

ANALYSIS OF LEAD WASTE ABSORPTION (PB) WITH ACTIVATED CHARCOAL ABSORBENT FROM PALM SHELL

SRI SURYANINGSIH*, FITRI LESTARI SHOLIAH, ANNISA APRILIA

*Department of Physics, Faculty of Mathematics and Natural Sciences, Padjadjaran University,
Jl. Raya Bandung -Sumedang Km.21, Jatinangor, Sumedang 45363, Jawa Barat
Phone (022) 779-6014*

Abstract. Some industries, especially the textile industry in general, use textile dyes containing lead heavy metals (Pb). Lead is a metal that is neurotoxin (toxic to the nervous system) and is accumulative because of its stable nature and has a long half-life, and it can be harmful to the environment and needs to be filtered using absorbent. This study aims to find out the absorption of activated charcoal palm shell as absorbent to eliminate lead waste. The method used by varying the concentration of H_3PO_4 and KOH activator solutions with concentrations of 20%, 30% and 40% respectively. Batch method is done to mix activated charcoal into Pb solution with variation of contact time of 30 minutes, 60 minutes, 90 minutes. SEM (Scanning Electron Microscope) and AAS ((Atomic Absorption Spectrophotometer) tests are conducted to find out the pore area and absorption. SEM test results showed that the best activator is KOH with a concentration of 40% compared to H_3PO_4 activators with the largest average pore area of 31.755,27 nm. AAS test results obtained the most absorption of 98.59% by activated charcoal KOH 30% at a contact time of 60 minutes.

Keywords: absorption, activators, palm shells, Pb acetate.

1. Introduction

Background

Activated charcoal is a charcoal with the configuration of carbon atoms that released from bonding with other elements and the pores are free from other elements or impurities, therefore activated carbon is recognize as carbon with a large surface area [1]. In general, activated charcoal can be made using coal and other material which is containing lignocellulose as raw element [2]. One of the potential materials that have an ingredient of lignocellulose to produce activated charcoal is the shell of oil palm [3].

The resulting activated charcoal can be used as a waste absorbent containing Pb. Lead or Pb is a metal which is neurotoxin (toxic to the nervous system) and is accumulative due to its stable nature and has a long half-life. Lead is very harmful to the environment, if the lead wasted into the environment has exceeded the specified threshold value [4]. The source of lead waste is around us such as textile factory [5], laboratory liquid [6], and motor workshop liquid waste [7]. To prevent pollution caused by lead, this study will be overcome by using activated charcoal as the absorbent.

This research is an analysis of lead waste absorption (Pb) with activated charcoal absorbent from the oil palm shell. In this study, oil palm shell as the main ingredient of charcoal carbonized at $380^{\circ}C$ which is then activated using an activator H_3PO_4 and KOH each to expand the pore surface and increase the absorption of charcoal against Pb contents.

*Email : sri@phys.unpad.ac.id

2. Research Methods

2.1. Research design

This research goes through the stages of the process as shown in the flow diagram at Figure 1.

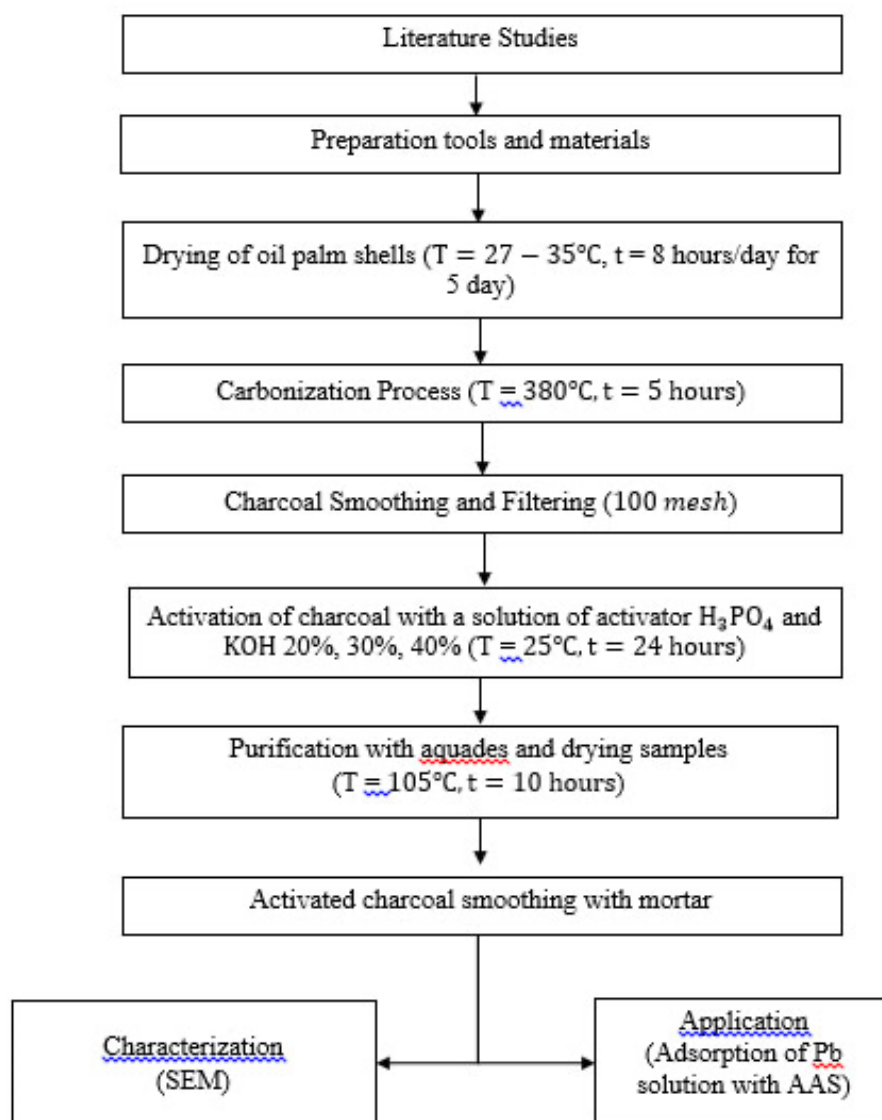


Figure 1. Research flow

2.1.1 Tools

Tools used in the manufacture of activated charcoal are: stopwatch, blender, sieve with a size of 100 mesh, measuring glass, pipette, furnace for drying, filter paper (Whatman 42, diameter 110 mm) simple furnace for carbonization, digital scale, ehlenhmeyer flask, magnetic stirrer, Scanning Electron Microscope (SEM) and Atomic Absorption Spectrophotometer (AAS).

2.1.2 Materials

The materials used in this study are as follows: palm shell, solution of H_3PO_4 (20%, 30% and 40%), solution of KOH (20%, 30% and 40%), solution of Pb acetate $Pb(CH_3COO)_2 \cdot 3H_2O$ in aquades, HCL and NaOH.

2.2 Procedure

2.2.1 Palm oil shell drying

The drying process is carried out for 8 hours with an average temperature range $27^\circ C - 35^\circ C$ within 5 days. Many of the water content lost in the dehydration process can be calculated using the formula:

$$\text{Air up (\%)} = \frac{m_0 - m}{m_0} \times 100\% \quad (1)$$

2.2.2 Carbonization process

Carbonization of oil palm shell with a temperature of $380^\circ C$ for 5 hours. The end product of the carbonization process is charcoal.

2.2.3 Smoothing and filtering

Once the charcoal is at room temperature the charcoal is smoothed using a blender. Then the charcoal flour that has been smoothed is filtered using mesh 100 to make it homogeneous.

2.2.4 Activation

The activation or soaking with activators H_3PO_4 20 %, 30%, 40% and KOH 20%, 30%, 40% with a ratio of charcoal and activator 1:3 at room temperature $25^\circ C$ for 24 hours. The following formulas are used to create different concentrations of activator solutions:

$$\text{Concentration Activator (\%)} = \frac{\text{gram activators}}{\text{gram solution}} \times 100\% \quad (2)$$

2.2.5 Active charcoal purification

The charcoal, then washed using filter paper and aquades until the pH is close to normal. The charcoal deposits located on the filter paper and put on a 50 ml bean glass for the drying process in the oven. Carbon leaching after activation is carried out to remove residual organic residues, contaminants, minerals and metal remnants left in the pore cavity. Metal oxides left in pores can affect the absorption of activated carbon in certain compounds. Absorption capability will also increase when the pH is lowered by adding mineral acids, because the ability of mineral acids will reduce the ionization of organic acids [8].

2.2.6 Drying activated charcoal samples

Activated charcoal that has been washed then dried inside furnace (Thermolyne) with temperature $105^\circ C$ for 10 hours until the activated charcoal becomes dry. Figure 2 shows the dryer.



Figure 2. Furnace tool for drying

2.2.7 Sample preparation

Waste as absorbent tested in this study is a solution containing metal Pb, solution made from $\text{Pb}(\text{CH}_3\text{OO})_2 \cdot 3\text{H}_2\text{O}$ compounds which is mixed with activated charcoal by batch method. Active charcoal from the study weighed 3.06 gram and put in a bean glass. Then, a solution of Pb acetate is added as much as 10 ml with a concentration of 5%. Next, stir with magnetic stirrer with time variation for 30 minutes, 60 minutes, and 90 minutes. Then the solution is filtered using filter paper to separate between the filtrate and its residue. Waste or filtrate is taken about 5 ml as a sample. Mixing with batch method is done on six activated charcoal samples with one sample having three variations of contact time, so that there are 18 samples to be tested.

2.2.8 Activated charcoal sample testing

Carbon Structure Test (SEM)

Detectors within Scanning Electron Microscope (SEM) detect reflected electrons and determine the location of high-intensity reflected beams. This direction will provide information on the surface profile of the object. Figure 3 (a) is an SEM tool used in research.



(a)



(b)

Figure 3. (a) Scanning Electron Microscope (SEM JEOL JSM-6510LA)
(b) Atomic Absorption Spectrophotometer (PerkinElmer AAnalyst 400)

Absorption Test (AAS)

The principle of atomic absorption spectrophotometer (AAS) analysis is based on the process of energy absorption by atoms located at the ground state. As a result of the radiation absorption process, electrons from these freely excitable atoms are unstable and will return to their original state

accompanied by emitting radiation energy with a certain wavelength and characteristics of each element. An image of the AAS tool shown by Figure 3 (b).

3. Results dan Discussion

3.1 Drying process results

In Table 1, mass decrease occurs due to the loss of moisture content in the shell of oil palm because it evaporates after being heated by the sun. By using formula (1) the lost of water content can be calculated as 5.35% of water lost.

Table 1. Results of palm oil shell dehydration process

Temperature (°C)	Time (Day)	Wet Weight (kg)	Dry Weight (kg)	Water Lost (%)
27 - 35	5	28	26.5	5.35

3.2 Carbonization process results

Based on Table 2 of the carbonization results showed a decrease in the mass of oil palm shell after carbonization, as for the decrease in the mass of the palm shell is 18.3 kg (69.05%), this mass loss proves the presence of other materials are reduced from the shell of oil palm such as cellulose, hemicellulose and lignin, also water content lost. Figure 4 shows a rough texture of charcoal after carbonization process.

Table 2. Carbonization process results

Temperature (°C)	Time (Hours)	Initial Mass(kg)	Final Mass(kg)	Lost Mass (%)
380	5	26.5	8.2	69.05



Figure 4. Oil palm shell charcoal after carbonization process

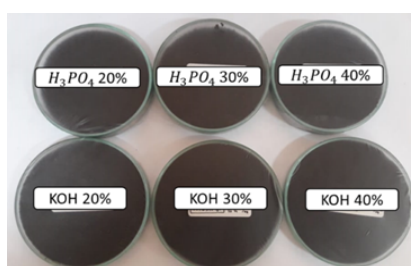
3.3 Activation process results

In Table 3, an increased activators concentration would increase the charcoal mass after washing process and obtained charcoal mass depreciation. Depreciation in charcoal can occur because in the process of activation, the impurities that cover the pores of charcoal become loosen (evaporate) as the temperature increases during drying after activation. In addition, the shrinkage in charcoal can also be caused because during the washing process there is a leak in the filter.

Table 2. Activated charcoal mass data during activation process

Activator	Concentration (%)	Initial Mass (g)	Mass after washing (g)	Mass after drying (g)	Charcoal Depreciation (g)
KOH	20	30	46.2483	29.554	0.4460
	30		48.0812	28.6311	1.3689
	40		56.7946	29.4051	0.5949
H ₃ PO ₄	20	30	49.1831	24.2876	5.7124
	30		52.0301	27.6149	2.3851
	40		88.0452	29.2807	0.7193

Figure 5 is an image of oil palm shell charcoal that has been activated with two variations of activator solution, namely KOH, and with the concentration of H₃PO₄ each activator varied also by 20%, 30%, 40%. In addition, the greater the concentration of activators given, the result of charcoal obtained has a smaller grain size of powder and the structure is getting smoother.

**Figure 5.** Results of charcoal activation process from palm oil shell

3.4 SEM test results

The Scanning Electron Microscope (SEM) test results will be seen in the image in the form of pores formed on activated charcoal palm shell.

Figure 6 shows image of the charcoal before the activation, it has few and small pores also on the pores that are much dirtyers. The following is an image of SEM for charcoal that has been activated by KOH solution with concentrations of 20%, 30% and 40%.

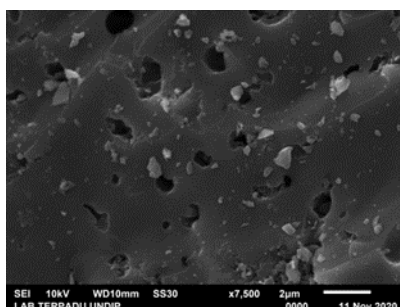
**Figure 6.** Photo of SEM charcoal morphology before activated magnification by 7500 times

Figure 7(a) the cleanest active charcoal surface concentration is at 20%, occurs because at a concentration of 20% there are not many KOH compounds that crystallize after the activation process. While in Figure 7(b) at 30% concentration there is KOH that crystallizes on the charcoal surface that covers its pores, as well as at 40% concentration (Figure 3.4 (c)), KOH crystals covering the pores so that are slightly visible, but at the pore area it's found to increase in line with the concentration value, as shown in Table 4.

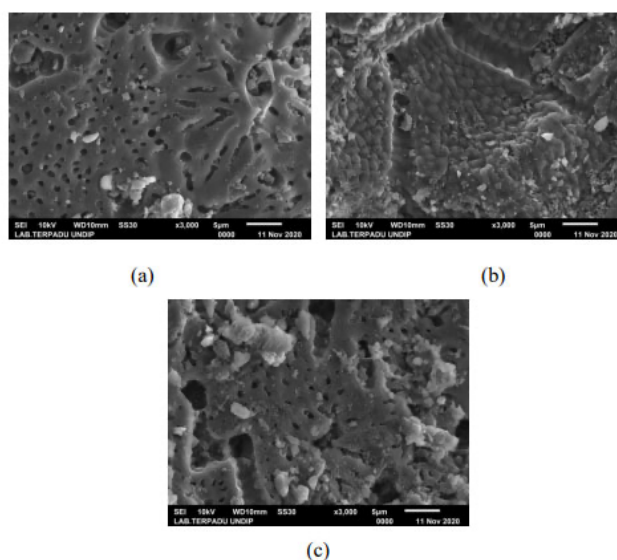


Figure 7. Image SEM: Morphology charcoal activated KOH, (a) 20 %, (b) 30%, (c) 40%

Table 4. Area of Active Charcoal Pores activated KOH and H_3PO_4

Charcoal Concentration	Pore Area (nm)	Charcoal Concentration	Pore Area (nm)
Pure	7959.87	Pure	7959.87
KOH 20%	15511.43	H_3PO_4 20%	13129.53
KOH 30%	22130.28	H_3PO_4 30%	27189.22
KOH 40%	31755.27	H_3PO_4 40%	24001.47

It can be concluded that the greater the concentration of KOH solution, the wider the pore obtained, but it will have more KOH crystals that cover the pores. Furthermore, there are photos of activated charcoal SEM H_3PO_4 shown by Figure 8. In Figure 8, the result of SEM activated charcoal solution H_3PO_4 at concentration of 30% shows that there are the largest pores but the number of pores is less than the SEM image for a concentration of 40%. When compared to SEM image from 20% concentration, 30% concentration is better, there are more H_3PO_4 crystals on the charcoal surface. H_3PO_4 crystals also can be clearly seen at 40% concentration SEM photos as well. H_3PO_4 activated charcoal pore area can be seen in Table 5.

SEM test results on activated charcoal H_3PO_4 can conclude that the increase of H_3PO_4 solution concentration would obtain more pores and more H_3PO_4 crystals that covers its pores. As seen at SEM image results, the 40% KOH activated charcoal is better than any H_3PO_4 activated charcoal, because it has a larger average pore area at 31,755.27 nm.

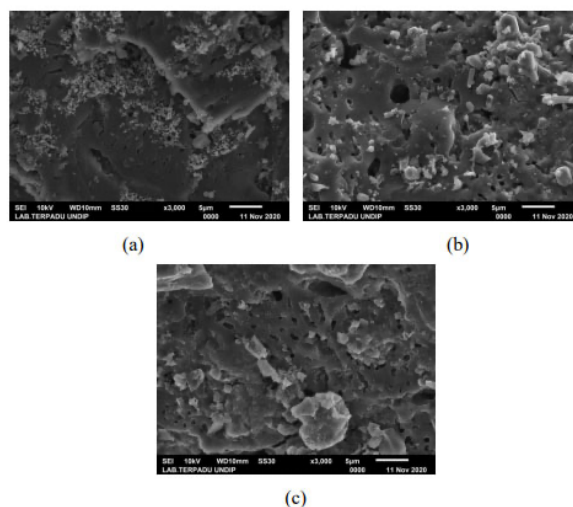


Figure 8. Photo SEM Activated Charcoal Morphology H_3PO_4 (a) 20 % (b) 30% (c) 40%

Table 5. AAS test results data

Concentration Activator (%)	Time (minutes)	Early Pb Rate (%)		Pb End Rate (%)		Absorbed Pb Levels (%)	
		KOH	H_3PO_4	KOH	H_3PO_4	KOH	H_3PO_4
20	30			47.2	34.8	52.8	65.2
	60		100	42.2	35.8	57.8	64.2
	90			41.2	86.8	58.8	13.2
30	30			31	21.8	69	78.2
	60		100	1.41	37.8	98.59	62.2
	90			18.6	89.4	81.4	10.6
40	30			26.2	4.8	73.8	95.2
	60		100	27	69.8	73	30.2
	90			11.8	63	88.2	37

Chemical activation using KOH activators and H_3PO_4 could erode carbon walls to form new pores thus increase porosity. Therefore, activated charcoal has more visible pores than charcoal before activated. But some pores that are partially formed were closed, because the presence of unwanted chemical element that contaminate the material samples

3.5. AAS test results

In Table 5, the activated charcoal absorption power with a concentration of activators of 20%, known for charcoal activated KOH experienced an increase in Pb concentration absorbed each increase in contact time, meaning the longer the contact time the more waste Pb acetate absorbed. While for activated charcoal H_3PO_4 decreased concentration of Pb absorbed every time of contact, means the longer the contact time the less waste Pb acetate absorbed, especially at the contact time of 90 minutes that only absorbs 13.2% of waste Pb.

The activated charcoal absorption power at a concentration of KOH 30%, which shows the longer the contact time the more Pb waste is absorbed. This indicates that charcoal is still optimally absorbing Pb waste, while in activated charcoal H_3PO_4 the longer the contact time the charcoal absorbs less waste Pb, which means the more towards saturation in 90 minutes of contact time.

The activated charcoal activators with a concentration of 40% showed temporary saturation at the contact time of 60 minutes, both samples experienced a decrease in the concentration of Pb waste absorbed, but again experienced an increase in the concentration of waste Pb at the contact time of 90 minutes.

The alignment of data showing that KOH activators are better at activating oil palm shell charcoal compared to H_3PO_4 activators judging from the average amount of active charcoal pore area, and the concentration of activated charcoal absorption of the Pb solution. From these three activators solution concentration variations, can be concluded that 40% KOH activated charcoal concentration is proven better from average value of the large pore area seen from SEM test results. And from AAS test results are also known that 40% KOH charcoal activated has a considerable concentration of absorption at 88,2 % with 90 minutes contact time.

4. Conclusion

As seen on the results of SEM photos, KOH and H_3PO_4 activators could change the morphological structure of the oil palm shell by increasing porosity. Increasing the activator solution concentration will increase the obtained average pore area. In addition to that, activator compounds that crystallize on the surface of activated charcoal has also occurred. By using 40% KOH, activated charcoal has the largest average pore area at 31,755.27 nm. Based on data on the concentration of activated charcoal absorption of Pb waste with testing using AAS, got that the best absorption rate at 98.59% are by using 30% KOH activated charcoal with 60 minutes contact time.

5. Acknowledgment

We would like to thank UNPAD Central Laboratory and UNDIP Integrated Laboratory as the place where activated charcoal testing is carried out as well as all parties who have contributed to the completion of this research.

References

- [1] R. Sudradjat and P. Gustan, *Arang Aktif: Teknologi Pengolahan dan Masa Depan*, Jakarta: Badan Penelitian dan Pengembangan Kehutanan, 2011.
- [2] A. Gracia-Garcia, A. Gregorio, D. Boavida and I. Gulyurtlu, *Production and Characterization of Activated Carbon from Pine Wastes Gasified in A Pilot Reactor*, Portugal: National Institute of Engineering and Industrial Technology Estrada do Paco do Lumiar, 2002.
- [3] E. Sari, S. Syamsiah, H. Sulistyono and M. Hidayat, *Biodelignifikasi Eceng Gondok untuk Meningkatkan Digestibilitas pada Proses Hidrolisis Enzimatik*, Reaktor, vol. 17, pp. 53-58, 2017.
- [4] Prabowo and A. Liberty, *Pembuatan Karbon Aktif dari Bonggol Jagung serta Aplikasinya untuk Adsorpsi Cu, Pb, dan Amonia*, Depok: Universitas Indonesia, 2009.
- [5] C. R. Priadi, A. P. N. Sari and S. S. Moersidik, *Absorpsi Logam Seng dan Timbal pada Limbah Cair Industri Keramik oleh Limbah Tanah Liat*, Reaktor, Vol. 15 No. 1, 2014.
- [6] M. Audiana, I. Apriani and U. Kadaria, *Pengolahan Limbah Cair Laboratorium Teknik Lingkungan dengan Koagulasi dan Adsorpsi untuk Menurunkan COD, Fe, dan Pb*, Jurnal Teknologi Lingkungan Lahan Basah, 2017.

- [7] E. S. Nadeak, N. Aldo and H. Horiza, *Analisis Kandungan Timbal (Pb) pada Limbah Cair Bengkel Kendaraan Bermotor di Kota Tanjungpinang Tahun 2014*, Jurnal Poltekkes Jambi Vol XIII Nomor 3, 2015.
- [8] S. T. Meiliata and T. Sinaga, *Pengenalan dan Proses Pembuatan Arang Aktif*, Medan: Sekolah Pascasarjana Universitas Sumatera Utara, 2003.