food industry for various applications. This mixture resulted in different CMC carbon aerogels based on the concentration of the CMC, duration of carbonisation, and the temperature of the carbonisation. These parameters have the potential to alter the pore structure as well as the pore size, resulting in different surface areas and different applications. During the process of preparing any chemical compounds, there are many contributing factors that will result in the generation of lower amounts of end product. Other than the obvious causes for mass loss, such as errors that occur during the experimental phase, the decrease in mass of the end product is often due to side reactions of undesirable nature as well as reactions that are incomplete. In the fabrication of products, the percentage of mass loss plays a significant role. For the production of chemicals, a lot of resources, such as funds and time, are spent in reducing the percentage of mass loss. A high percentage of mass loss can cause unnecessary waste of raw materials and unwanted costs in production [14]. This research is a comparative study that produced CMC carbon aerogels from different CMC concentrations and at different carbonisation temperatures and under different carbonisation durations to observe the changes in the mass loss of the produced CMC and, by extension, the changes and different potentials in its applications.

II. CARBON AEROGEL

Among low-density foams, carbon aerogels (CAs) constitute a unique class, and these aerogels are produced through reactions of an organic nature that involve sol-gel transitions. The structure of carbon aerogels is highly mesoporous and has a large surface area and mesopores that are few in number [15]. The aerogels that are converted into carbon aerogels are usually made of interconnected nanoparticles of carbon, and the diameter of these nanoparticles ranges from 3 to 20 nm. The conversion of organic aerogels into carbon aerogels involves a carbonisation process that is carried out in an atmosphere of inert gas (helium, argon, nitrogen) and at extremely high temperatures [16].

Carbonisation can be defined as the process of transforming biomass into material that is similar to charcoal and extremely carbonaceous. Carbonisation aims to increase char production by heating the biomass starting material in an environment that is deficient in oxygen or completely deprived of it [17].

Carbonisation is considered one of the earliest thermochemical reactions known to mankind. This thermochemical process was used to produce charcoal from wood, which was then used for the extraction of iron ore. Carbonisation takes place at a slow rate when biomass is heated to elevated temperatures, starting at 400 °C and continuing to rise higher [18].

III. THE MATERIALS AND METHOD

A. Materials

All the chemicals used in this experiment were purchased from a supplier.

Chemicals	Molecular formula	Manufacturer
Carboxymethyl cellulose Sodium	$C_8H_{15}NaO_8$	Evachem
Glycerol	$C_3H_8O_3$	Sigma-Aldrich
D-(+)-gluconic acid-lactone	$C_6H_{10}O_6$	Sigma-Aldrich

B. Methods

1) Preparation of CMC Aerogel: The first step involved placing different amounts of CMC (Sodium carboxymethyl cellulose) (5 g, 10 g, 15 g, 20 g) in different beakers and adding 2.5 g of glycerol and 10 g of GDL to each beaker. Next, the mixture was dissolved in 500 mL of deionized water and stirred vigorously for 5-10 minutes until a clear homogenous solution was achieved. The prepared solution had different concentrations of CMC based on the different amounts that were added, and the concentrations were 1%, 2%, 3%, and 4%, respectively. The solution was then placed in an ultrasonic bath for a duration of 3-4 hours to remove the air bubbles trapped in the liquid and to ensure the formation of a cross-linked structure within the solution. At this point, the mixture produced a gel-like substance, which was placed in suitable containers and stored for 72 hours in ambient temperatures to complete the cross-linking reaction and the gelation process. Next, the solution was placed in small plastic containers, and approximately 30 mL was poured into each small plastic container. The plastic containers were then placed in the freezer for 24 hours to prepare them for the freeze-drying step.

2) Freeze-drying the CMC Aerogel: Freeze-drying was carried out under vacuum parameters and at a temperature of 20 °C for 72 hours. The aerogel was then collected from the freeze-dryer and stored for the next step.

3) Carbonisation of CMC Aerogel: Four samples of the CMC aerogel of varying concentration (1%, 2%, 3%, 4%) of CMC based on the different CMC amounts that were added at first were placed in the tube furnace for 1 hour and at a temperature of 300 °C to produce carbonised CMC aerogel which was labelled and stored accordingly. This process was replicated for different temperatures of 400 °C, 500 °C, 600 °C, 700 °C and 800 °C to study the effect of temperature on the CMC carbon aerogel. The carbonisation step was repeated under the same experimental parameters but for a duration of 2 hours instead to test the effect of carbonisation time on the prepared CMC carbon aerogel.

III. RESULTS AND DISCUSSIONS

A. *Analysis of the prepared CMC aerogel*

We can observe a few noticeable differences on the different samples of the prepared CMC aerogel based on the different concentrations of CMC present in each sample. The samples with low CMC concentrations (1% and 2%) have an interconnected string-like physical structure that resembles a spider web. These samples also displayed an elastic consistency and were more susceptible to cutting rather than crumbling or breaking. On the other hand, the CMC aerogel samples that have higher CMC concentrations (3% and 4%) have a solid, brittle physical structure that would crumble and break under little pressure. These samples also have fewer gaps in their physical structure than those with low concentrations of CMC.

B. *Analysis of the prepared CMC carbon aerogel*

The prepared CMC carbon aerogel has a solid, brittle consistency and can crumble when placed under minimal pressure. The carbonized CMC aerogel can also be ground into powder easily, which makes it easy to store. A lamellar structure can be observed in the CMC carbon aerogel, which has been prepared by carbonising CMC aerogel [19].

A total of 48 samples of CMC aerogel were carbonized at varying temperatures, which are 300 °C, 400 °C, 500 °C, 600 °C, 700 °C and 800 °C, and for different time parameters which are 1 hour and 2 hours, this resulted in different mass loss in every sample based on the different experimental parameters. The following tables show the mass before the carbonisation, the mass after the carbonisation, as well as the mass loss for each of the 48 samples of CMC carbon aerogel.

C. *The different stages of mass loss of the CMC carbon aerogel*

The mass, as well as the volume of the CMC aerogel, is significantly decreased after the carbonisation, and that is due to the high temperatures of carbonisation, which led to the removal of hydrogen and oxygen atoms [9].

The mass loss in the CMC aerogel occurs in several stages as the temperature increases steadily over time. The first stage of mass loss occurs at 100 °C, and this is due to the residual water evaporating from the CMC aerogel. The mass loss is estimated to be about 5% of the total weight of the sample [20]. The mass loss that occurs at temperatures between 100 °C and 150 °C is due to the water desorption from the CMC aerogel [21].

The second stage of mass loss occurs at approximately 400 °C, and this indicates the start of the pyrolysis of the CMC aerogel samples, which is due to the breaking of the C–O bonds [20]. In temperatures between 390 °C and 400 °C, the weight loss is because of the reactions involving carbonisation and the breaking of C–O bonds [21].

Table 1. Carbonisation of CMC aerogel at 1 hour

% Of CMC	Temperature (°C)	Wt Before (g)	Wt After (g)	% Of mass loss
1	300	0.5776	0.2122	63.26
2	300	0.9221	0.4396	52.32
3	300	0.7552	0.3969	47.44
4	300	0.8795	0.4904	44.24
1	400	0.4822	0.1600	66.81
2	400	0.8135	0.3372	58.54
3	400	0.7571	0.3554	53.05
4	400	0.8588	0.4303	49.89
1	500	0.5199	0.1821	64.97
2	500	0.7149	0.3147	55.97
3	500	0.7372	0.3540	51.98
4	500	0.8347	0.4296	48.53
1	600	0.5044	0.1492	70.42
2	600	0.7323	0.2931	59.97
3	600	0.6889	0.3216	53.31
4	600	0.7794	0.3725	52.20
1	700	0.5290	0.1803	65.91
2	700	0.6624	0.2567	61.24
3	700	0.6152	0.2485	59.60
4	700	0.8614	0.3344	61.17
1	800	0.4567	0.1792	60.76
2	800	0.5602	0.3042	45.69
3	800	0.5142	0.2104	59.08
4	800	0.6579	0.2321	64.72

Table 2. Carbonisation of CMC aerogel at 2 hours

% Of CMC	Temperature (°C)	Wt before (g)	Wt after (g)	% Of mass loss
1	300	0.5180	0.2280	55.98
2	300	0.5408	0.2720	49.70
3	300	0.6420	0.3601	43.90
4	300	0.7453	0.4364	41.44
1	400	0.3826	0.1692	55.77
2	400	0.4782	0.2287	52.17
3	400	0.5810	0.3127	46.17
4	400	0.7421	0.4128	44.37
1	500	0.5518	0.1968	64.33
2	500	0.7152	0.3173	55.63
3	500	0.7395	0.3538	52.15
4	500	0.8116	0.4117	49.27
1	600	0.5408	0.1730	68.01
2	600	0.8221	0.3310	59.73
3	600	0.6052	0.2542	57.99
4	600	0.7671	0.3428	55.31
1	700	0.5284	0.1716	67.52
2	700	0.7287	0.2801	61.56
3	700	0.5830	0.1954	66.48
4	700	0.7786	0.2843	63.48
1	800	0.5559	0.1416	74.52
2	800	0.5339	0.1708	68.00
3	800	0.8505	0.2546	70.06
4	800	0.9282	0.2553	72.49

At 600 °C, the third stage of mass loss occurs due to the breaking of the C–H bonds, which leads the samples of CMC aerogel to turn into carbon, which is amorphous in nature. The carbonisation reaction at temperatures between 590 °C and 600 °C leads to the breaking of C–H bonds which lead to the final stage of mass loss [21]. The last stage of mass loss occurs at temperatures between 750 °C and 800 °C, which signifies the completion of the carbonisation process (The CMC aerogel fully decomposes into CMC carbon aerogel) [20].

Carbonisations at temperatures higher than 800 °C led to a decrease in the total pore volume as well as the surface area. [21] states that at temperatures higher than 800 °C, there is no further mass loss in carbon aerogels.

D. Comparison of mass loss of CMC carbon aerogel based on concentration of CMC

Figure 1 shows a bar graph comparing the mass loss of CMC carbon aerogel at different carbonisation temperatures and varying concentrations of CMC. From the graph, we can clearly observe that the mass loss in the CMC carbon aerogel decreases as the concentration of CMC increases in the temperatures varying from 300 °C to 600 °C and this is due to the physical properties of the CMC aerogels that were previously described, at lower concentrations (1% and 2%) the structure of the CMC aerogel is extremely porous and is made up of interconnected strings, this high surface area and physical structure leads to the decomposition and higher mass loss of CMC aerogels even at lower temperatures [22]. At temperatures of 700 °C and 800 °C we can observe that at 3% and 4%, there is a spike in mass loss which can be attributed to carbonisation process being completed and the CMC aerogel fully decomposing into CMC carbon aerogel [20]. At the higher concentrations of CMC, a higher amount of heat is required to fully carbonise and decompose the CMC aerogel samples. It is due to this, we can observe a spike in the mass loss at temperatures of 700 °C and 800 °C at the concentrations of 3% and 4% [23].

In Figure 2, we can observe a clearer distinction amongst the various temperatures of carbonisation, and that’s due to the longer time parameters (2 hours), which provide more time for a more stable, well-rounded carbonisation of the CMC aerogel. In the temperatures varying from 300 °C to 600 °C the pattern of mass loss follows that of the 1-hour carbonisation and shows a higher mass loss for lower concentrations of CMC (1% and 2%), and that’s because of the physical structure of the CMC aerogel which has a larger surface area at lower concentrations which leads to higher mass loss [22]. At higher temperatures (700 °C and 800 °C), we can notice a spike in the mass loss at higher concentrations (3% and 4%), and that’s due to the final stage of mass loss that occurs, which signifies the full decomposition of the CMC aerogel into CMC carbon aerogel and this means that samples with higher concentration of CMC loss mass at a higher rate than samples with lower concentrations of CMC [23].

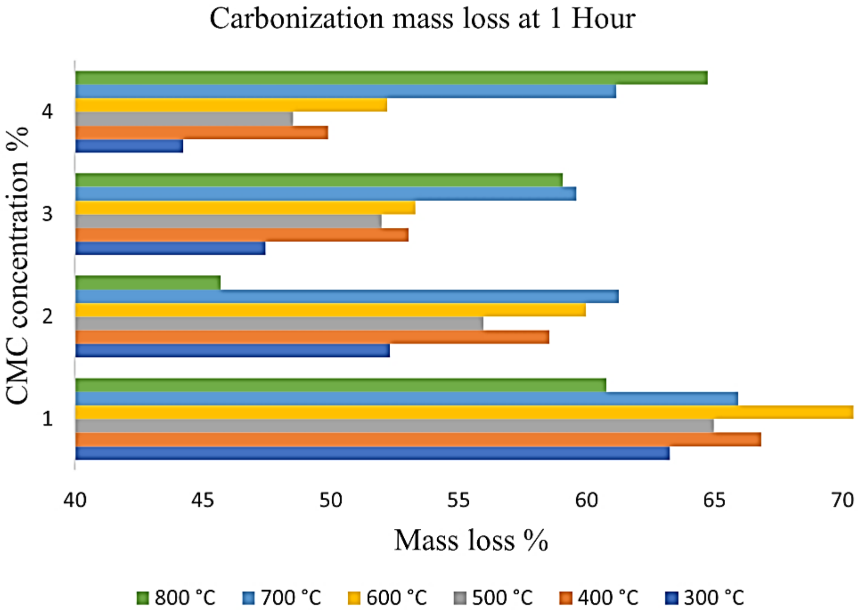

Figure 1. Carbonisation mass loss of CMC carbon aerogel at different temperatures and various concentrations of CMC at 1 hour

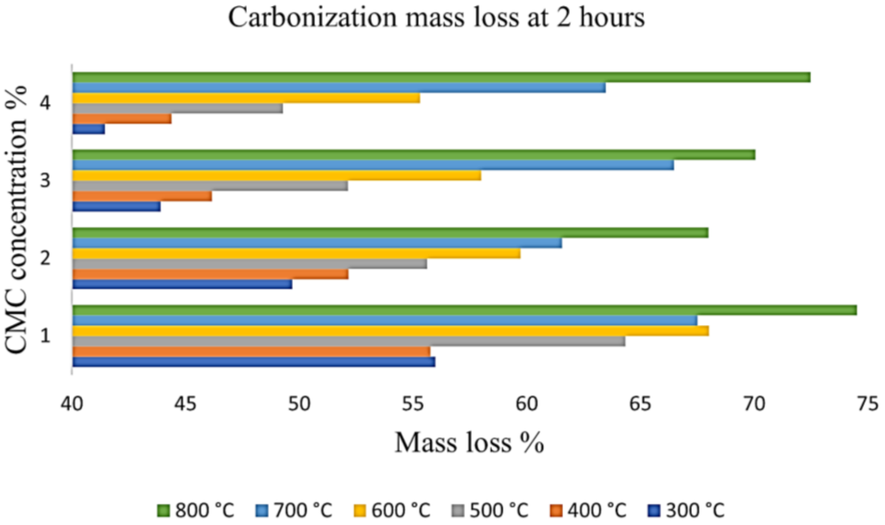

Figure 2. Carbonisation mass loss of CMC carbon aerogel at different temperatures and various concentrations of CMC at 2-hour

V. CONCLUSIONS

Concentration of CMC, duration of carbonisation, and variations in carbonisation temperatures have a significant effect on the mass loss of the prepared CMC carbon aerogel. In temperatures varying from 300 °C to 600 °C, mass loss decreases as the temperature increases, and this is due to the physical properties and characteristics of the CMC aerogel. At higher temperatures (700 °C and 800 °C), the mass loss at higher concentrations of CMC is greater due to the full decomposition of the CMC aerogel into CMC carbon aerogel.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this paper.

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