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Activated Carbon from Rice Husk with Various Activators for Lithium-Ion Battery Cathode Material Additive

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Abstract. Activated carbon is needed as an additive in the cathode and anode materials of the battery. Rice husk is a carbon source that allows it to be synthesized into activated carbon using an activator. Measurements using a simultane thermal analyzer (STA) showed that changes in the thermal pattern of rice husks occurred at temperatures of 400°C and 700°C. Dry and clean rice husks were carbonated at 400°C for 2 hours, activated with HCl, H3PO4, NaOH, or ZnCl2, then calcined at 700°C for 2 hours. The crystal structure analysis by X-ray diffraction (XRD) showed that carbon was formed and the results of morphological observations using a scanning electron microscope (SEM) showed the formation of activated carbon with an average pore diameter of 15 mm. The results of measurements using the impedance capacitance resistance meter showed the highest value 6.48x10-4 S.cm-1 was found in the use of 0.1 M NaOH activator, showing the most effective activator in comparison to other activators.

INTRODUCTION

Battery technology is developing very rapidly, both related to battery applications and battery component materials [1]. There are various forms of batteries such as coin, cylindrical, pouch and prismatic [2]. Lithium-ion battery is a battery that is functional in people's lives. Batteries are used in a variety of electronic devices and vehicles from laptops and cell phones to hybrids and electric cars [3-4]. This technology is increasingly popular because LIB some advantages of lithium-ion batteries such as: light weight, high energy density, and good ability to recharge [5]. LiNMC (lithium nickel manganese cobalt) type batteries are widely used for cars, motorcycles, and industrial vehicles [6]. The main components of a battery consist of a cathode, anode, separator, and electrolyte. Activated carbon in battery technology is used as an additive to cathode and anode materials [7]. Although its use is not much, this material is very important in improving battery performance, especially in terms of conductivity and capacity. Most research interest focuses on the new LiNi1-x-yCoxMnyO2. system (0< x<1.0<y<1) with NaFeO2 structure as cathode material, which is promising to replace LiCoO2. LiNi0.5Co0.2Mn0.3O2 (NCM-523) has a very good initial discharge capacity [8], but a higher at potential conditions there is a decrease in capacity, so it cannot be widely applied.

To get a good cathode material performance, activated carbon is added to the cathode core material. The addition of activated carbon can increase the capacity and conductivity of cathode and anode materials [9], so it is important to synthesize activated carbon with good quality. Activated carbon can be obtained from various biomass such as rice husk [10-11].

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Rice husk (RH) is an agricultural waste that is abundantly available in rice producing countries [12]. RH contains 80% volatile organic matter and the rest is silica. The chemical composition of RH ash varies from sample to sample which may be due to differences in geographical conditions, rice types, climatic conditions and types of fertilizers used [12]. Activated carbon has good adsorption property, can be produced from various raw materials, such as packaging paper, plastic bottles by-products of the wood industry (e.g. sawdust), straw, rice husks, seeds or bark of wood fruit sawdust, bark seeds, rice husks, shells, peat, bagasse, coal, lignite and animal bones [13-14].

Activated carbon has been used in industrial fields (water treatment, food and beverage, cigarettes, chemicals, soaps, scrubs, shampoos, paints and adhesives, masks, refrigeration equipment, automotive), in the health sector (absorb toxins in the digestive tract and pharmaceuticals).), environmental fields (absorb metals in wastewater, absorb pesticide residues in drinking water and soil, absorb gas emissions in the air, increase total organic soil, reduce microbial biomass and soil aggregation), fields (plant propagation by tissue culture, fertility of plant media , and prevent root rot), the field of renewable energy (as a battery electrode additive to increase the value of electrical conductivity both ionic and electronic)[15-16].

The process of making activated carbon is carried out in two stages. The first stage is the process of carbonization of raw materials in the kiln drum to produce carbon. The second stage is the carbon activation process to remove the hydrocarbons that coat the carbon surface thereby increasing the porosity of the carbon. The stages that occur in both processes are dehydration (the process of removing air), carbonization (the process of decomposition of organic cellulose into carbon elements, and the release of non-carbon compounds), activation (the process of formation and arrangement of carbon becomes larger) [17].

This work arms to synthesize activated carbon from rice husk with various activators and characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), and impedance capacitance resistance (LCR) meter.

METHODOLOGY

The materials and tools used in this experiment were rice husk, aquadest, HCl, NaOH, $ZnCl_2$, H_3PO_4, filter, oven, furnace, XRD, SEM, LCR-meter. Synthesis of activated carbon on rice husk material begins with washing using aquadest then drying at 105ºC for 24 hours using an oven. Dry husks were carbonated in a tube furnace at a temperature of 700ºC for 1 hour. The resulting carbon was then milled at 25 Hzs for 6 hours and sieved with a size of 230 mesh. Activation is done by soaking the carbon for 2 hours, then neutralized by washing using aquadest using the centrifuge method. After drying at 105ºC for 6 hours, the powder of activated carbon was obtained. Figure 1 shows the flow diagram of activated carbon synthesize from rice husk.

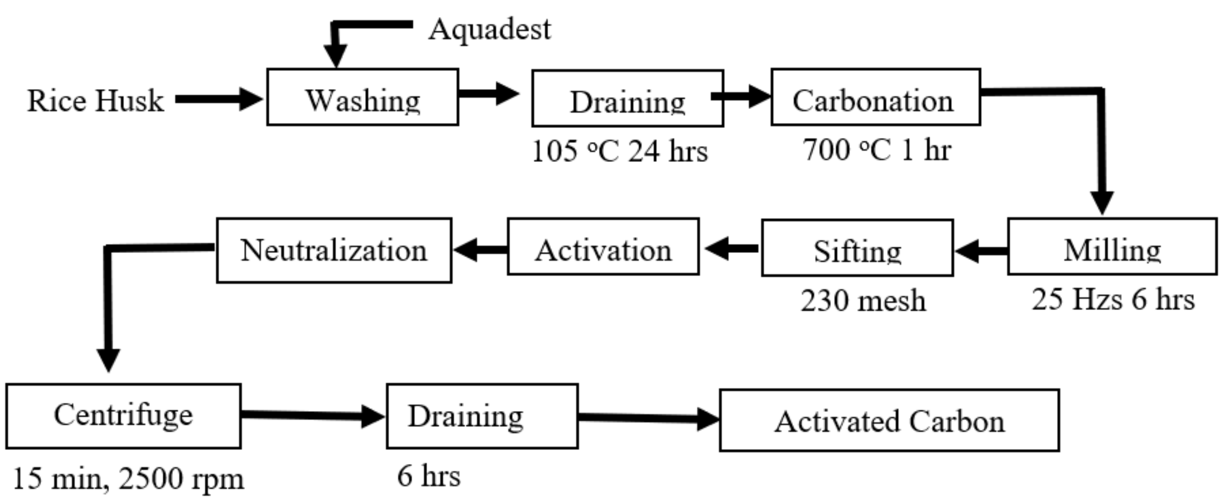

FIGURE 1. Flow Diagram of activated carbon synthesis from rice husk.

RESULTS AND DISCUSSION

There are various types of rice grown in Indonesia, the chemical composition of rice husks is also influenced by the conditions of the area where the rice is grown. The rice husks used as samples in this study came from the Tangerang area, Banten, as shown in Figure 2.

FIGURE 2. Rice Husk.

To obtain information on the behavior of rice husks to temperature changes, measurements were carried out using a simultaneous thermal analyser (STA). The results of STA measurements on rice husk samples are shown in Figure 3. The measurement results show that there is a significant change in mass at a temperature of 99.7-151.0°C, this indicates a process of evaporation of water contained in and on the surface of rice husks. At around temperature 462-700°C there is a decrease in mass this is due to the formation of carbon. Meanwhile, at a temperature of 700°C, there was no change in the mass of the sample at an increase in temperature of up to 1000°C, indicating that the carbon formation was optimum.

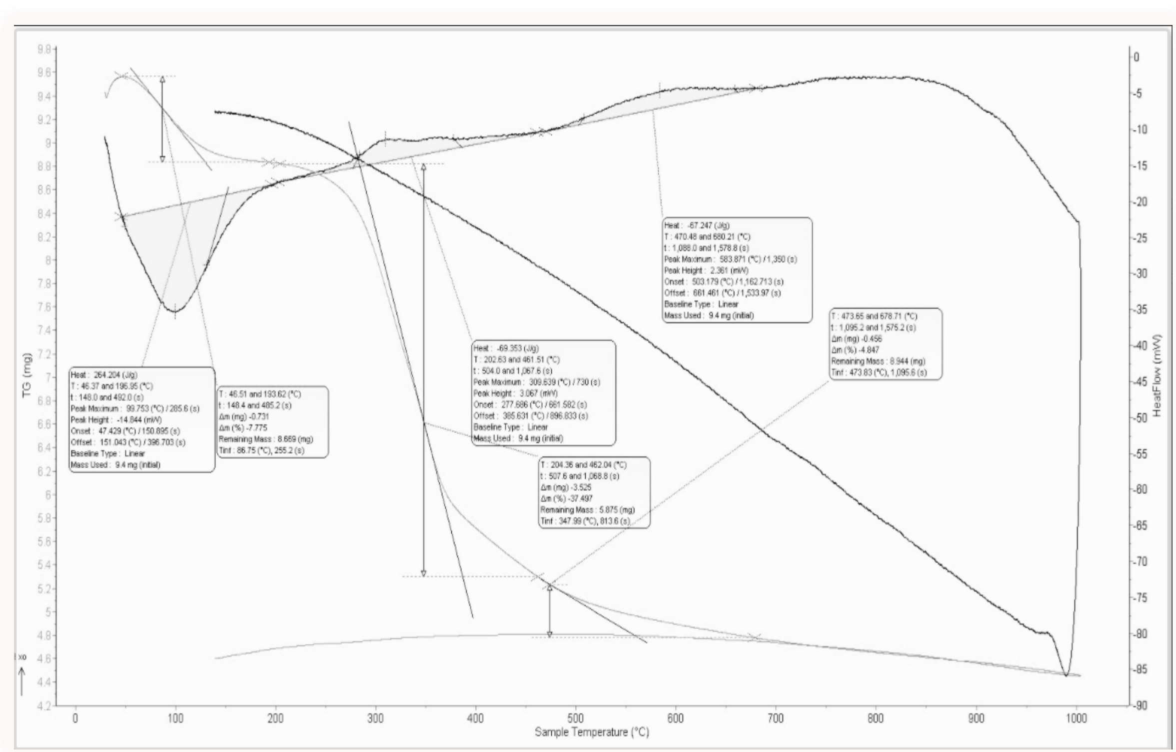

FIGURE 3. The results of the simulation thermal analysis of rice husks.

Analysis using X-ray diffraction (XRD) on rice husks before and after drying is shown in Figure 4 (a), showing that there is no difference in the diffraction peaks. This indicates that the rice husks used as samples did not contain any contaminants. Meanwhile, Figure 4 (b) shows the X-ray diffraction pattern from the carbonation which shows the formation of specific peaks of carbon. This supports the results of measurements using STA that at 700°C carbon is formed.

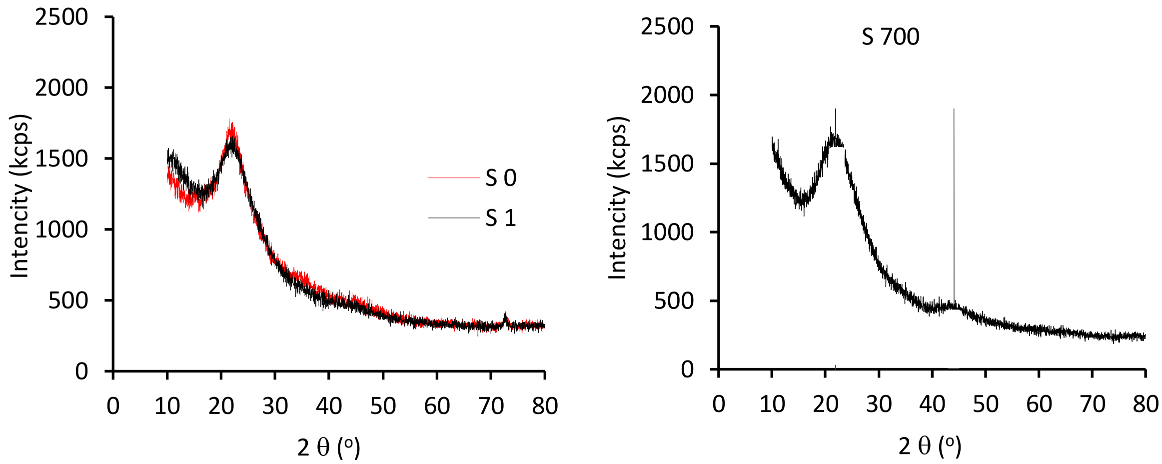

FIGURE 4. (a) The results of X-ray diffract-tion measurements of rice husks before (S0) and after (S1) washing and (b) X-ray diffraction pattern of carbonized rice husks.

Figure 5. show the results of measurements using X-ray diffraction on samples that have been activated using HCL, NaOH, $ZnCl_2$, and H3PO4 showed that the diffraction peaks indicate that all carbon activated with various activators still forms a carbon structure, although there is a slight shift in peaks in the activation results using NaOH, $ZnCl_2$, and H_3PO_4, while the results of activation using HCl have the same diffraction pattern as the diffraction pattern of activated carbon produced as a reference.

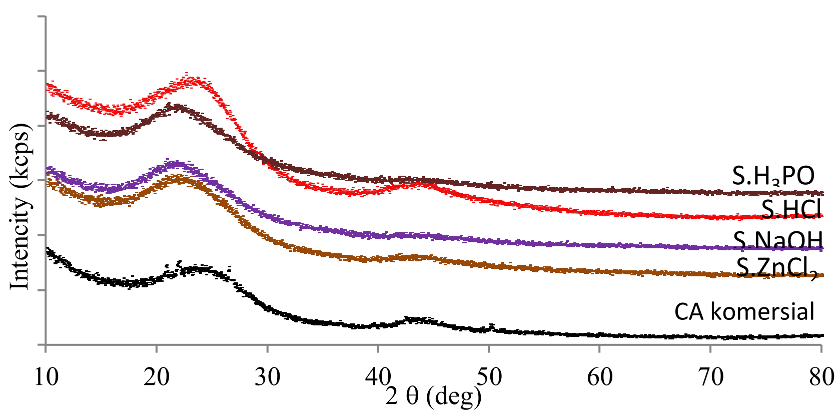

FIGURE 5. X-ray diffraction pattern of rice husk with various activators carbonized.

Morphological observations carried out using a scanning electron microscope (SEM) as shown in Figure 6. show that on the surface of dry rice husks there is a layer that covers the pores or the surface pores are too small so that they are not visible.

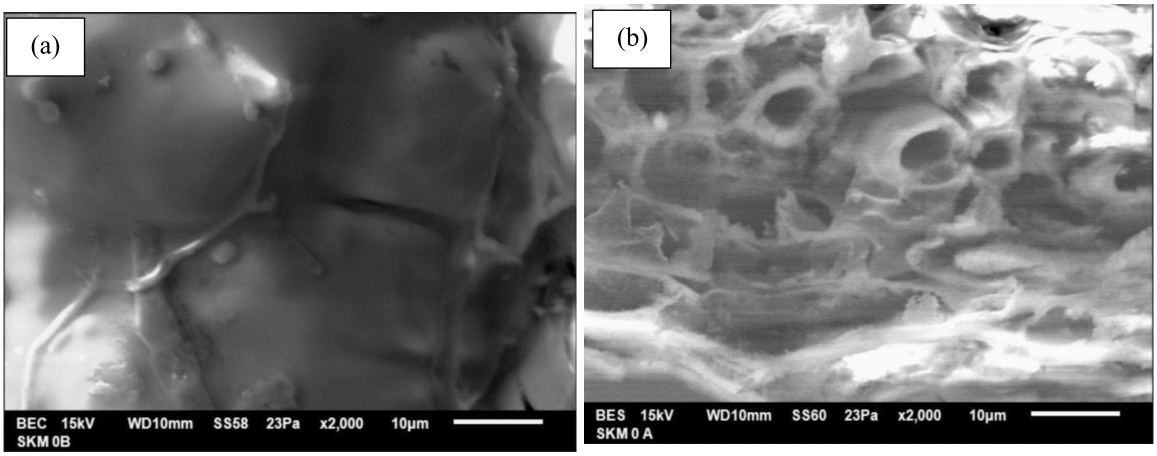

FIGURE 6. Scanning electron microscope observation of rice husk (a) before carbonization and (b) after carbonization

This is different compared to rice husks that have been carbonated, it is seen that there are pores with an average size of 5 μm. In the battery system, these pores function to accumulate lithium ions both at the cathode and the anode, so that a wider pore size is expected, thus facilitating the migration of lithium ions and increasing the amount of lithium adsorbed. For this reason, other treatments are needed to expand the pore size, one way is by using chemicals. HCl, NaOH, $ZnCl_2$, and H_3PO_4 were used in this study, each with a concentration of 0.1 M.

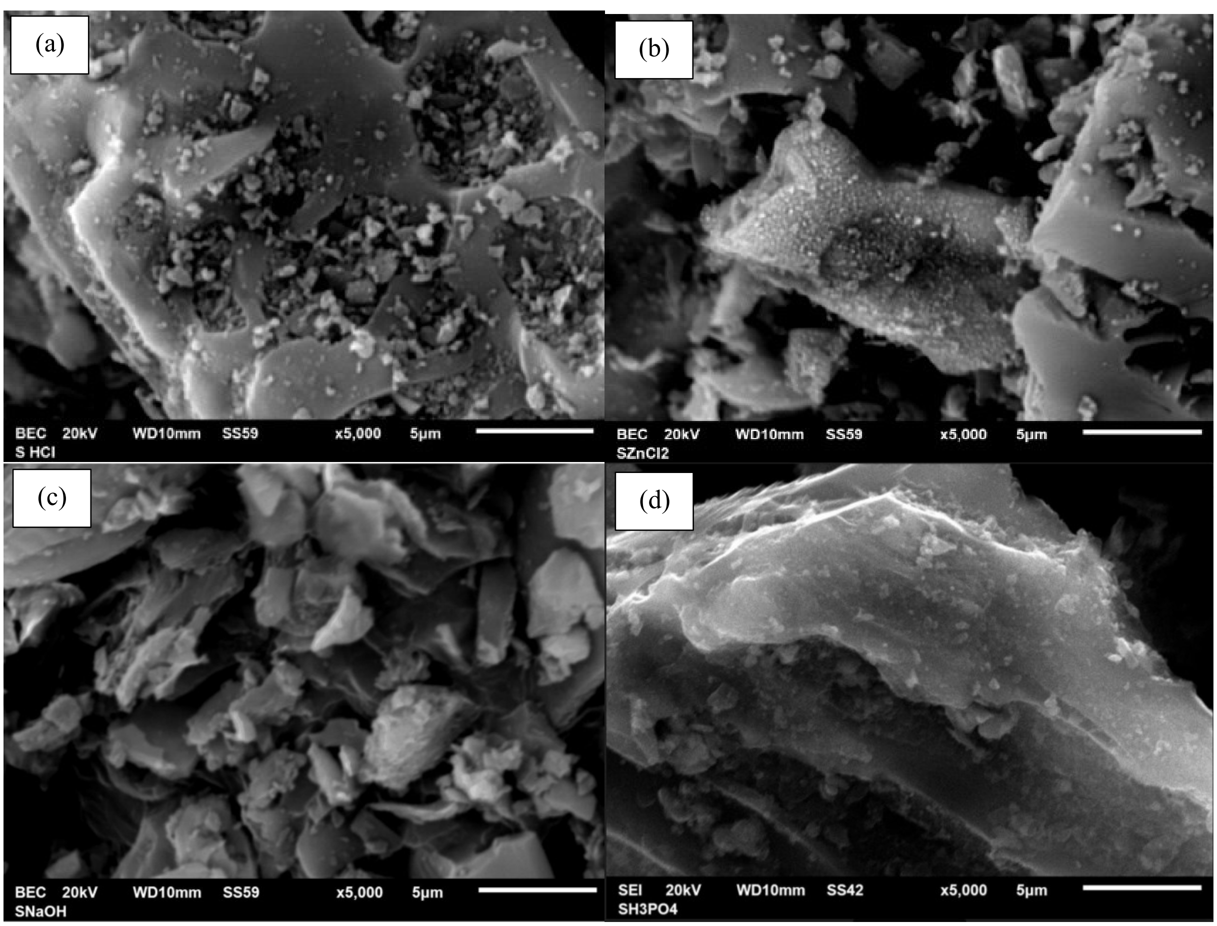

FIGURE 7. Observation of the carbon morphology of rice husk activated using (a) HCl (b) NaOH (c) $ZnCl_2$ (d) H_3PO_4.

Observations on the carbon morphology of rice husks activated using HCl, NaOH, $ZnCl_2$, and H3PO4 are shown in Figure 7. It shows that there is a significant pore enlargement compared to before activation. The average pore size of activated carbon as a result of activation is shown in Table 1.

TABLE 1. Effect of activator on pore size

Activators	**Average pore size after activation**
HCl	25 mm
$ZnCl_2$	12 mm
NaOH	3 mm
H_3PO_4	20 mm

Measurement of conductivity using an impedance capacitance resistance meter is shown in table 2. It can be seen that the conductivity of rice husks is the lowest and will increase twice after carbonation and will increase again after activation. Activated carbon material from rice husk which is activated using NaOH has a conductivity value of 6.4834×10^{-4}, this value is the highest compared to the results of activation with other activators.

TABLE 2. Conductivity values of the samples

Samples	**Conductivity ($S.cm^{-1}$)**
Rice Husk	8.6099×10^{-11}
Carbonation Results	3.0179×10^{-5}
HCl activator	4.1986×10^{-4}
H_3PO_4 activator	1.7943×10^{-4}
NaOH activator	6.4834×10^{-4}
$ZnCl_2$ activator	1.0130×10^{-4}

CONCLUSION

Activated carbon has been successfully synthesized from rice husks through the process of carbonation and activation using activator ingredients chloric acid, zinc bichloric, sodium hydroxide, or phosphoric acid. The use of NaOH as carbon activator from rice husk in this study was the most effective.

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