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To cite this article: Ruka Yulia *et al* 2024 *IOP Conf. Ser.: Earth Environ. Sci.* **1290** 012008

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Study of adsorption isotherms on removal of Cu (II) solution using activated carbon of sugar palm fruit shell (*Arenga pinnata*)

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Abstract. Sugar palm fruit shells (SPFS) are abundant agricultural waste in Indonesia especially in Aceh. It can be converted into activated carbon for heavy metal adsorption. This objective of this work is to study the maximum capacity of activated carbon derived from SPFS for copper ions adsorption. The sample size of 100 mesh was activated physically via heating at a temperature of 600 ºC for 2 hours. The Cu (II) metal ions of 30, 70, 100, 150, 200, 250, 300 ppm were prepared for the process. The prepared adsorbent was tested by adding a certain amount of copper sulphate concentration, 1 gr of adsorbent, and a contact time of 90 minutes. Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM-EDX) used in this work. The copper ions were calculated using Atomic Absorption Spectrophotometer (AAS). Both isotherm Langmuir and Freundlich models were utilized in this process. The Freundlich adsorption isotherm was found to be the fittest model isotherm with the adsorption capacitity of Cu(II) of 4,66 mg/g, mg/g, n = 1.02 with R^2 = 0,97,9713. The activated carbon from sugar palm fruit shell has the potency being used for removal of copper contaminants.

1. Introduction

Toxic heavy metals can be produced from various liquid wastes including industrial waste, agricultural waste, and from various other sources. As pollutants, heavy metals have become a world concern because their persistence and toxicity have a negative impact on living things. The existence of heavy metals in the environment beyond the threshold can disrupt the food chain, accumulate and can cause chronic disease.

The existence of heavy metals in liquid waste has become a challenge for the government and environmental monitoring agencies in reducing and remediating this contamination. Control of contamination of heavy metal levels in the environment has been regulated by the government in the permitted limit regulations because of their harmful effects. Because these heavy metals have different toxicities and cannot be decomposed naturally, their toxic effects can be permanent for a long time [1]. Therefore, the decreasing of dangerous metals such as Hg, Pb, Cr, Cd, Cu is very pivotal to maintain the stability and sustainability of life.

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Copper is one of the heavy metal that is produced from copper smelting, casting and power plants. The presence of copper above the threshold has an impact on toxicity such as discomfort stomach, melena, hipotension, liver and kidney disease [2]. Based on Ministry of Environment of Indonesia Goverment number 14 year 2014 about waste water quality standards reveal that the limit of copper levels in industrial wastewater was <0.005 mg/L[3] .

The adsorption technique has been convinced to be an excellent method for wastewater treatment because it offers benefits over conventional processes. Adsorption using agricultural waste have many advantages such as efficient, abundant, cheap, underutilized, simple and safe in removing heavy metals in wastewater. The adsorbent from agriculture waste is safe and has no adverse impact on life. Many researchers have been treated heavy metal wastewater by adsorption from agricultural waste like rice husk [4], grass waste [5], and teff straw agricultural wastes [6]. Reducing adsorbent dosage and minimizing disposal to get high adsorption capacity are still developed from the problems.

Isothermal adsorption models were utilized in wastewater treatment to estimate the ability of adsorbents in removing pollutants to a certain level. It describes the mechanism and equilibrium data through the interactions of the adsorbates on the surface. Calculation of the adsorption capacity at equilibrium is needed to predict adsorption efficiency and to understand the distinctive mechanisms from various adsorbents. The Langmuir and Freundlich isotherm are common equlibrium model in batch adsorption systems. Several researchers reported the adsorption of copper according to the Langmuir isotherm model from different adsorbent such as activated leaf based biomass [7] and activated teff straw [6]. While fumaria indica biomass [8] fits with isotherm Freundlich. In this research, sugar palm fruit shell was carbonated and activated physically with temperature of 600 ^{o}C without oxygen. So that, it needs to predict the mechanism of adsorben dan adsorbat occurred physically or chemically, the adsorbent contacts with heavy metal wastewater for a long time, an equilibrium between the absorbed and remained heavy metal in solution will be reached [9]. As long as the adsorbent is in interaction with the adsorbate , large amounts of heavy metals are expected to be retained by the active surface sites of the adsorbent.

The target of this study was to determine Freundlich and Langmuir isotherms used to calculate and estimate the adsorption capacity. Linearization method have been used for determination the Langmuir parameters similar as capacity adsorption(qm) and konstanta Langmuir(K_L) dan Freundlich parameters similar as capacity adsorption (n) and Freundlich constanta (K_F)

2. Materials and methods

2.1. Materials

Sugar palm fruit shell was obtained from Indrapuri, Aceh Besar District. 1000 ppm of copper solution was produced by dissolving copper sulphate (merck, Darmstadt, Jerman) in 1000 mL distilled water. To make variation of copper (II) concentrations were conducted by diluting certain volume of 1000 ppm copper sulphate with distalled water to obtain 10 – 300 ppm copper (II) aqueous solution.

2.2. Preparation of adsorbent

Sugar palm fruit shell was obtained from Samahani in Great Aceh , Indonesia. It was washed to remove the dust and the dirt. Then it was dried under a sunlight for 3 days. Finally, it was grained and sieved to be particle size of 60 mesh.

2.3. Carbonation and activation of the adsorbent

SPFS is put into the furnace (Suhatherm Furnaces) at 600 ^{o}C for 1.5 hours. Then, physical activation was carried out using a furnace with a heating temperature of 600 ^{o}C for 60 minutes to open the pores on the adsorbent surface. Then, it was explored by FTIR and SEM.

2.4. Batch adsorption experiment

The experiment were carry out using 1 g of SPFS activated carbon pour into 100 mL of various initial Cu (II) (merck, Darmstadt, Jerman) concentrations (30–300 mg/L). The admixture was shaken using shaker at 250 rpm for equilibrium contact time. Then, the mixture was filtered by filter paper (Sartorius). The filtrate was analyisis by Atomic Absorption Spectrometry (Shimadzu, IRPrestige-21). The Cu (II) concentration adsorbed per unit mass is calculated based on [6] as follow :

$$qe = \frac{(Co-Ct)}{m}.V \quad (1)$$

2.5. The adsorption Isotherms

The equations were illustrated by Langmuir and Freundlich isotherm. The Langmuir isotherm explicits the linear graph from plot of $\frac{Ce}{qe}$ and Ce (final concentration, mg/L). It can be shown from [6] by equation:

$$\frac{Ce}{qe} = \frac{1}{qm.Kl} + \frac{Ce}{qm} \quad (2)$$

This Freundlich isotherm indicates heterogeneous mechanisme of adsorption. It describes the relation between log qe versus log Ce to result a linear graph. It can be shown from below [4] :

$$\text{Log}\, q_e = \log \text{k}_\text{F} + \frac{1}{\text{n}} \log\ \text{C}_\text{e} \quad (3)$$

2.6. Fourier Transform Infrared (FTIR) analysis

The characterizing the functional groups of SPFS activated carbon SPFS was guided by FT-IR spectroscopy (Shimadzu, IRPrestige-21). The wavelengths were recorded in the area from 4000 to 500 cm^{-1}. The mixture of 0.2 mg adsorbent and 2 mg KBr was formed a pellet. Then, the pellet is analyzed by FTIR at 4000-500 cm^{-1}.

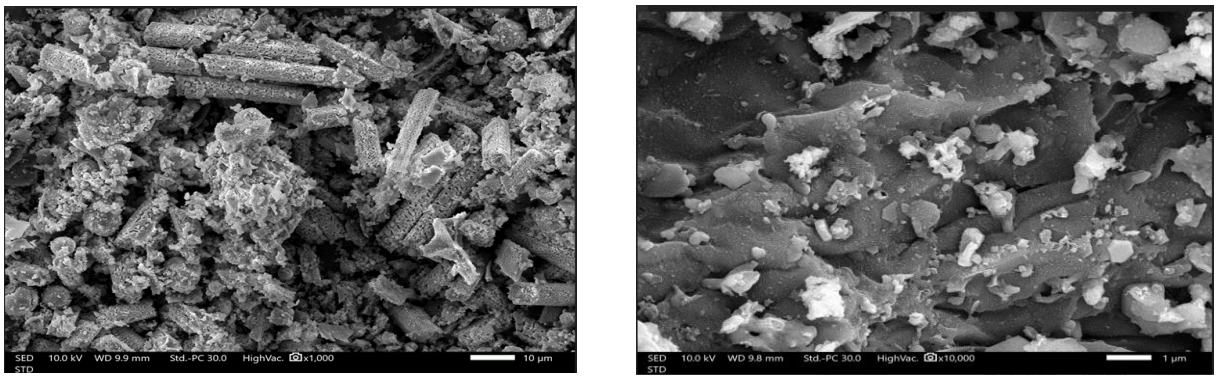
2.7. SEM-EDX analysis

SEM-EDX (JEOL, JSM-6510A) performed at 2 kV and point size of 40 to image the adsorbent. The adsorbent was freezed by the aluminium to dry. Then, sample was sprinkled by gold employing polaron during a half minutes. Then, stereoscan interprets the result.

3. Results and discussion

3*.1 SEM-EDX (Scanning Electron Microscope).*

The SEM-EDX characterizes the morphology and composition of surface activated carbon SPFS as adsorbent for adsorption process of Cu (II) in solution. The morphology of activated charcoal SPFS that has been physically characterized by SEM at 1000 x magnification is shown in Figure 1.



(a) (b)

Figure 1. Morphology of the surface activated carbon of SPFS by SEM (a) 1000 magnification; (b) 10.000 magnification

From Figure 1, it can be seen that the surface morphology of SPFS activated carbon is rod-shaped, uneven, irregular, and rough. be that the pores in the activated carbon are open. By opening the pores, it means that during the process, it can absorb the maximum Cu (II) in the solution. The more pores of the SPFS activated charcoal, the more optimal the absorption process will be during the adsorption process on Cu (II) ion [10]. It is similar to the result [11], study the various pore size of adsorbent which states the pore size significantly influenced the capacity of adsorbents. Cordia africana leaves activated carbon after activation also showed more the pore on the surface area the adsorbent that enhanced its performance [12]

The EDX analysis showed the activated carbon SPFS structure indicated the presence of C^{2+}, O^{2-}, Na^{+}, Cl^{+}, Ca^{2+}, Si^{2+}, P, Cl^{-}, K^{+}, Mg^{2+} peaks caused by the presence of nutrients in plants [5] with their respective percentages (% by weight) shown in Figure 2.

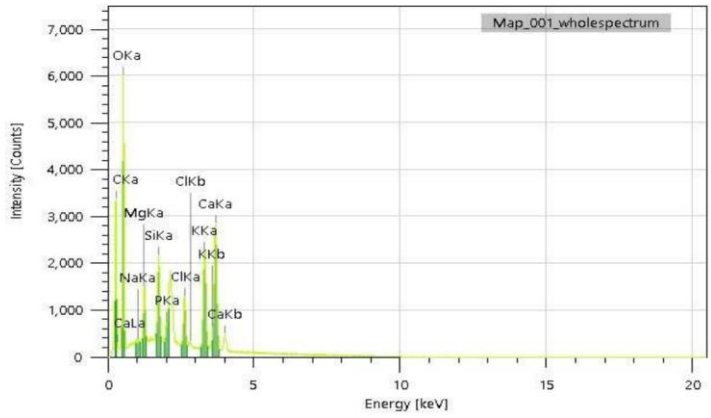


Figure 2. Composition of the surface activated carbon SPFS identified by EDX

3.2. Fourier transform infrared (FTIR) spectroscopy.

In this work, the types of functional groups on the surface of activated carbon from palm fruit shells, which were involved in Cu (II) adsorption, were determined using fourier transform infrared spectroscopy. It can be seen from Figure 3.

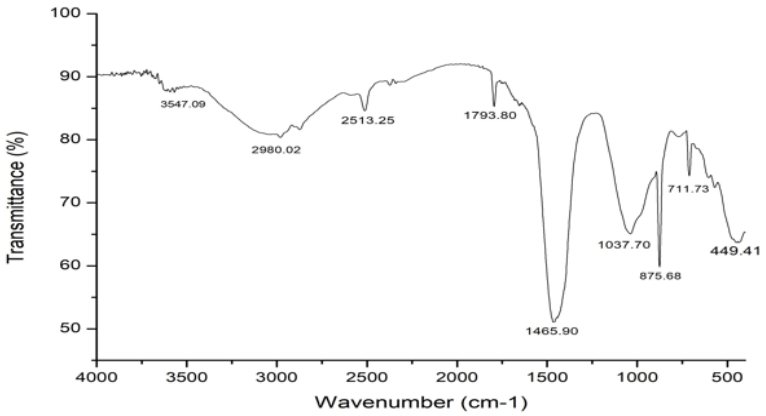


Figure 3. FTIR from activated carbon SPFS

As depicted in figure 3, the spectra of active carbon SPFS shown were peaks at 2980.02, 1465.90, 1037.70, 875.68, 711.73, 449.41 cm-1. The short and wide peak observed at a wavelength of 2873,94 cm^{-1} and 2980,02 cm^{-1} contributes to strecthing –C-H dari alkanes (-CH_3). The sharp and wide peak exposed at 1465,90 cm^{-1} indicates to C–C stretching vibrations [13]. In 1037,70 cm^{-1} wavenumber, C-O stretch vibration is found in this peak [14]. The short and narrow peak observed at 711,73 - 875,68 cm^{-1} accomodate the presence of polycyclic aromatic hydrocarbons and alkenes in aromatic rings [15].

3.3. The effect of contact time

The influences the occasion of the adsorbent contacts with Cu (II) ion in solution until the equilibrium of adsorption was implemented by contact time. It contributes to largest the adsorption capacity until the equilibrium time that inspected in Figure 4.

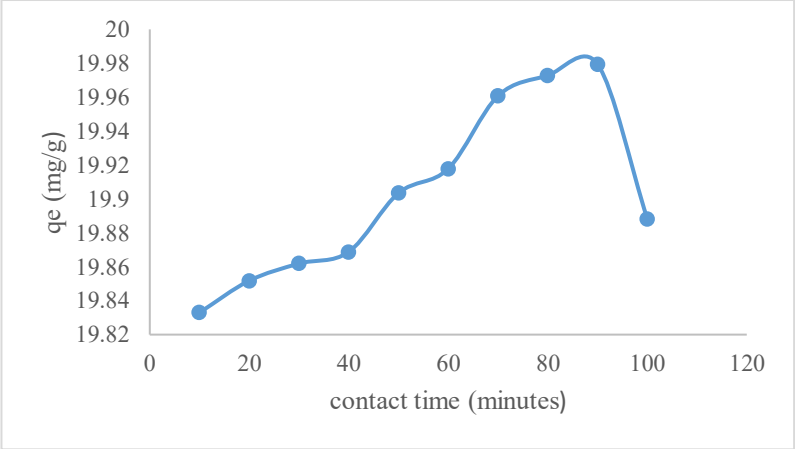


Figure 4. Effect of contact time on adsorption capacity of Cu (II) solution using activated carbon of SPFS (*Arenga pinnata*)

Based on the figure 4, the rate of adsorption increased rapidly from 10 to 90 min with the increasing from 19.883 to 19.979 mg/g. Then, it decreased until 100 min and increased along with the longer contact time prior to the equilibrium was attained in 90 min. At the time, The result is similar to what was reported [16] by using adsorbent nonwovens produced from lignocellulosic milkweed fibers. More active sites have not occupy the surface which provide greater opportunities for the adsorption process of Cu (II) ions with active sides of the SPFS activated carbon. In that time, active sites has adsorbed

most of the adsorbate in solution and reached the saturation [17]. The decreasing of adsorption capacity was occurred at 90 min to 100 min from 19.979 to be 19.883 mg/g because of desorption of the adsorbent. The desorption was also occurred in removal Cd (II) solution with activated carbon from banana peel after reaching the equilibrium time of 80 minutes [18].

3.4. Effect of initial Cu (II) concentration

In this study, the adsorption capacity of activated carbon SPFS was studied at the initial concentrations of 10 ppm until 300 ppm. The various amount of Cu (II) metal ion adsorbed in SPFS activated carbon was illustrate in Figure 5.

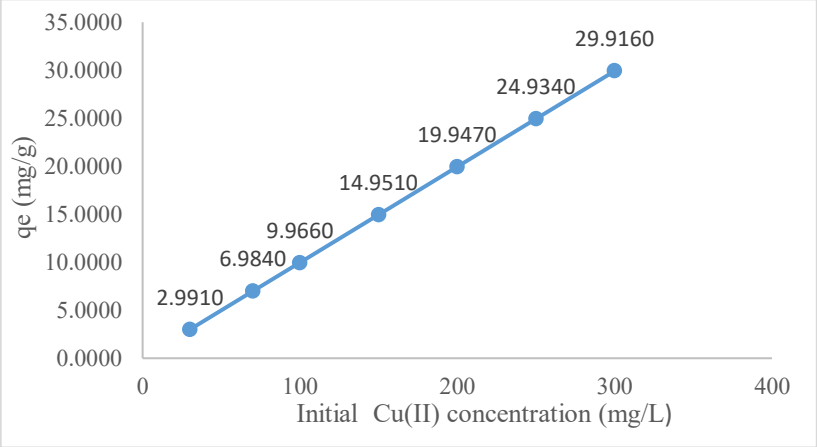


Figure 5. Effect of initial concentration of Cu(II) on adsorption capacity using activated carbon of SPFS (*Arenga pinnata*)

Figure 5 showed the initial Cu (II) concentration affected the performance of SPFS activated carbon. The adsorption capacity of SPFS activated carbon for Cu (II) ion concentration of 10 ppm to 300 ppm arise from 2,991 mg/g to 44,915 mg/g. The rise was influenced by concentration inclination of driving force toward mass transfer with increase together with initial concentration. At higher concentration, the more actives side was occupied until the saturation was reached [8]. At lower concentration, the ratio between active sites and adsorbat was higher.

3.5. Langmuir isotherm model

The Langmuir isotherm considers the metal ion absorbed on a homogeneous and monolayer surface with uniform active sites [5]. It reveals that the solute is distributed evenly on the adsorbent surface area by binding the functional sites to coverage all the adsorbent area [19]. No interaction occurred on the adsorbent [20] where all active sides of the adsorbent surface can only be proportionally filled by molecules, adsorption is reversible, and adsorbed molecules cannot move to other surfaces or interact with nearby molecules. The Langmuir model obtainned from this experiment was shown in Figure 6.

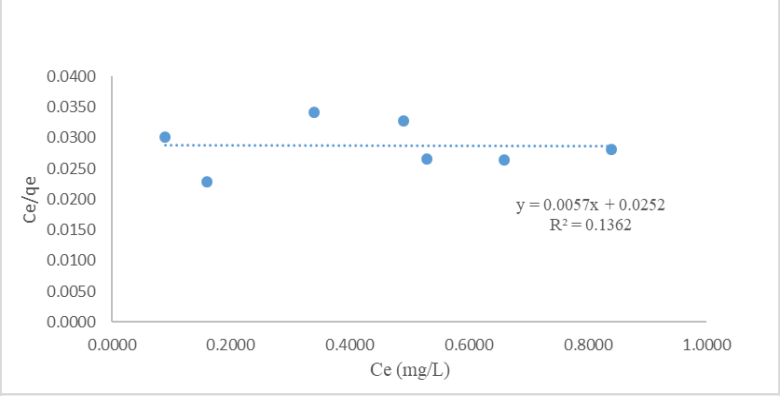


Figure 6. Langmuir adsorption isotherm for adsorption of Cu(II) ions using activated carbon SPFS (adsorbent mass 1 g; pH 4.5; initial metal concentration 30 mg/L - 300 mg/L).

Figure 6 showed that the linearization of the Langmuir isotherm, intercept and slope obtained through the Ce/qe versus Ce graph. The adsorption capacity, qm and Langmuir constant, K_L can be calculated from equation 1. The qm and K_L obtained at this study were 6.961 mg/g and 174.44 respectively. The obtained R^2 of the Langmuir isotherm was 0.1362. The value of R^2 obtained is still far from the value of 1, assuming that mechanism is not fit with the Langmuir isotherm.

3.6. Freundlich isotherm model

The Freundlich isotherm presume the adsorbent have heterogeneous surface. Adsorption was better when the concentration of Cu (II) solution with various affinities increase [5]. The Freundlich isotherm unindicated the saturation of adsorbent surface with the presence of Cu (II) ion. It is assumed that the active site was unlimited on the surface so that the multilayer formed when adsorption was happened. Every active side on the surface will be filled by many adsorbate molecules.

The Freundlich parameters from equation 3 include K_F capacity and 1/n parameters. If adsorption capacity is greater so that K_F value is the greater too and vice versa [21]. The n indicates the adsorption affinity, where the greater n value will be given the stronger interaction. If n value is greater than 1, adsorption was favorable [12]. The correlation coefficient (R^2) indicates the fitting data with the model isotherm.

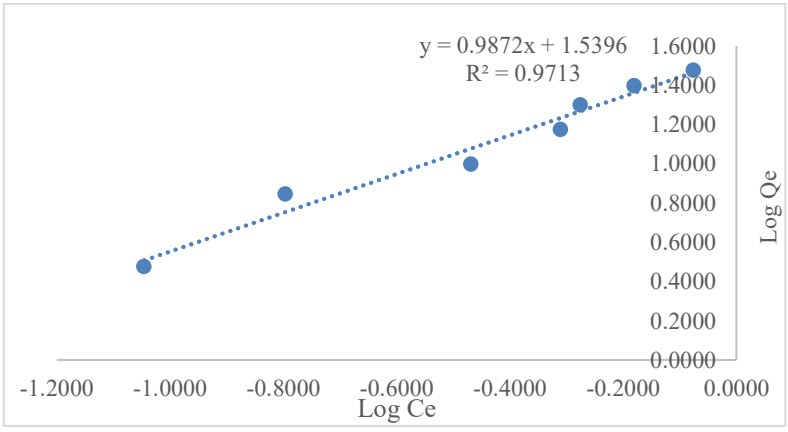


Figure 7. Freundlich adsorption isotherm for adsorption of Cu(II) ions using activated carbon SPFS (adsorbent mass 1 g; pH 4.5; initial concentration of Cu(II) metal 30-300 mg/L)

Based on Figure 7, the R^2 value was obtained at 0.9713 with a K_F of 4.66 mg/g, n = 1.02. The n was obtained in the value of 1-10, indicating a well adsorption occurs and the interaction between Cu (II) metal and the activated carbon SPFS is quite strong. The R^2 Freudlich equation is near to 1 than R^2 in the Langmuir equation indicating that was patterned multilayer surface. The results showed that the Freundlich isotherm fits to the empirical data which indicates that the process was approriate with heterogenous, multilayers and physisorption.

4. Conclusions

The equilibrium adsorptions of Cu (II) ion using SPFS activated carbon was figure out by Langmuir and Freundlich model isotherm. The Freundlich is the fittest isotherm model for Cu (II) adsorption using activated carbon SPFS with the correlation coefisient is 0.97. The n value obtained > 1 that represents the favorable adsorption occurred in this study. The adsorption capacity in this study obtained was 4.66 mg/g. To enhanced the performance of the adsorbent, modification with another material is still need to be studied.

Acknowledgements
The Ministry of Research, Technology, and Higher Education of the Republic of Indonesia provided funding for this study under Dissertation Research Grant Number 168/E5/PG.02.00.PL/2023.

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