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Analysis of Activated Carbon from Bituminous Coal as the Primary Battery Cathode Potential Using Fluoric Acid

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Author's contribution

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

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ABSTRACT

Activated carbon can generally be made from carbon-containing materials such as coal. The activation in activated carbon synthesis has different activation mechanisms and characteristics. The objective of this study was to analyze the quality and characterization of activated carbon in bituminous coal using a fluoride (HF) activator. The research method was carried out starting from the sample preparation stage, the chemical and physical activation carbonization stage, and the stage of testing the quality of coal-activated carbon. The results showed that each test obtained a moisture content ranging from 0.7% -1.61%. Ash content has a range of values ranging from 17.51% -25.74%, while volatile matter content ranges from 20.96% - 33.97%. The bound carbon content has a range of values ranging from 20.96% -33.97%, iodine numbers range from 3995.4455-4302, 0456 mg/g (all parameters follow SNI 06-3730-1995); and the degree of crystallinity obtained from the concentrations of 3.5 M and 5 M (duplo) has a degree of crystallinity of 26.6218 and 26.6014, respectively. Bituminous coal-activated carbon activated using Hydrogen

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Flouride (HF) with a temperature of 800 °C has optimal characteristics at a concentration of 5 M with a moisture content of 0.84. The volatile matter content is 18.60, while the iodine absorption capacity is 4.137.0704. The short carbon chain in degrees of crystallization is at an angle of 26.6014 2θ with two diffraction peaks forming a solid crystalline pattern. The surface morphology of activated carbon produces round, neat, and slightly impurities pore with a diameter of 8.81µm.

Keywords: Fluoric acid; bituminous coal; primary battery electrode; activated carbon.

1. INTRODUCTION

Community needs" in this globalization era" start from the tertiary and primary needs of the community. The increase in tertiary needs increases the need for goods like electronics. Electronics are an essential community need; some require batteries as a source of electricity. Batteries include devices that can convert chemical energy into electrical energy, which can be applied to several electronic devices [1]. In general, batteries can be grouped into two, namely primary batteries and secondary batteries. One example of a primary battery is a dry battery. The raw material for making batteries is activated carbon which acts as an electrode [2]. Coal is one of the uses of natural materials as a source of activated carbon in batteries.

Coal can be used as the primary raw material in the chemical and petrochemical industries. Until now, Indonesia has not been able to apply coal utilization technology like in other developed countries [3]. As a non-perishable natural resource, coal has real value for the national and regional economies and plays an essential role in achieving the prosperity and welfare of the people. Therefore, the management of coal mining must adapt to environmental changes to encourage regional autonomy, human rights, and the environment [4].

Coal can be used as activated carbon, where activated carbon has a texture with high porosity and a large surface area and has different functional groups depending on the manufacturing process [5]. Activated charcoal or commonly called activated carbon, has many advantages. The materials that can be used to make activated carbon such as" seed coat, shell, coal, wood, and others, but the nature of the activated charcoal will be different not only because of differences in raw materials but also influenced by the method of activation used" [6].

Activated carbon is generally an amorphous compound produced from several materials containing carbon elements which are explicitly needed to achieve high absorption depending on the size or volume of the pores and surface area [7]. Activated carbon from biomass can be made by physical activation using evaporation or gas CO_2 at high temperatures and chemical activation by using chemicals to form porous structures (Yahya et al. 2015).

Activated carbon can generally be made from carbon-containing materials such as coal and can generally be activated using H_3PO_4, KOH, and $ZnCl_2$. This activation in activated carbon synthesis has different activation mechanisms and characteristics, so the activated carbon produced is also different [8]. Novandara et al. (2020) used an activating agent previously using HF solutions with varying concentrations of 2%, 2.5%, 3%, 3.5%, and 4% by volume of immersion carried out for 5 hours at 30ºC, then the solution is filtered, and the coal residue is activated using a furnace with a temperature variable of 700; 750; 800; 850; 900°C for 2 hours. The results obtained showed that the best-activated carbon was obtained at a concentration of 4% HF and an activation temperature of 900°C with the absorption of iodine (I_2) of 810.75 mg/g, the water content of 1.9992%, volatile matter content of 0.192%, ash content of 5.408% and fixed carbon reaching 92.401%.

Patmawati and Kurniawan's [9] research describes the lignite coal carbonization process carried out at a temperature of 600ºC for 3 hours. The activation process is carried out using the H_3PO_4 activator and the combination of H_3PO_4-$NaHCO_3$. The best results were obtained by using the H_3PO_4-$NaHCO_3$. with an activation time of 6 hours. The bonded carbon value was 71.78%, and the iodine absorption was 505.01 mg/g.

Electrodes for various applications generally include applications for batteries. At the electrodes, electrons are transferred from the anode to the cathode, producing an electric current and a potential difference. The electrodes come from various capable metals" applied as a negative electrode with varying energy density

and electrode potential difference" [2]. Pahlevi et al. [2] previously used activated carbon electrodes using *betung* bamboo (Dendrocalamus asper) as activated carbon with electrolytes NaCl and NaOH. Betung bamboo, which will be carbonized at 500ºC for 2 hours in this furnace, is intended to get the best pore for SEM analysis and to increase porosity. The most significant pore measurement result for activated carbon is" 11.42m with 12% KOH activation. Then activation was carried out with 1 M KOH with a concentration of 10-12%. The selected electrolyte was 5-15 ml NaOH and 1 M NaCl."

Coal proximate analysis is carried out to determine the characteristics and quality of activated carbon so that the relative amount of moisture content, volatile matter (VM), ash (ash), and fixed carbon contained in the coal can be determined. This proximate analysis is the most basic test for determining coal quality [10]. The use of activated carbon in making battery electrodes is because activated carbon has high absorption, surface area, and high conductivity in battery applications. In addition, each sample used as a battery electrode has a different potential difference [2].

The activated carbon material prepared was characterized by several methods, including Scanning Electron Microscopy (SEM) and X-ray Diffractometry (XRD). The use of this method had previously been carried out by Febriyanto et al. [11] shows the result, namely the change from a sharp peak to a wide one at 20=24º. These changes indicate a change in the carbon structure from crystalline to amorphous.

In the research of Pahlevi. et al. [2], SEM (Scanning Electron Microscope) analysis using activated carbon from *betung* bamboo, activated carbon that has been activated using 10-12% KOH with a magnification of 5000 times shows many pores. The larger the pores of activated carbon that have been activated, the more significant the surface fraction to the volume of particles. Moreover, the greater the concentration of KOH, the greater the voltage and current generated in the battery. The amount of energy absorbed in a circuit affects the amount of electrolyte used and also affects the activation

3. RESEARCH METHODS

2.1 Tools and Materials

The tools used in this study included a Scanning Electron Microscope (SEM), X-Ray Diffraction, 48% technical hydrochloric acid (HF), sodium thiosulphate pentahydrate ($Na_2S_2O_35.H_2O$), sodium bicarbonate ($NaHCO_3$), iodine solution (I_2) 0.1 N, potassium iodide (KI), 1% starch indicator, distilled water, water one, aluminum foil, filter paper, tissue.

2.2 Procedure Preparation

Sample preparation was carried out by taking bituminous coal in the Lamuru area in Bone Regency. Then the samples were washed using distilled water and dried in the sun for two days (Kasturi et al. 2019). Afterward, the sample was ground and filtered using a 100-mesh sieve (Kusdarini et al. 2017).

2.3 Carbonization Stage

Samples that have been sifted are carbonized by heating using a furnace at 800^{o}C for 3 hours until activated carbon is obtained [5]. The activated carbon resulting from carbonization is then cooled using a desiccator for 60 minutes until the activated carbon is cold [12].

2.4 Phase Activation Chemically and Physically

Activated carbon that has been cooled is weighed using an analytical balance (digital) of as much as 50 grams. Furthermore, the activated carbon of coal is divided into five parts, each of which is activated using a solution of hydrofluoric acid (HF) in a ratio of 1:10 for each variable concentration 2; 2.5; 3; 3.5; 4 M with an immersion time of 8 hours at room temperature (30ºC) in a fume cupboard [5]. After that, the activated carbon was filtered using filter paper. Then the activated carbon is neutralized using distilled water until the pH of the activated carbon becomes neutral [5].

The coal-activated carbon is then activated physically, and each concentration that has been activated using a hydrofluoric acid (HF) activator is heated in a furnace at various temperatures of 700 and 800ºC for 2 hours [5]. Then the coal activated carbon is cooled using a desiccator. After that, testing or analysis of bituminous coal activated carbon quality is carried out.

2.5 Powder Structure Crystallinity Test of Activated Carbon Using X-Ray Diffraction

The characterization test was carried out using the X-Ray Diffraction (XRD) instrument to determine the crystallinity of a material, namely

activated carbon from bituminous coal from the Bone Regency area, which is related to the ability to absorb hydrogen. Then the instrument was operated with 40 kV, 30 mA, using radiation at a speed based on observation 2º/min (Kasmiani et al. 2018).

3. RESULTS

The quality of activated carbon processed from coal raw materials was tested using several analyses: moisture content, ash content, volatile matter content, bound carbon, and iodine absorption.

3.1 Water Content

The moisture content of coal-activated carbon is compared with temperature and various concentrations of activators. The results of the analysis of the quality of activated carbon can be seen in Table 1.

The results above show the water content value with a range of values ranging from 0.7% - 1.61%. These results state that the resulting water content value meets the specified Indonesian National Standard (SNI) (SNI 06-3730-1995), a maximum of 15%.

3.2 Ash Content

Ash content of coal-activated carbon with a comparison of temperature and activator concentration variations. The results of the analysis of the quality of activated carbon can be seen in Table 2.

The results above show the value of ash content with a range of values ranging from 17.51% - 25.74%. These results state that the resulting ash content does not meet the specified Indonesian national standard (SNI 06-3730-1995), a maximum of 10%.

Table 1. Moisture content of bituminous coal activated carbon

Temperature Activation (ºC)	Concentration (HF) (M)	Simplo Water Content (%)	Duplo Water Content (%)	Average Moisture Content (%)	Water content SNI (%)
700	3	0.38	0.56	0.47	<15
	3,5	0.46	0.04	0.25	<15
	4	0.76	0.64	0.7	<15
	4,5	0.14	0.14	0.14	<15
	5	0.1	0.08	0.09	<15
800	3	0.28	0.54	0.41	<15
	3,5	1.34	0.54	0.94	<15
	4	1.52	1,7	1.61	<15
	4,5	0.56	0.68	0.62	<15
	5	1.44	0.24	0.84	<15

Table 2. Ash content of bituminous coal activated carbon

Temperature Activation (ºC)	Concentration (HF) (M)	Simplo ash content (%)	Duplo ash content (%)	Average Ash Content (%)	Ash Content SNI (%)
700	3	25,92	25,49	25,70	<10
	3,5	25.00	21.56	23,28	<10
	4	20.75	23.07	21.91	<10
	4,5	26,92	23.52	25,70	<10
	5	25,49	21.15	23,32	<10
800	3	15.68	25,49	20.58	<10
	3,5	21.56	27.45	24.50	<10
	4	26.00	25,49	25,74	<10
	4,5	19.60	21.56	20.58	<10
	5	13.46	21.56	17.51	<10

3.3 Fly Substance Levels

Coal active volatile matter content with a comparison of temperature and variations in activator concentration. The results of the analysis of the quality of activated carbon can be seen in Table 3.

The results above show the value of volatile matter content with a range of values ranging from 20.96% -33.97%. These results state that the value of the volatile matter produced does not meet the specified Indonesian National Standard (SNI) (SNI 06-3730-1995), namely a maximum of 25%.

3.4 Bonded Carbon Content

The content of bonded activated carbon in coal is compared with temperature and various concentrations of activators. The results of the analysis of the quality of activated carbon can be seen in Table 4.

The results above show the value of bound carbon content with a range of values ranging from 20.96% -33.97%. These results state that the value of bonded carbon produced does not meet the specified Indonesian National Standard (SNI) (SNI 06-3730-1995), which is at least 65%.

3.5 Iodine Number

Calculate the iodine in activated carbon of coal with a comparison of temperature and variations in activator concentrations. The results of the analysis of the quality of activated carbon can be seen in Table 5.

Table 3. Levels of activated carbon flying matter in bituminous coal

Temperature Activation (ºC)	Concentration (M)	Simplo Fly Substance Content (%)	Duplo Fly Substance Content (%)	Average Flying Substance Content (%)	Fly Substance Levels SNI (%)
700	3	28.30	27.45	25,87	<25
	3,5	34,61	33,33	33.97	<25
	4	21.56	20,37	20.96	<25
	4,5	27.45	33,33	30,39	<25
	5	28,84	31.37	30,10	<25
800	3	23.07	18.86	20.96	<25
	3,5	21.15	33,33	27,24	<25
	4	22.00	28.30	25,15	<25
	4,5	13.72	36,53	25,12	<25
	5	17,64	19.56	18.60	<25

Table 4. Content of bonded carbon activated carbon bituminous coal

Temperature Activation (ºC)	Concentration (HF) (M)	Simplo Bonded Carbon Content (%)	Duplo Bonded Carbon Content (%)	Average Bonded Carbon Content (%)	Water content SNI (%)
700	3	45.78	47.06	46,42	>65
	3,5	40,39	45,11	47,25	>65
	4	57,69	56,56	57,12	>65
	4,5	45,63	43,15	44,70	>65
	5	45.97	47,48	46,72	>65
800	3	61.25	55,65	58.45	>65
	3,5	57,29	39,22	48,22	>65
	4	52.00	46,21	49,10	>65
	4,5	66,68	39.98	53,33	>65
	5	68,90	43,47	56,18	>65

Table 5. Bituminous coal activated carbon iodime number

Temperature Activation (ºC)	Concentration (HF) (M)	Simplo Iodine Number (mg/g)	Duplo Iodine Number (mg/g)	Average Iodine Number (mg/g)	Iodine Number SNI (mg/g)
700	3	4,263,9744	4.111,6896	4.187,8320	>750
	3,5	4,137.0704	4.368,8260	4,252,9482	>750
	4	4340,1168	3,781,7392	4060.9280	>750
	4,5	4. 238.5936	3,752,2974	3.995,4455	>750
	5	4.238,5936	4.187,7320	4.213,1628	>750
800	3	4.187,8320	3,781,7392	3,984,7856	>750
	3,5	4.187,8320	4.263,7440	4.225,7880	>750
	4	4.238,5936	4010.1664	4.124,3800	>750
	4,5	4,289,3552	4.162,4512	4.225,9032	>750
	5	4137.0704	4,467,0208	4,302.0456	>750

Table 6. Results of XRD testing of bituminous coal activated carbon

Concentration (HF) (M)	Degree of Crystallinity (2θ)	Peak Crystallinity (Å)
3.5M (duplo)	26.6218	3.34848
5M (duplo)	26.6014	3.35101

The results above show the value of the iodine number with a range of values ranging from 3,995.4455-4,302.0456mg/g. These results state that the resulting iodine value meets the specified Indonesian National Standard (SNI) (SNI 06-3730-1995), a maximum of 25%.

3.6 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 6.

The above results show that the degree of crystallinity obtained from concentrations of 3.5 M and 5 M (duplo) has a degree of crystallinity of 26.6218 and 26.6014, respectively.

4. DISCUSSION

Coal samples were taken from Masenrengpulu Village, Lamuru Subdistrict, Bones Regency, South Sulawesi Province, Indonesia. The coal is washed using distilled water to remove dust and dirt and dried in the sun to reduce the water content. The dehydrated sample is then crushed into fine granules using a crusher. Furthermore, the sieving process is carried out to homogenize the primary size. The 100 mesh size is used because if the coal particle size is too small, it can cause the coal to dissolve in the acid immersion. In addition to producing smaller coal powder sizes, it also facilitates the calcination or carbonization process [13].

4.1 Carbonization

Carbonization is a state of the process of decomposition of a substance or decomposition of organic materials to form carbon or commonly known as pyrolysis. Temperatures over 170ºC form carbon oxide (CO), carbon dioxide (CO2), and acetic acid. At 275℃, decomposition will form tar, methanol, and other products. This study used heating with a temperature of 800º. This C function is to evaporate the component substances such as water, silica, and volatile matter content, which is still absorbed in the pores. The carbonization process is also carried out to expand and increase the pores of the carbon. Heating is usually carried out at temperatures between 300-900℃ [5]. Non-carbon gases such as hydrogen, oxygen, and nitrogen are used instead of carbon in the carbonization process during pyrolysis in the boundary layer. In the carbonization process, it is necessary to secure hydrocarbon compounds such as cellulose and hemicellulose to produce carbon and supporting granules that contain absorption capacity (Hartini, 2014).

4.2 Phase Activation Chemically and Physically

Chemical activation can cause swelling of the adsorbent and open the cellulose structure. Determining the type of activator in the activated carbon purification process aims to remove impurities in the form of metal oxides such as

silica oxide (SiO_2). Types of chemicals that can be used as activators such as calcium chloride salt ($CaCl_2$), magnesium chloride ($MgCl_2$), zinc chloride ($ZnCl_2$), sodium hydroxide (NaOH), and sodium chloride ($NaCl_2$) [5]. The choice of hydrogen fluoride (HF) solution as an activator in the synthesis of activated carbon in this study is because the HF solution can dissolve 99.5% of the overall silica content contained in activated carbon so that the surface area of activated carbon will increase. HF activators have properties that quickly absorb water or are quite hygroscopic solid and can remove many impurities but do not damage the carbon structure in activated carbon [5]. This study used five concentration variations were used, namely 3 M, 3.5 M, 4 M, 4.5 M, 5 M simplo, and duplo.

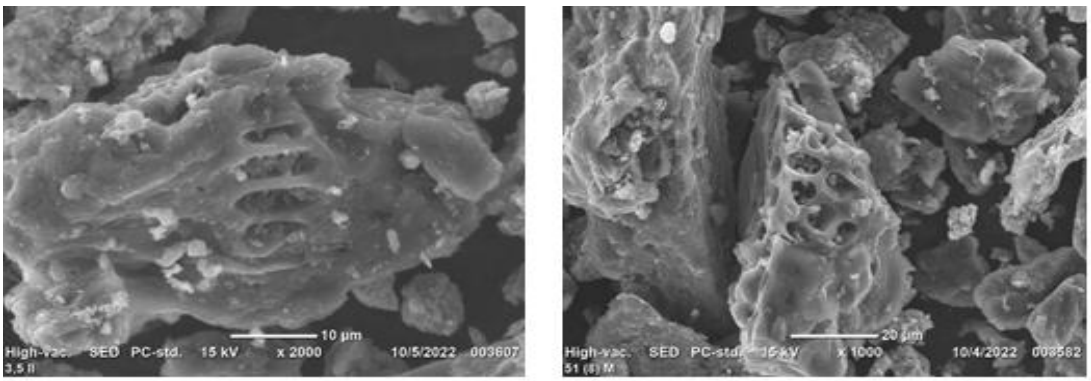
The process of chemical activation or immersion of the coal carbon in the HF solution for 8 hours is stirred using a magnetic stirrer to homogenize the interaction between the HF solution and the coal charcoal. The activated carbon is washed using distilled water until the pH is neutral (pH=7), which aims to remove any remaining impurities. After the rinse water is neutral, the coal carbon is filtered using filter paper to take the residue. After that, it is dried in the oven at 105^{o}C removes the water content contained in the resulting coal-activated carbon residue (Verlina, 2014).

Physical activation breaks the carbon chain of organic compounds using heat, steam, and carbon dioxide. Physical activation in this study aims to increase the volume and diameter of the pores formed in the carbonization process and create new pores. The carbon atom is not oxidized because the oxidizer only oxidizes the components that cover the porous surface of the activated carbon (Hartini, 2014). In this study, two temperature variations were used, namely temperatures of 700℃ and 800 ℃, which aim to determine the effect of temperature on the surface area and remove tar or impurities. At temperatures below 700ºC, the activation process with water vapor will take place very slowly, whereas temperatures above 1000^{o}C will cause damage to the hexagonal lattice structure of activated carbon [5].

4.3 Scanning Electron Microscope (SEM) Characterization

A scanning Electron Microscope (SEM) analyzes the porous surface structure of activated carbon from bituminous coal activated using hydrogen fluoride (HF) activator. This characterization aims to determine activated carbon's surface morphology and impurities. The effect of adding activator concentration on the morphology of activated carbon at concentrations of 3.5 M and 5 M can be seen in Fig. 1.

Based on the results of the SEM analysis in this study, it can be seen in Figs. a and b with a magnification of 5000x that there are differences in the pore structure of activated carbon activated by HF 3.5 M and HF 5.0 M with an activation temperature of 800℃. The activated carbon activated with HF 3.5 M has a small number of pores with a more oval shape and a pore diameter of 7.74-11.0 µm. Compared to activated carbon activated with HF 5 M, the pores formed are larger. Many form good pores but do not show homogeneity, and the pore depth formed is more significant, with a pore size of 1.40-8.81µm. It indicates that activated carbon activated with 5 M HF can increase the size and number of new pores.



(a) 3.5 M **(b) 5 M**

Fig. 1. Morphology of bituminous coal activated carbon to the addition of activator concentration, Figs. a (3.5) and b (5)

In Fig. 1a, the activated carbon activated with 3.5 M HF produces many impurities compared to the activated carbon activated with 5 M HF, which still contains a few impurities. The large number of impurities on the pore surface of activated carbon is due to chemical activation. The washing of activated carbon using distilled water does not take place ideally. In addition, after chemical activation, the drying process of activated carbon using an oven does not take place imperfectly; this causes impurities in the form of organic compounds, minerals, and oxides to not evaporate and remain in the activated carbon so that the substance will cover some or all of the pores impurity [14]. Following the more hydrocarbons or impurities that can be removed will increase the number of new pores in activated carbon[(6]. Pahlevi's research [2] also explained that the larger the pores of the activated carbon, the greater the surface to the volume fraction of the particles, and the greater the concentration of HF, the greater the voltage and current generated on activated carbon for its potential as a battery.

4.4 Characterization of X-Ray Diffraction (XRD)

Characterization using XRD in this study aims to analyze the effect of HF activator on activated carbon on the crystal form by paying attention to the peaks of the diffraction pattern curve. The degree of crystallinity of the activated carbon formed is the ratio between the crystalline phase and the amorphous phase formed in a material. The higher the degree of crystallinity, the structure of the activated carbon will be larger and farther from the amorphous structure. In addition, the degree of crystallization is also affected by the carbonization temperature and activator concentration. In this study, the samples used in testing the degree of crystallinity with the x-ray diffraction method were samples from the test results optimal and not optimal with a carbonization temperature of 800℃. Result Characterization can be seen in Figs. 2 and 3.

Based on the data showing that the addition of an activator can affect the broken and formed carbon chains, it can be seen in the figure that at low concentrations, which are activated with HF 3.5 M, the carbon chains at this concentration cannot completely break the carbon chains because at this concentration the carbon chains are still long compared to carbon that is activated with HF 5M. It shows that the higher the concentration can affect the breaking of the carbon chain so that the carbon chains formed are shorter, the crystalline form is denser, and the bonds are more substantial.

From Fig. 2 and Fig. 3, it can be seen the different degrees of crystallinity or crystal structure in the activated carbon of bituminous coal. It can be seen in the diffractogram pattern that there are minerals and crystalline phases visible from the presence of sharp diffraction peaks. This is in line with research by Hutapea et al. [15], which showed that using X-ray diffraction, the resulting activated carbon had a semicrystalline diffraction pattern with peaks at an angle of 2θ of 3.34848 Å and 3.34848 Å. The results of this study explain that the high and low peaks produced in XRD characterization are influenced by the activation process, which causes the plate to shift. Initially, the level of regularity is high (crystalline) to be irregular (amorphous).

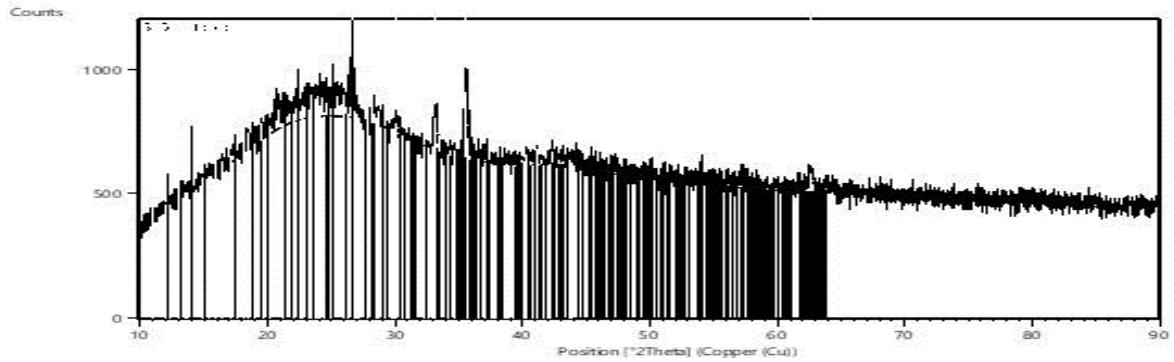


Fig. 2. Graph the relationship between the concentration of 3.5 and the peak of the diffraction pattern

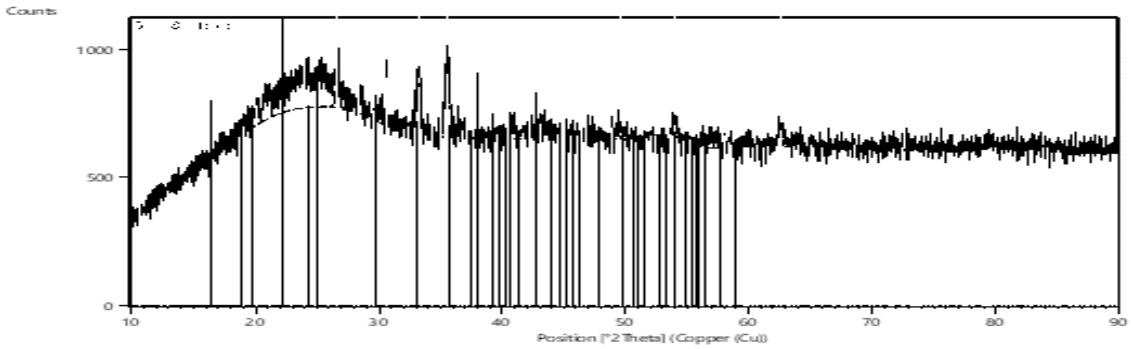


Fig. 3. Graph The relationship between the concentration of 5 and the peak of the diffraction pattern

The research results on manufacturing activated carbon from bituminous coal activated using hydrogen fluoride (HF) with the highest concentration and activation temperature indicate its potential for use as a cathode for primary battery electrodes. It is because the denser the crystal form, the more oxidation reduction at the cathode fast or the process of electron transfusion, and the more tenuous the carbon bond, the lower the voltage, and the resulting electric current are not substantial. It follows the research of Nurlia et al. (2020) with the results of characterization tests using XRD, obtaining the highest peaks of 2θ at 26.60 and 26.62. It indicates that at the peak of the carbon group fraction, short chains are formed, and the structure is regular so that the bonds are strong.

5. CONCLUSION

Bituminous coal-activated carbon using Hydrogen Flouride (HF) with a temperature of 800 °C has optimal characteristics at a concentration of 5 M with a moisture content of 0.84, a volatile matter content of 18.60, and an iodine absorption capacity of 4.137.0704. It has a carbon chain that has a short degree of crystallization at an angle of 26.6014 2θ with two diffraction peaks to form a solid crystalline pattern. The surface morphology of activated carbon produces round, neat pores and a small amount of dirt with a pore diameter of 8.81µm. It suggests that Activated carbon batteries can be used as the base material for cathodes on primary battery electrodes.

COMPETING INTERESTS

Author has declared that no competing interests exist.

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