International Journal on Advanced Science Engineering Information Technology

Vol.7 (2017) No. 3
ISSN: 2088-5334

The Making and Characterization of Husk Jengkol's Activated Carbon as Adsorbent

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***Abstract*— This research aims to make the adsorbent from the Activated Carbon at Husk Jengkol (ACHJ) and know the characteristics adsorbent ACHJ which have been processed. This study design is CRD (Completely Randomized Design) of 5x3 factorial and two replications. Factor A (size adsorbent) consists of 5 levels: A1 (20 mesh), A2 (40 mesh), A3 (60 mesh), A4 (80 mesh), A5 (100 mesh). Factor B (activator) consists of three levels: B1 (without activator / control), B2 (activator NaOH of 0.1 M), B3 (activator HCl of 0.1 M). The data was analyzed by analysis of variance (ANOVA) and a further test Duncan's New Multiple Range Test (DNMRT) at the 5% significance level. The results showed that there was a robust interaction between the size of the adsorbent and an activator that was used on all parameters of observation. The best adsorbent ACHJ has the size of 100 mesh with 0.1 M HCl activator, its characteristics: yield (23.69%), water content (2.84%), volatile content (1.13%), ash content (3.16 %), fixed carbon (92.87%), Iodine adsorption (1064.6647 mg / g) as well as having the largest pores of the adsorbent.**

***Keywords*— activated carbon of husk jengkol; activators; characteristics; size; adsorbent**

I. INTRODUCTION

Husk jengkol makes up 44% of the fruit jengkol and is a waste. [1] stated that the production of jengkol in Indonesia in 2009 amounted to 62.475 tons/year then the resulting with the total 49.087,5 ton/year of jengkol's husk. According to [2] the husk of jengkol, one kind of vegetables in Indonesia, contained cellulose, hemicellulose, and the number of other materials in which there are nitrogen, carbon, hydrogen, and oxygen. The compounds containing hydroxyl and carboxyl groups can bind the pollutants and makes it is good to be used as an adsorbent.

Adsorbent from agricultural waste is better to develop because of the availability of raw material is abundant, low cost, and the preparation process was simple [3]. Purpose adsorbent them is to purify the air and gas, purify the solvent, deodorizing in refining vegetable oils and sugar [4], to adsorb a variety of organic compounds, such as dyes, heavy metals [5].

As the growing industry which is followed by the need for the adsorbent, so that the needs of the activated carbon as one type of adsorbent will also continue to increase. To overcome this issue, it is necessary to diversify the sources of raw materials adsorbent so as to balance the needs of the industry on the adsorbent.

According to [6] activated carbon is a good adsorbent and is widely used for various applications due to their high surface area and porous. In the production of activated carbon, activation is an important process because this process will determine the quality of the resulting activated carbon both surface area and adsorption power. According to [7] material containing lignin and carbohydrates, activation is done chemically with acids. Chemical activation is more economical due to lower activation temperature and activation time shorter [8]. Activators are commonly used are HCl and NaOH.

According to [9] HCl is able to reduce the content of inorganic materials activated carbon and expands the pores of activated carbon. NaOH can expand the surface and increase the volume of the resulting microporous activated carbon, have a fairly low price and not corrosive [10]. Therefore, in this study, the husk of jengkol used as raw material for the manufacture of activated carbon with an activator HCl 0.1 M and 0.1 M NaOH ACHJ has evaluated its adsorbent characteristics.

II. MATERIAL AND METHOD

Preparation and analysis of ACHJ adsorbent have been conducted in the laboratory of Technology and Process Engineering of Agricultural Products, Chemical Laboratory, Biochemistry Agricultural Products Department of Agricultural Technology and Environmental Engineering Laboratory, the University of Andalas from February - June 2016.

A. Materials and Equipment

Materials used were obtained from the traders of husk jengkol in Raya's Market Padang, West Sumatra, Indonesia. Chemicals needed were NaOH (Smart Lab Indonesia), HCl (Smart Lab Indonesia), iodine (HIMEDIA Lab, India), $Na_2S_2O_3$. $5H_2O$ (Merk, Germany), starch (Merk, Germany) and aquadest obtained from a study carried out.

The tools used in this study were a SEM (Scanning Electron Microscopy) Hitachi S-3400N, Furnace Neycraft, Retsch Sieve Test No. 20, No. 40, No. 60, No. 80, and No. 100, and other supporting materials.

B. Design of Experiments

This study design is CRD (Completely Randomized Design) of 5x3 factorial with two replications.

Factor A (Size Adsorbent)
A1 = adsorbent 20 mesh size
A2 = adsorbent 40 mesh size
A3 = adsorbent 60 mesh size
A4 = adsorbent 80 mesh size
A5 = adsorbent 100 mesh size
Factor B (Activator)
B1 = Without activator (control)
B2 = Activator 0.1 M NaOH
B3 = Activator 0.1 M HCl

The data were performed with analysis of variance (ANOVA) and a further test Duncan's New Multiple Range Test (DNMRT) at the 5% significance level.

C. Implementation Research

1) Carbon Making Process : Jengkol husk washed and cut to a size of 2x2 cm and dried in an oven at a temperature of 105^oC for 3 hours. Then it burned in a furnace temperature of 300^oC for 30 minutes. The char was cooled at room temperature for 24 hours, grounded and sieved with20, 40, 60, 80, and 100 mesh.

2) Activation Process: Carbon activated by soaking each of them in HCl 0.1 M and 0.1 M NaOH for 24 hours, then washed with running water until pH 7. dried in an 110^oC oven for 3 hours. Retrieved adsorbent ACHJ and conducted one treatment without activation (as a control).

3) Observations: Observations were made on the adsorbent ACHJ include yield, moisture content, volatile content, ash content, fixed carbon, and Iodine adsorption. The procedure referred to the observation of the Indonesian National Standard (SNI 06-3730-1995), and adsorbent surface observations made by SEM (Scanning Electron Microscopy).

D. The observation of adsorbent's surface by SEM (Scanning Electron Microscopy)

The samples were observed affixed using glue on the spot conducting material. Then, it conducted the coating with Pt and Au where the sample was not damaged when scanning process. Samples were stored in a vacuum chamber before it was observed. Equipment connected to a computer for SEM photograph at the desired magnification.

III. Result and Discussion

A. Yield

The influence of the size of the adsorbent and the type of activator to yield an adsorbent ACHJ can be seen in Fig. 1. The size and type of activator adsorbent significant effected on the yield of the resulting adsorbent. The yield of the adsorbent was affected by the moisture content of leather jengkol, loss of organic compounds during the carbon process, lost product when sifting process, loss of product during the activation process (soaking and filtration).

The process of carbon produces the carbon which was increasing in line with the rising temperatures and the long activation and vice versa [11]. The use of activators of 0.1 M NaOH and 0.1 M HCl significantly affected to the yield ACHJ adsorbent. According to [9], chemicals are added in the activation of activated carbon can slow the reaction rate in the oxidation process. Thus, in addition to functioning as an activator, NaOH 0.1 M and HCl 0.1 M also served as a protective char from high temperatures.

B. Water Content

The influence of the size of the adsorbent and the type of activator to the water content of the adsorbent ACHJ can be seen in Fig. 2. There was a significant interaction effect between the size of the adsorbent and the type of activator to the water content of the adsorbent produced. In Fig. 2, it is known that the smaller the size of the adsorbent, the water content of the adsorbent is getting low. This is because of the surface of the adsorbent increasingly broad so as to accelerate the process of evaporation of water, hence the water contained in the adsorbent becomes less.

The resulting of adsorbent was highly hygroscopic so that the amount of the water content of the adsorbent was affected by humidity environment, so the more humid the air, the adsorbent will adsorb more water vapor in the air. In accordance with the statement of [12], the water content of activated carbon is affected by the hygroscopic properties of activated carbon, the amount of water vapor in the air, the long process of cooling, grinding and sieving.

Based on the Indonesian National Standard (SNI) in 1995, the water content of the adsorbent produced meets the required standards (<15%).

C. Volatile Content

The influence of the size of the adsorbent and the type of activator to the levels of volatile content the adsorbent ACHJ, it can see in Fig. 3. There was a significant interaction effect between the size of the adsorbent and the type of activator to the levels of volatile content of the adsorbent produced. The smaller the size of the adsorbent, the higher levels of volatile content because more moistures were trapped in the adsorbent. The volatile content existed on activated carbon usually trapped moisture content during the storage [13] and gas [14]. The more levels of volatile content generated, then the pore structure formed on ACHJ the better.

A non-activated adsorbent (control) had a higher level of volatile content and higher than the activation adsorbent. It was due to the control of adsorbent that has port was not open yet, so the content of water vapor and other vaporized materials were still trapped in the pores of the adsorbent.

Activated carbon adsorbent has very high hygroscopic properties that need to be packaged properly with airtight.

D. Ash Content

The influence of the size of the adsorbent and the type of activator to the ash content of the adsorbent ACHJ can be seen in Fig. 4. There was a significant interaction effect between the size of the adsorbent and the type of activator to the ash content of the adsorbent produced. From the data obtained, which mostly affected to the ash content of the adsorbent was a type of activator. Adsorbent without activation (control) has a higher ash content than adsorbent activated. According to [15] activation process reduces the mineral content of activated carbon. Mineral has a bind to an activator (NaOH 0.1 M and 0.1 M HCl) and form salt that soluble into the water, as can be seen in Tables 1, 2, and 3.

The entire adsorbent produced full theSNI standards (<10%). The best adsorbent has the size of 100 mesh, and an activator of 0.1 M HCl with ash obtained contains mineral oxides: Fe (0.47%), Na (0.74%), K (0.64%), Mg (0.73%), and Ca (1.59%).

E. Fixed Carbon

The influence of the size of the adsorbent and the type of activator to the fixed carbon ACHJ that bind adsorbent can be seen in Fig. 5. There was a significant interaction effect between the size of the adsorbent and the type of activator to the fixed carbon of the resulting of adsorbent bonded. The values fixed carbon was influenced by moisture content, ash content, and volatile content contained in the adsorbent. The higher levels of fixed carbon to activated carbon, the better the quality of the carbon produced, because the more the amount of fixed carbon in the carbon, the more activated carbon pores are formed [9].

According to the Indonesian National Standard (SNI) in 1995, the levels of fixed carbon/pure activated carbon min. 65%. Carbonation and activation of the process of making the adsorbent are able to eliminate the element of Hydrogen, Oxygen, or other substances in the pores, so it contains only carbon that becomes the dominant element of the adsorbent. All the adsorbent produced in this study has a value of carbon bonded / pure activated carbon> 80%.

F. Iodine Adsorption

The influence of the size of the adsorbent and the type of activator to the absorption of iodine adsorbent ACHJ can be seen in Fig. 6. There was a significant interaction effect between the size of the adsorbent and the type of activator to the absorption of iodine adsorbent produced. The smaller the size of the adsorbent, the greater Iodine adsorption because the surface of the adsorbent is expanded. An activated adsorbent having iodine absorption of greater than adsorbent without activation (control) was caused by the surface of the adsorbent without activation, and it is smaller than the surface of the adsorbent which has been activated. According to [7] the activation process causes the carbon atoms are selectively removed, thus forming pores, cracks, dented, so the activated carbon has a wider inner surface.

An activated adsorbent with 0.1 M HCl adsorption of its Iodine was greater than the adsorbent which was activated by 0.1 M NaOH; it is influenced by the ability of each different activators to expand the surface area of the adsorbent. According to [9], activation of the use of chemicals was also able to expand the surface of activated carbon, but the ability of a chemical to expand the surface area of activated carbon is different. According to [16], activated carbon adsorption capacity is affected by the pore size and surface chemistry.

According to the Indonesian National Standard (SNI), 1995 absorption of iodine-activated carbon min. 750 mg / g, There are 2 treatments that do not meet these standards, such as the adsorbent with 20 and 40 mesh size without activation. The adsorbent which has a value of the highest Iodine adsorption is 100 mesh size adsorbent with an activator HCl 0.1 M.

G. SEM (Scanning Electron Microscopy)

The SEM images ACHJ adsorbent that has been activated with 0.1 M NaOH, 0.1 M HCl, and without activation (control) at the size of 100 mesh obtained significantly different surface. The differences can be seen in Figs. 7, 8, and 9.

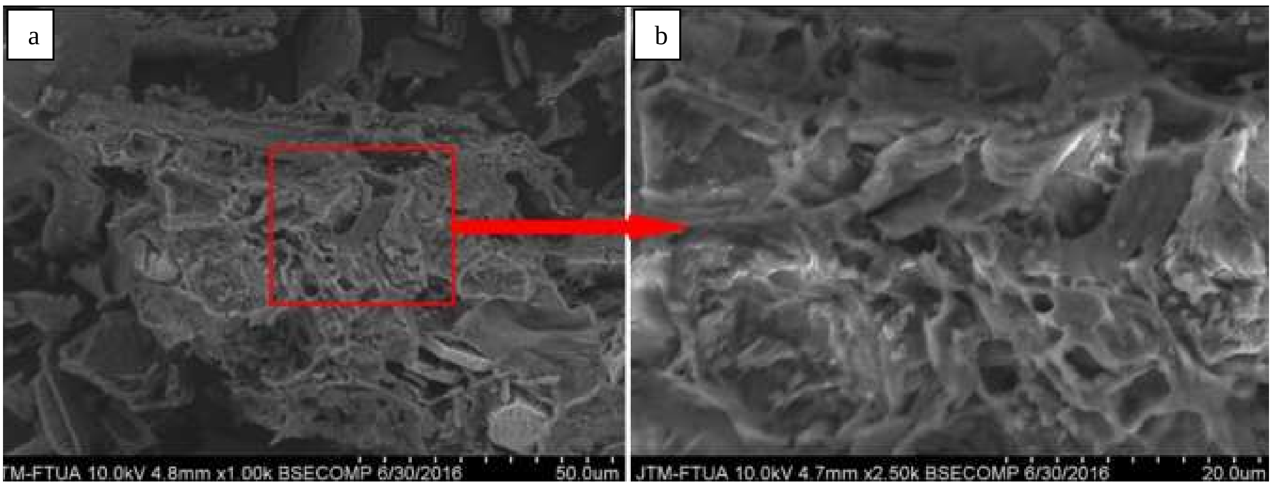

Fig. 7 SEM photos adsorbent ACHJ without activation (control) (a) magnification of 1000x (b) magnification 2500x

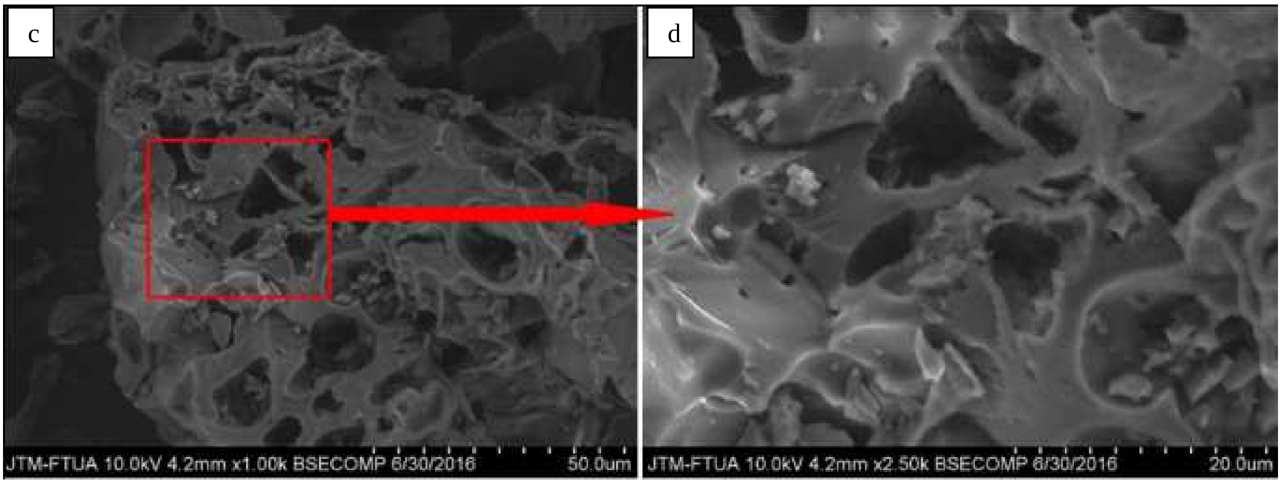

Fig. 8 SEM photos adsorbent with activator ACHJ at 0.1 M NaOH (c) magnification 1000x (d) magnification 2500x

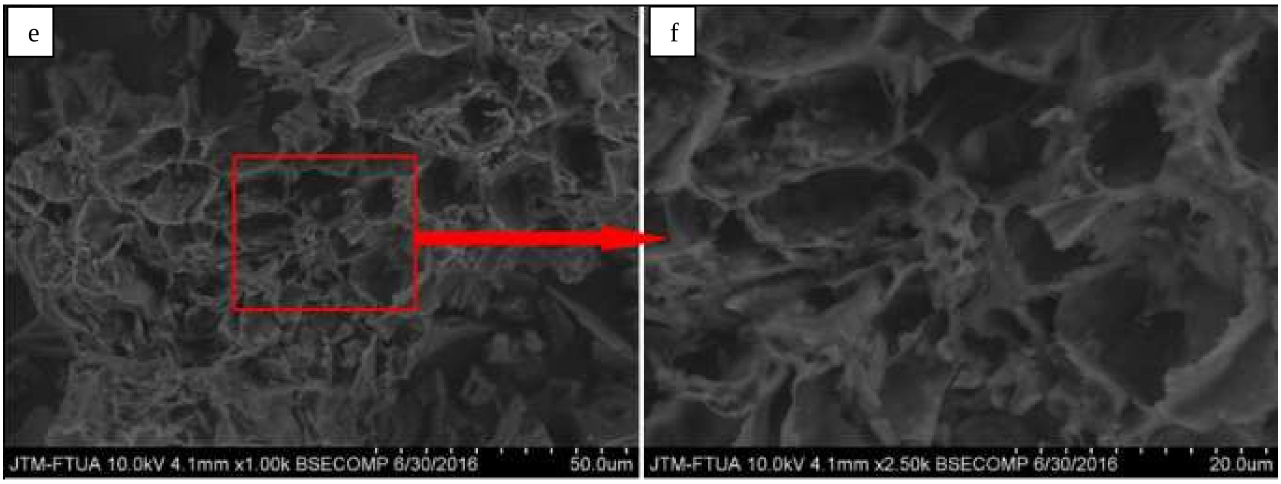

Fig. 9 SEM photos adsorbent with activator ACHJ at HCl 0.1 M. (e) magnification 1000x (f) magnification 2500x

The activation process is a process which is important because this process will determine the quality of the resulting adsorbent. Based on the picture above, the chemical activation process can expand the surface area of the adsorbent. Carbon control has pores that are less than with activated carbon. Carbon which is activated by 0.1 M HCl has pores more than the 0.1 M NaOH. According to [17], activated carbon is a unique material because it has a pore size molecular scale (nanometer). The pore has a strong force of Van der Waals.

Husk jengkol solid form before the charred as many volatile components before charred and activated. Authoring process (carbonation) causes the loss of some of the carbon component so that the pores of the carbon expands. This is evident in their morphological structure that can be seen in SEM [18].

According to [15] analyzed the chemical composition of activated carbon based on the percentage of C, H and O. The chemical composition of the activated carbon depends on the raw materials, methods of activation, the amount and concentration of activators, and the specific surface area. Meanwhile, the relation between the chemical composition and the preparation conditions and the mechanism of activation is rarely discussed. Thus, changes in the chemical composition of the material during the activation process yet can not be explained and still being researched.

The comparison of the value of EDX (Energy Dispersive X-rays) on each of the adsorbent can be seen in the following table. EDX value is taken randomly at three different point pores in each adsorbent.

TABLE I
VALUES EDX ADSORBENT ACHJ WITHOUT ACTIVATION (CONTROL)

No	Parameter Test	Spectrum I (%)	Spectrum II (%)	Spectrum III (%)
1.	C	62.97	70.45	99.40
2.	O	11.68	12.86	-
3.	Fe	1.57	16.69	0.50
4.	Na	1.36	-	0.17
5.	K	18.56	-	-
6.	Mg	-	-	-
7.	Ca	3.86	-	-
Total		100	100	100

TABLE II

VALUES EDX ADSORBENT ACHJ WITH ACTIVATOR 0.1 M NAOH

No	Parameter Test	Spectrum I (%)	Spectrum II (%)	Spectrum III (%)
1.	C	69.64	68.71	67.20
2.	O	22.33	26.44	12.95
3.	Fe	3.71	-	-
4.	Na	1.63	2.10	1.46
5.	K	1.91	0.56	6
6.	Mg	0.78	-	-
7.	Ca	-	2.18	12.39
Total		100	100	100

TABLE III

VALUES EDX ADSORBENT ACHJ WITH ACTIVATOR 0.1 M HCL

No	Parameter Test	Spectrum I (%)	Spectrum II (%)	Spectrum III (%)
1.	C	80.70	81.49	76.53
2.	O	16.99	15.34	15.25
3.	Fe	1.40	-	2.81
4.	Na	0.64	1.24	-
5.	K	0.25	-	-
6.	Mg	-	1.93	1.69
7.	Ca	0.03	-	3.73
Total		100	100	100

The value of EDX obtained is different from each treatment depending on the position of the selected EDX spectrum. EDX value describes the content of the element at the point of the spectrum in a specific area in the adsorbent pores. Test parameters are used only for certain elements as shown in Tables 1, 2, and 3.

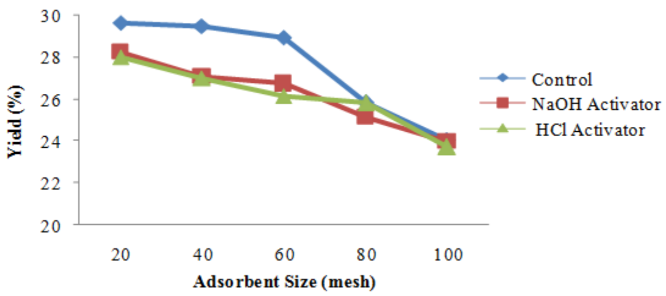

Fig. 1 Effect of adsorbent size and type activators against yield on adsorbent ACHJ

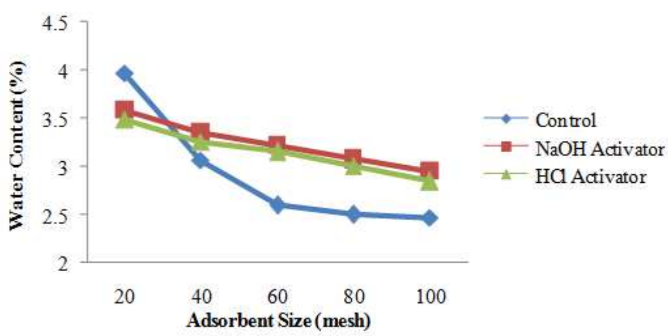

Fig. 2 Effect size and type activators adsorbent against water content on adsorbent ACHJ

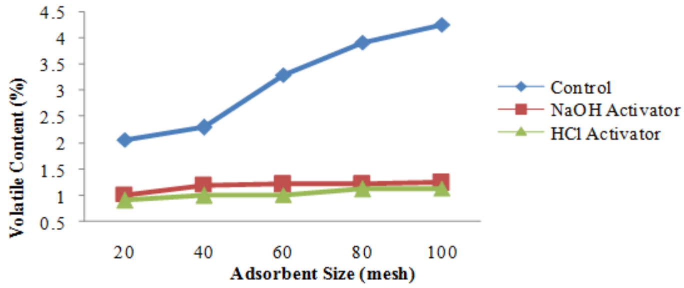

Fig. 3 Effect of size and type activators adsorbent against volatile content on adsorbent ACHJ

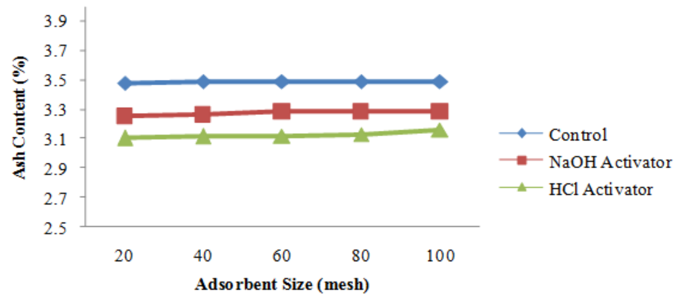

Fig. 4 Effect size and type activators adsorbent against ash content on adsorbent ACHJ

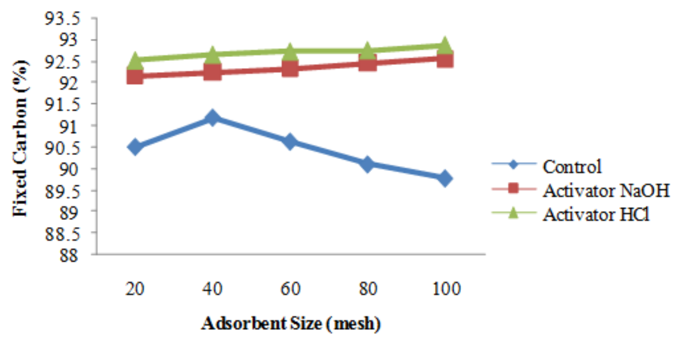

Fig. 5 Effect size and type activators adsorbent against fixed carbon on adsorbent ACHJ

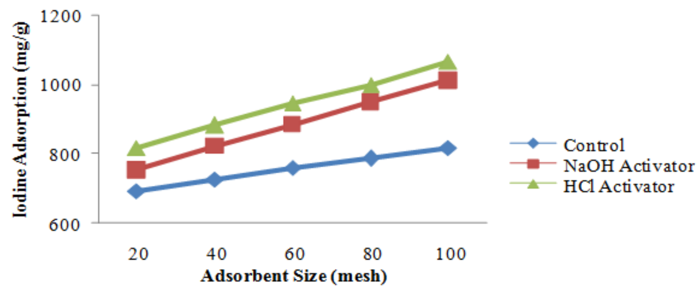

Fig. 6 Effect size and type activators adsorbent against iodine adsorption in adsorbent ACHJ

IV. CONCLUSIONS

Based on the research that has been done, it can be concluded as follows: The results show that there is a strong interaction between the size of the adsorbent with an activator that is used in all parameters of the observation. The best adsorbent is an adsorbent ACHJ with the size of 100 mesh and an activator of 0.1 M HCl with characterization adsorbent such as: yield (23.69%), water content (2.84%), volatile content (1.13%), ash content (3.16%), fixed carbon (92.87%), Iodine adsorption (1064.6647 mg / g) and has the highest adsorbent pores. All parameters fulfil the standard of SNI 1995. Rated EDX adsorbent contains C (80.70, 81.49 and 76.53%) and other minerals.

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