Asian Journal of Advanced Research and Reports

Volume 17, Issue 2, Page 39-45, 2023; Article no.AJARR.96308
ISSN: 2582-3248

Test Bituminous Coal Activated Carbon by Use Hydrochloric Acid (HCl) Activator as Electrode Material

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Author's contribution

The sole author designed, analysed, interpreted and prepared the manuscript.

Article Information

DOI: 10.9734/AJARR/2023/v17i2468

Open Peer Review History:
This journal follows the Advanced Open Peer Review policy. Identity of the Reviewers, Editor(s) and additional Reviewers, peer review comments, different versions of the manuscript, comments of the editors, etc are available here: https://www.sdiarticle5.com/review-history/96308

Original Research Article

Received: 08/12/2022
Accepted: 14/02/2023
Published: 22/02/2023

ABSTRACT

Activated carbon is widely used in various electrochemical applications, one of which is an electrode material. The price is low, and the essential ingredients are easily obtained from various natural materials. The material properties of carbon are very strongly influenced by the method of manufacture. Thus, the selection of synthesis procedures, type of precursor, type of activator, heating rate, and temperature of combustion or pyrolysis makes it easy to control the final product/porous carbon but can also design the type of carbon targeted. Supercapacitor electrodes are a technology developed from conventional capacitors. In this study, bituminous coal was used to turn activated carbon using hydrochloric acid (HCl) activator. This study aimed to determine the characteristics and potential of activated carbon from bituminous coal using hydrochloric acid (HCl) activator as a base material in the manufacture of supercapacitor electrodes. The coal sample is converted into activated carbon, starting with preparing the coal through the carbonization process. Furthermore, the coal powder was crushed and sieved to obtain a uniform size. The coal powder was immersed in the HCl solution, and the HCl solution's concentration varied by: 2.5 M; 3M; 3.5M; 4M; 4.5 M done in duplo. Soaking was carried out for 12 hours, the solution was filtered, and the

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coal residue was activated using a furnace for 1 hour. The results obtained show that activated carbon has the best concentration in characterizing, namely the concentration of 3.5 M (duplo) with water content, ash content, volatile matter content, bound carbon content, and iodine number comply with SNI standards. 340.1168 mg/g, degree of crystallinity at an angle of 2θ.

Keywords: Hydrochloric acid (HCl); supercapacitor electrodes; bituminous coal; coal activated carbon.

1. INTRODUCTION

The potential for energy and mineral resources in Indonesia is quite significant. Mining materials are still an attraction in Indonesia, namely coal, which can be used as a primary energy source for the community. One of the fossil coal materials applications is supercapacitor electrodes that can be used in society. There are 20 provinces of coal resources in Indonesia, one of which is in the Lamuru region, Bone district, South Sulawesi province, which can be used as mining products in the industrial sector [1].

The management of coal mines in the Lamuru region has existed since 1978, and permits have been granted since 2008. Based on the results of geoelectrical resistivity measurements and geological data substantiation, it is stated that there are variations in the resistivity values of coal in the Lamuru area, Bone Regency, which are classified as having low resistivity values, which range from 10 – 20 ohms so that the classification is included in the bituminous type [2]. Bituminous ($C_{80}OH_5O_{15}$) is a type of coal with physical characteristics of a shiny black color, which has a caloric content of between 5833 kcal/kg – 7777 kcal/kg, with the element carbon (C) 68% – 86%, a little ash content, and a moisture content of 8% – 10% of its weight. Therefore, this type of coal can be used as a raw material for making activated carbon [3].

The process of coal formation is influenced by temperature and pressure, which contains elements such as carbon, hydrogen, and sulfur because coal is a heterogeneous substance that burns and is formed from many components with different properties. The formation process takes a long time to form coal in a tertiary manner. In addition, coal is formed through a deposition process, and there will be a peat and coalification process which is influenced by microbes, temperature, time, and pressure to produce coal with different contents according to the factors that influence it [4].

The raw material for making activated carbon can come from fossil fuels in the form of coal. Production of activated carbon from raw materials resulting from these fuels will encourage the use of coal, which is currently felt to be limited. The structure of activated carbon consists of a microcrystalline structure consisting mainly of free carbon, usually obtained by special treatment and has a high absorption capacity. Therefore, researching manufacturing coal-based activated carbon is necessary to obtain operating conditions that provide maximum recovery and quality [5].

There are three stages of making activated carbon: the dehydration process, the carbonization process, and the activation process. The dehydration process is carried out by heating to a temperature of 170°C. The carbonization process is carried out by burning raw materials using limited air with temperatures between 300°C and 900°C [5]. Furthermore, the activation process is a process of treating carbon to open pores on carbon. The activation process can be done using physical or chemical activation [5]. This chemical activation process involves mixing the original material with KOH, $ZnCl_2$, H_3PO_4, H_2SO_4, HCl, and others. Physical activation involves using CO gas_2 and water vapor or a mixture of the two. The steps for making activated carbon can be applied to electrochemical applications [6].

The use of activated carbon occurs in many electrochemical applications, one example of which can be used as an electrode material. It is because the raw materials for manufacture are easy to obtain from various types of natural materials, easy to synthesize, and can be obtained in the form of powder, fiber or fibers and composites, large surface area, and adjustable pores. Carbon electrodes are easy to polarize, stable in different solutions such as acids, bases, and aprotic, and stable over a specific temperature range. Carbon electrodes can be used as a raw material for making supercapacitors [7].

Supercapacitors utilize the electrode surface and electrolyte solution to achieve large capacitance. Supercapacitors are also more interesting because the electrodes' primary material is activated carbon. Activated carbon can be made from various raw materials with relatively high carbon content, such as fossil fuels, namely bituminous coal [8]. The devices for making supercapacitors include electrodes, separators, electrolytes, and charge collectors because supercapacitors are based on an electrochemical mechanism that stores electrical energy directly as a charge. Its performance can be seen by the immense power density and energy density depending on the nature of the electrodes and electrolytes [9].

Supercapacitors promise a large energy storage capacity due to their high-power density, high energy density, and long cycle life. In making supercapacitors, characterization is needed first. The characterization is carried out by using the method of examining the composition of the mineral content of coal obtained from coal mining results of analyzing the quality of activated carbon so that the quality of the activated carbon is known in bituminous coal by analyzing water content, ash content, volatile matter content, bound carbon content, as well as iodine numbers [5].

In previous studies analyzing the quality of activated carbon in lignite-type coal, the resulting water content ranged from 5.83–9.16%. Ash content ranged from 6.66 – 8.33% and volatile matter content ranged from 20 – 21.66%, bound carbon content ranged from 70 – 73.33%, and iodine absorption ranges from 1,205.65 - 1,827.50 mg/g using hydrochloric acid (HCl) activator which meets the Indonesian National Standard (SNI) for requirements activated carbon. The effect of variations in concentration dramatically affects the quality of activated carbon for the value of water content and iodine absorption because the higher the concentration of the activator used, the water content in the activated carbon decreases, and the absorption of iodine increases [10].

2. RESEARCH METHODS

2.1 Tools and Materials

The tools used in this research are Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), Potassium Iodide (KI), Filter Paper, Iodine Solution (I_2) 0.1 N, Sodium Thiosulfate Pentahydrate ($Na_2S_2O_3.5H_2O$), Sodium Bicarbonate ($NaHCO_3$), Tissues and Water.

2.2 Powder Structure Crystallinity Test of Activated Carbon Using *X-Ray Diffraction* (XRD)

Furthermore, characterization tests were carried out using the X-Ray Diffraction (XRD) instrument to determine the crystallinity of a material, namely bituminous coal from the Bone Regency area, related to the ability to absorb hydrogen from activated carbon products resulting from chemical and physical activation. Then the instrument was operated at 40 kV, 30 mA, using radiation at speed based on observations of the degree of crystallinity (2θ) per minute [11].

2.3 Activated Carbon Surface Morphology Test Using Scanning Electron Microscopy (SEM)

The morphology test on the surface of the activated carbon using a Scanning Electron Microscopy (SEM) instrument produced an electron beam by an electron gun which was focused on one point on the surface of the sample (activated carbon of bituminous coal) by two condenser lenses (objective lens). Then the electrons are focused into a minor diameter of about 10-20 nm. Then, the pores and surface properties of activated carbon were clearly described using Scanning Electron Microscopy (SEM) [10].

3. RESULTS

The quality of coal-activated carbon is an analysis to determine the quality of coal-activated carbon by looking at several parameters contained in the Indonesian National Standard (SNI 06-3730-1995) for activated carbon. The analysis was carried out in simplo and duplo ways using bituminous coal activated carbon samples.

3.1 X-Ray Diffraction (XRD) Test

The results of testing the activated carbon of bituminous coal using X-Ray Diffraction (XRD) can be seen in Table 1.

Table 1. Results of XRD testing of bituminous coal activated carbon

HCl concentration (M)	Degree of Crystallinity of Activated Carbon (2θ)	Peak Crystallinity of Activated Carbon (Å)
2.5M (duplo)	35.4912	2.52940
3M (duplo)	26.6389	3.34647

Table 2. SEM test results for bituminous coal activated carbon

HCl concentration (M)	Activated Carbon Pore Diameter (µm)
2.5M (duplo)	3.56
3M (duplo)	4.91

The above results show that the degree of crystallinity obtained from concentrations of 2.5 M and 3 M (duplo) has a degree of crystallinity of 35.4912 and 26.6389, respectively.

3.2 Scanning Electron Microscopy (SEM) Test

The results of testing the activated carbon of bituminous coal using Scanning Electron Microscopy (SEM) can be seen in Table 2.

The above results show that the pore diameters obtained from concentrations of 2.5 M and 3 M (duplo) have macro-sized pore diameters of 3.56 µm and 4.91 µm, respectively.

4. DISCUSSION

The chemical activation process begins with weighing a sample of coal carbon powder to obtain the weight of the sample. After the weighing is complete, make reagents with five different concentration variations. The reagent or activator used was hydrochloric acid (HCl) activator with a concentration of 2 M; 2.5M; 3 M; 3.5 M; 4 M in simplo and duplo. The choice of the activator material is to expand the cavity or pore volume of the activated carbon because the activating molecules will dissolve the impurities in the carbon pores. After the manufacture of the hydrochloric acid activator (HCl) was completed, the samples of coal carbon powder were soaked and allowed to stand for 8 hours. The purpose of the immersion is to remove impurities present in the sample. The soaked sample was then filtered using filter paper and washed using distilled water until the pH became neutral. After being neutral, it is then dried using an oven so that the sample becomes an activated carbon powder, and then proceed with the physical activation process.

The physical activation process is carried out with temperature variations of 700℃ and 800℃ to expand the charcoal's pore structure, removing volatile substances and removing tar or impurities in the charcoal. As stated by Amir et al. [12] that physical activity is an activation process carried out at high temperatures and the help of CO_2 as well as to break the carbon bonds of organic compounds

4.1 Characterization of Crystallinity of Coal Activated Carbon Structure Using X-Ray Diffraction (XRD)

Characterization of coal-activated carbon using X-Ray Diffraction (XRD) is a test carried out to determine crystal size, lattice strain, chemical composition, and other conditions that have the same order [13]. The selection of samples in this test is based on the results of the optimum and minimum conditions when testing the quality of activated carbon to compare the characterization of activated carbon. Data from the results of the characterization test using the XRD instrument can be seen in Figs. 1 and 2.

The optimum condition for the sample based on the quality of activated carbon lies in an activated carbon sample with a concentration of 3 M (duplo) at 700℃. While the minimum condition sample lies in an activated carbon sample with a concentration of 2.5 M (duplo) at 700℃. The XRD characterization results curve indicates that the carbon yield obtained has an amorphous structure in the activated carbon sample with a concentration of 3 M (duplo) while the carbon yield obtained in the activated carbon sample with a concentration of 2.5 M (duplo) has a crystalline and amorphous structure. Oko et al. [14] state that an irregular amorphous structure causes sloping peaks in amorphous while an ordered crystal structure causes sharp peaks of crystals.

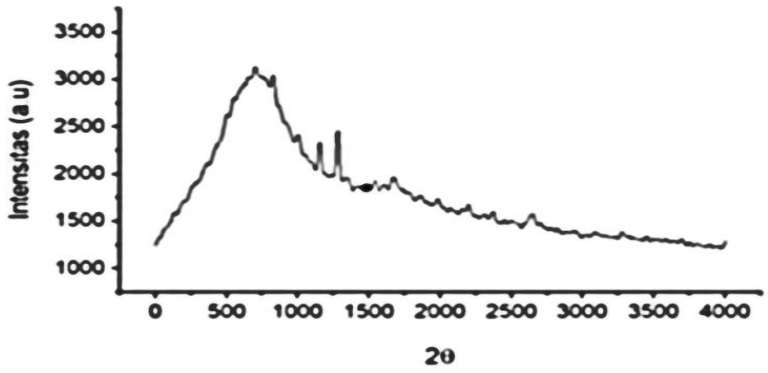


Fig. 1. Activated carbon crystallinity curve using x-ray diffraction temperature 700℃

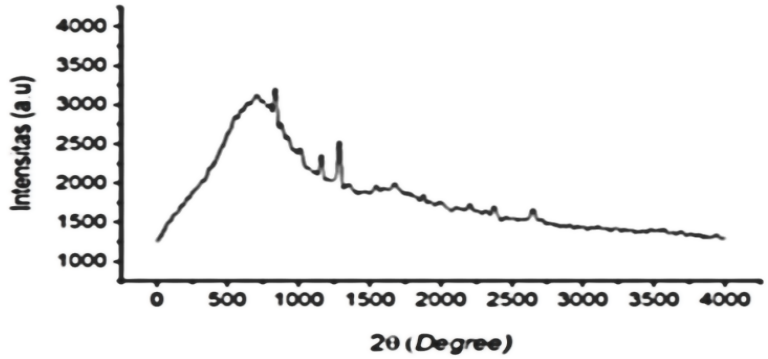


Fig. 2. Activated carbon crystalline curve using x-ray diffraction temperature 800℃

From the data above shows measurements using instruments *X-Ray Diffraction* (XRD) obtained a value of 2θ with a degree of crystallinity of 35.4912º at a concentration of 2.5 M at a temperature of 700℃ (minimum sample conditions) with a crystallinity peak of 2.52940 Å where the activated carbon sample contains a chemical compound, namely 4-chloro-o-anisinic acid ($C_8H_7CLO_3$) and 1,2,3,7,8,9-hexachlorodibezo-p-dioxin ($C_{12}H_2CLO_2$). At the same time, the activated carbon sample with a concentration of 3 M at 700℃ has a degree of crystallinity with a value of 26.6389º(optimum sample conditions) with a crystallinity peak of 3.34637 Å where the activated carbon sample contains a chemical compound, namely diclone ($C_{10}H_4cl_2O_2$) and sodium hydroxide hydrate ($NaOH.nH_2O$).

It can be seen that there are differences in the two samples, namely that there are more carbon atoms in the activated carbon sample with a concentration of 2.5 M (duplo) compared to the carbon atoms in the sample with a concentration of 3 M (duplo). It indicates that the more carbon atoms, the easier it is to heat up because carbon atoms have a high boiling point.

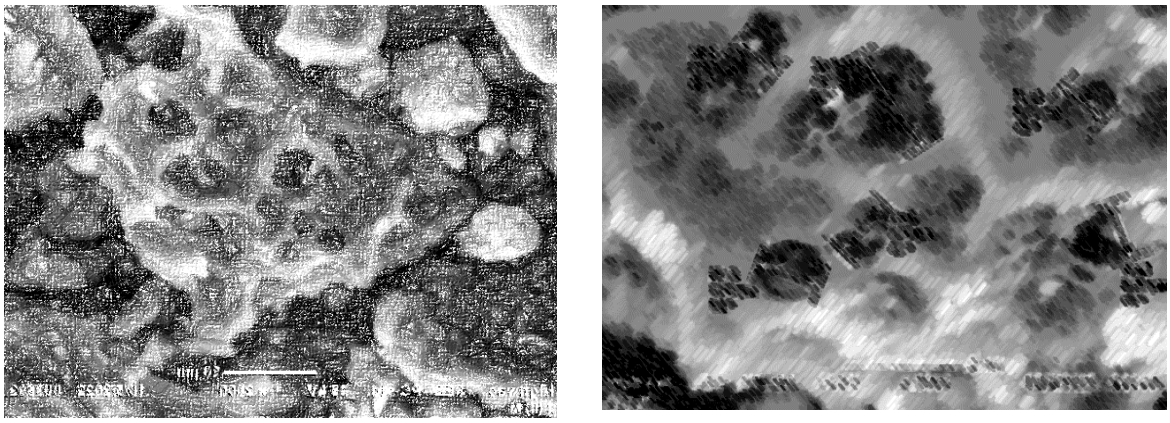
4.2 Surface Morphology Characterization of Coal Activated Carbon Using Scanning Electron Microscopy (SEM)

Morphological characterization of the activated carbon surface of coal using Scanning Electron Microscopy (SEM) is a characterization of the porous structure that has porosity on the surface of activated carbon. In addition, SEM is used to determine the pore surface of coal-activated carbon [15]. Test result By using Scanning Electron Microscopy (SEM) can be seen in Figs. 3 and 4.

The results show a significant difference in the activated carbon pores in bituminous coal activated carbon samples with concentrations of 3M (duplo) and 2.5M (duplo). Fig. 3 shows an open pore structure with a pore diameter of 4.91 µm in large but irregular pores and a pore surface area that belongs to the mesoporous type, and there are impurities present in the carbon pores. The cause of the irregular pore structure is that when the washing process is stirred, excessive collisions occur on the stir bar and the surface of the activated carbon, so the pore structure is irregular. Fig. 4 shows a closed,

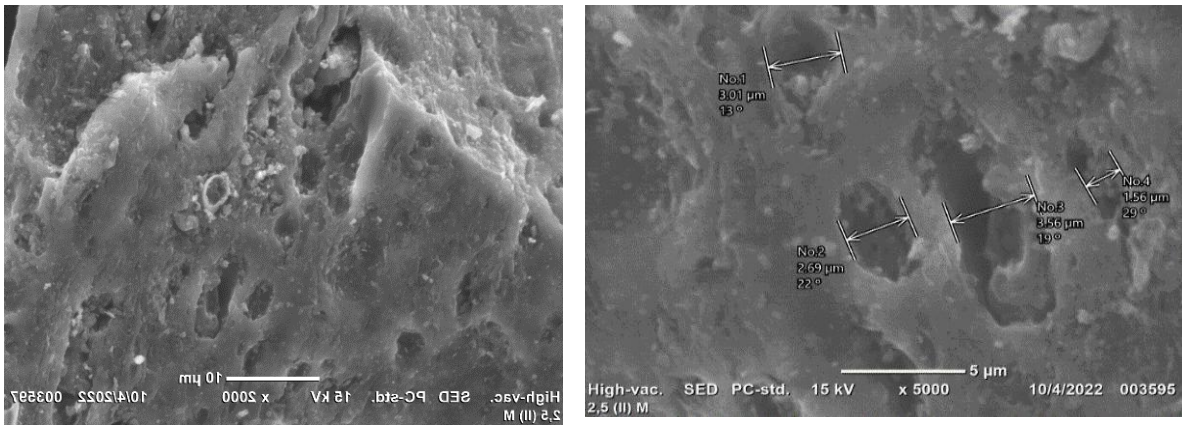
irregular, slightly pore structure with many impurities on the pore structure's surface, and the largest pore diameter is only 3.56 µm. A previous study conducted by Nurrahman et al. [10] stated that the larger the pore diameter, the higher the surface area of activated carbon. Therefore, the surface area is needed to determine activated carbon's feasibility.

From the research that has been done to determine the quality of activated carbon, testing for water content, ash content, volatile matter content, bound carbon content, and iodine number testing has been carried out, and characterization of activated carbon has been carried out using X-Ray Diffraction (XRD) and Scanning Electron Microscopy instruments. (SEM). From the data above, it shows that the quality of activated carbon from bituminous coal has a quality that meets the standards of quality that have been determined to be used as supercapacitor electrodes because the quality of supercapacitor electrodes from carbon electrodes is influenced by carbonization temperature, activation, and activated carbon materials used to produce the appropriate data. with standard (SNI 06-3730-1995). This effect directly affects the increased surface area.



(a) (b)

Fig. 3. (a) SEM Test Results of Activated Carbon at 2000x Magnification of 3 M Concentration Samples (duplo), (b) SEM Test Results at 5000x Magnification of Activated Carbon Samples of 3 M Concentration (duplo)



(a) (b)

Fig. 4. (a) SEM Test Results of Activated Carbon at 2000x Magnification of 2.5 M Sample Concentration (duplo), (b) SEM Test Results of Activated Carbon at 5000x Magnification of 2.5 M Sample Concentration (duplo)

5. CONCLUSION

Coal-activated carbon activated with hydrochloric acid activator (HCl) concentration of 3 M at 700 has a degree of crystallinity at an angle of 26.6389 2θ which is amorphous and has a pore diameter of 4.91 µm and has a lot of pore structure so that it can be used as one of the parameters as a base material supercapacitor electrode.

COMPETING INTERESTS

Author has declared that no competing interests exist.

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Peer-review history:
The peer review history for this paper can be accessed here:
https://www.sdiarticle5.com/review-history/96308