

The Effect of Impregnation Ratio on the Yield of Corn Cob Activated Carbon by Chemical Activation

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ABSTRACT

Agricultural waste biomass as a precursor for the synthesis of activated carbons has increased in recent years. In spite of transportation cost, agricultural waste biomass is one of an environmentally friendly, readily available and known as renewable sources compared to conventional activated carbon which is very expensive and exhaustible. That is the main reason activated carbon derived from agricultural waste biomass has been widely used in a variety of applications in the environment and industry for the separation, retrieval, modification, and removal of diverse substances in the liquid and gas phases. The purpose of this research is to produce activated carbon (AC) from corncob through carbonization followed by chemical activation with sodium hydroxide. Initially, the corncob was ground and sieved into a powder form with a mesh size of 500 μm . Later, the powder was chemically activated with sodium hydroxide at different impregnation ratios (1:1, 1:2, and 1:4) under activation temperatures of 200 °C. The percentage of carbon yield and ash content were evaluated using a mathematical approach. The results revealed that the sample impregnated at ratio of NCAC 1:4 showed the lowest ash content (6.58 %) with highest carbon yield of 20.52 %. As the impregnation ratios of sodium hydroxide increases, these entrapped chemicals in the activated corncob contributed in high carbon yield and reduced ash content.

Keywords: Activated carbon; chemical activation; biomass; corncob; sodium hydroxide Introduction.



INTRODUCTION

Activated carbon (AC) or also known as activated charcoal, is a crystalline form of carbon described to have small, low-volume pores, high porosity with a large surface area. It also can be recognized as a foam solid with a limited volume of particle structure along with a huge surface area contained within a rigid granule. Balci *et al.* [1] and Mudoga *et al.* [2] revealed that the chemical composition of activated carbon makes it capable of adsorbing organic molecules and other non-polar chemicals from gas and liquid streams.

The first industrially produced activated carbon factory was developed in Germany in the early twentieth century for use in the sugar refining industries. However, in the early 1900s, a slew of new factories sprang up to produce activated carbons, mostly for decolourization and also has been utilized in gas masks during World War I to protect against harmful gases and vapours. Presently, biomass-derived activated carbon has been widely used in the production of porous carbon.

With a highly developed surface area of activated carbon, they are extensively used in many different applications such as removing toxic substances, chemical purification, catalysis, supporting active phases and also as bulk catalysts. Moreover, the application of activated carbons continues to grow through the development of technology with emerging technologies in electrochemical or energy storage applications for use as supercapacitors. Jankowska *et al.* [3] reported that, half a million tons of activated carbon are produced per year worldwide. With 80 percent of these are employed for applications in liquid phase and 20 percent in solid phase [4].

Activated carbon could be derived from different materials such as coal, lignite, anthracite or synthetic macromolecular systems, willow peat, biomass, coir and petroleum pitch. When it comes to raw materials selection, the steadiness of the source is absolutely vital with the cost and quality of the raw materials. Currently, biomass is playing a replacement source for organic fuels, chemicals and next-generation materials. According to Yahya *et al.* [5], corncob is a common biomass waste from agriculture, and one of the most important sources of income for the agro-based

industries. Lignocellulosic materials are a complex composition, and it contains cellulose, hemicellulose and lignin. Corncob primarily consists of structural component content like cellulose, hemicellulose and also lignin, which indicated corncob is potentially and effectively used as a precursor for activated carbon production [6].

The conversion of these materials into a form of activated carbon is straightforward and extensively utilized. It is a favorable environmental technology to convert lignocellulosic biomass into a type of activated carbon. Indeed, it is possible to convert such worthless waste into a useful product by analyzing agricultural waste in the development of activated carbon. Biomass waste also contributes significantly to creating a healthier environment by using activated carbon formed in the treatment of pollutants from liquid and gas streams from home and industrial sources [7,8].

The synthesizing activated carbons can be either physical or chemical activation. The reaction of the carbon structure during the physical activation is created through carbonization with carbon dioxide (CO_2), oxygen (O_2), air, or steam. Typically, physical activation of biomass would consist of one-step or two-step in a single procedure. In a one step process, all samples will undergo heat-treated at the temperature around 200 to 900 °C under the carrier gas. Next, in a two-stage process at high temperatures, biomass is carbonized under an inert atmosphere for a specified period of retention so that the carbon biomass will interact with triggering agents, such as steam, CO_2 or under-heating air [9]. A study from Altintig & Kirkil [10] suggested that around 350-550 °C is the most suitable temperature if the air is employed, and for CO_2 or steam, the suggested temperature is from 800-1100 °C.

In chemical activation, the raw materials are impregnated with certain chemicals agents such as sodium hydroxide, phosphoric acid, potassium carbonate, potassium hydroxide, aluminum chloride, sodium carbonate, zinc chloride, and sulfuric acid [11-13]. The advantages can be seen with an increase in carbonization ability, and the formation of network biomass with desired pore structure. Mostly, activating agents in the form of liquid and solid have been practically used in impregnation process of the biomass. Depending on the structure and conditions, each chemical interacts differently on different parent materials, but they all have an impact on the

carbonization process [14,15].

According to Agriculture Department of Malaysia, in 2019 there reported the production of corn in Malaysia reached up to 58,000 tons. More surprisingly, in the subsequent year, it has increased about 3.45 % to 60,000 tons. Typically, this corn waste is left on the farm or found littering the market streets [16]. Excess waste from corn production, especially corncob waste gives a big impact on the environment, especially disposal problems facing the country. Due to this matter, more attention has been focused on transforming corn waste into valuable products. At the same time, there is insufficient data in the literature which reports on the study of sodium hydroxide as an activating agent in the production of activated carbon. Thus, further investigation regarding the potential of activating agent from alkaline material like sodium hydroxide is crucial.

METHODOLOGY

Experimental work

This section describes the method used in producing activated carbon from agriculture waste (Corncob) collected from local farm around Arau, Perlis. Sweet Corn hybrid 926 was chosen in this study as it is common and widely available. Basically, the preparation of activated carbon includes three processes; pre-carbonization, carbonization and chemical activation. Chemical activation is the main method used in corncob wastes to produce AC. Figure 1 shows an outline of experimental work on the preparation of activated carbon from corncob waste using sodium hydroxide (NaOH) as an activator agent.

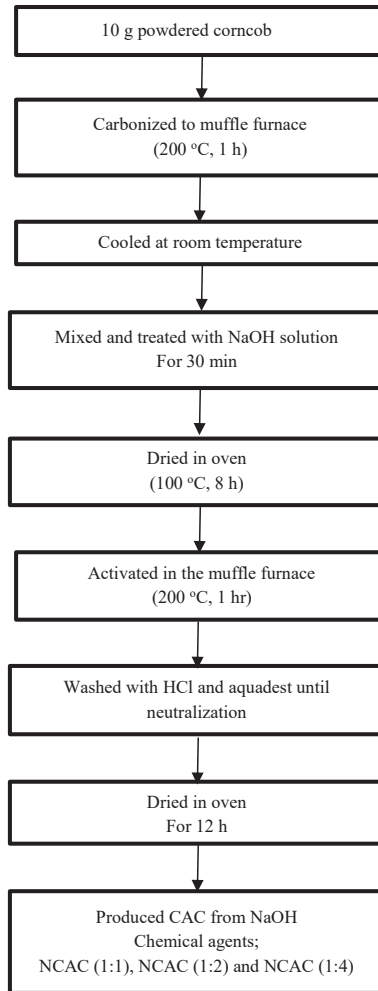


Figure 1: An outline of experimental work

Sample preparation

As shown in Figure 2, the corncob was cut into small pieces and undergo a standard method of oven-drying; particularly at 100 °C for 12 hours to remove the moisture content [17].



Figure 2: Raw materials preparation process

The dried corncobs were tested for moisture content before undergo grinding process. After grinding, the corncob powder was sieved to mesh size of 500 μm and kept in an airtight container before use. Later, the corncob powder was prepared by placing 10 g of powdered corncob in each three separated crucibles and carbonized in a muffle furnace at 200 $^{\circ}\text{C}$ for 1 hour [17]. After carbonization, the produced char as shown in Figure 3 was cooled to room temperature prior chemical activation.

The produced char was impregnated with NaOH solution at room temperature at different impregnation ratios (1:1, 1:2 and 1:4 (Corncob: NaOH)). Impregnation ratio can be defined as the dry weight of powdered corncob to the dry weight of NaOH. Table 1 depicts the dry weight of NaOH diluted to a 50 ml deionized water with respect to the ratio given.

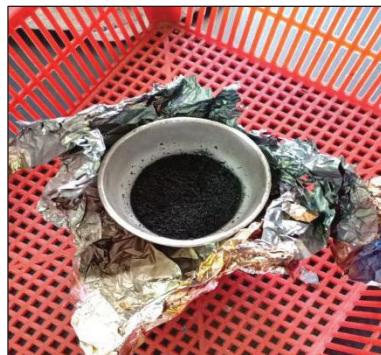


Figure 3: Produced char after the first carbonization phase

Table 1: Dry weight of NaOH being diluted

Ratio	Deionized water (ml)	Weight of NaOH (g)
1:1	50	10
1:2	50	20
1:4	50	40

The char was placed in a beaker for the chemical activation phase, and continuously mixed with the sodium hydroxide solution by using a magnetic stirrer. After mixing, the impregnated sample was left for 45 minutes for pores opening. The solutions were then transferred into the crucible and dried in oven at 100 °C for overnight. Next, the sample was subjected to muffle furnace for 200 °C for 1 hour. As in standard method, the samples that were subjected to high temperatures will activate the carbon by forcing the samples to open up and have more microscopic pores. At this stage, corncob powder have been converted into corncob activated carbon (CAC) [18]. After cooling, the produced corncob activated carbon was then washed with HCl and aquadest to neutralized the CAC. They were rinsed repeatedly to guarantee that no chemical residues remain on the samples. This step ensures no residues of contaminants that might become trapped in the pores of activated carbon. The washed CAC was then oven dried at 100 °C for 24 h that yielded CAC and labelled as NCAC (1:1), NCAC (1:2) and NCAC (1:4), stored in an impenetrable container covered with parafilm to prevent humidity.

Percentage of yield and ash content

CAC yields were measured on a chemical-free basis and can be used as a process efficiency measurement for the chemical activation process. The carbon yield percentage was determined from the final weight of the resulting corncob activated carbon after carbonization at 100 °C for 1 hour divided by the weight dried corncob. From equation (1), the yield of CAC can be easily calculated [19].

$$Yield(\%) = \left(\frac{\text{Weight of AC after carbonization}}{\text{Weight of the dry raw material}} \right) \times 100 \quad (1)$$

The ash content can be determined by placing 10 g of powdered corncob in a weighing boat. Next, the sample was transferred into a crucible

to be subjected to the muffle furnace at 200 °C for 1 hour, then was cooled at room temperatures and reweighed. Hence, the percentage of ash content can be determined using equation (2) as follows [20];

$$Ash (\%) = \left(\frac{Weight\ of\ sample\ after\ ash\ process}{Weight\ of\ sample\ before\ ash\ process} \right) \times 100 \quad (2)$$

RESULTS AND DISCUSSION

Table 2 depicts the result of the average carbon yield and ash content for each activated carbon samples. It shows that the concentration of NaOH played a major role in the preparation of AC. Based on the data, the carbon yield (%) of the NaOH activated samples increased from 13.62 to 20.52 % as the impregnation ratio increases from 1:1 to 1:4. Carbon yield showed an increment of 34.43 % as the impregnation ratio increases from 1:1 to 1:2. Meanwhile, 12.07 % enhancement of carbon yield was observed when the impregnation ratio increases to 1:4. The percentage of yield increases as the concentration of NaOH increases. Higher fixed carbon content of activated CAC with greater impregnation ratio reflects a corresponding reduction in the ash content of the samples from 11.44 % (1:1) to 6.58 % (1:4). This is because the chemical activator, NaOH acts to protect the internal carbon structure and prevents the product from excessive burn-off [5].

Table 2: Percentage of yield, ash content and its parameter

Sample code	Particle size (µm)	Percentage of yield (%)	Ash content (%)
NCAC 1:1	500	13.62	11.44
NCAC 1:2	500	18.31	9.22
NCAC 1:4	500	20.52	6.58

Figure 4 compares the percentages yield of carbon and ash content under the effect of different impregnation ratios. Referring Figure 4, the yield increases at higher impregnation ratio. Sample NCAC 1:4 showed the highest percentages yield up to 20.52 % compared to other samples achieved at 1:1 (13.62 %) and 1:2 (18.31 %). To sum up, a greater impregnation ratio of sodium hydroxide will create more of these samples in higher yields. During NaOH activation, the process resulted in more potential locations penetrating and occupying the activating agent. This improves the opening and expansion of pores. A similar result was reported by Anisuzzaman et

al. [21] during the synthesis of AC from cattail leaves.

In contrast, the percentages of ash content (Figure 5) showed an opposite trend with an increasing in impregnation ratio. Sample NCAC 1:4 exhibited lowest ash content of 6.58 %. According to a study conducted by László, K *et al.* [22], some of the chemicals that remained in the activated corncob after washing contributed to the ash level in the CAC. In other words, when the NaOH impregnation ratios increases, the entrapped chemicals in the corncob were activated and contributed to the final product having a high ash content and a low surface area [23].

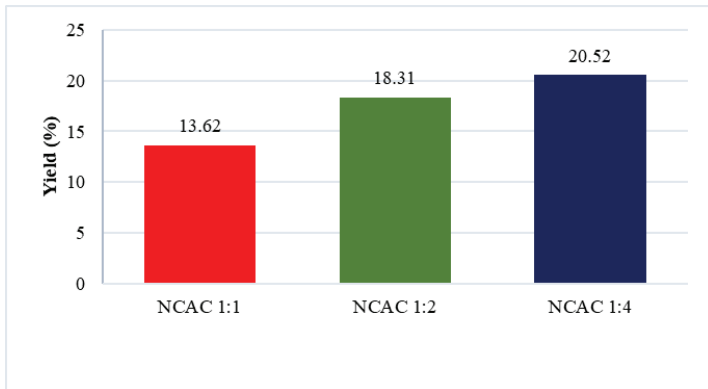


Figure 4: Carbon yield at different impregnation ratios

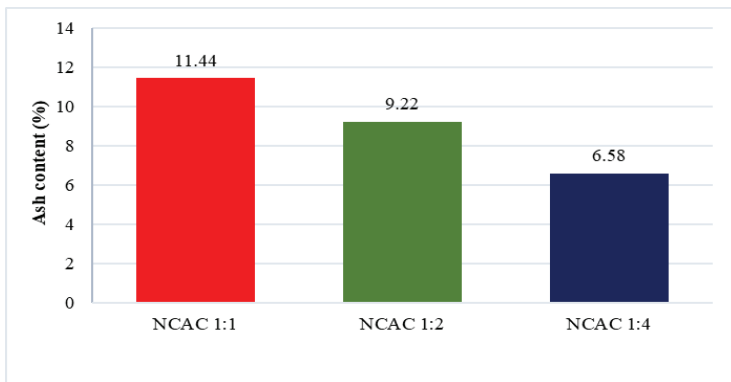


Figure 5: Ash content at different impregnation ratios

CONCLUSION

This study shows that corncob waste serves as a precursor for the preparation of activated carbon due to its high concentration of carbon. In this work, the chemical activation technique was chosen, employing sodium hydroxide (NaOH) as the activating agents. The corncob was impregnated with NaOH at different mass ratios (1:1, 1:2, 1:4) and undergo the activation process. Based on the chemical analyses of the resultant carbon, the percentage of carbon yield and ash content were noticed to yield different results on the effect of the impregnation ratios. The highest carbon yield was obtained at 20.52 %, while the lowest ash content value was at 6.58 %. High percentage yield in corn cob activated carbon was due to the NaOH activation, which caused more potential sites to penetrate and occupy the activating agent. Thus, it enhances pore opening and widening. In terms of economic point of view, increasing the impregnation ratio of NaOH is still consider cost effective as it is preferred by the industry because of its lower cost compare to other chemical agents. In summary, it can be concluded that the chemical properties of the final corncob activated carbon are substantially impacted by both the parameters and the production method used during the preparation

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