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The Analysis of Low-Cost Pb(II) Adsorbents using Batch Method of Solid-Phase Spectrophotometry

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Abstract

Heavy metal pollutants contained in wastewater can cause health problems for living things around. Minor to fatal health problems can occur due to heavy metal poisoning, mainly caused by Pb(II) metal.. This study aimed to determine the optimum mass combination of rice husk and zeolite to adsorb Pb(II) metal ions in simulated wastewater, and to determine the sensitivity of the analysis method. This study used Solid Phase Spectrophotometry (SPS) to determine the decrease in Pb(II) metal ion levels after being adsorbed by activated carbon from rice husks and zeolites. This study used an experimental method with simulated wastewater samples containing Pb(II) at several concentrations. Pb(II) adsorption processes by rice husk and natural zeolite used various adsorbents' mass ratios. The adsorbents were characterized by using Fourier-Transform Infra-Red (FTIR) Spectrophotometry. Pb(II) analysis during adsorption processes used a single beam UV-visible Spectrophotometer for Solid-Phase Spectrophotometry. This study indicates that the combination of adsorbent from rice husk and natural zeolite can properly adsorb Pb(II) ions with an adsorption capacity of 0.75 μg g^{-1} and 0.025 μg L^{-1} for the LoD of the instrument.

Keywords*:* Low-cost adsorbent, Pb(II), solid-phase spectrophotometry.

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1. INTRODUCTION

The danger of liquid waste released in the waters has widely proven (Chanani *et al.*, 2015; Kasmaei, Naderi, & Bahrami, 2017; Moradi *et al.*, 2015; Yosofi *et al.*, 2017). Heavy metals in wastewater will be released into the waters and negatively impact the surrounding life. Research shows that some of the heavy metals contained in liquid waste have become major pollutants, they are zinc (Zn), nickel (Ni), chromium (Cr), copper (Cu), and lead (Pb) (Liu *et al.*, 2019; Lingamdinne *et al.*, 2018). These metals can cause various health problems even in relatively low concentrations. Heavy metal is poisonous, especially Pb(II) (Veselaj *et al.*, 2019). The Pb(II) is a heavy metal pollutant that is widely produced in various industries contained in their wastewater. Pb(II) is very dangerous for the living things in the ecosystem. It can cause various health problems, even in small amounts. Unlike other organic compounds, Pb(II) ions cannot be degraded biologically, so Pb(II), which is discharged into water bodies continuously, will end up with Pb(II) accumulation. The amount of this accumulation will continue to increase over time as the process of wastewater disposal continues. Therefore, Pb(II) released in the waters must be handled before it increases harm to life in the surrounding aquatic ecosystem immediately. It can cause severe problems in the human body (Lingamdinne *et al.,* 2017; Lingamdinne *et al.,* 2016), metabolism malfunction (Atkovska *et al.,* 2018) and can decrease children's cognitive thinking ability because it can affect the nerve activity (Ryan *et al.,* 2004), even more, at specific concentrations it can cause death.

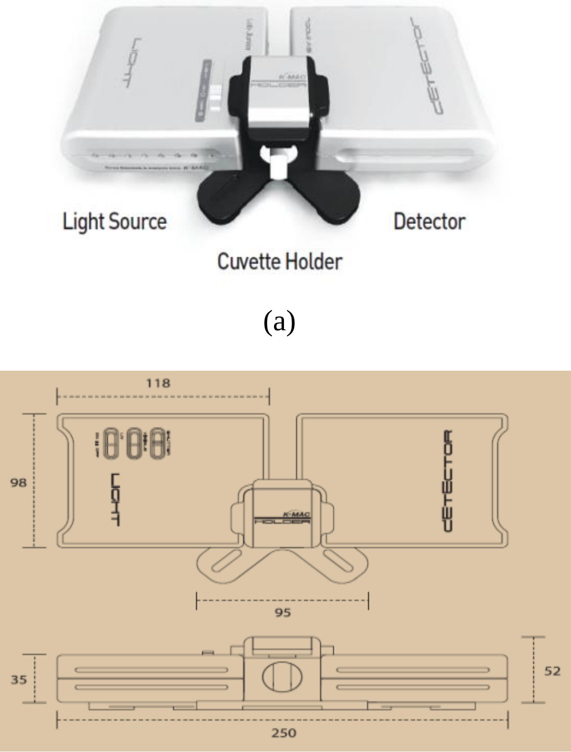
Water purification is being carried out by many researchers using natural materials such as natural zeolites or its modification (Kussainova *et al.,* 2018), moss (Koz & Cevik, 2014), tea residue (Dizadji & Anaraki, 2011), coconut shell (Rohmah, Masykuri, Saputro, & Mahardiani, 2018), water hyacinth (Saputro *et al.,* 2018), corncobs *et al.,*s with high purification capacity such as nano sorbent, graphene, and other polymeric materials (Liu et al., 2013; Yang *et al.,* 2015; Li & Ge, 2018). These high capacity purification materials are still relatively expensive, while the need for water purification is increasing due to the increasing amount of wastewater. Meanwhile, Indonesia is a country that can produce a various agricultural product, especially rice. Paddy field plays an important role to supply the main comestibles in Indonesia, yet it also produces enormous amounts of organic wastes such as rice husk, rice straw, rice groats, and rice bran. Rice husk contains lots of silica *et al.,* 2016) which means that it can be used as an adsorbent, so does the natural zeolite; both are low-cost adsorbent.

This research aims to determine the use of rice husk and natural zeolite as adsorbent of Pb (II) ion in simulated wastewater, the optimum mass combination of rice husk and zeolite to adsorb Pb (II) metal ions, and to determine the sensitivity of the analysis method. SPS in determining the decrease in Pb (II) metal ion levels after being adsorbed by activated carbon from rice husks and zeolites. Rice husk was chosen as an alternative to the low-cost and environmentally friendly adsorbent. The utilization of rice husks can also reduce the abundant waste of rice husks in Indonesia.

2. MATERIALS AND METHOD

Tools and Materials

Pb(II) ion adsorption analysis was performed using a single beam UV-Visible Spectrophotometer for the SPS by K-MAC Lab Junior, Satoda Science, Japan (Figure 1) and AG Muromac 50W-X2H+ form resin (100-200 mesh). The optical entrance of the SPS used had a diameter of 0.5 mm, which mounted in SMA-coupling. The lamp used in this SPS was a LED, a monochromator from the grating with 600 lines mm^{-1}, a light detector from a CCD array with the power of 2048 bit, which can measure the wavelength from 400 nm to 800 nm, and a data processor using laptop PC. An analysis of functional groups of adsorbents was also performed using Fourier-Transform Infrared Spectroscopy (FTIR) by Shimadzu. Rice husks taken from rice mills in Waru Village, Slogohimo, Wonogiri, Central Java were chosen as the first adsorbent, which would be combined in different ratio of mass with natural zeolite taken from Wonosari, Klaten, Central Java. The simulation sample used is a 1000ppm solution of $Pb(NO_3)_2$, diluted into several concentrations. The two adsorbents will be activated using two different solutions; they are 10% H_2SO_4 solution for activating the natural zeolite and 20% $ZnCl_2$ solution for activating the rice husk.



(a)

(b)

Figure 1. (a) Solid-phase spectrophotometer Junior series by K-MAC, Satoda Science, Japan; (b) The sketch of the inner part of solid-phase spectrophotometer Lab Junior series by K-MAC, Satoda Science, Japan (Source: K-Mac, Korea Materials and Analysis Corp)

Production of Adsorbent

The adsorbent used in this study consisted of natural zeolite and rice husk in several combinations of mass. Before contacting it with the simulated wastewater, these two materials are prepared in advance. The existing rice husk was washed using distilled water to remove the impurities then it is heated at 105 ^{o}C for 24 hours to remove the water. Furthermore, rice husks washed and free of water were being heated in muffle furnaces for 1.5 hours at 350 ^{o}C. The ash that had been produced was then mashed using mortar and pestle then sieved. Meanwhile, natural zeolite was mashed using mortar and pestle and then sieved. Both the rice husk ash and natural zeolite then sieved with the 100 mesh sieve.

The Activation of Adsorbents

The materials, rice husk ash and natural zeolite, were activated in advance before been contacted with the sample. Activation of rice husk ash was carried out using a 20% $ZnCl_2$ solution, while natural zeolite was activated using a 10% H_2SO_4 solution. Both of these adsorbents were immersed in their respective solutions for 24 hours. The mixtures then were filtered and washed using distilled water until they are neutral. The two adsorbents' water content is removed by heating it for 24 hours at 105 ^{o}C in the oven. The prepared adsorbents were then characterized using FTIR.

Resin Preparation

The resin used in the analysis was 100-200 mesh of AG Muromac 50W-X2H+. The resin was prepared by dissolving it in the aquades and left for a few moments until it transformed into fluffier form.

The Pb(II) Calibration Curve Determination

Calibration curves were determined using Solid-phase Spectrophotometry (SPS). The sample used is the standard solution Pb(II) at concentrations of 0 μgL^{-1}, 40 $\mu g\ L^{-1}$, 80 $\mu g\ L^{-1}$, and 160 μgL^{-1} that added with 1 mL of $(NH_4)_2MoO_4$ and 0.06 mL of resin. The mixture produced was being stirred for 20 minutes. Then, the samples produced were analyzed using SPS in two wavelengths, they are 484, and 615 nm and the difference was determined in the formula of $\Delta A = A_{485nm} - A_{615nm}$. ΔA found will be made as a Pb(II) standard curve that provides A versus concentration data.

The Pb(II) Species in Simulated Liquid Waste Determination

The determination of Pb(II) species refers to the research of Sulistyo Saputro, Yoshimura, Matsuoka, Takehara, and Narsito (2009) by mixing simulated wastewater samples of 20 mL with 1 mL of $(NH_4)_2MoO_4$ and 0.06 mL of resin. The mixture produced was being stirred for 20 minutes. Then, it had been analyzed using SPS in two wavelengths, they are 484, and 615 nm and the difference was determined in the formula of $\Delta A = A_{485nm} - A_{615nm}$. ΔA will be substituted into the standard curve equation Pb(II) so that Pb(II) concentration will be found.

The Determination of Adsorbent's Optimum Combination

The determination of optimum mass combinations of adsorbent was done by taking each 20 mL sample of Pb(II) 50 $\mu g\ L^{-1}$ simulated wastewater, then contacted with three different absorbent mass combinations of rice husk active carbon (RH) and natural zeolite (NZ) ratio of 1: 1, 1: 2, and 2: 1 then stirred to increase its contact area for 20 minutes. The filtrate obtained was then added with $(NH_4)_2MoO_4$ for 1 mL and resin for 0.06 mL then stirred for 20 minutes and analyzed using SPS in two wavelengths, are 484, and 615 nm and the difference was determined in the formula of $\Delta A = A_{485nm} - A_{615nm}$ which the ΔA will be substituted into the standard curve of Pb(II) so that the concentration of Pb(II) will be known.

Determination of Detection Limit

The detection limit was determined by taking 20 mL each of five blank solutions then added with $(NH_4)_2MoO_4$ for 1 mL and resin for 0.06 mL. The mixture then stirred for 20 minutes. The mixture was analyzed using SPS in two wavelengths, they are 484, and 615 nm and the difference was determined in the formula of $\Delta A = A_{485nm} - A_{615nm,}$ which the ΔA will be substituted into the standard curve of Pb(II).

3. RESULTS AND DISCUSSION

Production of Activated Adsorbent from Natural Zeolite and Rice Husk

Activated carbon made from rice husk contains large amounts of silica to be used as an adsorbent (Lima *et al.*, 2011). Zhang *et al.*, (2014) and Lee *et al.*, (2013) found that rice husk contained more than 12% of silica were found by using X-Ray Fluorescence (XRF) is presented in Table 1.

Table 1. The element components of rice husk

Element Component	Wt.%	
	Zhang et al., (2014)	**Lee et al., (2013)**
Mg	0.060	0.084
Al	0.038	0.084
Si	15.367	13.000
P	0.065	0.130
S	0.130	0.110
K	1.209	0.140
Ca	0.399	0.110
Fe	0.014	0.038

Besides silica, rice husk also contains a -OH group and a porous surface to cause interactions with the adsorbate, for example, the metal ions. Besides, natural zeolite has a great composition of silica and alumina. Zeolites can separate molecules based on their configuration and size. These adsorbents are activated using a 10% H_2SO_4 solution to activate rice husk and a 20% $ZnCl_2$ solution to activate natural zeolite. Activation was done to clean up the surface of the adsorbent and to increase the adsorption capacity. The spectra obtained from FTIR analysis from rice husk charcoal and natural zeolite before and after activation generally do not show any difference. The bonds that are read before and after activation are still the same, for example, C=C aromatic group, -OH, C=O, and Si-O from Si-O-Si. The slight difference that arises after the activation process is that the after-activation porous is wider than before-activation.

The Analysis of Fourier Transform Infrared (FTIR) Spectra

Figure 2 shows the functional group spectra for rice husk charcoal adsorbent before and after activation using FTIR. Based on the FTIR analysis results, it was obtained about the functional groups contained in the rice husk charcoal both before and after activation. The functional groups are determined based on the FTIR spectra's wavelength, which is presented in Table 2.

Table 2. The functional groups contained in the rice husk charcoal both before and after activation

Wavelength (cm^{-1})		Functional groups along with the mode of vibration
Before activation	**After activation**	
408.93	408.93 430.14	Si-O bending
466.79	462.94	Si-O-Si bending
798.56	796.64	Si-O stretching of Si-Si
1098.51	1098.51	Si-O stretching of Si-O-Si
1610.63	1610.63	C=C aromatic group
1708.04	1706.11	C=O group
3408.36	3416.08	O-H stretching from Si-OH

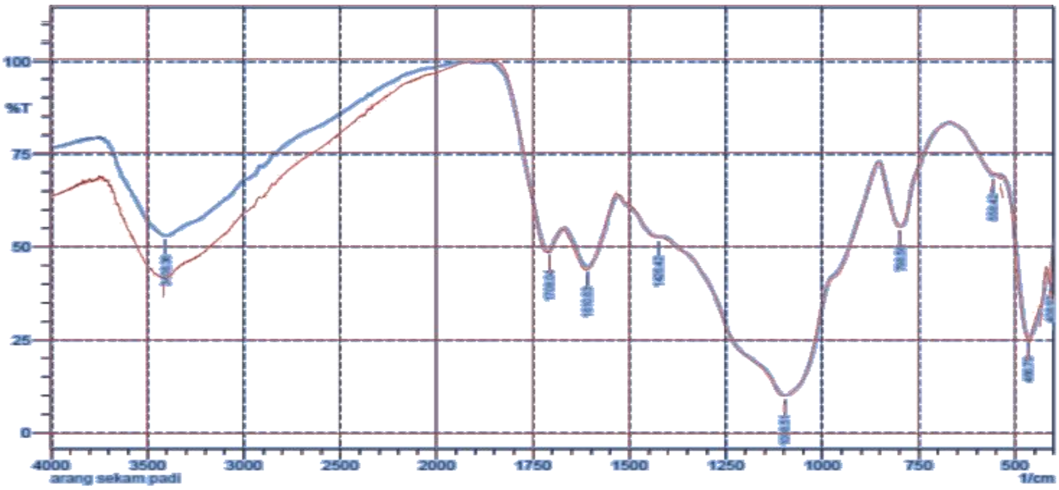


Figure 2. FTIR spectra of rice husk charcoal before and after activation (blue line: spectra of rice husk charcoal before activation, red line: spectra of rice husk charcoal after activation)

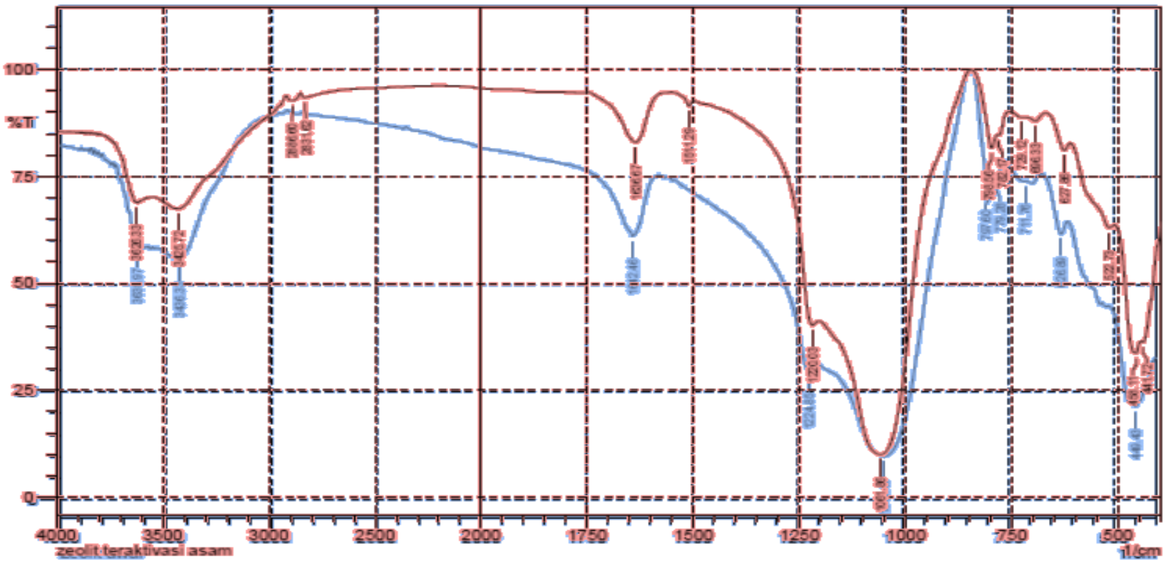


Figure 3. FTIR spectra of natural zeolite (blue line: spectra of natural zeolite before activation, red line: spectra of natural zeolite after activation)

Based on the FTIR spectra of rice husk charcoal adsorbent after activation, it generally does not show any difference before activation. The adsorbent's active group became wider, from the pre-activation spectra to the after-activation spectra from low to high concentrations. From the spectra, it can be concluded that the rice husk charcoal adsorbent before and after activation contains C = C aromatic, -OH, C = O groups and the presence of Si-O from Si-O-Si. Figure 3 shows the functional group spectra of natural zeolite adsorbent before and after activation. From the figure, it can be concluded that it contains a cluster of –OH, Al-O, Na-O, K-O, and Si-O.

Table 3. The functional groups contained in the natural zeolite both before and after activation

Wavelength (cm^{-1})		Functional groups, along with the mode of vibration
Before activation	**After activation**	
449.43	441.72	Na-O vibration
797.60	798.56	K-O vibration
1046.43	1061.86	Al-O vibration
1642.46	1636.67	Si-O vibration
3436.33	3425.72	-OH stretching

Based on the FTIR analysis results, it was obtained about the functional groups contained in the natural zeolite both before and after activation. The functional groups are determined based on the FTIR spectra's wavelength, which is presented in Table 3. The results from the natural zeolite spectra and zeolite after activation showed not much difference. It is just that the zeolite after activation has peaks that are visible compared to natural zeolites. Besides, the activated zeolite may have a wider surface area than the zeolite before activation. The pore distribution of activated natural zeolite has increased in mesoporous; this indicates a better adsorption ability due to a broader surface area.

Determination Results of Pb(II) Levels in Simulated Liquid Waste

Table 4. Determination of Pb(II) levels in simulated liquid waste

Sample	[Pb(II)] calculation (µg L^{-1})	ΔA	[Pb(II)] actual (µg L^{-1})
Sample Pb(II)	75	0.289	75.63

To determine the concentration of Pb (II) solution in the simulation waste, initially, a standard curve of Pb (II) solution was made with variations of 0 µg L^{-1} , 40 µg L^{-1}, 80 µg L^{-1}, and 160 µg L^{-1}. The equation $y = 2.226x + 0.147$ is obtained from the standard curve, where y is ΔA and x is the concentration. This equation is used to determine Pb (II) concentration in the simulated wastewater, where the concentration was 75.63 µg L^{-1}. From these results, it can be seen that the expected solution concentration is not too far from the solution that has been made with a dilution treatment.

Determination of the Optimum Combination of Adsorbent in Pb(II) Removal

Based on the results of the determination in Table 5 and Figure 4, the mass combination that produces the most optimal absorption percentage is the mass composition ratio of 1 (RH): 2 (NZ) of 74.43%, then then the ratio of 2 (RH): 1 (NZ)) amounting to 61.54%, and the smallest percentage of absorption is the ratio of 1 (RH): 1 (NZ) of 34.08%. The combination of activated carbon from rice husk and zeolite resulted in a relatively large percentage of absorption. This result is possible because of the adsorption process with the adsorbent pores and the ion exchange from the zeolite so that the Pb (II) metal ion is adsorbed more optimally. Besides playing a significant role in the adsorption process, zeolite also acts as a cation exchanger. This process is possibly done because zeolites are usually equipped with more than one side for cation exchange, so Pb (II) metal ions are not only adsorbed but also in the cation exchange process.

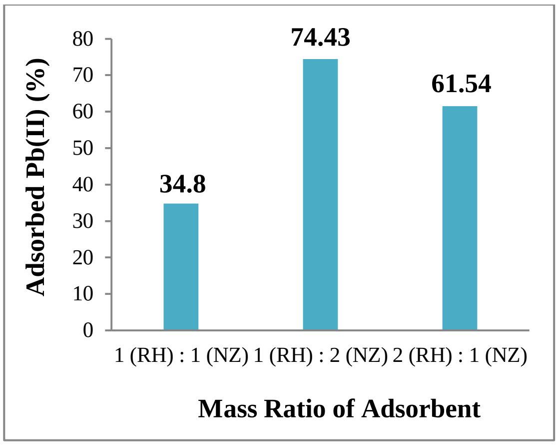


Figure 1. Determination of the optimum combination mass of adsorbent

Table 5. The influence of the composition of the mass to ion adsorption of Pb(II) ions

Mass Ratio of RH : NZ	Initial [Pb(II)] (µg L^{-1})	Final [Pb(II)] (µg L^{-1})	Adsorbed [Pb(II)] (µg L^{-1})	Adsorbed (%)
1 : 1	75.63	49.86	25.77	34.08
1 : 2	75.63	19.34	56.29	74.43
2 : 1	75.63	29.09	46.54	61.54

Determination of Detecting Limit

Table 6. The determination result of the limit of detection value

Blank solution	1	2	3	4	5	Average
Δ A	0.0903	0.1021	0.0582	0.0832	0.0665	0.0751
Concentration	-0.03	-0.02	-0.04	-0.03	-0.04	-0.0299
Std. Deviation						0.0083
Detecting Limit						0.025

The detection limit is the concentration of the most minor or lowest analytes in the sample detected. However, it does not need to be quantized. The resulting value does not meet the criteria for accuracy and precision—acceptance limit values for accuracy less than 5%. Suppose the analysis is carried out using a tool or instrument. In that case, the detection and quantization limits are determined by measuring the blank response several times, then the standard deviation of the blank response is determined (Torowati & Galuh, 2014). From the following data results, the detection limit analyzed by SPS has been met, amounting to 0.025 (2.50%). According to Torowati and Galuh (2014), the acceptable Limit of Detection (LOD) value is less than 5%.

4. CONCLUSIONS

The combination of low-cost adsorbent made from rice husk and natural zeolite (1:2) can reduce Pb(II) ions' concentration in wastewater. Analysis of wastewater containing Pb(II) ions below 1 ppb can be done well using a single beam UV-Visible Spectrophotometer for the SPS by K-MAC Lab Junior, Satoda Science, Japan with an adsorption capacity of 0.75 µg g^{-1}. SPS can produce a sensitive result in analyzing Pb(II) ions below 1 ppb, and SPS can also be determined as the cost-efficiency instrument.

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