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Effect of phosphoric acid composition in the production and characterization of coal activated carbon

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Abstract: Liquid waste containing heavy metals must be treated before being released into the environment. This study investigated the potential of coal-derived activated carbon as an adsorbent in AMD (Acid Mine Drainage) treatment. Coal was activated with H3PO4 with five weight ratios (10%, 20%, 30%, 40%, and 50%), carbonized for 120 min at 650 °C, and subjected to the proximate analysis of activated carbon. The results showed that the water content, ash content, volatile matter content, and fixed carbon were generally in accordance with Indonesian standards (SNI). The results of FT-IR and SEM on BET surface area and total pore volume of activated carbon were 296.4 m2/g and 0.1562 cc/g, respectively, indicative of sizeable pores, cracks, channels, and active groups. The iodine outnumbered the minimum limit in the SNI, and the activated carbon quantity consistently increased with H3PO4 ratios. The iodine produced with 10%, 20%, 30%, 40%, and 50% H3PO4 was 956.95 mg/g, 967.13 mg/g, 1,221.63 mg/g, 1,206.346 mg/g, and 865.32 mg/g., respectively.

1. Introduction

Activated carbon is extensively utilized in the purification of drinking water and treatment of wastewater and air pollution. It efficiently eliminates metals and chlorinated chemicals, which bring unpleasant taste and odor, exhibits the most powerful physical attraction, and possesses the most porous space for adsorption[1][2][3]. As a result, it boasts a huge surface area for capturing pollutants. Activated carbon is derived from various sources, including almonds, wood, coconut and walnut shells, and coal. Activated carbon is recognized for its affordability, well-established pore architectures, and high capacity for adsorption [4][5].

Previous studies have reported activated carbon from varied sources, such as bituminous coal with 1,198 mg/g iodine yield [6] [7], and a mixture of H3PO4-NH4HCO3 and bituminous coal raw material with 1,238 mg/g iodine yield [8]. The activation process using ZnCl2, K2CO3, NaOH, and H3PO4 with sub-bituminous coal raw material was conducted by Hardianti et al., resulting in 1,238.5mg/g iodine [9] An activated carbon contains spaces occupied by free water molecules, enabling adsorbent properties for wastewater [10]. Studies show that chemically activated carbon produces a higher uptake of metal ions than activated carbon without activation [11]. A study has used medium-rank coal as the main active carbon material [12] and other research has reported chemical activation using low-rank coal, which has abundant reserves but low calorific value, thus less profitable as an energy source [6][8][13][14].

Adsorption is a process in which a component moves from one phase to another surface, resulting in a change in concentration on the surface. The absorbed substance is known as adsorbate, whereas the

absorbing substance is adsorbent. Activated carbon raw materials derived from plants, wood, animals, waste, and minerals containing carbon can be converted into activated carbon through a carbonization combustion process [15]. These materials include bones, softwood, husks, corncobs, coconut shells, and coal. Around 60% of a large coal reserve in Indonesia is of low quality and less valuable as a fuel source. The use of low-rank coal as raw material in producing activated carbon is reportedly promising due to its availability and economy; low-rank coal with phosphoric acid activation produced iodine numbers below 1.000 mg/g [16]. In this research, activated carbon is made using sub-bituminous coal with phosphoric acid activation using medium-ranked coal and only a single activator of phosphoric acid. It highlights the opportunity of utilizing medium-rank coal and phosphoric acid as the sole activator to produce an adsorbent with high adsorption capacity.

2. Method

2.1 Coal Preparation

The coal sample was weighed (3 kg), and the size was reduced using a jaw crusher. The sample was oven-dried at 30oC-35oC for 6h, and then the size was reduced again using a hammer mill to pass the 60-mesh sieve. The sample was divided using a mechanical divider, a splitter, or both, or the coning or quartering method. Exactly 2/8 parts of the sample were weighed for treatment, air dried at 30°C for 6h, and analyzed for coal quality based on parameters, including the total moisture, volatile matter, coal ash, fixed carbon, total sulfur, and calorific value.

2.2 Preparation of Carbon Activation Using Coal

The coal was weighed for treatments with five coal to H3PO4 ratios (based on weight, in percentage), namely 90:10 (AC1), 80:20 (AC2), 70:30% (AC3), 60:40 (AC4), and 50:50 (AC5). Then, coal and H3PO4 were incorporated into a container, mixed well until smooth, and let stand for one day (24 h). The dried residue for carbonizing activated carbon was placed inside a steel box, then into a muffle furnace with nitrogen gas flowing for 1.5 h at 650oC, let cool for 1h by allowing nitrogen gas flow in the muffle furnace. The activated carbon was washed in distilled water (80oC-90oC) with consistent stirring until the pH reached 5, then filtered using filter paper and funnel.

2.3 Characterization of Coal-activated Carbon with Phosphoric Acid Activation

Coal-activated carbon with phosphoric acid was characterized in several analyses. Iodine number testing determined the ability of activated carbon to adsorb substances with smaller molecular sizes, expressed in the amount of iodine yield. The surface composition and morphology (pore shape) of the sample were identified from various signals produced by the scanning electron beam method, Scanning Electron Microscope (SEM). These signals were provided by Fourier-Transform Infrared Spectroscopy (FTIR) analysis, as well as Brunauer-Emmett-Teller (BET) analysis for pore area, diameter, and volume.

3. Result and Discussion

3.1 Coal Quality Analysis

Data obtained in coal quality testing included moisture, coal ash, volatile matter, fixed carbon, total sulfur, and calorific value. As presented in **Table 1**, the coal rank was determined by changing the unit of calorific value analysis results to Btu/lb, demonstrating that the coal used in this study was categorized as the sub-bituminous coal class B with a calorific value of 9,500-10,500 Btu/Lb, classified as medium rank coal.

Table 1 Coal Quality Analysis

Parameter Analysis	Analysis Result	Unit	Base	Methods
Moisture	16,58	%	Adb	ASTM D.3173
Ash	2,9	%	Adb	ASTM D.3174
Volatile Matter	43,82	%	Adb	ASTM D.3175
Fixed Carbon	36,7	%	adb	ASTM D.3172
Total Sulfur	0,38	%	adb	ASTM D.3177
Calorific Value	6052,38	Cal/g	adb	ASTM D.5685

3.2 Proximate Analysis of Coal Activated Carbon

Based on the analysis (see Table 2), the moisture content and adsorption of iodin met the SNI standard, whereas volatile matter content, ash content, and fixed carbon were below the SNI. Low moisture content indicated little bonding with water vapor, and the pores of activated carbon were only slightly covered by the remaining moisture content, thus enlarging the surface area of the carbon. Ash content, an additional factor, can affect pore clogging and surface area; the higher the ash content, the more pores clogged, and the smaller the surface area [17][18][19].

Table 2 Proximate Analysis of Coal Activated Carbon

Requirement Type	Analysis Results					Quality Standards SNI 06-3730-1995
	AC1	AC2	AC3	AC4	AC5	
Water Content	3,8%	13,3%	6,8%	13,2%	13%	Max. 15 %
Ash Content	5,7%	9,9%	11,7%	1,8%	12,2%	Max. 10 %
Volatile Matter Content	20,6%	22,1%	24,6%	23,9%	25,4%	Max. 25 %
Fixed carbon Content	59,9%	54,7%	56,9%	61,6%	49,4%	Min. 65 %
Iodine number	956,9 mg/g	967,1 mg/g	1221,6 mg/g	1206,3 mg/g	1191,09 mg/g	Min. 750 mg/g

The high volatile content was due to the binding of carbon, water vapor, and non-carbon compounds such as nitrogen, hydrogen, and oxygen on the activated carbon surface, leading to a lower adsorption capacity. On the other hand, low fixed carbon content indicated that water vapor reacted with carbon atoms to produce carbon dioxide and carbon monoxide. Although the fixed carbon content was lower than the standard (65%), the other parameters were appropriate, so this activated carbon is arguably of good quality.

3.3 Iodine Number Analysis of Coal-activated Carbon

The adsorption capacity of activated carbon from this study was tested using the iodine number method to identify how many milligrams of iodine were adsorbed by 1 gram of activated carbon, expressed mg/g. The iodine numbers of activated carbons with 10%, 20%, 30%, 40%, and 50% H_3PO_4 were 956.95 mg/g, 967.13 mg/g, 1221.63 mg/g, 1206.346 mg/g, and 1,191.09 mg/g, respectively. These results were above the minimum standard of SNI, namely 750 mg/g (Table 2).

Figure 1 shows that the iodine number of activated carbon increases with the percentage of H3PO4 in the activation process. When activated, H3PO4 can catalyze the breaking of bonds in biopolymers found in coal. It can also link together the pieces of these biopolymers through cyclization and condensation, forming phosphate and polyphosphate bridges [20][21]. As a result, the acid fills crevices, prevents the material from shrinking, and produces a porous structure when washed away with water [22]. An adverse trend is observed in the adsorption capability following a particular H3PO4 composition. The decrease in adsorption capacity at high H3PO4 concentration is attributed to the formation of phosphate and polyphosphate linkages, which create a barrier that covers the pores. Excess acid expands the previously existing pores in activated carbon, resulting in more reactive sites created and carbon converted into a mesoporous structure instead of new pores formed [23].
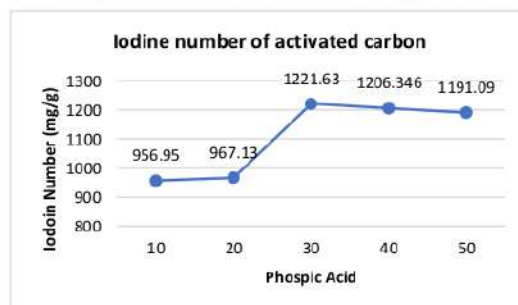

Figure 1. Graph of Phosphoric Acid Composition against Iodine Number

3

3.4 Scanning Electron Microscope (SEM) Analysis

In Figure 2, the coal surface in the SEM test shows considerable voids and clogged dirt. The surface texture appears quite rough, with larger voids, scattered on the activated carbon that has been activated with phosphoric acid. The surface texture of activated carbon (B) looks rough compared to that of coal (A) due to the reaction of functional groups on the surface with phosphoric acid [24].
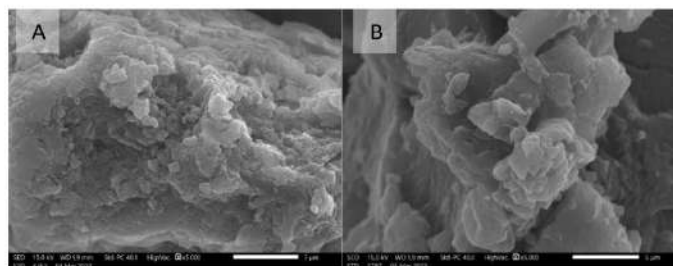

Figure 2. Scanning Electron Microscope Results (A) Coal, (B) Coal Activated Carbon With Phosphoric Acid Activation (AC3)

3.5 Brunauer-Emmett-Teller (BET) analysis

In the BET test results, the surface area of 60 mesh coal was 2.876 m2/g, but when activated, the carbon increased to 296.379 m2/g, indicative of an enlarged surface area (AC3). The pore volume of coal in the activated carbon using phosphoric acid increased from 0.0039 cc/g to 0.1562 cc/g. The pore diameter of 60 mesh coal was 27.3252 Å when measured but dropped to 10.5419 Å after activation and carbonization. A larger surface area will increase the pore volume, whereas a small pore diameter will increase the number of pores and enlarge the surface [25].

3.6 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

Figure 3 the adsorption peaks of coal and activated carbon using phosphoric acid. At a wavelength of ±2908.37 cm-1, there was a hydroxy OH group that can bind metal ions and increase pH. Carboxylic groups were presented at a wavelength of ±1635.92 cm-1, ±1594.51 cm-1, and ±1445.71 cm-1, and ether groups were presented at a wavelength of ±1286.17 cm-1. The adsorption peak of activated carbon with coal may increase or decrease due to the phosphoric acid activation process [24].
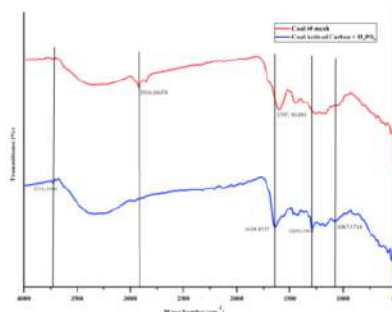

Figure 3. Fourier Transform Infrared Spectroscopy (FTIR) of coal activated carbon using phosphoric acid (AC3)

4. Conclusion

The coal for activated carbon was categorized as the sub-bituminous B coal class. In the proximate test of coal-activated carbon, the moisture content and adsorption capacity of iodine met the national standard (SNI), but substandard values were observed for ash content, volatile matter content, and fixed carbon content. Despite this partial standard accomplishment, the activated coal has demonstrated a good quality due to contributions of other parameters: the maximum capacity and large adsorption

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power. The largest number yield was 76% in the ratio of 10% H3PO4, while the largest iodine number was 1,221.63 mg/g in the ratio of 30% H3PO4. Conclusively, the surface area increased when coal was utilized as the activated carbon, demonstrated by the 296.4 m2/g of BET surface area and 0.1562 cc/g of total pore volume of activated carbon. Further SEM analysis showed that the surface had a substantial number of pores, cracks, channels, and active groups. The adsorption peaks of coal and activated carbon using phosphoric acid, as a result of FTIR analysis, fluctuated due to the phosphoric acid activation process.

5. Acknowledgments

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