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The Characterization Of Conversion Low-Rank Coal From East Kalimantan Indonesia Through Physical - Chemical Activation To Be Adsorbent

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ABSTRACT

One of the largest coal reserves in Indonesia is in the Kalimantan region, specifically in East Kalimantan. Generally, there are three types of coal, namely anthracite, bituminous, and lignite. Lignite is a type of coal that is widespread and has low economic value as an energy source. Its relatively low calorific value makes this type of coal unsuitable for use as fuel. One way to increase the added value of low-rank coal is to convert the coal into active carbon which can act as an adsorbent medium. The adequate carbon value found in this type of coal makes it possible to convert it into an absorbent material. One of the important parameters of the absorbent material is the iodine number. The process of converting coal into an absorbent material is through an activation process which is carried out through a carbonization stage at a temperature of 600°C for 3 hours then followed by chemical-physical activation of the carbonization material which is soaked in a chemical mixture of phosphoric acid and sodium carbonate in 2.5 M for 8 hours. and physically activated at 800 °C for 1.5 hours. The results of increasing the carbon content were obtained when the low-rank coal was initially 38.68%w, then increased in the carbonization process to 68.62%w, and at the time of activation, the amount of carbon reached 73.79%w, indicating that the low-rank coal conversion process was fulfilled in terms of the quality of the results obtained.

1. Introduction

The adsorbent is an absorbent media that is widely used in various purification industries. Adsorbents are used to absorb several unwanted impurities found in a solution. One type of adsorbent used is activated carbon. Activated carbon is mostly made from biomass such as coconut shell charcoal, wood, etc [1]. The use of coal as an adsorbent need to be developed because the carbon elements use of adsorbents, particularly activated carbon, is crucial in various purification industries for effectively removing impurities from solutions. However, there is untapped potential in utilizing low-rank coal from Indonesia, especially in East Kalimantan, where abundant coal

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resources are currently underutilized due to their low economic value as a fuel. By activating the carbon element in coal, it can be transformed into active carbon, offering a cost-effective solution for absorbing impurities such as color and dissolved metals. With the right activation methods, low-rank coal can be harnessed to significantly enhance its adsorption capacity, making it a valuable resource for purification processes.t contained in coal can be utilized through an activation process to become active carbon. Coal in Indonesia, especially East Kalimantan, has abundant coal potential compared to other provinces in Indonesia, as a producer that is used as a source of energy in the form of fuel. There is still a lot of coal in East Kalimantan in the low-ranking category which is inefficient as fuel and is not used optimally because of its poor quality, low calorific value, and high sulfur. and ash content. Good coal has a calorific value above 5000 Cal, coal that has a high calorific value is very suitable for use as an energy source [2-3]. However, this does not mean that low-rank coal cannot be optimized for use, this low-rank coal could be used as active carbon which is an absorbent medium because it contains carbon. When used as active carbon, low-rank coal can be useful for absorbing impurities such as color and dissolved metals. Activation of coal originating from low-rank coal can be done by carbonizing it first to increase the amount of carbon, then it can be activated chemically or physically to optimize the active surface as an absorbent material, in this case, the aim is to increase the adsorption capacity of low-rank coal. It is important to know that coal is a heterogeneous solid organic and inorganic component, so coal may contain not only carbon elements but also several minerals contained therein. Low-rank coal can provide added value by using it as an adsorbent through by activation process, the activation is a process to increase the adsorption capacity by physical activation techniques and also by chemical activation techniques, for chemical techniques using chemicals (activators) which aim to build porosity and increase surface area. The addition of these chemicals will greatly affect the absorption capacity of the adsorbent, the type of chemical used can be acid, wet, salt, or a combination of one of them [4-5].

A material containing carbon elements can be called activated carbon because it is amorphous carbon which has a large surface area and internal volume so it has a high adsorption capacity. As an adsorbent, activated carbon is considered a very good medium in the process of adsorbing a dissolved substance, this is because many very small pores allow chemical and physical bonds to occur which can help the adsorption process of various other substances around it so that they can be adsorbed well on the surface. the activated carbon. The presence of these pores is very influential, the greater the number of pores, the greater the surface area of the activated carbon, and the better its absorption capacity will be. As has been said previously, the pores of activated carbon are greatly influenced by the activation method. There are at least two ways that can generally be used to increase its surface area, the first is the activation process through a physical process, namely with the help of high temperatures, and the second is through a chemical process where activation is carried out with the addition of certain chemicals, these chemicals can be in the form of acids or bases and a combination of both. To convert low-rank coal into active carbon so that it can be used as an adsorbent, in this research, the lowest-rank coal originating from East Kalimantan was used [6-9]. The carbonization technique is carried out first on low-rank coal to enrich the amount of carbon, after which the low-rank coal is physically activated at a temperature higher than the carbonization temperature. To improve the quality, chemical activation is also needed as chemical such as H_3PO_4, HCl, HNO_3, $ZnCl_2$, and alkali compounds. [10]. This research will use chemicals as activating ingredients, namely H_3PO4, and NH_4HCO_3. The results to be achieved in this research are focused on the characterization of the components contained in coal which undergo the carbonization process stages and subsequent physical-chemical activation using H_3PO_4-$NaHCO_3$. It is hoped that this research can contribute to the opportunity for low-rank coal to increase its economic value as a coal downstream product in the form of an adsorbent.

2. Methodology

2.1 The conversion process

The raw material is low-rank coal originating from East Kalimantan. For the activating agent, a chemical solution is used H_3PO_4 and NH_4CO_3 in a ratio of 1:1, with a concentration of each chemical of 2.5 M. Before the coal is used, it is analyzed first to determine the classification of the coal. The results were found that coal which has a calorific value of 3800 Cal is low grade caloric. This indicates that the coal used is truly low-rank coal. Next, the coal can be converted into active carbon or what is called an adsorbent, where the conversion process is shown in the following Figure.

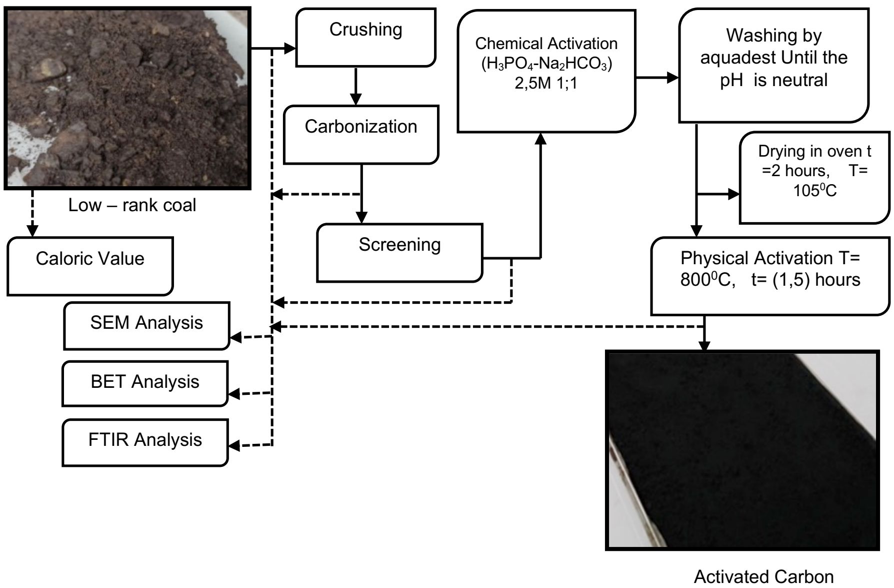

Fig. 1. Flow diagram activation process

The process of converting coal into an adsorbent is through a physical and chemical activation process which starts with the following sequence as shown in the picture above

i. First, the coal is crushed then carbonized to increase the amount of carbon, then screened to get a uniform size.
ii. Next, the coal is chemically activated by adding a mixture of chemical solutions, namely sodium bicarbonate and phosphoric acid, in the same ratio, namely 1: 1 with a concentration of 2.5 M. The mixture after the coal and chemical solution are mixed will then be left or soaked for 8 hours.
iii. After the soaking process is carried out, the coal is then washed repeatedly using water until the pH is neutral, then dried using an oven until the material remains constant.
iv. The final step is physical activation which is carried out at a temperature of 800^0C in a furnace for 1.5 hours.

2.2 Analysis by Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX)

The samples are ground low-rank coal, coal that has been carbonized and activated coal, and 2 g of each sample into the sample holder provided with carbon tape. Later, the remaining sample that is not attached is cleaned from the carbon tape, and then the sample is inserted into the SEM unit. In SEM-EDX measurement analysis, each sample is analyzed based on its area. The electron beam from the resulting sample shooting area is directed directly towards the sample. The beam stream emitted by electrons is focused using Columb optical electrons, and the electron beam formed will hit the sample, then converted into images through graphic SEM analysis with EDX analysis.

2.3 SAA Analysis by Brunaeur-Emmet-Teller (BET)

In principle, the Surface Area Analyzer (SAA) works based on degassing and analysis methods. SAA works by removing the absorbing gas (degassing) in a vacuum chamber for 3 hours. Next, the sample was put into a quartz container, which was placed appropriately, then liquid nitrogen was poured in, then the analysis was carried out using Quanta chrome Nova Win. The data obtained during analysis is in the form of pore diameter, surface area specificity, and pore volume. It is important to pay attention to using a sample weight of no more than 0.1 g in this test. Besides that, the BET method is used to determine the diameter and pore volume of a material using the BJH (Barrett-Joyner-Halenda) method. The BET test results are in the form of a graph of the relationship between P/Po and the BET transformation. Based on the graph obtained, it will provide an overview of the surface area, pore size, and total pore volume.

2.4 FTIR Analysis

FTIR is a technique for obtaining the infrared spectrum of absorption or emission of solids, liquids, or gases. In simple terms, the working principle of FTIR is to identify compounds, detect group functions, and analyze mixtures and samples being analyzed. infrared (IR) spectroscopy or FTIR (Fourier Transform Infrared spectroscopy). This spectroscopy is based on molecular vibrations. Infrared spectroscopy is a method that observes the interaction of molecules with electromagnetic radiation that is in the wavelength region at wave numbers 7,500 – 350 cm^{-1}. The working principle of an infrared spectrophotometer is photometry. Light from the source. Infrared light is a combination of different wavelengths. The light passing through the interferometer will be focused on the sample. The beam transmitted by the sample is focused on the detector. Changes in light intensity produce interference waves. These waves are converted into a signal by a detector, amplified by an amplifier, and then converted into a digital signal.

3. Results

3.1. Elements detection

With carbonization and activation treatment, it can cause an increase in the amount of volatile materials. This can apply to all types of raw materials that contain carbon bonds. This event can result in the remaining carbon chain structure becoming open, allowing the formation of pores in new materials. The activation process of low rank coal into activated charcoal which is carried out using phosphoric acid and sodium phosphate at a temperature of 800 °C for 1.5 hours is intended to open the small pores and reduce the hydrocarbon coverage on the surface of the activated carbon. The expansion of the pores of the material in this sense is the number of pores increasing per unit volume of the material due to the evaporation of degraded components and the release of volatile substances. So, with the reduction of volatile hydrocarbon compounds, the surface pores of the active carbon material become more clearly visible [11-13].

The results obtained from SEM-EDX analysis on low-rank coal samples show that the amount of carbon is initially low, and changes after carbonization treatment and activation, this is influenced by the opening of the pore structure. It can also be observed that several elements are also reduced or even lost from their initial conditions of sulfur, lead, nitrogen, and potassium. The changes in the composition of these elements can be seen clearly in Table 1 below

Table 1
Comparison of elements composition on conversion process by SEM analysed

Element name	Element Symbol	Comparison materials, Weight Conc (%)		
		Low rank coal (LRC)	LRC Carbonized	LRC Activated carbon
Carbon	C	52.56	63.85	73.79
Oxygen	O	32.32	23.30	16.99
Silicon	Si	4.65	2.99	2.49
Aluminium	Al	3.35	2.41	-
Nitrogen	N	3.31	2.60	3.82
Iron	Fe	1.74	2.25	2.04
Sulphur	S	0.90	1.12	-
Potassium	K	0.38	-	0.14
Calcium	Ca	0.33	0.98	-
Magnesium	Mg	0.31	0.50	-
Lead	Pb	0.15	-	0.17

3.2. Porosity Analysed

In the process of converting activated carbon derived from low-rank coal through the stages of carbonization and activation using a combination of physicochemical methods, several volatile compounds will evaporate and water bound to the coal, the carbonization process also enriches the rich carbon residue [14-15], this can be seen in the data generated from the SEM analysed in Table 1 above. The next increase in surface area is by going through the stages of physical - chemical activation. In the activation process, the carbon structure becomes more porous, this is influenced by the substitution of elements found in coal where the activating chemicals replace and eliminate several elements deposited in coal [16], as shown in Table 1 above. The effect of temperature also causes the loss of several elements and the formation of several new functional groups which can increase the pore size as shown in Figure 4. This can be seen in the comparison of the pore sizes in the process of converting coal to activated coal. as shown in Table 2.

Table 2
Comparison of elements composition on conversion process by SEM Analysed

Parameter	Unit	Comparision materials		
		Low rank coal (LRC)	LRC Carbonized	LRC Activated carbon
1.Surface Area Results				
Multipoint BET	m²/g	7.87198	175.483	250.005
BJH adsorption	m²/g	0.267226	22.9632	14.4723
BJH desorption	m²/g	0.163533	4.41063	8.81875
2. Pore Volume Results				
BJH adsorption cumulative micropore volume	cc/g	0.00175022	0.0423353	0.029541
BJH desorption cumulative micropore volume	cc/g	0.00104758	0.0100267	0.0150389
Total Pore Volume	cc/g	-0.00501579	0.123836	0.152025
3. Pore Size Results				
BJH adsorption pore radius	nm	13.0992	1.68674	1.68753
BJH desorption pore radius	nm	13.098	1.6945	1.70471
Average Pore Size	nm	-1.27434	1.41137	1.21617
Weight Sample	g	0.0519	0.0674	0.0702
Specific surface area Result		151,6759	2,603.6053	3,561.3248

The pores on the surface of the activated carbon will influence the surface area of activated carbon. The surface area of this activated carbon will determine the adsorption process. Adsorption is a surface phenomenon, the ability of activated carbon to adsorb many organic molecules that are in solution caused high pore structure, which provides a large surface area. Surface area can be analysed by gas media adsorption and solution media adsorption [17-19]. The surface area analysed with gas media is the amount of gas absorbed by activated carbon, is usually determined by the adsorption of nitrogen gas known as the Brunauer-Emmett-Teller (BET) method. Besides using the BET method, there is also a method Another way to determine the surface area is by adsorption of iodine solution, namely by measuring the portion of adsorbate that is lost from solution. For example, the portion of iodine adsorbed from a known solution its concentration is proportional to the surface area of the pores having a diameter of more than 10 Å [20].

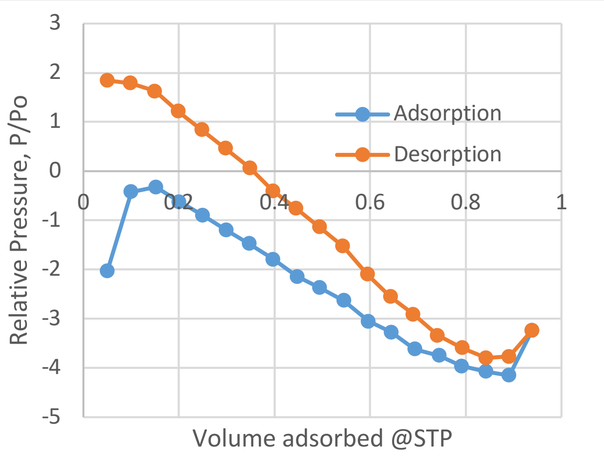

Fig. 2. The comparison adsorption and desorption Low rank coal

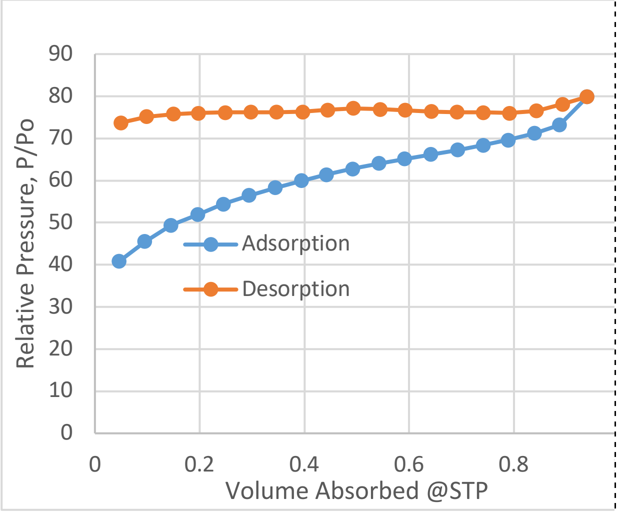

Fig. 3. The comparison adsorption and desorption Coal carbonized

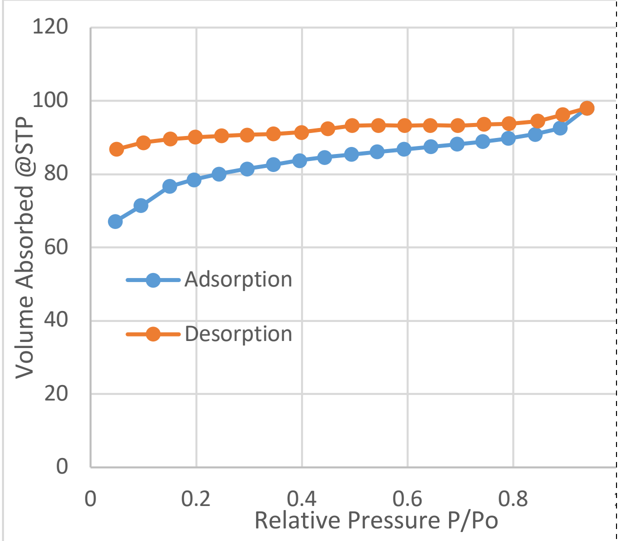

Fig. 4. The comparison adsorption and desorption Coal Activated

It should be noted that the higher the surface area adsorbent pore, the better performance of the adsorbent. The higher the pore surface area of the adsorbent indicates the smaller the size of the carbon particles active. By reducing the size of the carbon particles, the pore size is formed between the carbon particles and inside the particles The carbon is also small so the pore surface area on the adsorbent will be large. The smallest pore size of activated carbon desired so that the adsorbent has a high performance is the pore size on the micropore scale (1-2 nm). Pore size will affect the adsorption event. Adsorption is defined as a process in which a solid surface absorbs gas/fluid molecules due to a chemical and physical style. In the adsorption process, molecules attach to the carbon surface with weak forces known as Van der Waals forces due to intermolecular interactions. Activated carbon can be likened to a material that has a surface with millions of pores like a sponge. Activated carbon and adsorbed gas do not undergo chemical changes [21]. The figure 3 and 4 shows the adsorption and desorption events, the picture indicates that the activated carbon which has gone through the process of carbonization and chemical activation shows the pore size type which is in the micro and meso pore sizes. With such a range of pore sizes, Van Der Waals forces will make the adsorbate stored in the pores become stronger with very high density with such a pore distribution there will be good Van der Waals bonds, namely the presence of intermolecular attractions due to adjacent chemical groups. Van der Waals bond will work optimally with a tiny pore size (micropore) which is about 1.1 nm, the Van der Waals forces give an enormous energy to adsorb molecules adsorbate onto the pore walls of the adsorbent. However, if the pore size is too small, it will not be able to accommodate the adsorbate molecules because the adsorbent becomes too large solid so unable to move within the pore, this is because there are not enough cavities for the occurrence of intermolecular van der Waals bonds [22-23]. Which shows a decrease in the adsorption/desorption volume for each increase in relative pressure. The surface area of activated carbon is affected by the internal structure of the pores on the surface.

3.3. Functional Group Analysed

The FTIR testing describes the presence of functional groups at the stage of carbonization and coal activation, this functional group is important because it is related to the porosity size of solid material, the more functional groups in a solid material, the better the porosity size. Below is a picture that shows the comparison of functional groups in the initial low-rank coal conditions, and low rank coal after carbonization and after activation.

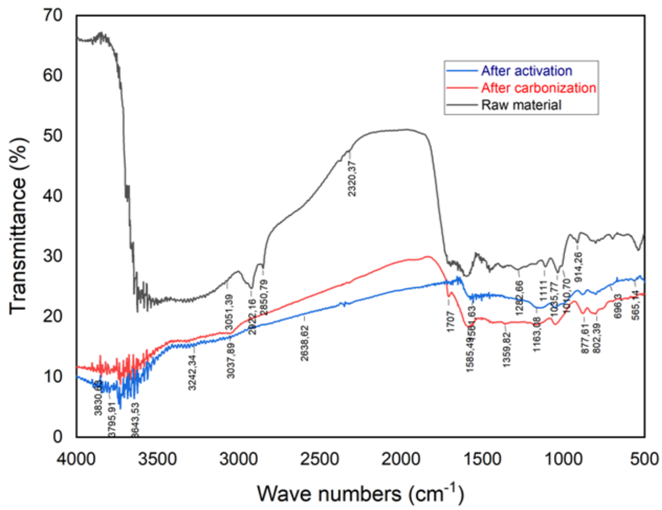

Fig. 3. The comparison of presence of functional group of Low - rank coal, Coal carbonized, Coal Activated

The peak appears at a wavelength of 675-995 cm^{-1} which may indicate the presence of a CH Alkene functional group which usually appears at a wavelength of 3010-3095 & 675-995 cm^{-1}, this occurred in all three types of experiments, both on uncarbonized coal data. as well as after carbonization and after being treated with activation but there was a very wide difference in the transmittance percent where the activated coal was lower than the carbonization and the initial low-rank coal. The same thing also happened at the wavelength of 690-900 cm^{-1} which possibly indicates the presence of an aromatic ring CH functional group which usually appears at wavelengths 3010-3100 & 690-900 cm^{-1}, as well as a peek at wavelength 1050-1300 cm^{-1} which may indicate the presence of a CO functional group Alcohol/ether/acid carboxylate/ester which usually appears at that wavelength and peaks at 1500-1600 cm^{-1} which may indicate the presence of a functional group C=C aromatic ring which usually appears at that wavelength, but in all cases above, it shows a difference in percent transmittance which shows that there is an activation effect on the coal structure where after activation has coal, the percent transmittance is lower than that of coal. Other [24]. the effect of carbonization and activation also shows the formation of new functional groups and the loss of other functional groups, namely in carbonization and after activation, alkane functional groups are removed because in low-rank coal before carbonization and activation, FTIR data shows peaks appear at a wavelength of 2850-2970 cm^{-1} which may indicate the presence of the CH Alkane functional group which usually appears at wavelengths 2850-2970 & 1340-1470 cm^{-1}. Furthermore, specifically after the activation of the FTIR data, it shows the emergence of a new functional group, namely the appearance of a peak at a wavelength of 1690-1760 cm^{-1} which may indicate the presence of a functional group C=O Aldehydes/ketones/carboxylic acids/esters which usually appear at these wavelengths, the presence of functional groups in active carbon is considered to play a role in the adsorption process through ionic bonds [25].

4. Conclusions

From the results of the research carried out, it can be concluded that the characteristics of low-rank coal which is converted into an adsorbent are that there are several minerals that cover the pores of the coal, but with carbonization and activation techniques it is known that a number of these mineral elements can be reduced, including the amount of Pb and K that were originally found in the coal. low-rank coal becomes zero in coal after carbonization. The same thing was also found in the levels of S, K, Pb, and Mg which were originally found in low-rank coal, becoming zero when converted into adsorbents. The carbon element increased after the conversion was carried out, where the initial carbon content was 38.68%, increased to 68.62% after going through the carbonization stage, and then increased to 73.79% after going through the activation stage. Activated carbon that has gone through a carbonization and chemical activation process shows pore size types, namely micro and meso pore sizes. It is worth suspecting that based on the results of functional group analysis the resulting activated carbon indicates the presence of carboxyl (-COO), hydroxyl (-OH), and carbonyl (=O) functional groups, where these functional groups play a very important role in improving the adsorption performance of activated carbon.

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