The synthesis of corncobs (zea mays) active charcoal and water hyacinth (eichornia crassipes) adsorbent to adsorb Pb(II) with it's analysis using solid-phase spectrophotometry (sps)

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Abstract. This research aim to examine the effect of the combination between corncobs and water hyacinth to adsorb lead (II), the most effective combination have determined by compared the ratio of corncobs adsorbent and water hyacinth to the increasing adsorption of the Pb(II), prove the effectiveness of the solid-phase spectrophotometry (sps) to determine the levels of Pb(II) as the result of the corncobs active charcoal adsorption and water hyacinth in the level of μg/L.The research method used is experimental method. The data collecting technique is carried out by several stages, which are carbonization using muffle furnace at a temperature of 350˚C for 1.5 hours, activation of the corncobs charcoal and water hyacinth using HCl 1M and HCl 5M activator, contacting the adsorbent of corncobs active charcoal and water hyacinth with liquid waste simulation of Pb(II) using variation of corncobns and water hyacinth, 1:0; 0:1; 1:1; 2:1; 1:2, analysis of Pb(II) using an sps, characterization of corncobs active charcoal adsorbent and water hyacinth using FTIR. Research results show that the combined effect of activated charcoal corncobs and water hyacinth can increase the ability of the adsorbent to absorb Pb(II), the optimum adsorbent mass ratio of 1:1 with the absorption level of 90.33%, SPS is an effective method to analyze the decreasing level of Pb(II) as the adsorbtion result of the corncobs active charcoal and water hyacinth in the level of μg/L, with the limit of detection (LOD) of 0.06 μg/L.

1. Introduction

Technological developments have an impact on the development of the industry growing by leaps and bounds. This led to a growing number of industrial waste that pollutes the environment even groundwater that is widely used for the community's daily needs, such as cooking, washing and bathing. One of the heavy metals to become donors in the decline in the quality of human life, namely lead or plumbum [1].

Currently many developed methods to reduce the impact of water pollution by industrial waste containing heavy metals, one is with the method of adsorption. Adsorption is the heavy metal ion absorption method using specific adsorbents. Adsorbents used can be either a corncobs charcoal and water hyacinth.

The corncobs charcoal contain cellulose, hemicellulose, lignin and extractive substances which can be activated carbon [2]. Activated carbon can be used as adsorbent for heavy metals Pb^{2+} with

activation first. In addition to the corncob, water hyacinth is also the aquatic plants that grow very rapidly and are considered very detrimental to society because it causes depletion of oxygen in the water and result in more fish that die because of the difficulty gets oxygen. Water hyacinth has good properties, among others, can absorb heavy metals, sulfides, compounds other than protein it contains more than 11.5% and contains cellulose that is bigger than lignin, ash, fat and other substances [3].

To activate charcoal from corn cobs and water hyacinth needed an Activator accordingly. The selection of the appropriate Activator will make the absorption of heavy metals are more optimally.In this research using corncob activator is HCl1 M and the water hyacinth activator is HCl 5 M.

The analysis is used to find out the capabilities of active charcoal adsorbent Hyacinths and corncob absorb heavy metals Pb is solid phase spectrophotometry (SPS).Solid Phase Spectrophotometry is a method of analysis using the spectrophotometer UV rays-looks. This method is based on measuring the sample directly through the absorption phase ion exchanger resin that absorbs the sample in the form of complex [4]. Solid phase spectrophotometry (SPS) has sensitivity better than previous instruments [5]. SPS showed that teak sawdust and zeolite can be used as adsorbents to adsorb Cr(VI) metal ions with the adsorption capacity 1.19 µg/g analyse by solid-phase spectrophotometry (SPS)[6]. The advantages of this method of analysis compared to other methods is the simple operation, high sensitivity and accuracy, which reached the level µg/L, so it can detect the metal ions dissolved in very small concentration [7]. Solid Phase Spectrophotometry are extremely sensitive and highly accurate because it can detect up to the size of the ppb. Higher sensitivity can be obtained by using 20 cm^3 volume of the sample [8]. The advantages of this method of analysis compared to other methods is the simple operation, high sensitivity and accuracy, which reached the level µg/L, so it can detect the metal ions dissolved in very small concentration [8]. The determine the reduction levels of Cd(II) metal ions as an adsorption result of the activated rice husk charcoal in the level of µg/L with the limit of detection (LOD) was0.059 µg/L [9]. Thus, researchers interested in conducting research of the application of activated carbon from corncob and water hyacinth waste as adsorbent of Pb(II) with an analysis using SPS.

2. Experimental

2.1. Tools and materials
The used tools were a UV-visible spectrophotometer from K-MAC, FTIR spectrophotometer from Shimadzu, muffle furnace, oven, pan, analytical balance, volumetric flask, volume pipette, beaker glass, measuring cup, flask, Erlenmeyer, watch glass, stirrer glass, drop pipette, aliquoting devices which are assembled using a syringe, blender, mortar and pestle, a 100 mesh sieve, porcelain bowls, stirrer bar, and a magnetic stirrer. Materials used are rice husk, Pb(NO$_3$)$_2$, HCl 1 M, HCl 5M, resin AG Muromac 50W-X2, H$^+$form(100-200 mesh), distilled water, chloroform, Whatman filter paper, blue litmus, and dithizone.

2.2. Research procedure

2.2.1. Production of adsorbent. Corncobs and water hyacinth cleaned and washed with distilled water, then it is dried at 105°C then charred in a muffle furnace at a temperature of 350°C for 1 hours. Results obtained are mashed and then sieved with a 100 mesh size.

2.2.2. Adsorbent activation. Corncobs soaks in a solution of HCl 1 M and water hyacinth soaks in a solution of HCl 5 M for 24 hours at room temperature. Filter and rinse the residue with distilled water until the filtrate become neutral, and then dried in an oven at 110°C for 24 hours. Adsorbent was tested by FTIR before and after activation.

2.2.3. Resin preparation. Muromac resin AG Muromac 50W-X2 H$^+$form (100-200 mesh) dissolved in distilled water and silence a few moments until the resin becomes more fluffy.

2.2.4. Determination of Ob(II) calibration curve. A 20-mL of standard solution of Pb(II) 0 μg/L, 2 μg/L, 4μg/L, and 8μg/L respectively were added with 1 mL of 0.005% dithizone, and 0.06 mL resin. Stir it for 20 minutes and analyzed using UV-vis spectrophotometer at a wavelength of 483 nm and 558 nm and then absorbance difference of the two wavelengths determined, $\Delta A = A_{483nm} - A_{558nm}$, which ΔA will be made standard curve (ΔA vs concentration).

2.2.5. Determination of Pb(II) species in simulated liquid waste. Simulated liquid waste solution of 50 μg/L was taken 20 mL then added 1 mL of 0.005% dithizone, and 0.06 mL resin. Then, stir it for 20 minutes and analyzed using UV-Vis spectrophotometer at a wavelength of 483 nm and 558 nm. ΔA obtained will be substituted in equation Pb(II) calibration curve (ΔA vs concentration), so that the Pb(II) ions in the effluent can be known.

2.2.6. Determination Of The Most Effective Adsorbents Comparison. Incorporating active charcoal and corn cob charcoal active water hyacinth with a comparison of 1:0; 0:1; 1:1; 2:1; 1:2 into the beaker of solution already contains sewage Pb (II) simulation of as many as 25 mL. Stirer for a solution that contains a mix of active charcoal and waste Pb (II) simulation for 30 minutes. The solution obtained is filtered with a filter paper and diluted from 5 mL filtrate into a 50 mL. Take 20 mL and then adding 0.06 mL resin, 2 mL and 1 mL of H_2SO_4 dithizon. Stirer for 20 minutes. The filtrate obtained tested with solid-phase spectrophotometry with 483 nm wavelength and 558 nm. Test comparison with most effective adsorbents using Fourier Transform Infra-Red (FTIR).Determination of detection limit. Taking five blank solution 20 ml, then each added 1 mL of of 0.005% dithizone, and as much as 0.06 ml resin, then stir it for 20 minutes. Furthermore, it analyzed by UV-vis spectrophotometer at a wavelength of 483 nm and 558 nm. ΔA obtained will be substituted in the equation of the calibration curve.

3. Result and Discussions

3.1 Production of activated charcoal from corncobs and water hyacinth

The process of making corn cob active charcoal and water hyacinth consists of 3 stages, namely the stage of dehydration, and the carbonation stage stage of activation. At the stage of dehydration corn cobs and dried water hyacinth with an oven at a temperature of 105 ° C for 1 hour to eliminate the water levels.The carbonation stage done by inserting the corncob and water hyacinth into the furnace at a temperature of 350 ˚ C for 1 hour because higher temperatures can increase the rate of reduction of impurity and the volatile compounds that fills pore adsorbents, thus optimizing the establishment of active pore [10].Next up is stage activation charcoal corn cobs and water hyacinth. The main purpose of the activation process is to add or develop pore volume and pore diameter enlarging that had formed on the carbonization process as well as to make some new pore [11].

3.2 Fourier transform infra red spectra analysis

Based on FTIR Spectra analysis against a mix of active charcoal adsorbent corncobs and water hyacinth before activation, after activation and after pengontakkan done, obtained results are not too much difference. It's just that there is no vibration after activation of the C-O-C and C-H amine and vibration CH_3. On the infrared spectra of active charcoal adsorbent blend of corn cobs and water hyacinth after activation or after pengontakkan does not show a difference too much, just that there is no vibration after activation of the C-O-C and C-H amine.While after contacting the absence of C-H vibration amines and CH3. Infrared Spectra analysis results, it can be concluded that the fusion of active charcoal adsorbent and cob of corn contains an aromatic C-H cluster, C-O-C, C = O, C-H,-CH3 amines, C = O, and aromatic C-H.
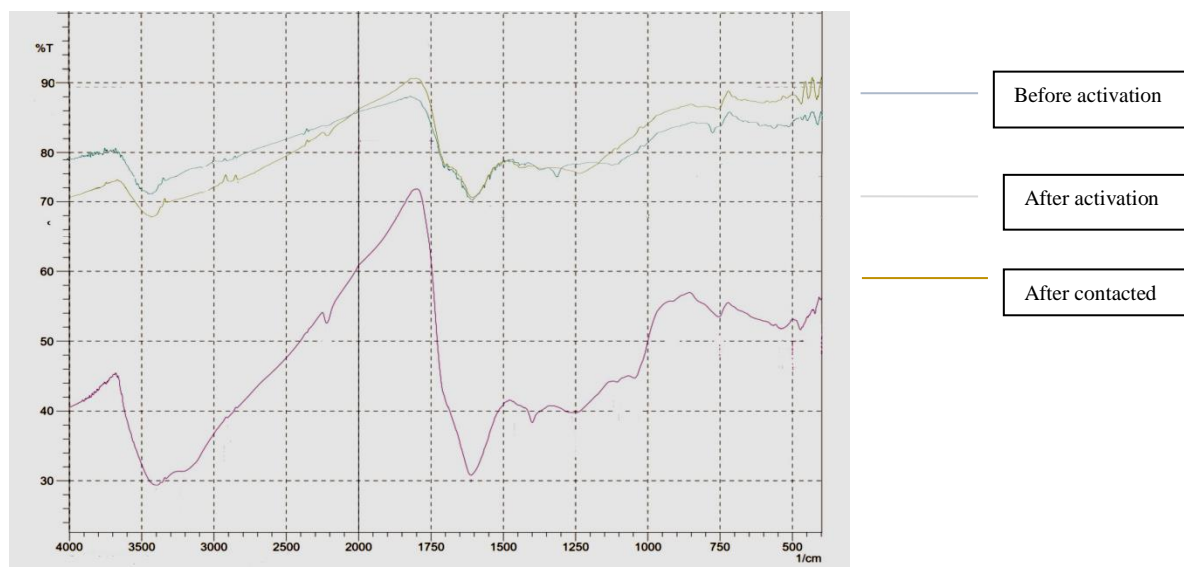
Figure 1. FTIR Spectra

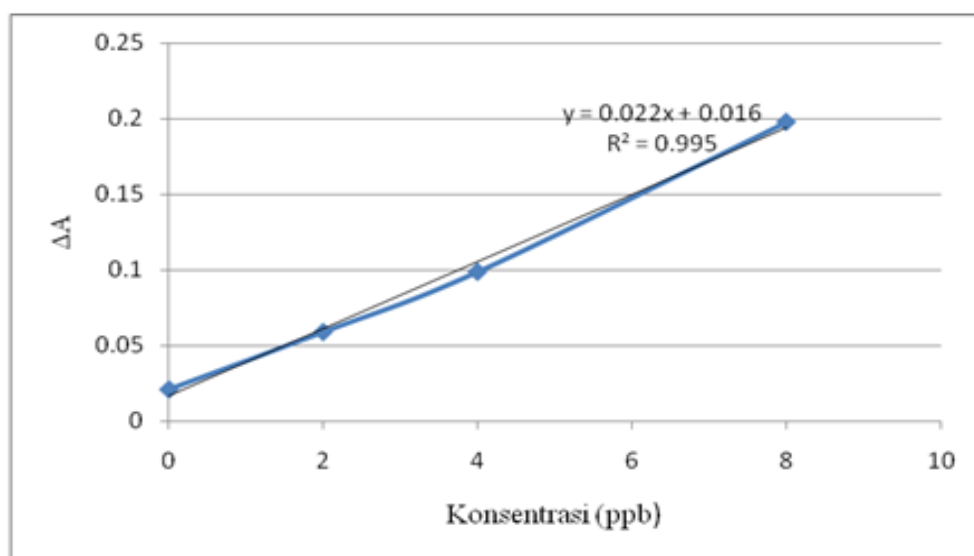*3.3 Determination of Pb(II) calibration curve*

Figure 2. Calibration curve of Pb (II) solution.

From the graph in Figure 2 are obtained one equation that is y = 0.022 x + 0.016, where y is and x is ΔA concentration. The equation which will be used to determine the concentration of Pb (II) contained in the waste liquid simulation. Determination results of Pb(II) levels in simulated liquid waste.

Table 1. Determination results of Pb(II) levels in simulated liquid waste

Sample	[Pb(II)] (µg/L)	ΔA	[Pb(II) actually] (µg/L)
Sample Pb(II)	4.99	0.126	49.99

3.4 Comparison of most effective adsorbents

Table 2. Optimum adsorbent mass comparison

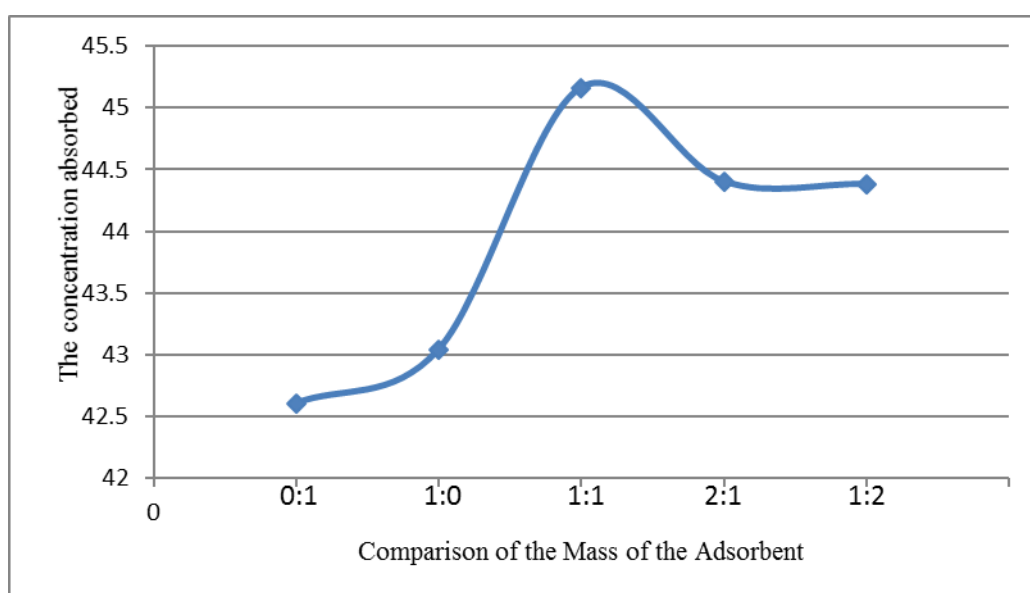| Mass Comparison	Initial [Pb(II)] (µg/L)	ΔA	Final [Pb(II)] (µg/L)	Adsorbed [Pb(II)] (µg/L)	Adsorbed [Pb(II)] (%)
0:1	49.99	0.178	7.39	42.60	85.21
1:0	49.99	0.169	6.96	43.04	86
1:1	49.99	0.122	4.84	45.16	90.33
2:1	49.99	0.139	5.59	44.40	88.81
1:2	49.99	0.139	5.61	44.38	88.77

Figure 3. Optimum adsorbent mass comparison

Comparison of the mass of active charcoal adsorbent corncob: copper ion absorption occurs 1:1 of heavy metals Pb (II) highest i.e. 45.16 amounting to µ g/L or 90.33%.Determination of detecting limit.

Table 3. Determination of detecting limit

Blank	ΔA	Concentration	Standard deviation	LOD
A	0.104	4.03		
B	0.104	4.02		
C	0.103	3.98		
D	0.104	4.02		
E	0.104	4		
Average		4.01	0.02	0.06

In the experiments that have been conducted obtained rating LOD 0.06 µ g/L. This shows that at a concentration of 0.06 µ g/L sample is still legible.

4. Conclusion

Influence of combination of active charcoal adsorbent corncob and water hyacinth is able to improve the ability of adsorbents to absorb heavy metals Pb (II), comparison of the mass of active charcoal adsorbent COB corn and water hyacinth are the most effective 1:1 with the absorption of heavy metals Pb (II) of 90.33% and Solid-Phase Spectrophotometry (SPS) is an effective method for determining the levels of heavy metals ions, Pb (II) results of active charcoal adsorbed corncorb and water hyacinth in the level µg/L.

Aknowledgement

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