UTILIZATION OF SOLUTION $(NH_4)_2$ CO_3 ON COCONUT SHELL CARBON PURIFICATION AS AN ACTIVE CARBON MATERIAL

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Abstract

One of the economically valuable products made from coconut shells is shell charcoal. Shell charcoal is obtained from pyrolysis (carbonisation). Pyrolysis technology is the combustion of biomass in conditions without oxygen. This pyrolysis process is in the form of liquid, gas and solid. The solid product of this process is charcoal (char) which is then called carbonization. In this process, carbonized coconut shells will produce solids in the form of carbon, as well as distillate which is a heterogeneous mixture of liquid smoke and tar. This solid form of carbon has the potential to be processed into activated carbon. Activated carbon can be used for various industries, including the pharmaceutical industry, food, beverage, water treatment, and others. Not much has been done to manufacture activated carbon, in terms of the raw material potential and its use as well as the market potential which is quite large. In terms of quantity, it can be seen that the manufacture of activated carbon has bright prospects for meeting domestic needs as well as non-oil and gas export commodities (Setiaji, 2005). Activated carbon is a product that is widely used domestically, where nearly 70% of activated carbon products are used for refining in the sugar, coconut oil, pharmaceutical and chemical industry sectors. In addition, activated carbon is also widely used for water purification processes and other industries (Pari, 1995). This study will use a solution $(NH_4)_2$ CO_3 1M and 2M to act as activators in a chemical way, namely by soaking the carbon before being activated. It is hoped that the metal attached to the charcoal can be exchanged by NH cations$_4^-$. Therefore, it is necessary to investigate the concentration of $(NH_4)_2CO_3$ 1M and 2M to produce good quality activated carbon, seen from the physical and chemical characteristics of the carbon produced, one of which is the reduction of Fe metal as a carbon impurity mineral. The aims of the research are: 1. Determine the conditions $(NH_4)_2$ CO_3 as an activating agent that produces carbon from coconut shell pyrolysis which is free of Fe metal. 2. Enlarge the absorption capacity of activated charcoal with activation treatment at 5000C. From the research data, it was found that the content of Fe metal in charcoal without activation was more than the content of Fe metal in activated charcoal. The higher the

concentration of the solvent, the more Fe metal content is exchanged with ions, in this case NH ions$_4^+$. The higher the washing temperature is raised, the higher the mobility of the ions from the solution which facilitates the exchange of ions in it, and the higher the concentration of the solution $(NH_4)_2$ CO_3 washing, the affinity for ion exchange that occurs in it will be even greater. The absorption capacity of activated charcoal for iodine is lower than the absorption capacity that has been determined according to the Industrial Standards in Indonesia.
Keywords: Shell Charcoal, carbon, pyrolysis, carbonization

INTRODUCTION

Coconut plant (*Cook nuts. L*) grows a lot in Indonesia, especially in North Sulawesi Province so that North Sulawesi is known as a region *Nyiur Melambai*, because the distribution of coconut plants in this area is very wide. This plant is also versatile plant because almost all parts of the plant can be used for human life. The main component that has the highest value from coconut is the fruit part, where the coconut fruit consists of the shell (endocarp), fruit flesh (endosperm) and fruit juice (Mandey, 2007). The utilization of coconut fruit is mainly only for the flesh of the fruit to be used as copra, oil and coconut milk for household needs, while other by-products such as shell have not been used much. The use of coconut shells, a small part as fuel for household needs, smoking, copra, handicrafts, and others (Ketaren and Jatmiko, 1978), even though coconut shells can be processed into various high-value processed economic products. One of them is coconut shell to be used as shell charcoal.

Indonesia has a very large comparative advantage over coconut shells, but the coconut shell business in Indonesia still faces various obstacles so its potential cannot be utilized properly. Shell charcoal is obtained from pyrolysis (carbonisation). Pyrolysis technology is the combustion of biomass in conditions without oxygen. This pyrolysis process is in the form of liquid, gas and solid. The solid product of this process is charcoal (char) which is then called carbonization. In this process, carbonized coconut shells will produce solids in the form of carbon, as well as distillate which is a heterogeneous mixture of liquid smoke and tar. This solid form of carbon has the potential to be processed into activated carbon. Activated carbon can be used for various industries, including pharmaceutical, food, beverage, and water treatment. Not much has been done to manufacture activated carbon, in terms of the raw material potential and its use as well as the market potential which is quite large. In terms of quantity, it can be seen that the manufacture of activated carbon has bright prospects for meeting domestic needs as well as non-oil and gas export commodities (Setiaji, 2005).

Activated carbon is a product that is widely used domestically, where nearly 70% of activated carbon products are used for refining in the sugar, coconut oil, pharmaceutical and chemical industry sectors. In addition, activated carbon is also widely used for water purification processes and other industries (Pari, 1995). The raw materials that can be made into activated carbon are all carbon-containing materials, whether derived from plants, animals or minerals. These materials are various types of wood, animal bone rice husks, coal, coconut shells, coffee bean skins and others (Pari and Sailah, 2001).

The process of making activated carbon is carried out in two stages, namely the carbonization and activation stages. Carbonization is a pyrolysis stage of a carbon-containing material without the presence of oxygen and other chemicals, while activation is required to convert the carbonized product into an absorbent that has high porosity and a large surface area (Jankowska*et al*, 1991). To remove metal oxides in the charcoal that cover the pores, you can wash the charcoal. Various chemicals for washing charcoal have been carried out, among others, with water and HCl. When washing with water, many metal oxides attached to the surface are still not lost (Mandey, L. L: 2007). While washing using HCl on a large scale can cause pollution. This study will use a solution $(NH_4)_2\ CO_3$ 1M and 2M act as activators in a chemical way, namely by soaking the carbon before being activated. It is hoped that the metal attached to the charcoal can be exchanged by NH cations$_4^-$. Therefore, it is necessary to investigate the concentration of $(NH_4)_2CO_3$ 1M and 2M to produce good quality activated carbon, seen from the physical and chemical characteristics of the carbon produced, one of which is the reduction of Fe metal as a carbon impurity mineral. The research objective is

1. Determine conditions $(NH_4)_2\ CO_3$ as an activator that produces carbon from the pyrolysis of coconut shells that is free of Fe metal. 2. Increase the absorbency of activated charcoal with activation treatment at a temperature of 5000C.

RESEARCH METHODS

Research Place

This research was conducted in the Chemistry Laboratory Faculty of Mathematics and Natural Sciences, Manado State University.

Tools and materials.

The tools used, a set of tools for activation (electric furnace), *disk mill*, sieve 100 mesh (USA Standard Testing Sieve), desiccator, electric balance AND GR-200, thermocouple, Furnace type 21100tube (maximum temperature 1600^0 C), Buchner filter with *Rotary Vacum Pump, hot plate* magnetic and steering wheel, magnet, a set of ASS brand Perkin Elmer model 2380, model oven*Gravity Vonvection*, chemistry laboratory glassware.

The material used is coconut shell charcoal from the coconut shell charcoal factory in Ranotongkor Village, Tombariri District, Minahasa Regency, $(NH_4)_2\ CO_3$, HCl 37%, gas N_2, distilled water from the Chemistry laboratory, aquades from the Chemistry laboratory, KI, Fe $(NO_3)_3$, 9 H_2O, Amylum, HNO_3, kertas saring Whatman No 42, $Na_2S_2O_3$, glaswolle than I_2.

METHODS

1. Content of Sample Preparation for Reducing Tar in Coconut Shell Charcoal.

50 grams of coconut shell charcoal finely ground and then filtered through a 100 mesh size sieve. Filtered coconut shell charcoal is put in a column made of glass which is then put into an electric furnace. N gas$_2$ flowed from the gas cylinder through the bottom of the column with a flow rate of 20 ml/min. The activation process was carried out at a temperature of 500^0C. Activation lasts for 3 (three) hours, then dried. The activated charcoal is then used to determine the content of Fe metal which is analyzed by atomic absorption spectrophotometer.

2. Sample Preparation to Determine Reduction of Fe Metal Content in Coconut Shell Charcoal by Washing $(NH_4)_2$ CO_3 1M At Room Temperature.

   Activated coconut shell charcoal is heated in an oven at about 110^0C for one hour. Then the shell charcoal was weighed 1.00 grams and put into a 100 ml beaker then added 25 ml of 1 M ammonium carbonate solution and then stirred with a magnetic stirrer for 1 hour. Then filtered with Whatman No 42 filter paper, the resulting charcoal residue is ashed by heating in a furnace at a temperature of 600^0C for two hours, then cooled and moistened with HNO_3 concentrated, and then heated again to 600^0C for 1.5 hours. The ashes were dissolved in 25 ml of 1 M HCl solution and this solution was prepared for analysis of the Fe content for an activation temperature of 500^9C.

3. Sample preparation to determine the reduction of Fe metal content in coconut shell charcoal by washing $(NH_4)_2$ CO_3 2m at room temprature.

   Activated coconut shell charcoal is heated in an oven at about 110^0C for one hour. Then the charcoal was weighed 1.00 grams and put into a 100 ml beaker. Then 25 ml of 2 M ammonium carbonate solution was added and stirred with a magnetic stirrer for 1 hour. Then filtered with Whatman No 42 filter paper, the resulting charcoal residue is ashed by heating in a furnace at a temperature of 600^0C for two hours, then cooled and moistened with HNO_3 concentrated, and then heated again to 600^0C for 1.5 hours. The ashes were dissolved in 25 ml of 1 M HCl solution and this solution was prepared for analysis of the Fe content for an activation temperature of 500^9C.

4. Sample preparation to determine the reduction of Fe content in coconut shell charcoal by washing $(NH_4)_2$ CO_3 1M At 54 Temperature^{0}C.

   Coconut shell charcoal is activated at a temperature of 500^0C heated in an oven at a temperature of 110^0C for one hour, then weighed 1.00 grams added 50 ml of 1M ammonium carbonate solution and then heated to 54^0C, and stirred using a stirrer for 1 hour. Then the mixture was filtered using a Buchner filter with a vacuum pump, and Whatman No 42 filter paper. The resulting charcoal residue was ashed according to procedure III.1.2.

5. Sample preparation to determine the reduction of Fe content in coconut shell charcoal by washing $(NH_4)_2$ CO_3 2M At 54 Temperature^{0}C.

   Coconut shell charcoal is activated at a temperature of 500^0C heated in an oven at a temperature of 110^0C for one hour, then weighed 1.00 grams added 50 ml of 2M ammonium carbonate solution and then heated to 54^0C, and stirred using a stirrer for 1 hour. Then the mixture was filtered using a Buchner filter with a vacuum pump, and Whatman No 42 filter paper. The resulting charcoal residue was ashed according to procedure III.1.3.

6. Analysis of Fe Metal with Atomic Absorption Spectrophotometer.

   Analysis of Fe metal in coconut shell charcoal includes the metal content in the initial charcoal or before activation, the metal in the charcoal after being activated and the metal in the charcoal after being activated and washed with ammonium carbonate solution, washing was carried out both at room temperature and at 54^0C. Fe metal was analyzed by Atomic absorption spectrophotometer.

7. Iron Analysis.

Preparation of standard solutions and standard solutions

Weighed 7.2340 grams of Fe (NO_3) $3.9H_2O$, then dissolved in a procedure such as making a 1000 ppm K standard solution. Into the solution added 2 ml of HNO_3 concentrated. From this 1000 ppm Fe standard solution, standard solutions were prepared with concentrations of 0.5 ppm, 1 ppm, 2 ppm, 4 ppm, 6 ppm, 8 ppm and 16 ppm.

8. Carbon Adsorption Test Against Iodine.

a. Preparation of 0.1 M Iodine Solution

Weigh 12.510 grams of KI and dissolve it in 15 ml of water, then put it into a 500 ml measuring flask, and add 6.5 grams of I_2, shaken until dissolved. The volume is made up to 500 ml by adding aquabides. Before use the solution is stored in a cool and dark place.

b. Preparation of 0.1 M Sodium Thiosulfate Solution

Weigh 26 grams of $Na_2S_2O_3$ and 0.2 grams of Na_2CO_3, both were dissolved in 1000 ml of aquabides. The solution was added with 10 ml of isoamyl alcohol and shaken regularly, then stored in a cool place.

c. Activated carbon adsorption test for Iodine

Samples in the form of coconut shell charcoal powder which have not been activated, which have been activated, and which have been washed at various temperatures and with various washing variations must be preheated in an oven at 110^0C for 1 hour. Then cooled. Then 1.00 gram of each sample was weighed and put into 50 ml of 0.025 M Iodine solution. After shaking for 15 minutes at room temperature, then allowed to stand for 5 minutes and filtered through filter paper. Filtrate was taken as much as 10 ml and titrated with Na solution$_2S_2O_3$ 0.1 M. After the brown colour of the solution changed to light yellow, 1% starch solution was added as an indicator. Titration was continued until the blue colour disappeared and each titration was carried out 2 times. The adsorption test for iodine was also carried out at concentrations of 0.05 M and 1 M. Calculation of the amount of iodine absorbed can use the following formula:

$$Iodine\ absorbed\ (mg/g = \frac{(10-V)XMX126{,}9X5}{IN}$$

Information:

V = Volume of sodium thiosulphate solution for titration

M = Molarity of thiosulfate solution

W = Sample weight in grams

9. Water Rate

Testing the water content is carried out by taking a test sample of activated carbon with a weight of about 1 gram as the initial weight (a). The test sample was dried in an oven at 110^0C for approximately 2 hours. Before weighing, the test sample is put into the desiccator to reach a balanced temperature and then weighed. Weighing is done until the weight is constant (b) (ASTM 2006).

$$Water\ level(\%) = \frac{a-b}{a} X100\%$$

10. Ash Rate

Ash level testing is done by taking a test sample weighing 1 gram as the first weight (a) then put into a porcelain cup, an empty cup weighed as weight (b) and a cup filled with charcoal dried at a temperature of 600^0C for 4 hours. Then the furnace is opened for a minute to complete the ashing process. The test sample is put in a desiccator and weighed as weight (c) (ASTM 2006).

$$Ash\ content(\%) = \frac{c-b}{a} X100\%$$

11. Volatile Substances

Testing the ash content is carried out by taking a test sample weighing 1 gram (a) and then heating it in an electric furnace until the temperature reaches 950^0C, after reaching the temperature, let it cool first in the furnace, then put it in a desiccator and weigh it (b). If there are white parts in the sample, the test must be repeated (ASTM 2006).

$$Volatile\ matter\ rate(\%) = \frac{a-b}{a} X100\%$$

RESULTS AND DISCUSSION

1. Determination of Fe Metal Content in Coconut Shell Charcoal Before and After Activation.

The activation process carried out on coconut shell charcoal is intended to remove the tar content attached to the surface of the charcoal. In proving this intention, it is necessary to conduct research on metal content in this case Fe metal in charcoal without prior activation or with activation.

To determine the content of Fe metal in activated coconut shell charcoal, it was destructed at 500°C^{0}C and flowed with gas N_2 from a gas cylinder through the bottom of the column with a flow rate of 20 ml/min. Then the activated (SA) and non-activated (TA) charcoal were analyzed using the Atomic Absorption Spectrophotometry method. The results of the analysis obtained the data presented in table 1.

Table 1. Content of Fe Metal in Coconut Shell Charcoal before and after Activation.

No	Sample	A1	A2	Average value	Concentration (ppm)
1	FACING	0,157	0,158	0,157	6,562
2	on	0,157	1,155	0,156	6,499

From the research data, it was found that the content of Fe metal in charcoal without activation was more than the content of Fe metal in activated charcoal.

2. Decreased content of Fe metal in Coconut Shell Charcoal after being activated and washed with $(NH_4)_2$ CO_3 1M And 2M At Room Temperature

To reduce the metal content in coconut shell charcoal, the activated charcoal is washed. The washing process is carried out on charcoal (which has been calcined at a temperature of 500^0C while flowing Nitrogen gas) using a solution $(NH_4)_2$ CO_3 1M and 2M at room temperature. In addition to room temperature, it was also carried out at 54^0C.

The reaction on washing by solution $(NH_4)_2$ CO_3 is actually an ion exchange reaction. With the functional groups present on the acidic carbon surface, the carbon will be able to act as a cation exchanger. When carbon is reacted with aqueous $(NH_4)_2$ CO_3 the carbon will be able to bind NH $ions_4^+$, because in solution $(NH_4)_2$ CO_3 releases NH $ions_4^+$, so that metals that can be exchanged in charcoal will be exchanged with NH $ions_4^+$ This.

This ion exchange is influenced by several things, namely metal cations and their cation valence. For the metal cation Fe^{3+} the ion exchange affinity is relatively small, this is due to the valence of the Fe $cation^{3+}$ including trivalent. The size of the metal cation Fe^{3+} is also relatively small, namely the radius of the metal cation Fe^{3+} is 0.53 Angstroms.

From the results of research and calculations, it is obtained that the higher the concentration of the solvent, the more Fe metal content is exchanged with ions, in this case NH $ions._4^+$. As presented in Table 2 I below.

Table 2. Content of Fe Metal in Coconut Shell Charcoal After Activation and Washing with $(NH_4)_2$ CO_3 1M and 2M at Room Temperature

No	Sample with Concentration	A1	A2	Average value	Concentration (ppm)	Rate Decrease (%)
1	1M	0,119	0,119	0,119	4,957	5,44 %
2	2m	0.112	0,113	0,112	4,687	5,44 %

3. Decrease in Fe Metal Content in Coconut Shell Charcoal After Activation and Washing with $(NH_4)_2$ CO_3 1M and 2M At 54 Temperature^{0}C

The treatment carried out in this process was the same as the treatment for reducing the Fe metal content which was carried out at room temperature, but the washing temperature was different namely 54^0C. This is to compare the results between washing in cold conditions and in hot conditions. From the research results and washing calculations at a temperature of 54^0C decreased Fe metal content in coal experienced an increase.

The factors that influence the washing or dissolving process are temperature and type of solvent. The type of solvent in this study used ammonium carbonate because, for a large scale or factory, waste from the materials used can increase soil fertility.

If the temperature of a solution is increased, the mobility of the ions from the solution will be higher. If the ion mobility is high, it will be more easily exchanged with other ions. According to Van't Hoff the effect of temperature on solubility can be expressed as follows:

$$\frac{d\ In\ S}{dT} = \frac{D\ H}{RT^2}$$

Information:

S = Solubility of the substance

T = Temperature

R = General gas constant (1.9872 cal/K)

$D\ H$ = Heat of solubility (Cal/mol)

If $D\ H$ is positive and the temperature has increased the solubility of the substance will increase. If the solubility is large, the metal cations are easily exchanged,

Table 3. Fe Metal Content in Coconut Shell Charcoal After Activation and Washing With $(NH_4)_2\ CO_3$ 1M and 2M At 54 Temperature ^{0}C

No	Sample with Concentration	A1	A2	Average value	Concentration (ppm)	Rate Decrease (%)
1	1M	0,118	0,117	0,117	4,895	6,39 %
2	2m	0.110	0,110	0,110	45682	6,39%

4. Reduction of Fe Metal Content in Coconut Shell Charcoal After Activation And Washing With $(NH_4)_2\ CO_3$ 1M and 2M At Room Temperature And Temperature 54^{0}C

The treatment carried out in this process is the same as the treatment for reducing the Fe metal content carried out in procedure 4, but with the same concentration (1M) and different washing temperatures, namely at room temperature and 54^{0}C. From the research data and calculations shown in Table 4 below, it turns out that the higher the washing temperature is increased, the ions from the solution will have higher mobility which facilitates ion exchange in it, and the higher the concentration of the washing solution, the affinity ion exchange that occurs in it will be greater.

Table 4. Fe Metal Content in Coconut Shell Charcoal After Activation and Washing With $(NH_4)_2\ CO_3$ 1M and 2M At Room Temperature and 54^{0}C

No	Sample with Concentration	Temperature	A1	A2	Average value	Concentration (ppm)	Rate Decrease (%)
1	1M	Like	0,119	0,119	0,119	4,957	1,25%
2	1M	54^{0}C	0,118	0,117	0,117	4,895	1,25%
3	2m	Like	0,112	0,113	0,112	4,678	2,24%
4	2m	54^{0}C	0,110	0,110	0,110	4,582	2,24%

5. Determination of Absorption of Activated Charcoal Against Iodine

Determination of the adsorption capacity (adsorption) of activated charcoal to iodine is a general requirement for assessing the quality of the resulting activated charcoal. Determination of the absorption capacity of the resulting activated charcoal. Determination of the absorption capacity of activated carbon for iodine is expressed as the number of milligrams of iodine absorbed in 1 gram of solid activated carbon. The Indonesian Industrial Standard has set a requirement for Iodine absorption, namely 200 mg/global warming, while the International Industrial Standard issued even higher, namely 500 mg/g. From the results of research and calculations, it is shown that the absorption capacity of activated charcoal for iodine is lower than the absorption capacity which has been determined according to the Industrial Standards in Indonesia. Data on the absorption capacity of activated charcoal on iodine with three repetitions are shown in Table 5 below.

Table 5. Absorption of Activated Charcoal Against Iodine

Iodine Concentration (M)	Repetition	Volume Natrium Thiosulfat (ml)	Iodine Number (mg/g)
0,025	1 x	0,12	156,69
0,025	2 x	0,18	155,76
0,025	3 x	0,23	154,97

6. The decrease in total Fe metal content observed before activation and after activation and washing with $(NH_4)_2$ CO_3 1M and 2M At Room Temperature And Temperature 54^{0}C

From the research data and calculations obtained from the observed decrease in the Fe metal content, it was found that the higher the washing temperature and the greater the concentration of the washing solution, then the decrease in the metal content in charcoal is greater than without activation and washing, the smaller the concentration obtained. Thus the content of tar attached to the surface of the charcoal can be eliminated or reduced, which results in the metals covering the carbon pores being easily washed away. Then the activated charcoal can be used.

CONCLUSION

The content of Fe metal in charcoal without activation is more than the content of Fe metal in activated charcoal, the higher the concentration of the solvent, the more Fe metal content is exchanged with NH ions$_4^+$. The higher the washing temperature is raised, the higher the mobility of the ions from the solution which facilitates the exchange of ions in it, and the higher the concentration of the solution $(NH_4)_2$ CO_3 washing, the affinity for ion exchange that occurs in it will be even greater.

The absorption capacity of activated charcoal for iodine is lower than the absorption capacity that has been determined according to the Industrial Standards in Indonesia.

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