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Preparation and Characterization Composites of Activated Carbon from Cassava Peel (*Manihot Utilisima*) - Copper (II) Oxide (CuO) as a Thermoelectric Material

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ABSTRACT

Electrical energy is one of the energies that is needed by humans. One of the efforts to save electrical energy is by using the thermoelectric method. Thermoelectric material is a material that can convert heat energy into electrical energy through the Seebeck effect. In this study, the thermoelectric material was made from activated carbon composites of cassava peel-CuO. Activated Carbon-CuO composite material is made by mixing various mass composition ratios (grams). Composite material is tested for electrical conductivity, thermal conductivity and the resulting electrical voltage (Seebeck effect). To see the crystal shape and size of the composite material, characterization was carried out using X-Ray Diffraction (XRD) and to determine the band gap of the composite material, characterization was carried out using UV- Diffuse Reflectance (UV-DRS). The results of the test show that the composite material with a ratio of grams of activated carbon: grams of CuO is the best material as a thermoelectric material because it has a high electrical conductivity value of $0.4167 \text{ M}\Omega\text{-cm}^{-1}$ with a low heat conductivity of 20.3057 J / s and with the mains voltage (Seebeck effect) of 0.019036 mV / K . The results of characterization using XRD showed that the addition of activated carbon to CuO did not damage the crystal form of CuO and the crystal size (D) 23,4240 and crystal size (d) 0,2521 of the composite material, namely nm and nm. The results of characterization using UV-DRS show that the band gap of the composite material is 1,63 eV. Based on the test results and

characteristics, it is concluded that the CuO composite material can be used as a thermoelectric material.

Keywords: Activated Carbon, Copper (II) Oxide, Composites, Thermoelectric Materials.

INTRODUCTION

Electricity is energy that humans really need. All objects at this time are activated and moved with electrical energy. Until now, electricity sources still rely on fossil fuels as the main source, such as coal and other petroleum fuels which will run out from time to time if they are used continuously. Therefore, alternative and renewable energy is needed as an effort to save fossil fuels and as the creation of new electrical energy using the thermoelectric method.

Thermoelectric materials are useful for converting heat energy into electrical energy with a seebeck effect (Zhang et al , 20017). The material used in this thermoelectric component is a material that can convert heat energy into electrical energy or when given an electric voltage there is a temperature difference (Sutjahja, 2010). Ideally, thermoelectric materials have a seebeck effect and high electrical conductivity and low thermal conductivity (Zheng, 2008). One of the materials that can be used for thermoelectric materials is Activated Carbon. Activated carbon is a

porous material that contains 85% - 95% carbon as a result of heating carbon-containing materials at high temperatures (Gultom, 2014). Activated carbon has good conductivity as a thermoelectric material (Shapley, 2012). Activated carbon is classified as easy to prepare and the price is cheap. However, activated carbon has a very small electrical conductivity, namely 0.001 S / m (Shapley, 2012) so that an alternative is needed to obtain a material with low thermal conductivity and high electrical conductivity.

In this study, the authors wanted to use cassava peels as an active carbon ingredient. Activated carbon from cassava peels functions as a matrix (binder) and CuO as a filler. Based on this background, the authors are interested in conducting research on cassava peel activated carbon composites - CuO as a thermoelectric material.

MATERIALS & METHODS

The thermoelectric method is a material that belongs to the semiconductor group that can convert heat energy into electrical energy using the principle of the seebeck effect and can convert electrical energy into heat energy using the Peltier effect. ⁽¹⁾ In 1934 Jean Charles Athanase Peltier conducted an experiment on the reverse of the Seebeck effect. He glued two pieces of metal and then flowed them with electricity. When electrified, heat absorption occurs by the two metal joints and heat release occurs in the other metal. When the flow is reversed in the absorption and release of panas will copy g turn anyway. This phenomenon is then called the Peltier effect. ⁽²⁾

Activated carbon is a porous material containing 85% -95% carbon which comes from incomplete combustion of materials containing carbon at high temperatures (Mulyana and Turmuzi 2014). Activated carbon has been widely used in many applications such as gas purification, decaffeination, gold refining, metal extraction, water purification, medicine, and filters in compressed air. This is due to the

characteristics of carbon that is porous and has a large surface area (Sulaiman et al. 2018). Activated carbon is one of the most important sinks from an industrial point of view. This absorbent is mainly used for the separation and purification of a mixture of liquid and gas phases. ⁽³⁾ Activated Carbon is made in 2 steps. The first stage is the carbonization stage, namely the carbon production stage and the second stage is the activation stage (Rachmat, Mawarani, and Risanti 2018). There are two activation processes, namely chemical activation and physical activation. Chemical activation is called the one-step method of manufacturing using chemicals. Physical activation involves carbonization of carbonated materials followed by activation of charcoal produced from the activating agent (Sudaryanto et al. 2006). The use of chemical activation is more beneficial in terms of a short period of time compared to physical activation and uses lower temperatures between 300°C - 700°C (Ahmida et al. 2015).

The activators used to activate activated carbon are HCl, KOH, and ZnCl₂ which have the advantage of facilitating recovery by washing with water and absorbing more water and expanding the pores on the carbon surface. (Moreno-Piraján and Giraldo 2010). There are several things that must be considered in making activated carbon. Activated carbon has several conditions which are described in the table.

Table 1. Indonesian Activated Carbon Quality Requirements (Sni 06-3730-1995)

Test type	Requirements
Water content	Max 15%
Substances Evaporates	Max 25%
Ash content	Max 10%
Fid carbon	Min 65%

Cassava peel is a biomass from agricultural residues that has not been widely utilized and has a high potential as heavy metal absorbent. Cassava peels can bind heavy metals because they have non-reducing cellulose which is effective in binding metal ions. ⁽⁴⁾ Cassava peels have

quite a lot of carbon elements, namely 59.31% and the ability to adsorb metal ions because they contain protein, non-reducing cellulose, and crude fiber.

Several transition metal oxides have the potential to be used as semiconductor materials and catalysts such as: ZnO, CuO, MnO₂, TiO₂, Fe₂O₄, and others. CuO is a copper oxide compound which is a p-type semiconductor material with a band gap of 1.2 eV.⁽⁵⁾ CuO as a semiconductor has many uses, such as: gas sensors, catalysts, batteries, high temperature semiconductors, solar energy convection, and emission fields.⁽⁶⁾

Composites come from a mixture of the main ingredients as a binder and a support material as a reinforcement. The main material is in the form of a matrix while the reinforcing material is in the form of fibers, particles, flakes, or other forms (Campbell, 2004). XRD is a tool that can be used to provide information on the structure of the characterization of crystalline materials of a polymer, crystal texture and other crystal parameters such as crystal grain size, crystallinity, and crystal defects used for X-ray diffraction. The XRD principle is the X-rays produced of a certain wavelength of a metal by varying the angle of reflection until an elastic reflection occurs which can be detected.⁽⁷⁾ The DR-UV principle is that if a material with a thickness of x reacts with photon energy, the material will absorb or dissipate the photon energy. So that the radiation flux can occur in positive or negative.⁽⁸⁾

MATERIALS & METHODS

A. Making Activated Carbon from Cassava Bark

Cassava peels are obtained in the South Solok area. Cassava peels that are dried in the sun to dry reduce the water content contained so that good activated carbon is formed

B. Carbonization and Activated Carbon Activation

In the carbonization stage, the sample was weighed 500 grams, pyrolysis was carried

out in a furnace at 300°C and 320°C for 1 hour. The carbon from the cassava skin is mashed using a mortar and almonds, then filtered using a 100 mesh sieve. Activation was carried out by immersing 6 grams of cassava peel carbon in 25 mL of the activator reagent (HCl, ZnCl₂ and KOH) with a concentration of 4N for 24 hours. Then the activated carbon was filtered using Whatmann filter paper and washed with distilled water until neutral conditions were obtained, then heated at 105°C for 1 hour. Then the variation of the optimized activated carbon reagent concentration was carried out from 2N, 4N, and 6N to produce the optimized activated carbon. The optimized activated carbon reagent and activated carbon reagent concentration were obtained by applying the measurements of the Indonesian National Standard, SNI 06-3730-1995 (moisture content, ash content, vapor content and bonded carbon). The activated carbon obtained was tested with the following parameters:

a. Water Content Analysis

Activated carbon was weighed 1 gram and put in a dry porcelain crucible, then heated in an oven at 105°C for 1 hour. The activated carbon is then cooled in a desiccator and weighed, the water content can be calculated by the following equation

$$\text{water content} = \frac{a-b}{a} \times 100\%$$

where :

a = initial charcoal weight (grams)

b = weight of charcoal after drying (grams)

b. Ash Content Analysis

Carbon was weighed 1 gram and put into a crucible porcelain container that had been dried, then put into a furnace and heated using a furnace at 600 ° C for 2 hours. When all carbon has turned to ash, cool in a desiccator and weigh it for permanent weight. The ash content can be calculated by the following equation :

$$\text{Ash content} = \frac{\text{ash weight}}{\text{initial carbon weight}} \times 100\%$$

c. Vapor Content Analysis

Activated carbon is weighed 1 gram and put into a dried porcelain container, then heated after the furnace temperature reaches below 100°C to a temperature of 330°C then turned off. The activated carbon is transferred to a desiccator and cooled. The vapor content can be calculated by the following equation :

$$\text{Steam Content} = \frac{a-b}{b} \times 100\%$$

where :

a = initial weight of charcoal (grams)

b = weight of charcoal after heating (grams)

d. Analysis of Bound Carbon Content

The carbon content attached to activated carbon is obtained from the reduction of the missing parts on heating, namely the vapor content and ash content .

$$\text{Bonded carbon content} = 100\% - (A + B)$$

where :

A = ash content (%)

B = vapor content (%)

CuO-Activated Carbon Composite Molding

Pure activated carbon obtained from cassava peels is mixed with CuO powder compared to activated carbon, namely:

Table 2 . Composition of Mass Comparison (Gram) of CuO Activated Carbon Composite Material

Komposisi material (gram)	KA	KC1	KC2	KC3	KC4	KC5	CuO
Karbon Aktif	10	7	6	5	4	3	0
Tembaga (II) Oksida	0	3	4	5	6	7	10

The mixture is refluxed at 90°C while stirring using a magnetic stirrer for 1 hour using distilled water, then filtered using filter paper then oven at 105°C for 1 hour and calcined at 400°C for 3 hours.

D. Testing of Activated Carbon-CuO Composites

Electrical conductivity testing

The electrical conductivity test is carried out by applying an electric voltage of 5 volts to

one metal rod in the material and then measuring the resulting electric current using a multimeter. To determine the electrical conductivity, the resulting electric current is treated using the formula:

$$R = \frac{V}{I}$$

$$\rho = \frac{RA}{L}$$

$$\sigma = \frac{1}{\rho}$$

Where:

R is the resistance (MΩ)

V is the voltage (Volt)

I is the current (μA)

L is the length (cm)

A is the cross-sectional area (cm²)

ρ is the resistivity (MΩ.cm)

σ is the electrical conductivity (MΩ⁻¹.cm⁻¹)

Heat conductivity test

The thermal conductivity test can be done by applying the heat on the underside of the printed material (T₂) with temperature variations of 40 ° C, 60 ° C, 80 ° C using a hot plate, after 15 minutes of heat the top position of the material is measured using a thermometer (T₁). To determine the thermal conductivity, the data obtained is processed using the formula:

$$Q = m \cdot \Delta T$$

$$H = \frac{Q}{t}$$

Where:

H is the conductivity (J /s)

Q is heat (J)

t is (s)

m is mass (kg)

c is the specific heat (J kg. °C)

ΔT is the difference in temperature (°C)

Testing the electric voltage produced by Cassava Peels

Testing of oak Seebeck done by transferring heat to the bottom side of the material sample and reference material (CuO pure) with variations in temperature of 40°C, 60°C, 80°C, which is connected with a slab of aluminum, passed hand over the material sample and reference material by the plate copper, each of which is connected directly to the multimeter to measure the voltage generated.

Characterization of Cassava Skin-CuO Activated Carbon Composite

X-Ray Diffraction (XRD) Analysis

The sample used for XRD analysis is a composite powder form. This analysis functions to determine the crystal form of the resulting composite.

Diffuse Reflectance Analysis - Ultra Violet (DR-UV)

The sample used for DR-UV analysis was a composite result in the form of a powder. The sample is entered into the sample holder. Then analyzed by computer to produce a spectrum. This analysis serves to observe the (band gap) resulting from the sample.

RESULT & DISCUSSION

Preparation of Activated Carbon from Cassava Bark

Sample preparation is the first stage to start this research. Cassava peels cleaned of impurities and cut into small pieces. Once clean and minimized, the cassava peel samples are then dried by drying them in the sun for 2-3 days. The aim is to reduce the water content of the sample so as to reduce the appearance of smoke during pyrolysis.

Carbonization and Activation of Carbon from Cassava Bark

Temperature Variation

Variations in carbonization temperature, namely 300°C and 320°C. To find out which carbon complies with (SNI 16-3730-1995), a characteristic test of activated carbon is carried out, namely testing moisture content, vapor content, ash content and bonded carbon content. The water content test aims to determine the remaining water content in the carbon after the carbonization process is carried out. Testing of vapor content is intended to determine the amount of substances or compounds that have not evaporated at carbonization temperature but evaporated at 310°C at carbonization temperature 300°C and 330°C at carbonization temperature 320°C. The ash content test aims to

determine the metal oxide content that is still present in the carbon after going through the carbonization process, and the bound carbon content aims to determine the carbon content after going through the carbonization process. The bound carbon content is calculated from the value of vapor content and ash content .

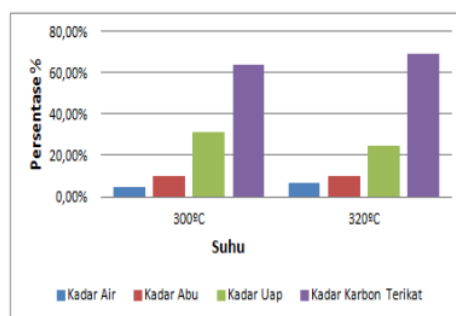


Figure1 .Graph of cassava peel carbonization temperature variations

In this graph it can be seen that at the carbonization temperature of 300°C the resulting carbon has a moisture content of 4.78%, an ash content of 9.91%, a vapor content of 31.5% and a bound carbon content of 63.72%. The carbonization value at 300 ° C does not yet meet the Indonesian National Standard (SNI 06-3730-1995) because the vapor content and carbonization temperature bound carbon content do not meet the standards. At the carbonization temperature of 320°C, the results obtained were 7% moisture content, 10% ash content, 24.4% vapor content, and 69.6% bonded carbon content. With this the charcoal produced in the carbonization stage at a temperature of 320°C meets the Indonesian National Standard (SNI 06-3730-1995) where according to SNI the maximum moisture content is 15%, the maximum ash content is 10%, the maximum vapor content is 25% and the bound carbon content is at least 65%.

Variation of Activation Reagents

The carbon from the cassava peel at 320°C was activated with several activating reagents. The activation stage is the stage of

forming carbon into activated carbon. The carbon from the cassava peel is activated by reagent HCl, KOH, ZnCl₂ for 24 hours using a 4N concentration and tested the moisture content, vapor content, ash content, and bound carbon content.

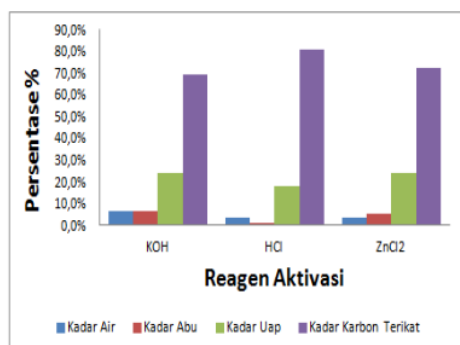


Figure 2 . Graph of cassava peel carbon activation with various concentrations

In the graph, it can be seen that the activation with HCl produces 3.4% moisture content, 1.3% ash content, 18% vapor content, and 80.7% bound carbon content. The test results of moisture content, ash content, vapor content, and carbon content bound to activated carbon using HCl activator meet the Indonesian National Standard (SNI 06-3730-1995). Activated carbon resulted from activation using KOH resulted in a moisture content of 6.5%, an ash content of 6.5%, a vapor content of 24.4% and a bonded carbon content of 69.6%. The results of the characteristic test of activated carbon using KOH show that it meets SNI 06-3730-1995 but the value is still below HCl. As well as activated carbon using ZnCl₂ activator produces 3.9% moisture content, 5.4% ash content, 24% vapor content, and 72.1% bound carbon content. The results of this characteristic test show that the activator using ZnCl₂ solution meets SNI 06-3730-1995 but its value is still below HCl (attachment). It can be concluded that the best activation solution for activating cassava peels is HCl, because the characteristic test is the best and meets the Indonesian National Standard (SNI 06-3730-1995).

Variation in the concentration of the activation reagent

After obtaining HCl as a good activation solution, activation was carried out using HCl with various concentrations ranging from 2N, 4N, 6N, and 8N. The aim of this concentration variation was to find the optimal concentration for the activated carbon activation process.

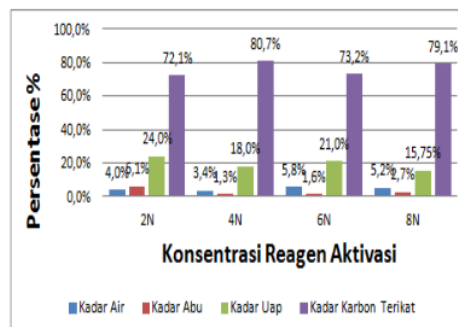


Figure 3 . Graph of cassava peel carbon activation concentration variation

In the figure it can be seen that the most optimum HCl activation reagent for activated carbon activator from cassava peels is 4N HCl which has a water content of 3.4%, 18% vapor content, 1.3% ash content and 80.7% bound carbon content according to with the quality standard of activated carbon SNI 06-3730-1995. The results show that with the increasing concentration of the activating reagent, the activated carbon produced will be better but if the concentration is too large it will reduce the results of the activated carbon obtained because too many activating substances from chemical compounds will cause clogged pores of activated carbon.

Compositing Activated Carbon with CuO

The process of making activated carbon-CuO composites is carried out by mixing the best activated carbon from the previous activated carbon HCl 4N with pure CuO powder with various mass ratios (grams). The mixing resulted in a ratio (KC10:0, KC7:3, KC6:4, KC5:5, KC4:6, KC3:7, KC0:10). This mixing was carried out for 1 hour by stirring using a stirrer to

homogenize the composite mixture. The composite result is molded into the mold by adding glycerol dropwise to mix it into a paste. Glycerol functions as an adhesive and composite thickener, making it easier for the printing process.

Testing of Activated Carbon-CuO Composites

Thermal Conductivity Testing

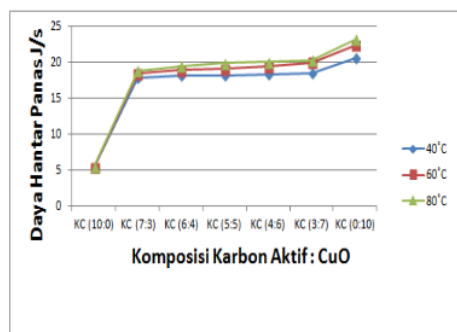


Figure 4 . Heat conductivity test graph

Based on the figure, it can be explained that the higher the temperature, the higher the heat conductivity of a material. Activated carbon has a low thermal conductivity, namely 5.45 J / s while CuO has a high thermal conductivity, namely 23.192 J / s. The addition of activated carbon to the composite material can reduce the heat conductivity of the CuO because activated carbon has a very good ability to absorb heat. The smaller the thermal conductivity of a material, the better it absorbs heat, the more heat energy that can be converted into electrical energy. ⁽⁹⁾ From the graph, it is concluded that 3 grams: 7 grams of CuO activated carbon has the best heat conductivity, namely 20.3057 J / s. This proves that the addition of activated carbon can reduce the heat conductivity of CuO.

Electrical Conductivity Testing

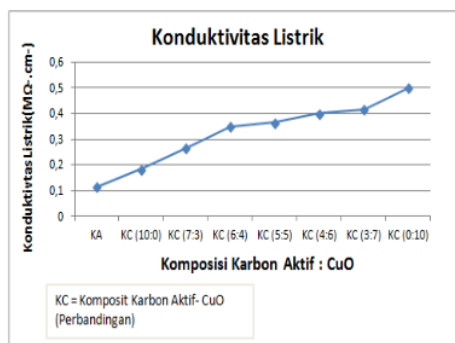


Figure 5 . Electrical conductivity test graph

Based on the figure, it can be concluded that carbon has a very low electrical conductivity, namely 0.116 MΩ .cm-, after activation the electrical conductivity increases to 0.1833 MΩ .cm-, and CuO 0.5 MΩ .cm-. carbon and activated carbon have low electrical conductivity because they are not semiconductor materials or conductors that can conduct electricity. The addition of CuO to activated carbon will increase the electrical conductivity of activated carbon in the composite material. In thermoelectric materials, the higher the electrical conductivity value, the better the value of the thermoelectric material. ⁽⁹⁾ From the graph below, it can be concluded that the ratio of 3 grams of activated carbon: 7 grams of CuO produces a high value of electrical conductivity, namely 0.4167 MΩ . Cm-.

Electrical Voltage Testing (Seebeck Effect)

Testing the Seebeck effect menggunakan CuO purely as a reference material which is a p-type metal oxide. ⁽⁵⁾ In this test, the material that generates a positive voltage is used as an N-type thermoelectric material and the material that produces a negative electric voltage is used as a P-type thermoelectric material

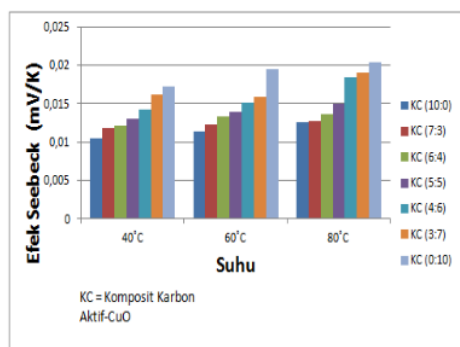


Figure 6 . Mains voltage test graph

Based on the figure, it shows that carbon without activation has a very small electric voltage (Seebeck effect), namely 0.00979 mV / K, after being activated the electric voltage (Seebeck effect) rises to 0.0125 mV / K, it is still under voltage (effect Seebeck) CuO is 0.02039 mV / K. The addition of CuO can increase the electrical conductivity of the composite material. The more the mass composition of CuO which is composite with activated carbon, the higher the electrical conductivity. Based on the graph, it can be seen that the composite material with a ratio of 3 grams of activated carbon: 7 grams of CuO has the best value for electrical voltage (Seebeck effect), namely 0.019036 mV / K at 353K. The higher the electrical conductivity value of a material, the better it is used as a thermoelectric material. ⁽⁹⁾

Characterization of CuO-Activated Carbon Composites UV-DRS analysis

Testing using UV-DRS aims to find the band gap value of the composite. Based on the test data, the correlation between % reflectance and UV irradiation wave length was obtained. The data obtained is processed to produce an estimate of the band gap energy value of the composite being tested. The result of band gap energy produced by activated carbon and activated

carbon-CuO composite (3: 7) can be seen in the figure.

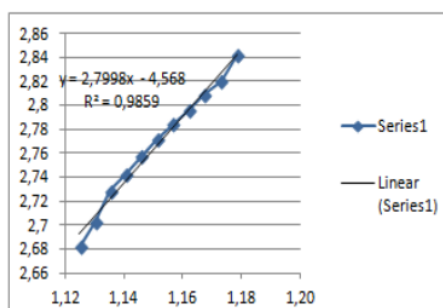
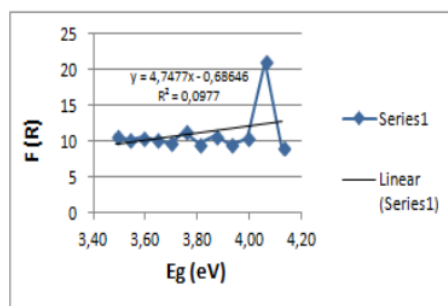


Figure 7 . (a) UV-DRS graph of activated carbon, (b) UV-DRS graph of activated carbon-CuO composites (3: 7)

Based on the figure, it can be seen that the band gap energy value of activated carbon-CuO 3: 7 is 1.63 eV seen with the recession line $4,568 / 2,7998 = 1.63$, and CuO theoretically has a band gap value. ie 1.1-1.2 eV. ⁽⁵⁾ This can be explained that the addition of CuO to activated carbon can increase the band gap value of the material, because activated carbon is not classified as a semiconductor or conductor material so it has a small band gap value.

XRD analysis

Characteristics with XRD aims to determine the crystal size and crystal lattice of a sample. The samples that were characterized by XRD were the activated carbon samples of cassava peels

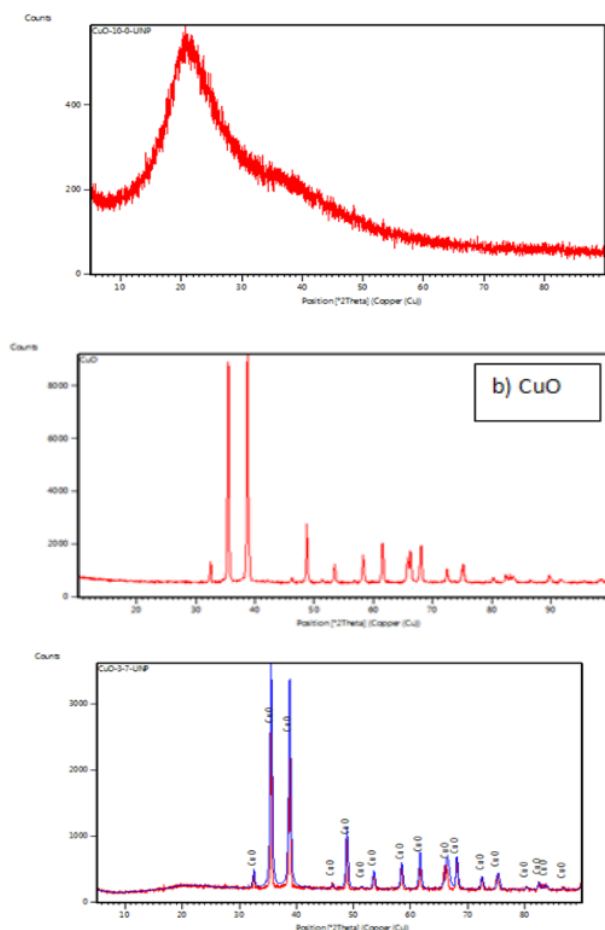


Figure 8 . (a) XRD spectrum of activated carbon, (b) Spectrum of CuO, (c) Conposit spectrum of activated carbon of CuO (3: 7)

In Figure 8 (a) it can be explained that the XRD spectrum of activated carbon does not have a specific peak because activated carbon is an amorphous material. In the XRD spectrum image (b) pure CuO appears to have a clear crystal specific peak, while (c) it can be seen from the XRD spectrum that the CuO-Activated Carbon Composite (3: 7) has the same peak as pure CuO. From the XRD spectrum results, it can be concluded that the addition of activated carbon to CuO does not affect or change the shape of the CuO crystalline spectrum. Based on the calculation of the XRD spectrum of activated carbon composite-CuO obtained the highest peak value at the position 2θ ie 38,7272 using a formula Debye Scherrer obtained crystal size (D)

and the crystal lattice (d) is 22.3008 nm and 0, 1565 nm.

CONCLUSION

Based on the research conducted, it can be concluded that:

1. Activated carbon from cassava peels with an optimum temperature of 320°C and activated with 4N HCl is activated carbon that meets SNI 06-3730-1995
2. Composite material with a ratio of activated carbon 3 grams:7 gam CuO is the best composite material with an electrical conductivity value of 0.4167 MΩ-Cm-, a value of heat conductivity 20.3057 J / s, and a value of electric voltage (Seebeck effect) 0 , 019036 mV / K.

3. XRD characterization results showed that the crystal size (D) and the crystal lattice (d) of the material Kompo sit is 23,4240 nm and 0, 2521 nm . The results of characterization with UV-DRS show that the band gap of the composite material is 1,63 eV

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