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The Influence of NaOH Activator Concentration on the Synthesis of Activated Carbon from Banana Peel for Pb(II) Adsorption

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<i>Keywords :</i> Activated carbon, NaOH activator, banana peel, adsorption	Abstract. Lead has been known as one of the heavy metals with a high adverse effect to the environment and human health. This study reports the activity of activated carbon from biomass of banana peel as an adsorbent to resolve the hazardous lead-contaminated wastewater. The influence of the activator was studied via the alteration of NaOH concentration from 1, 3, and 5 M, where the sample was denoted as AC-1, AC-3, and AC-5. Some techniques, including FTIR, XRD, and SEM were applied to characterize the sample with the highest adsorption capacity. FTIR result affirmed
	the presence of hydroxyl group on the activated carbon with NaOH 1 M (AC-1) that was beneficial for adsorption. XRD and SEM confirmed that the activated carbon possessed crystalline and amorphous phases with sheet-like morphology. Regarding Pb(II) adsorption, the higher concentration of activator caused the decline of adsorption capacity as the contact time prolonged. The highest adsorption capacity and efficiency were obtained using 1 M NaOH activator with a contact time of 1 hour, which was 3.71 mg/gram and 97.86%, respectively.
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1. Introduction

Industrial waste and fossil fuel manufacture mainly contribute to high concentration of heavy metal in the environment. It's low solubility in water, the accumulation of heavy metals is feasible to happen in the water stream, which is hazardous to the ecosystem and human health. Lead (Pb) is considered the most toxic heavy metal owing to its non-biodegradable property and high affinity to sulfur, which attack the bond in enzyme [1][2]. Lead (Pb) in water will form $Pb(OH)_2$ and accumulate in the bodies of animals and humans [3]. The presence of lead in human body can cause intellectual deficiency, growth problems, and paralysis. This carcinogenic metal commonly intoxicates the human body through the consumption of contaminated food and drink [4][5][6].

Various methods have been developed to address the high concentrations of Pb in the environment, including adsorption, ion exchange, membrane filtration, and sedimentation [7]. Adsorption is believed to be the most promising technique due to its feasible applicability, low-cost operation, high activity, and efficiency [8][9]. The materials commonly employed as a Pb adsorbent is activated carbon [10][11][12][13][14]. Activated carbon has a high surface area, internal porous structure, and uniform pore size distribution. It can adsorb cation, anion, and molecules of organic compounds in the form of liquid and gas [15]. Many studies reported that microporous and macroporous activated carbon possess excellent stability and thermal conductivity. Activated carbon can be prepared by a simple heating process of material such as biomass (lignocellulosic biomass or organic wastes), which may play a powerful role not only in replacing fossil-based materials, but also in enhancing sustainable bio-economy [16][17][18]. Inexpensive and abundant biomass such as logs, woods, fruit, and nut peels had been applied as a precursor. One potential alternative for activated carbon feedstock is banana peel. In the Madura region, the banana peel has abundant availability and low cost, making it a promising feedstock for activated carbon.

Previous studies reported the high adsorption activity over activated carbon synthesized from various agricultural, coal mining, and many other wastes. Activated carbon was successfully prepared from Water Hyacinth (Eichhornia crassipes) for the adsorption of Pb(II) dan Hg(II) by Waly et al., (2021) [19]. The highest adsorption capacity of Pb(II) and Hg(II) was 310.9 mg/g and 252.5 mg/g respectively. The following condition obtained this result: pH 5.5, the temperature of 25°C, and 60 minutes contact time. Neolaka et al., (2021) [12] studied the activation of activated carbon from Schlechera oleosa on the adsorption of Pb(II). The result showed Langmuir isotherm behavior with the highest adsorption capacity of 1.634 mg/g. Kharrazi et al., (2021) [11] investigated the synthesis of activated carbon from Elm sawdust and its application to the adsorption of Pb(II) and Cr(VI). Before to the adsorption, the carbon was treated with alkali and alkali earth metal for Pb adsorption capacity enhancement. The acid treatment improved the adsorption activity on the Cr(VI) on the activated carbon. HCI-treated biomass produced microporous carbon with a volume of 0.443 cm³/g, and the amount of Cr adsorbed was 190 mg/g. Meanwhile, the highest adsorbed Pb was 1430 mg/g.

In this study, we aim to investigate the adsorption activity of activated carbon prepared from banana peel on the Pb (II). The influence of the activator was studied by altering the concentration of NaOH. The sample's highest adsorption capacity was characterized by some techniques, including FTIR, XRD, and SEM. Pb (II) adsorption over activated carbon was observed as a function of reaction time.

2. Experimental Section

2.1 Materials and Equipment

The tools used in this research include a set of beaker glass, oven, furnace, and filter paper. While the materials used are NaOH and PbNO₃ were purchased from Merck. The Banana peel originated from the plantation on Madura Island, Indonesia.

2.2 Experiment flow chart

The research flow chart is shown in Figure 1



Figure 1. Research flow chart

2.3 Synthesis of activated carbon from banana peel waste

The banana peels were dried under sunlight to remove the moisture altogether. Afterward, the dried banana peel was calcinated at 400 °C for 1.5 h. The resulted carbon was crushed and smoothed to 125 mesh. The activation was carried out by the addition of NaOH with various concentrations of 1, 3, and 5 M. The mixture was stirred at 600 rpm at ambient temperature for 2 h. The activated carbon was washed to reach neutral pH. The water content was removed from the activated carbon through drying in the oven at 110 °C for 2 h. The sample collected was denoted by AC-1, AC-3, and AC-5, where the number expresses the NaOH concentration applied for activation.

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2.4 Characterization of activated carbon

Activated carbon with the highest adsorption capacity was characterized by XRD, FTIR, and SEM. Crystallinity and phase identification carbon was analyzed using XRD (Bruker D2 Phaser. A Nicolent Avatar 360 FTIR determined the functional group of activated carbon. The sample was tested in a KBr pellet with a wavelength range of 4000-500 cm⁻¹. The morphology of activated carbon was measured using SEM (Zeizz Evo MA10).

2.5 Pb(II) adsorption using activated carbon

The solution of Pb 50 ppm was analyzed with AAS to determine the exact concentration. About 50 mg activated carbon sample and 200 ml of Pb(II) 50 ppm were mixed in a beaker glass under a stirring condition of 600 rpm. As the comparison, the contact time was altered to the range of 1-3 hours with intervals of 0.5 hours. 10 ml of Pb solution was taken through a filtering process using filter paper. The actual concentration of Pb before and after adsorption was determined by the AAS technique. The amount of Pb adsorbed was calculated using the following formula [20]:

$$Qe = \frac{(Co - Ce) V}{m} \ge 100$$

Where Co is Pb concentration before the adsorption (ppm), Ce represents the Pb concentration after the adsorption (ppm), V is the volume of Pb(II) solution (liter), and m is adsorbent mass (gram).

3. Results and Discussion

This study prepared carbon from banana peel by combustion at 400°C for 1.5 h. The chemical activation was carried out by adding NaOH with various concentrations (1,3 and 5 M). This treatment created a high surface area of activated carbon, which is beneficial for adsorption. The activity of activated carbon from banana peel was investigated by Pb(II) adsorption. The surface of carbon material is composed of carbon elements interacting in covalent bonds, contributing to its nonpolar properties. The pore structure determines carbon surface area as porous material [21]. Figure 2 illustrates the activated carbon synthesized from banana peel.



Figure 2. Activated carbon with concentration variation of NaOH activator

The X-Ray Diffraction (XRD) result of activated carbon (AC-1) is depicted in figure 3. Sample AC-1 was analyzed using an angle of 2 θ between 5°-100°C. XRD characterization is used to identify the phase and crystallinity of a sample [22]. The crystallinity of the material is indicated by the intensity level of the peaks generated from XRD. The specific feature of carbon was represented by a

peak at $2\theta=32.28^{\circ}$. However, the peak was very broad and formed a hump at $2\theta=20^{\circ}-50^{\circ}$. This feature indicated the amorphous phase of activated carbon.



Figure 3. AC-1 sample diffractrogram

Fourier Transform Infra-Red (FTIR) spectrum of the AC-1 sample is shown in Figure 4. Characterization using FTIR was used to analyze the functional groups of activated carbon. FTIR result describes several features at wavelength of 3420 cm⁻¹, 1573 cm⁻¹, 1439 cm⁻¹, 1056 cm⁻¹ and 868 cm⁻¹. The Peak at 1573 cm⁻¹ was assigned to the C=H vibration and C=C stretching [13]. A broad peak at 3420 cm⁻¹ represented the presence of hydroxyl group. This hydroxyl resulted from the interaction of NaOH as an activator on the carbon surface [23]. The activation process also generated a C=C bond indicated by a feature at 1439 cm⁻¹. The peak in the range of 1400-1500 cm⁻¹ also depicted the number of carbon [6]. The peak at 1056 cm⁻¹ represented the existence of C-O-C group. Another peak appeared at 868 cm⁻¹, which was attributed to the bending vibration of C-H aromatic [12].





The morphology of the AC-1 sample is illustrated in figure 5. The result confirmed the sample as porous material with a rough surface. The morphology was not uniform with the form of sheet-like and round pore. This morphology affirmed that the activation removed the volatile organic molecule and water from carbon pores.



Figure 5. Morphology of AC-1 sample

Atomic Absorption Spectroscopy (AAS) techniques determined the Pb(II) concentration before and during the adsorption process. The concentration of feed Pb(II) solution was found to be 37.947 ppm. The adsorption of Pb(II) over activated carbon from the banana peel as a function of contact time is illustrated in Figure 6. The adsorption process was conducted for 3 hours, where the solution was taken out periodically every 30 minutes for AAS analysis. The figure also explains the effect of NaOH concentration as an activator. The highest adsorption capacity was achieved over the carbon sample activated by NaOH 1 M (AC-1) for 1 h adsorption. With the same contact time, AC-3 and AC-5 adsorbed only 3.69 mg/g and 3.67, respectively. The carbon activated by adding NaOH 1 M required more extended time to adsorb Pb(II) selectively compared to rests. Moreover, the adsorption capacity declined with the extended contact time, where the adsorption capability exceeded the saturation point. The adsorbate was accumulated on the active site of carbon. weakening the interaction between the adsorbate and the active site leading to the desorption process [24]. During the desorption, ion Pb(II) was released from the active site to the feed solution so that the pore became smaller [25]. Based on the figure, the adsorption capacity presumably reached equilibrium during the stirring process or the first hour of contact time. This phenomenon was related to the relationship between the adsorption rate and mechanism driven by the physical electrostatic interaction of-Adsorbate-adsorbent surface. This interaction was provided by the active site, where the adsorption proceeded rapidly in less than one hour [26].



Figure 6. Graph of the relationship between contact time and adsorption capacity

Figure 7 illustrates the relationship between adsorption efficiency and the function of contact time. The adsorption efficiency of Pb(II) declined as the contact time extended, because the activated carbon had reached equilibrium. The activated carbon could not to adsorb Pb(II), and/or both adsorption and desorption underwent at the same rate. After one hour, the highest adsorption capacity was attained over AC-1 with 98.86%. Contrary, the lowest capacity resulted over AC-5 after 3 h contact. This inferred that the prolonged contact time and higher activator concentration lowered the adsorption capacity of activated carbon on Pb(II) adsorption.





4. Conclusion

The Biomass of the banana peel was successfully converted to activated carbon. Characterization using FTIR confirmed that the synthesized activated carbon possessed hydroxyl, which is beneficial for Pb adsorption. XRD and SEM results showed the amorphous and crystalline phases of activated carbon with nonuniform-like sheet morphology. The highest adsorption efficiency and capacity were 97.86% and 3.71 mg/gram, respectively. This result was attained over carbon activated by adding NaOH 1 M after 1 h contact time.

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