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Study into the Effectiveness of Using Activated Carbon of Kluwak Shell (Pangium Edule Reinw) as Adsorbent of Heavy Metals in Wastewater

Yuyun Sukawati Rusma[1*], Paulina Taba[2], St Fauziah[2], Muhammad Zakir[2], Farid Samawi[1], Alimuddin Hamzah Assegaf[3]

[1] Department of Environmental Management, Graduate School, Hasanuddin University, Makassar 90245, Indonesia

[2] Department of Chemistry, Faculty of Mathematics and Natural Sciences, Hasanuddin University, Makassar 90245, Indonesia

[3] Department of Geophysics, Faculty of Mathematics and Natural Sciences, Hasanuddin University, Makassar 90245, Indonesia

* Corresponding author's e-mail: yuyunsukawati2111@gmail.com

ABSTRACT

The purpose of this study is to determine how well activated carbon made from kluwak shell (Pangium edule Reinw) works as an adsorbent to remove Pb(II) and Cu(II) heavy metal ions from wastewater used in laboratories. Kluwak shell was selected as an agricultural waste material due to its high carbon content, which makes it a promising material for activated carbon that can lower Pb(II) and Cu(II) heavy metal levels. Finding the ideal contact time, pH, and adsorption capacity as well as the kinetics and adsorption isotherm model are the main goals of this study. Using 25% KOH for carbonization and chemical activation, Fourier Transform Infrared (FTIR), Scanning Electron Microscopy (SEM), and Surface Area Analyzer (SAA) for characterization are some of the research methods used. FTIR results showed the presence of O-H, C-H, C = C and C-O functional groups, while SEM showed more open pores after activation. SAA analysis indicated an activated carbon surface area of 3.11 m^2/g, pore volume of 0.006 cm^3/g, and pore diameter of 4.08 nm, categorized as mesoporous. The optimum condition for adsorption of Pb(II) ions is at pH 5 with contact time of 10 minutes and adsorption capacity of 21.83 mg/g. As for Cu(II) ions, the optimum condition is at pH 4 with a contact time of 20 minutes and adsorption capacity of 10.82 mg/g. The adsorption of metal ions is in accordance with the Langmuir isotherm model and pseudo second-order kinetics. The adsorption effectiveness of Pb(II) and Cu(II) ions from laboratory wastewater was 1.69 mg/g and 1.73 mg/g. It is concluded that activated kluwak shell can be used as Pb(II) and Cu(II) metal adsorbent.

Keywords: adsorption, Pb(II), Cu(II), Laboratory wastewater, kluwak shell.

INTRODUCTION

Environmental pollution is a critical issue that affects human beings [Geca et al., 2022]. One source of environmental pollution is laboratory wastewater [Audiana et al., 2017]. In general, laboratory wastewater is smaller than industrial waste. Still, it is generated in the long term. In that case, it requires serious handling because if it is not managed correctly, it can pollute the environment through direct disposal into waterways, which can damage soil structure, disrupt ecosystem balance, and cause health problems [Sukmawardani and Amalia, 2019; Sunarti and Sutejo, 2021]. Laboratory wastewater contains heavy metals and hazardous organic matter such as lead (Pb) and copper (Cu). These heavy metals have high toxicity effects and can be accumulated in human bodies, which cause serious damage to health [Chaemiso and Nefo, 2019; Zhang et al., 2023]. Adsorption is one method of removing heavy metals [Priyadi et al., 2015]. Adsorption uses simple equipment [Khairy et al., 2022], is low cost, is easy to apply, and does not cause side effects [Adeyemo et al., 2017]. The procedure is dependent on the adsorbent's surface area, which may be raised by activation and carbonization [Sahendra et al., 2021]. Biomass agricultural waste such as kluwak (*Pangium edule Reinw*), which grows in tropical forests in Indonesia [Faisal and Pato, 2021; Sirajuddin et al., 2020], produces kluwak shells after harvesting which have not been optimally utilized so that they can be used as an efficient adsorbent material [Adhar et al., 2022]. Kluwak shell contains high carbon and 70.52% cellulose, which can be activated into activated carbon with good adsorption ability towards heavy metals [Oktaviandra et al., 2020]. Several studies have explored the potential of kluwak shell as a starting material to produce adsorbents with the ability to efficiently remove organic and inorganic pollutants such as research by [Salsabila, 2022] in the absorption of Cd (II) metal obtained ability to adsorption of 3.673 mg/g and research [Yuliani et al., 2023] in the absorption of Methylene Blue attained the ability to adsorb of 47.05 mg/g. Previous research shows a critical gaps in literature regarding a comparative study about kluwak shells in removing heavy metals in wastewater effluents, especially Pb(II) and Cu(II). These metals were chosen because they can have adverse effects on living beings, causing damage to the the brain, liver, kidney, and even death. The objectives we have in addressing this research gap are to determine how well using activated carbon works from kluwak shells in decreasing heavy metal levels, especially Pb(II) and Cu(II), contributing insights into sustainable wastewater treatment in utilizing agricultural waste from kluwak shell as a substitute medium for treating wastewater.

MATERIALS AND METHODOLOGY

Materials

Kluwak shell obtained from Kahu District Bone Regency South Sulawesi, Indonesia, Laboratory wastewater (obtained from the Science Research and Development Laboratory FMIPA Unhas), dobly distilled water, potassium hydroxide (KOH), lead (II) nitrat ($Pb(NO_3)_2$), copper (II) sulfate pentahydrate ($CuSO_4.5H_2O$), pH paper (E-Merck), Whatmann paper no. 42.

Methods

Preparation of activated carbon

The kluwak shells were nested in a furnace at 350 °C for two hours. The resulting carbon was cooled and pulverized using a crusher and then sieved using a 100-mesh sieve. The activation of Kluwak shell carbon was 25% KOH in a ratio of 1:4 b/v (carbon: KOH). Stirring the mixture was done at a hot plate Stirrer for four hours at an 80 °C temperature. After letting the mixture stand for a full day, a Buchner funnel was used to filter it. The resulting precipitates were neutralized by washing them in distilled water, dried for two hours at 150 °C in the oven, and cooled in a desiccator [Yuliani et al., 2024]. (Figure 1)

Characterization of activated carbon

Kluwak shell carbon and activated carbon were examined using both SEM and FTIR spectroscopy are utilized. The FTIR examines changes in the vibrational frequencies of surface functional groups and functional groups of carbon and activated carbon. To examine the surface morphology of carbon and activated carbon, we obtained SEM images using a Hitachi Flexsem 1000.

Determination of optimal condition

The parameters applied to metal ions Pb(II) and Cu(II) are contact time, pH, and initial concentration of metal ions in solution; the pH level is adjusted from 2 to 6. Contact time ranges between 1 and 60 minutes, while the starting metal ion concentration in the solution is set in the range of 25 to 200 mg/L. After filtering the mixture, the filtrate was gathered and measured using an atomic absorption spectrophotometer (AAS) [Tandigau et al., 2018; Ermadani et al., 2023].

Adsorption experiment

Kluwak shell activated carbon was weighed as much as 0.1 gram, and 50 mL of wastewater was added to each Erlenmeyer. Shaker was used to stir the mixture with the optimum time of Pb and Cu and filtered. Next, the absorbance of the filtrate was measured using an AAS. Measurement of the effectiveness of adsorption potential is seen in formula (1) as follows (Abbou et al., 2021):

$$q = \frac{C_0 - C_e}{m} \times V \quad (1)$$

where: q represents the adsorption capacity (mg/g), V is the quantity of the solution

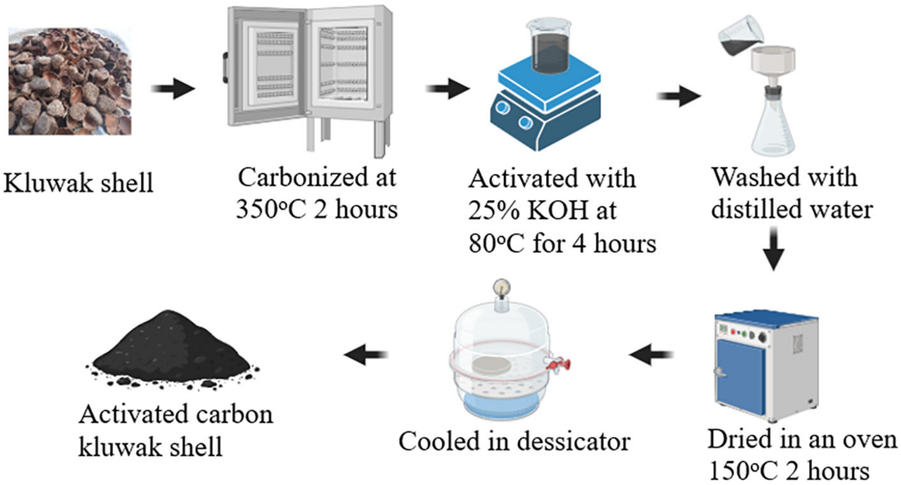

Figure 1. Preparation of activated carbon samples

(L), m is the weight of adsorbent (g), C_0 is the starting concentration (mg/L), and C_e is the concentration during the experiment (mg/L).

RESULTS AND DISCUSSION

Activated carbon characterization

Fourier transform infrared (FTIR) characterization

In analyzing the FTIR spectrum of kluwak shell carbon (Figure 2a) and kluwak shell activated carbon (Figure 2b), several prominent peaks associated with specific functional groups can be identified. The peak at 3400–3500 cm^{-1} represents the existence of hydroxyl (OH) groups, while the peak around 2900 cm^{-1} is related to the aliphatic C-H stretch (Figure 2a). The peak at 1600 cm^{-1} can be attributed to aromatic ring C = C vibrations, and the peaks around 1000–1300 cm^{-1} indicate C-O is alcohol vibrations, ethers, or esters. Some changes indicate carbon activation and the addition of new functional groups. The peak at 3400–3500 cm^{-1} still shows the hydroxyl groups' existence, and the peak around 2900 cm^{-1} still shows aliphatic C-H stretching [Queiroz et al., 2020] (Figure 2b). The peak at 1600 cm^{-1} is more

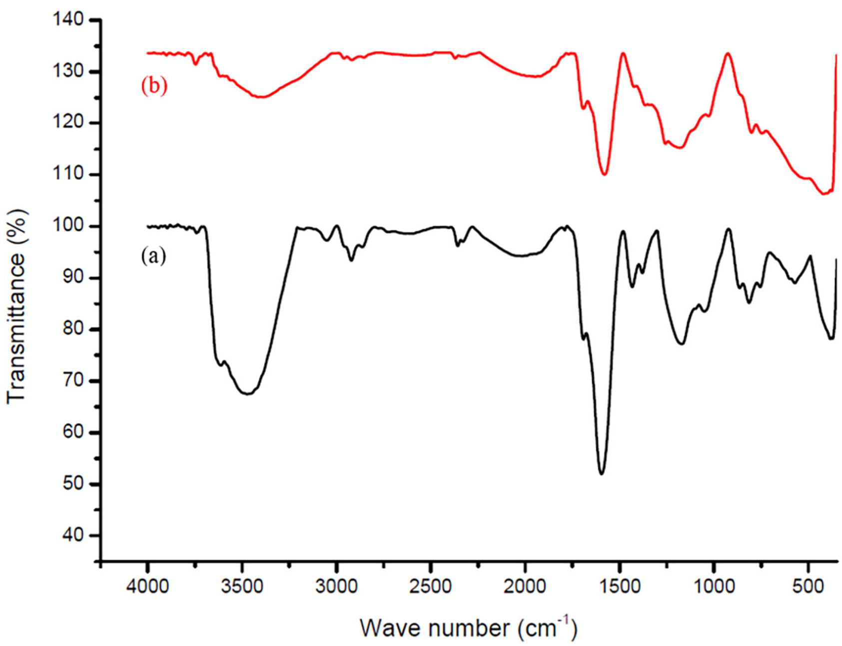

Figure 2. FTIR spectra of (a) kluwak shell carbon (b) kluwak shell activated carbon

pronounced, indicating a more conjugated aromatic C = C bond. The presence of carbonyl groups (C = O) is indicated by a new peak at roughly 1700 cm^{-1}, and peaks at about 1000–1300 cm^{-1} indicate modification of the chemical structure during the activation process [Pehlivan, 2017].

Scanning electron microscope (SEM) characterization

Figure 3a shows the surface structure of kluwak shell carbon before activation. This structure still has a rough surface with irregular particle shapes and small pores and is evenly distributed throughout the surface. However, Figure (3b) shows the surface structure of kluwak shell carbon after activation. The surface structure looks smoother with larger and more defined pores. Activation using activating agents is likely to have resulted in an expansion in the size and quantity of pores and a reduction in non-carbonaceous material. Activated carbon using KOH generally increases the specific surface area and pore volume [Sudaryanto et al., 2006; Luo et al., 2013]. Activated carbon that has been activated has a maximum metal ion absorption performance because the surface or pores of activated carbon are more open, so it absorbs more ions compared to carbon before activation (Noor et al., 2020).

Brunaeur-Emmet-Teller (BET) method characterization

Characterization using N adsorption-desorption was used to measure the activated carbon's surface area and pore size using the Brunaeur-Emmet-Teller (BET) method. Kluwak shell activated carbon has a surface area of 3.11 m^2/g, a volume of pores 0.006 cm^3/g, and a pore diameter of 4.08 nm. In accordance with the International Union of Pure and Applied Chemistry's (IUPAC) classification, the pore size of the kluwak shell's activated carbon was mesopores due to its small pore size, varying from 2 to 50 nm [Alcaraz et al., 2018]. As a comparison, Muhajir et al. (2021) obtained a surface area of carbon activated by coconut shell of 23.24 m^2/g. Research conducted by Allwar (2016) using oil palm shells obtained a surface area of 325.40 m^2/g. The surface area of activated carbon obtained is small, possibly due to the different carbonization processes [Hidayat et al. 2019].

Effect on contact time

The optimum time of adsorption by kluwak shell activated carbon is determined by calculating the maximum quantity of ions made of metal that the adsorbent can absorb. From the outcomes of the study using time (Figure 4) variations of 1, 5, 10, 15, 20, 30, 40, 50, and 60 minutes, the quantity of Pb(II) ions that were adsorbed at the optimum contact time at 10 minutes was 10.92 mg/g and 7.99 mg/g of Cu(II) ions had been adsorbed after 20 minutes. The ideal time for Pb(II) and Cu(II) ion adsorption shows different outcomes based on the kind of adsorbent employed. In a study conducted by Mawlood et al. (2024) using sunflower seed coat activated carbon, the optimum Pb(II) was reached at 30 minutes. In the research of Rahimnejad et al. (2018) using activated carbon derived from kiwi skin, Pb(II) ions are most effective adsorbed for 180 minutes at a rate of 0.15 mg/g.

Fillaeli et al. (2019) used sea pandanus leaf as a source of activated carbon for Cu(II) adsorption and obtained an optimum time of 60 minutes with the quantity of ions adsorbed of 2.94 mg/g. Siaka and Sahara's research (2018) utilized the

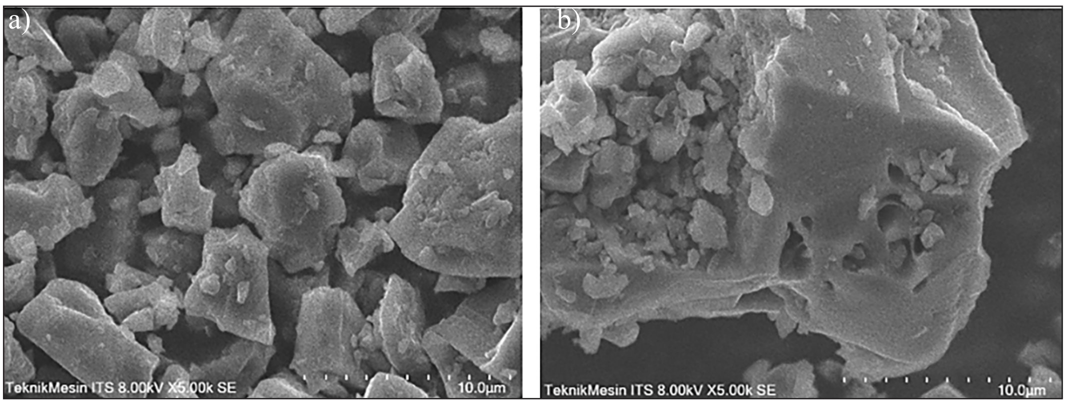

Figure 3. Surface morphological structure of (a) kluwak shell carbon (b) kluwak shell activated carbon

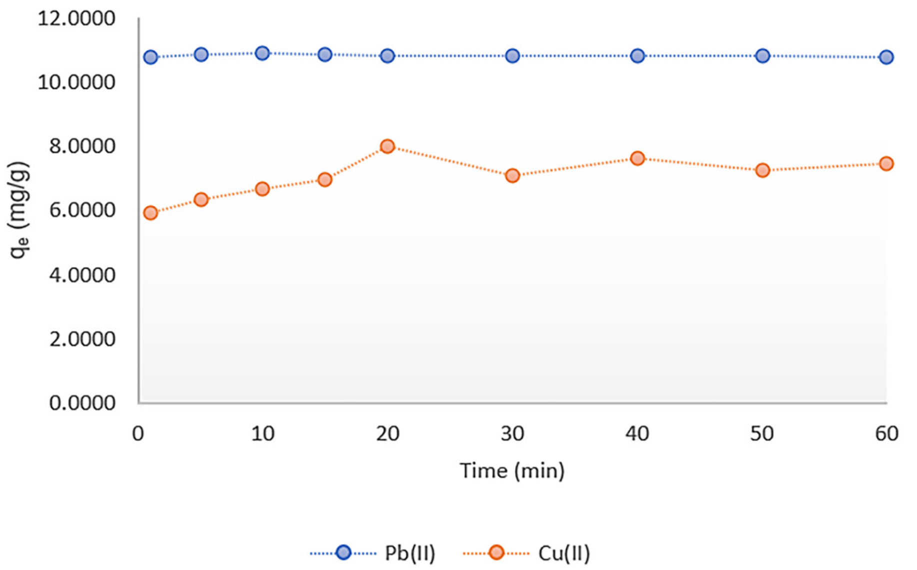

Figure 4. Effect of contact time on the amount of Pb(II) and Cu(II) ions adsorbed by kluwak shell activated carbon

Marigold stem to produce activated carbon in adsorbing Cu(II) ions and obtained that 40 minutes was the ideal duration with the number of adsorbed ions of 0.20 mg/g. The optimum times obtained in this study for Pb(II) and Cu(II) ions were 10 minutes and 20 minutes.

Effect on pH adsorption

The acidity (pH) is essential in adsorption because changes in the pH of the adsorbent's surface charge is modified by the solution. Based on Figure 5, it is clear that Pb(II) ions at pH 5 experience a significant increase, and in this condition, the greatest amount of Pb(II) ions adsorbed, namely 14.91 mg/g. The optimum pH for the adsorption of Cu(II) ions is pH 4, with the number of ions adsorbed at 4.98 mg/g.

Pb(II) and Cu(II) ion adsorption at low pH (pH < optimum pH) tend to be small due to competition between H^+ ions of Pb(II) and Cu(II) to interact with the exterior of activated carbon, leading to the ions' rejection. Pb(II) and Cu(II) adsorption of ions at pH > than the ideal pH is ineffective because of precipitation. The number of Pb(II) and Cu(II) ions decreases in solution because they form hydroxide compounds $Pb(OH)_2$ and $Cu(OH)_2$ [Sahara et al., 2017].

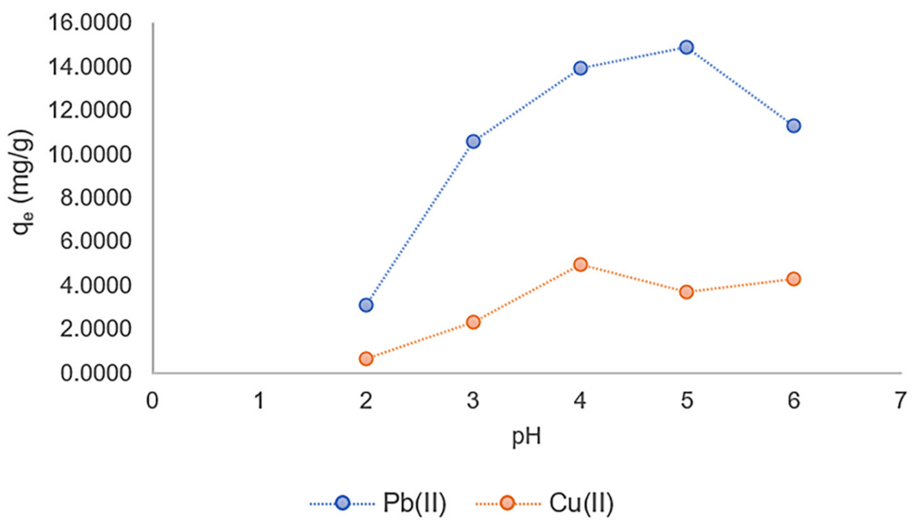

Figure 5. Effect of pH on the amount of Pb(II) and Cu(II) ions adsorbed

Moyo et al. (2013) used tasseled corn-activated carbon to adsorb Pb(II) ions and obtained the optimum pH at pH 5, Considering the number ions are adsorbed at 37.31 mg/g. Aminah et al. (2022) used durian skin waste biomass in adsorbing Pb(II) ions and reported the optimum pH at 5, and the number of ions adsorbed was 4.12 mg/g. Research conducted by Adriansyah et al. (2018) using coffee skin to adsorb Cu(II) ions obtained the ideal pH of pH 4, corresponding to 62.5 mg/g of adsorbed ions. Shirendev et al. (2021) utilized modified activated carbon and obtained were able to achieve pH 4, which is the ideal pH for Cu(II) ions, with 1.94 mg/g of ions adsorbed.

Adsorption isotherms

Figures 6 and 7 show that the optimum adsorption condition by activated carbon kluwak shell Pb(II) and Cu(II) ions occurs at 200 mg/L adsorbate solution. At low concentrations, the magnetite active site is only slightly filled with Pb(II) metal ions, so the adsorption process continues to increase to a concentration of 200 mg/L. The greater the adsorbate concentration, the greater the amount of substance collected on the adsorbate surface. Adsorption capacity can be determined by two models of isotherms, Langmuir isotherm (Fig. 6) and Freundlich isotherm (Fig. 7), respectively, for the adsorption of Pb(II) and Cu(II) ions.

These two isotherms describe different sorption mechanisms on the adsorbent surface. According to the Langmuir isotherm model, sorption takes place on a homogeneous surface with uniform sorption energy for all adsorbed molecules [Muliani et al., 2023]. It describes monolayer sorption on the adsorbent surface. Based to the Freundlich isotherm model, sorption takes place on surfaces that are heterogeneous with varying sorption energy. It describes multilayer sorption and is often utilized to explain sorption on non-uniform surfaces [Anggriani et al., 2021]. Based on the Table, The isotherm of adsorption for Pb(II) and Cu(II) ions is Langmuir isotherm because The (R^2) coefficient of correlation that was found to be almost identical to the of one. The results of the comparison of Langmuir and Freundlich isotherms on Table 1.

Adsorption kinetics

Adsorption kinetics studies the uptake rate of ions or molecules from a solid phase by an adsorbent. The rate in which Pb(II) and Cu(II) ions are

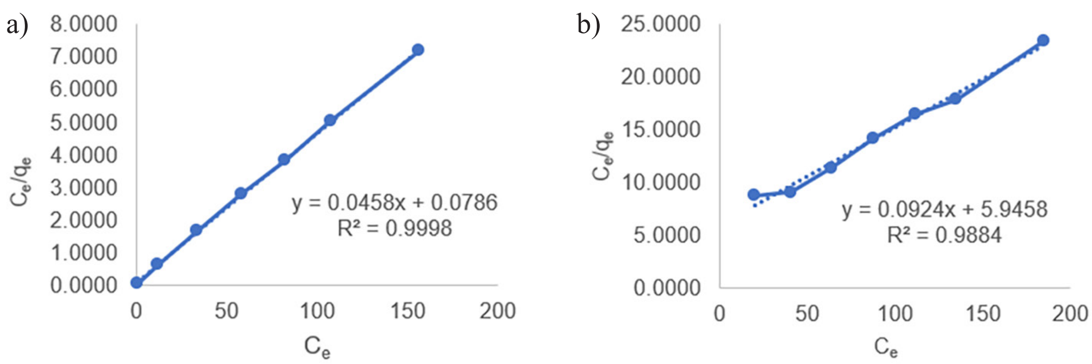

Figure 6. Isotherm Langmuir (a) Pb(II), (b) Cu(II)

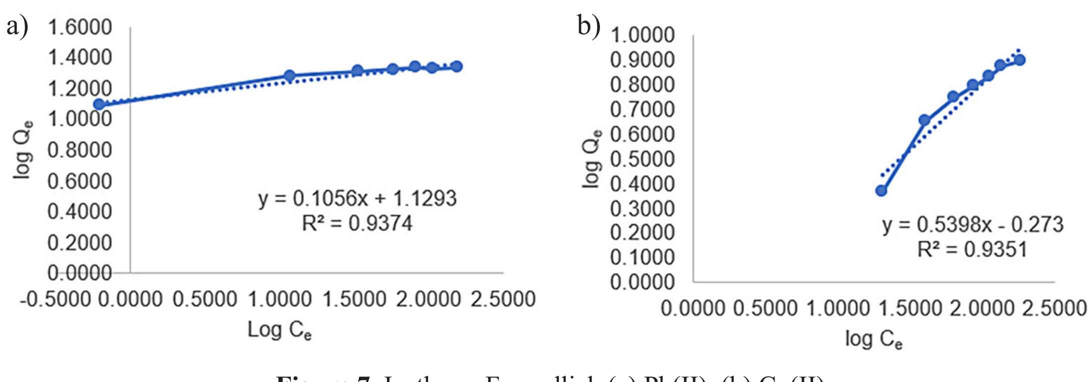

Figure 7. Isotherm Freundlich (a) Pb(II), (b) Cu(II)

Table 1. Langmuir and Freundlich isotherm parameters

Parameters	Langmuir			Freundlich		
	Q_o (mg/g)	*b* (L/mg)	R^2	K_f (mg/g)	*n* (L/mg)	R^2
Pb(II)	21.83	0.5827	0.9998	13.4679	9.4697	0.9374
Cu(II)	10.82	0.0155	0.9884	0.5333	9.4697	0.9351

Table 2. Kinetics models of Pb(II) and Cu(II) ions adsorption

Parameters	Pseudo first order			Pseudo second order			q_e eksp (mg/g)
	K_1	R^2	q_e (mg/g)	K_1	R^2	q_e (mg/g)	
Pb(II)	0.2058	0.3989	0.0466	3.6403	1	10.9289	10.9178
Cu(II)	0.0396	0.9402	2.0114	0.1097	0.9844	7.9808	7.9878

Table 3. Comparison of adsorption Pb(II) and Cu(II) by agricultural wastes as adsorbents

Ions	Adsorbent	pH	Time (min)	q_e (mg/g)	Reference
Pb(II)	Coal-based	5	60	162.33	Yi et al., 2016
	Nutmeg shell	5	40	1.93	Ishar, 2024
	Coconut shell	5	240	1.07	Maruni et al., 2022
	Kluwak shell	5	10	1.69	This study
Cu(II)	Sorghum stem	6	10	6.74	Anisa et.al., 2024
	Sugarcane bagasse	6	90	3.65	Pranata et al., 2022
	Cocoa pods husk M45	6	90	28.41	Pabbenteng et al., 2020
	Kluwak shell	4	20	1.73	This Study

absorbed by kluwak shell activated carbon is ascertained by calculating the adsorption kinetics. Determination of adsorption one can use pseudo-first-order or pseudo-second-order equations to study kinetics. [Melichova and Hromada, 2013]. The correlation coefficient (R^2) for Pb(II) and Cu(II) ion adsorption applying pseudo-second-order kinetics is close to 1, and the q_e value derived from the model which has pseudo-second-order is closest to the trial q_e value. This indicates the suitable adsorption kinetics model for Pb(II) and Cu(II) ions follows pseudo-second order. Comparison of Pb(II) and Cu(II) ion kinetics models observable in Table 2.

Adsorption effectiveness of Pb(II) and Cu(II) ions in waste water

Table 3 shows a comparing the adsorption of Pb(II) and Cu(II) by Agricultural wastes as adsorbents. Table 3 indicates that the capacity for adsorption of various adsorbents made from agricultural waste is different. The findings indicated that activated carbon with kluwak shell has significant potential as a heavy metal adsorbent. The data obtained from the experiment of the kluwak shell can effectively adsorb ions of Pb(II) and Cu(II) in wastewater. The morphological structure and chemical composition of kluwak shell activated carbon contributes as an adsorbent of Pb(II) and Cu(II) metal ions, as well as The interactions of kluwak shell activated carbon with the function groups of Pb(II) and Cu(II) ions of metals. Cheap, easily accessible raw materials and a comparatively simple modification procedure creates a substitute to adsorbing the environment's heavy metal pollution. This study shows that kluwak shell activated carbon has great potential as a heavy metal adsorbent and makes an important contribution to environmental pollution management efforts. Continuing this research is expected to be a more effective and sustainable solution when dealing with future issues related to heavy metal pollution.

CONCLUSIONS

This study successfully created an economical kluwak shell adsorption method for eliminating heavy metals from wastewater. The optimum times for Pb(II) and Cu(II) ion adsorption by kluwak shell (Pangium edule Reinw) activated carbon adsorbent were 10 and 20 minutes at pH 5 and pH 4, respectively. With an adsorption capacity of 21.83 mg/g and 10.82 mg/g, kluwak shell activated carbon adsorbs Pb(II) and Cu(II) ions, satisfying pseudo second-order kinetics and Langmuir isotherm. The efficiency of Pb(II) and Cu(II) ion adsorption in laboratory wastewater containing Pb(II) ions 1.69 mg/g and Cu(II) ions 1.73 mg/g. Researchers recommend that using kluwak shells in large quantities can lower the waste's concentrations of the metals Pb(II) and Cu(II) and reduce the amount of kluwak shell waste in the environment.

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