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Production of Activated Carbon from High-Grade Bituminous Coal to Removal Cr (VI)

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Abstract

Activated carbon was produced from high-grade bituminous coal, potentially removing Cr metal in textile industrial waste. The purposes of this study were 1) getting activated carbon characteristics, 2) studying the impact of reagent concentration and activation temperature on the activated carbon characteristics, and 3) getting the isotherm adsorption equation for activated carbon developed by Freundlich and Langmuir on Cr metal. This research used an experimental method with a laboratory scale, which means the manufacture of activated carbon and a test of adsorbs power of activated carbon to the Cr metal were conducted in the laboratory. Activated carbon manufacture through carbonization process of chemical activation used reagent $(NH_4)_3PO_4$, neutralization, filtration, physical activation, and cooling. At the same time, it tested the adsorption power of the activated carbon to Cr metal through activated carbon contact with a solution containing some Cr^{6+} ion. The update in this research was using reagent $(NH_4)_3PO_4$ and the test of adsorption power of activated carbon to Cr^{6+} ion contained in the artificial waste. This research showed activated carbon that has been activated using reagent $(NH_4)_3PO_4$ 0.5 – 2 M at physical activation temperature of 825°C and has met the standard of SNI number 06-3730-1995. The best-activated carbon was produced from chemical activation using $(NH_4)_3PO_4$ 1 M solution and physical activation at 825°C. The best-activated carbon characteristics contained 1.27% water; 17.17% content of volatile matter, 9.39% was ash content; 73.17% contained fixed carbon, and the iodine value was 1248.30 mg/g. The best Equation of Langmuir isotherm adsorption of activated carbon to the Cr^{6+} produced Constant Ar = -90.0901 and Kc = -0.0075.

Keywords: *Cr; Freundlich; Langmuir; high grade bituminous; activated carbon; $(NH_4)_3PO_4$*

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INTRODUCTION

Some textile industrial wastes are still containing more Cr metal than the waste quality standard, so it pollutes river water, plants, and groundwater around the waste disposal (Biju et al., 2022; Brilian & Muntalif, 2019; Dwi Martina Dewi, 2016; Paul, Goswami, Pegu, & Bhattacharya, 2020). The chromium content in river water comes from textile industry waste. This waste contains Cr between 0.157-0.77 mg/L (Brilian & Muntalif, 2019). Meanwhile, the maximum limit for total chromium content in textile industrial waste based on East Java Governor Regulation Number 52 of 2014 is 0.2 mg/L (Governor, 2014). River water contaminated with chromium can endanger the health of people who consume river water. (Kusdarini et al., 2021; Kusdarini et al., 2019; Kusdarini et al., 2020). One of the methods to decrease chromium content in wastewater was the adsorption method.

Adsorber can be produced from organic materials, like coal, still abundant in Indonesia. Several studies have been conducted to develop activated carbon from coal. Previous studies showed that activated carbon from bituminous coal with low grade had met the standard of SNI number 06-3730-1995 as a parameter of water and ash contents, but this standard has not met the parameter standard of volatile and fixed carbon content and the maximum specific surface of activated carbon was 86,213 m_2/g (Kusdarini et al., 2017). Another study showed activated carbon from lignite coal has a reasonably good surface area of 1288.8 mg/g. However, this activated carbon has not been shown to meet all the parameters of the SNI standard number 06-3730-1995. Another study showed activated carbon from lignite coal has a reasonably good surface area of 1288.8 mg/g. However, not all parameters of the SNI standard number 06-3730-1995 are met by this activated carbon. (Suliestyah & Astuti, 2021).

Further, the study has shown that activated carbon from anthracite coal has a reasonably good maximum specific surface area of 3136.80 m_2/g, and the capacity of maximum adsorption to CH_4 gas was 19.31 mmol/g. The primary use of anthracite coal as fuel, so the lower grade coal for activated carbon was interested in conducting (Liu et al., 2022). Previous research has shown that activated carbon, made from coal with low carbon content, could absorb basic blue dye. The adsorption capacity was 8.0 to 9.3 mg/g, and the activated Carbone doses were 2 mg/mL, pH 9, and a contact time of 120 minutes with a q max between 17 to 30 mg/g (Shaida et al., 2022). The research on activated carbon manufacture from coal gangue and bamboo explained that the combination of zeolite and activated carbon had an adsorption capacity to the Cu^{2+} of 104.9 mg/g and Rh-B was 83.4 mg/g (Li et al., 2021). The study of activated carbon manufacture from coal was interesting, but several results showed that there were parameters that had met SNI standards or other standards, and some of the results have not tested for the parameters and adsorption capacity for chromium pollutants.

The process of activated carbon making consists of three main stages: dehydration, carbonization, and activation. Dehydration is water removal from raw materials by heating to 170ºC. Carbonization is the combustion of the raw material using little air at 200 to 900 ˚C, making any decomposition of organic compounds compiling the material structures become methanol, acetic acid vapor, tar, and hydrocarbons. The solid materials left after the canonization process were carbon in charcoal with a narrow specific surface. The quality standard of activated carbon in Indonesia is SNI 06-3730-1995 (Table 1).

Table 1. Activated carbon requirements based on Indonesian National Standard (SNI) 06-3730-1995 (BSN, 1995)

Requirements Type	Parameters
Water content	Maximum 15%
Ash content	Maximum 10%
Evaporation level	Maximum 25%
Bonded carbon content	Minimum 65%
Iodine number	Minimum 750 mg/g
Absorption of Benzene	Minimum 25%

The research on activated carbon manufacture with the novelty of using high-grade bituminous coal as raw material, reagent of $(NH_4)_3PO_4$, and activation temperature was higher than in previous studies (around 800ºC) was interesting.

MATERIALS AND METHOD

Tools and Materials

The materials used to make activated carbon were coal, ammonium phosphate, sodium hydroxide, iodine, sodium sulfite, starch, and chromic acid. The coal was a bituminous type, as shown in Figure 1. The tools used were analytical balance, porcelain dish, grinder, dropper, filter paper, measuring flask, burette, clamp, beaker glass, funnel, furnace, aluminium foil, measuring cup, cans, and Erlenmeyer.

Figure 1. Crushed coal

Coal Proximate Analysis

Coal parameter tests consisted of water content tests (ASTM Designation D.3173-92), volatile matter content (British Standard BS.1016), and ash content (ASTM Designation D.3174-98). Then, the fixed carbon content was being calculated based on the formula:

Fixed carbon content = 100% - (water content + volatile matter content + ash content (1)

Activated Carbon Manufacturing

The procedure of activated carbon manufacturing consists of several stages, such as 1)sample preparation, which means pulverizing coal, sifting with a 60 mesh sieve, and the coal used was only passing 60 mesh; 2) coal heating in the furnace for 3 hours at the temperature of 600ºC; 3) coal charcoal cooling and then coal charcoal is divided into four parts; each activated uses 0.5 M $(NH_4)_3PO_4$ solution; 1 M; 1.5 M and 2 M at the room temperature for eight hours; 4) coal charcoal is neutralized at the temperatures of 625ºC, 725ºC, and 825ºC for 3 hours.

Activated Carbon Analysis

Activated carbon tests consisted of water content test (ASTM Designation D.3173-92), volatile matter content (British Standard BS.1016), ash content (ASTM Designation D.3174-98), and iodine value (ASTM D4607-94), dan SEM-EDX. The Fixed carbon content was calculated based on Equation (1).

Test of Activated Carbon Adsorption Power on Cr^{6+} Ion

The first step was making a solution of CrO_4 from a solid of CrO_3 with several concentrations of 20 ppm, 105 ppm, 90 ppm, 75 ppm, and 60 ppm. A gram of activated carbon was contacted with 1000 mL CrO_4 for each type of concentration and stirred at 400 rpm for 4 hours. Then, the concentration of CrO_4^{2-} solution was contacted with activated carbon, which was analyzed with Cr content (SNI 6989.71:2009). The constants K and q of the Freundlich equation must be found to complete the Freundlich equation (Kusdarini et al., 2018).

$$\frac{x}{p} = K_{fr}\, C_s^{\frac{1}{q}} \qquad (2)$$

Equation 6 was the amount of Cr^{6+} ion which was adsorbed per unit mass of active carbon (ppm/g), Cs was a concentration of Cr^{6+} ion in the adsorbate after it runs into desorption process (ppm), K_{fr} and q were empirical constants (Basu, Ghosh, & Saha, 2018; Kusdarini et al., 2018; Kusdarini, Purwaningsih, & Budianto, 2021). K_{fr} and q constants can be found in equation 3.

$$\text{Log}\,\frac{x}{p} = \log K_{fr} + \frac{1}{q}\log C_s \qquad (3)$$

Plotting data used the Freundlich equation to obtain K_{fr} and q constants data that form an equation of activated carbon isotherm adsorption to Cr^{6+} ion contained in artificial waste. The Langmuir isothermal adsorption equation is presented in Equation (4).

$$\frac{K}{m} = \frac{1}{K_c A_r} + \frac{K}{A_r} \qquad (4)$$

Where A_r and K_c are coefficients, m is the weight of Cr^{6+} ions adsorbed per unit weight of activated carbon (ppm/g), K is the concentration of Cr^{6+} ions in solution after activated carbon is adsorbed. Then, the graph of K/m vs K was plotted to obtain the constants A_r and K_c.

RESULTS AND DISCUSSION

Coal Characteristics

The characteristics of coal raw materials used in this research are explained in Table 2.

Table 2. Characteristics of coal raw materials based on proximate analysis

Parameters	Content (%)
Water content	4.32
Volatile matter content	14.25
Ash content	8.37
Fixed carbon content	73.06

Table 2 showed that coal was used as raw materials in this study, including bituminous coal, with a fixed carbon content of 73.06%, volatile matter content was 14.25%, and 4.32% for water content (Hessley et al., 1986; Kirk & Othmer, 1979).

Characteristics of Activated Carbon

Analysis results of water content, volatile matter content, ash content, fixed carbon, iodin number, and Surface morphology from activated carbon are shown in Figures 2, Figure 3, Figure 4, Figure 5, Figure 6, and Figure 7.

Water Content

Figure 2 showed that the water content of activated carbon was between 0.21-6.49%, although all of the water content samples have met the SNI standard was 15% for the maximum (BSN, 1995), but activated caron water content would decrease quite sharply if there were an activation temperature of 825ºC compared to 625ºC and 725ºC, which was 93-94%. The water content of activated carbon is activated at a temperature of 825oC, which is 0.21-0.65%. The moisture content of activated carbon is smaller than that of activated carbon-based on low-grade bituminous coal (Kusdarini et al., 2017). The moisture content of activated carbon from peat and coal activated with H_3PO_4, H_2SO_4, and $ZnCl_2$ at a temperature of 900ºC was 4.1-4.3% (Budihardjo et al., 2021). Empty palm oil bunches waste was activated using H_3PO_4 activators at an activation temperature of 700ºC, around 3% (Budianto et al., 2021). The water content of activated carbon from coir and coconut shell, activated using NaOH activator at an activation temperature of 600ºC, was 2-18% (Budianto et al., 2021). However, the activated carbon of water content was higher than activated carbon made from mangrove charcoal, and it was activated using phosphoric acid at an activation temperature of 650 to 724ºC was 0.11-0.38% (Budianto et al., 2019).

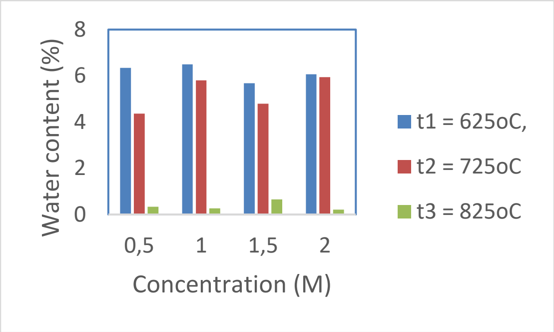
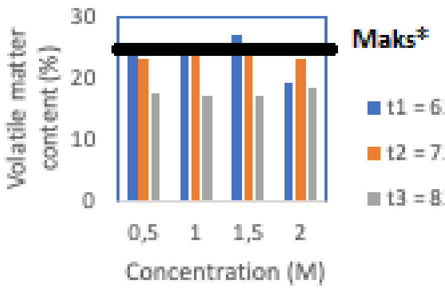


Figure 2. Percentage Diagram of activated water content, which was physics activated to the temperatures of 625ºC, 725ºC, and 825ºC

Volatile Matter Content

The volatile matter content of activated carbon was 17.11 - 27.03%, so most samples have met the SNI standard, which was a maximum of 25% (Figure 3) (BSN, 1995). The volatile matter content of activated carbon was lower than activated carbon made from low-grade bituminous coal, was 39.1-45% (Kusdarini et al., 2017), and young coconut coir and shells were 5-50% (Budianto et al., 2021). The volatile matter content of activated carbon was almost the same as activated carbon made from charcoal mangroves was 19.8-23.4% (Budianto et al., 2019). Furthermore, the volatile matter content of activated carbon was higher than activated carbon made from empty palm oil bunches waste, around 0.62-0.8% (Budianto et al., 2021).

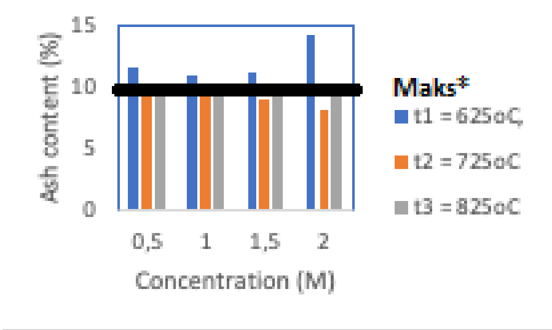


**SNI 06-3730-1995*

Figure 3. Percentage Diagram of activated carbon volatile matter, which was physics activated at the temperatures of 625ºC, 725ºC, and 825ºC

Ash Content

Activated carbon of ash content was around 8.03-11.03%. Ash content met SNI standards at activation temperatures of 725ºC and 825ºC, and the maximum was 10% (BSN, 1995). It showed that activation temperatures affected ash content, and the concentration of activator $(NH_4)_3PO_4$ did not significantly affect ash content (Figure 4). Activated carbon ash content was almost the same as activated carbon made from low-grade bituminous coal, and it was 4.7-10% (Kusdarini et al., 2017), and mangrove charcoal was 6.8-10% (Budianto et al., 2019). However, activated carbon ash content was higher than activated carbon made from empty palm oil bunches waste, around 2-5.88% (Budianto et al., 2021).

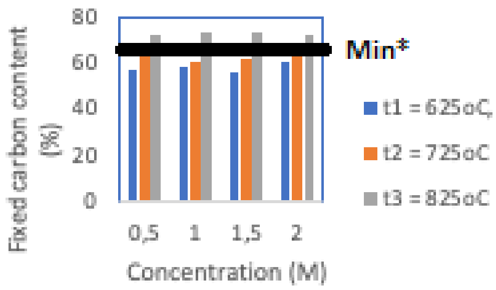


**SNI 06-3730-1995*

Figure 4. Percentage Diagram of activated carbon ash content, which was physics activated at temperatures of 625ºC, 725ºC, and 825ºC

Fixed Carbon Content

The fixed carbon content of activated carbon ranged from 56.08 to 73.17%. Figure 4 showed that the activation temperature affected fixed carbon content, and concentration changing of activator $(NH_4)_3PO_4$ had little effect. Activated carbon was activated at the temperature of 825ºC and has met SNI standards by at least 65% (Figure 5) (BSN, 1995). Activated carbon of fixed carbon content was higher than activated carbon which was made from low-grade bituminous arranging from 40.2-48.5% (Kusdarini et al., 2017), and it was lower than activated carbon which was made from mangrove charcoal, between 68.13 to 70.25% (Budianto et al., 2019) and the empty palm oil bunches waste was range from 84.64-97.20% (Budianto et al., 2021).



**SNI 06-3730-1995*

Figure 5. Percentage Diagram of activated carbon the fixed carbon content which was activated at physics temperatures 625ºC, 725ºC, and 825ºC

Iodin Number

Activated carbon, the iodine number ranged from 1238-1259 mg/g, meeting the SNI standard of at least 750 mg/g (BSN, 1995) (Figure 6). The concentration of the activator reagent did not significantly affect the iodine number. But activation temperature affected the iodine number. The higher the activation temperature was obtained, the higher the iodine number affected, so the optimal activation temperature was 825°C. Activated carbon of iodine number was higher than activated carbon which was made from mangrove charcoal with was maximum of 1020 mg/g (Budianto et al., 2019), low-grade bituminous coal was 1173-1239 mg/g (Kusdarini et al., 2017), and the empty palm oil bunches waste was 945 mg/g (Budianto et al., 2021).

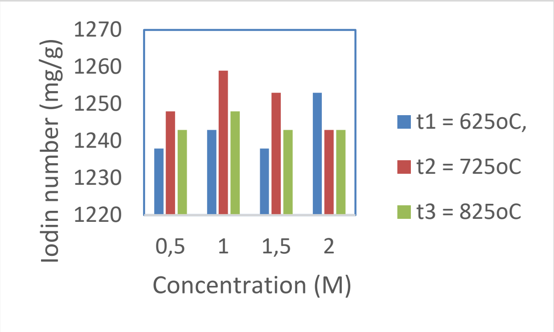
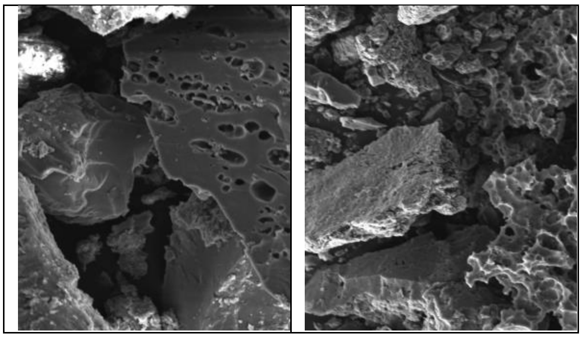


Figure 6. Percentage diagram of activated carbon the iodine number which was physics activated at temperatures of 625°C, 725°C, and 825°C

Surface Morphology with SEM-EDX

The comparison of surface morphology of coal and activated carbon is presented in Figure 7. SEM-EDX photos of the activated show that activated carbon pores are broader and more numerous than before activation.



(a) (b)

Figure 7. (a) SEM-EDX photo of carbon before activation (1.5 K X); (b) SEM-EDX photo of activated carbon (1.30 K X)

Freundlich Equation

Chrom content in CrO_4 solution before it was contacted activated carbon (initial concentration) and after it was contacted activated carbon (final concentration) was presented in Table 3.

Table 3. Chrom concentration in the CrO_4 solution

Samples	Initial Concentration (ppm)	Final Concentration (ppm)
1	120	76.1
2	105	53.4
3	90	44.5
4	75	35.3
5	60	29.1

Freundlich Equation was described in Figure 8.

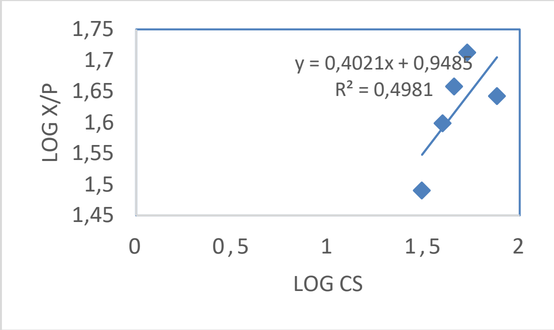


Figure 8. The correlation between log (x/p) and log C_s in the Freundlich Equation

Based on equation (3) and regression linear equation in Figure 8, the calculations were obtained the value of 1/q = 0.4021 and log K_{fr} = 0.9485 so q = 2.4870 and K_{fr} = 8.8818. This study obtained Freundlich Equation which was quite accurate because R = 0.7058 or close to 1. Furthermore, the result of the Freundlich Equation, which was produced by the Equation, was

$$\frac{X}{p} = 0.9485\ C_s^{\frac{1}{2.4870}} \qquad (5)$$

Langmuir Equation

The Langmuir adsorption isotherm equation for activated carbon concerning Cr^{6+} ions was obtained from the K/m vs K graph plot. The plot is presented in Figure 9.

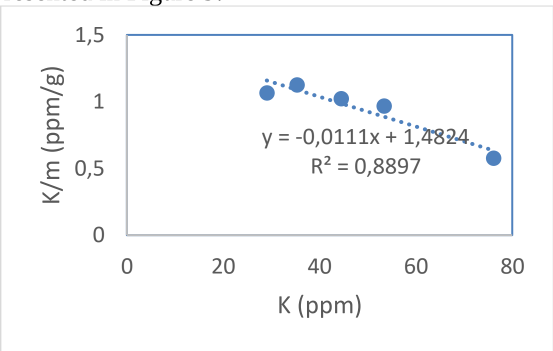


Figure 9. Plotting between K/m vs K to get the Langmuir equation

Based on Equation (4) and the Equation obtained from Figure 9, it can be determined that the constants Ar = -90.0901 and Kc = -0.0075. The Langmuir adsorption isotherm equation is presented in Equation (6).

$$\frac{K}{m} = 1.4824 + \frac{K}{-90.0901} \qquad (6)$$

The correlation coefficient of the Langmuir equation is 0.8897 so that the relationship between the independent and dependent variables is strong (Figure 9).

CONCLUSION

The activator used $(NH_4)_3PO_4$ 0.5 M, 1 M, 1.5 M, and 2 M with the temperature of physical activation was 825ºC at the making of activated carbon from high-grade bituminous coal produce activated carbon which was met SNI standard number 06-3730-1995. The best-activated carbon was produced by an activator used of $(NH_4)_3PO_4$ 1 M, and the characteristics were 1) water content of 0.27%, 2) volatile matter was 17.17%, 3) 9.39% of ash content, 4) fixed carbon content was 73.17%, and 5) the iodine number of 1248.302 mg/g. In the use of activator carbon with several concentrations, such as 0.5 M, 1 M, 1.5 M, and 2 M, the concentration of the activator did not significantly affect the parameters of activated carbon.

While, at the activated temperature, the activation temperature variables were 625ºC, 725ºC, and 825ºC. Activation temperature significantly affected the activated carbon characteristics. Then, the activated carbon of carbon adsorption isotherm to Cr with the Freundlich equation produced q = 2.4870 and K_{fr} = 8.8818 constant. The Langmuir isotherm adsorption equation of activated carbon to Cr resulted in constants Ar = -90.0901 and Kc = -0.0075. The correlation coefficient of the Freundlich equation is 0.4981, and the correlation coefficient of the Langmuir equation is 0.8897, so the use of the Langmuir equation is more valid.

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