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Characterization of Eggshell Combination with Activated Corn Cob as Ion Pb^{2+} Adsorbent through a Batch Process

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Abstract

This study aims to determine the characteristics of the combination of corn cobs and eggshells as Pb metal adsorbents. In this research, adsorption using the Batch method. This research uses an experimental approach in the laboratory. This experiment has four stages: adsorbent preparation, adsorbent activation, characterization of adsorbent functional groups using FTIR, and characterization of adsorbent to adsorb the ion Pb2+. The adsorption isotherm was determined using the Langmuir, Freundlich, Temkin and Dubinin-Radushkevich isotherm patterns. While the determination of adsorption effectivity through optimum conditions on variations in adsorbent composition, adsorbent mass, contact time, and the effect of initial concentration of Pb metal on adsorption capacity are carried out using AAS. The results showed that the adsorbent combination of corn cobs and eggshells contained OH, CaCO32- groups, cellulose and protein constituent groups that played a role in the adsorption process, using the Langmuir isotherm pattern, which means the adsorption process takes place chemically. The optimum composition ratio of the adsorbent corn cobs and eggshells is 1: 2, the optimum mass of adsorbent was 0.2 g, and the contact time optimum is 60 minutes, with the optimum percentage of Pb2+ adsorbed in those conditions are 94.49%, 98.69 %, 99.03%, respectively. The more the initial concentration of Pb metal, the greater the adsorption capacity, and in this study, the largest adsorption capacity at a concentration of 30 ppm with a value of 1.393445 mg/g.

Keywords: *Adsorption, Corn cob, Egg shell, Pb metal*

1. Introduction

Industrial waste is one of the potential sources of heavy metal pollution for the waters. It is known that heavy metals are dangerous elements on the earth's surface, so heavy metal contamination in the environment is a big problem today. Specific issues in the background, especially their accumulation in the food chain and their presence in nature, as well as the increasing amounts of heavy metals that cause poisoning to soil, air and water, is increasing [1]. Pb is one of the dangerous heavy metals. Metal lead (Pb) is found in the electroplating industry waste, dry battery industry, paint industry, and the cosmetic soda industry. Lead compounds (Pb) can have a toxic effect on many organs in the human body [2]. Therefore, the Decree of the Minister of Environment No. 51/MENLH/10/1995 concerning the quality standard of liquid waste for industrial activities for Pb 0.1 mg/L [3]. Many methods are used in treating water contaminated with heavy metals, namely precipitation, solvent extraction, reverse osmosis (RO), cation exchange, and chemical reduction-oxidation [4]. Coagulation, evaporation, and membrane separation [5]. However, the conventional method requires a very high cost, so that it is less effective in its use. Therefore, it is necessary to have a compelling alternative way to separate heavy metals, namely the adsorption method, because it has many advantages, including low cost, high adsorbate binding capacity and environmental friendliness [6]. The adsorption method is the absorption process by certain solids against certain substances that occur on the surface of the meaning [7]. This study uses a batch or static system. In this batch system, the adsorbent powder is put in a container containing a solution containing heavy metals to be adsorbed and then stirred so that the adsorption process can take place optimally. The Batch system is adequate for large-scale adsorption and better interaction between adsorbent and adsorbate. The adsorbent used in this study came from nature, namely corn cobs and eggshells, because they have an economical price and good adsorption capabilities [8]. Corn cobs are organic wastes that have not been widely used and contain 41% cellulose and 36% hemicellulose with OH- groups that can be used as adsorbents [9]. In addition to corn cobs, waste can be used as an adsorbent for heavy metals, namely eggshells. High eggshell waste can be utilized more optimally so that eggshells become household waste where the actual content of eggshells is calcium carbonate (CaCO3). In addition, eggs contain 10% eggshell, which is household waste. Chicken eggshells also have the potential to be adsorbent because the eggshell includes about 98% CaCO3 (calcium carbonate) and has 10,000 - 20,000 pores, so it is estimated that it can absorb a solute and can be used as an adsorbent [10]. This study combines adsorbents from corn cobs waste and chicken eggshells, where this combination is a combination of organic and inorganic adsorbents. Combination intake is expected to complement each other between the two adsorbents so that this adsorbent combination can strengthen the interaction with the adsorbate. This study aims to determine the functional groups of the combination of corncob and eggshell adsorbents, the optimal composition of the adsorbent, the optimum mass of corncob and eggshell adsorbents in adsorption of Pb metal, and the optimum time and the pattern of adsorption isotherms that occur in corncob and eggshell.

2. Experimental

2.1. Materials

The adsorbent used was corn cobs obtained from the shelled corn business in the Karanganyar area, and the eggshell adsorbent came from food stalls and household waste. Meanwhile, Pb metal ions used were simulated artificial solutions made from 1000 mg/L Pb mother liquor obtained from the Chemical Sub-Laboratory of UNS.

2.2. Adsorbent preparation

Corn cobs are washed, dried, ground, or blended to form a powder and then sieved through a 100-mesh sieve. Meanwhile, the eggshells were mashed with a mortar and pestle and then sieved through a 100-mesh sieve. Then the eggshell powder was heated in the oven for 1 hour at a temperature of 110^0C.

2.3 Adsorbent Activation

Eggshells were immersed in 0.1 M HCl solution for 24 hours. Then the adsorbent was filtered and rinsed with distilled water until a neutral pH (pH 7) was reached and then oven-dried for 180 minutes at 100^{o}C. Next, dry corn cobs were mixed with 150 mL of 0.1 M HCl solution for 3 hours at room temperature, then filtered with filter paper and washed with distilled water, after which it was dried in an oven overnight at 60^{0}C.

2.4 FTIR characterization analysis

Un-activated and activated eggshells and corn cobs were characterized by Fourier Transform Infrared Spectroscopy (FTIR) to determine the functional groups present in the adsorbent.

2.5 Preparation of sample solution

Prepared the sample solution by taking 10 ml of 1000 ppm Pb stock solution and entering it into a 100 ml volumetric flask. Dilute with distilled water to the mark.

2.6 Testing effectiveness of the adsorbents

Determination of the effectiveness of adsorption by using experiments on variations in adsorbent composition, variations in contact time, variations in adsorbent mass and the effect of initial concentration on adsorption capacity according to the steps below:

2.6.1 Variation of Adsorbent Composition

They determined the adsorbent composition by taking 10 ml of Pb sample solution with a concentration of 12 ppm and then inserting it into a vial and weighing 0.1 grams of eggshell and corncob adsorbent with a ratio of 1:1; 1:2; 2:1. The solution was then stirred at 100 rpm for 30 minutes using a shaker. Then filtered with Whatman paper no 42, and the filtrate is ready for analysis. Finally, analysis of metal ion determination was carried out using Atomic Absorption Spectrophotometer (AAS).

2.6.2 Adsorbent Mass Variation

Determination of the optimum adsorbent mass was carried out by taking 10 ml of Pb sample solution with a concentration of 12 ppm and then entering it in a vial. I weighed 0.10 grams of corncob and eggshell adsorbent with a ratio of optimum conditions with a mass of 0.05 grams, 0.15 grams, 0.20 grams, and 0.25 grams. The solution was stirred at 100 rpm for 30 minutes. Then filtered with Whatman paper no 42, and the filtrate is ready for analysis. Analysis of metal ion determination was carried out using Atomic Absorption Spectrophotometer (AAS)

2.6.3 Variation of Adsorption Time

Determination of the optimal time is done by taking 10 ml of the sample solution of Pb with a concentration of 12 ppm and then inserting it into a vial and then contacted with eggshell and corncob adsorbent with the ratio of optimum adsorbent composition and optimum adsorbent mass. Then the solution was stirred at 100 rpm for 10 minutes, 20 minutes, 30 minutes, 40 minutes and 50 minutes using a shaker. Then filtered with Whatman paper no 42, and the filtrate is ready for analysis. Finally, analysis of metal ion determination was carried out using Atomic Absorption Spectrophotometer (AAS).

2.6.4 Variations in the effect of initial concentration on adsorption capacity

Determination of the effect of concentration was carried out by taking 10 ml of Pb sample solution with concentrations of 5, 15, 20, 25 and 30 ppm and then put it in a vial and then contacted with eggshell and corncob adsorbent with the ratio of optimum adsorbent composition and optimum adsorbent mass. Then the solution was stirred at 100 rpm with the best time using a shaker. Then filtered with Whatman paper no 42, and the filtrate was ready for analysis. Finally, analysis of metal ion determination was carried out using Atomic Absorption Spectrophotometer (AAS).

3. Results and discussion

In the preparation process until activation, there are differences in colour and texture of the adsorbent. After the initial preparation process, the corn cobs are light brown in the form of powder. After completing the corn cobs in a fine powder and light brown (beige), it was activated and dried in the oven. In this study, acid activation was carried out. Acid activation can function to delignify the lignin content in corn cobs. Lignin covers the surface of the adsorbent, which can interfere with the adsorption process, so it needs to be removed because it will inhibit the binding process of the OH group with cations on heavy metal Pb. The delignification process is carried out with solid acid HCl. Cl- in hydrochloric acid content will disrupt the stability of the bond between cellulose, lignin, and hemicellulose. This bond will be broken and cause lignin decay. Lignin in tissue functions as an adhesive between cells, and when lignin decays or is lost, the bonds between cells will become loose [12]. At the same time, the eggshell before being prepared is brown in rough shape. After mashing, the eggshells are in the form of refined grains, creamy white. Then, after completing the activation process with a solution of HCl, the eggshell is creamy white, clean, and in the form of excellent granules. After that, it is activated by using HCl acid to enlarge the pores of the eggshell to increase the ability to absorb. Acid activation with HCl causes deionization, which causes an increase in the surface area of eggshell waste due to reduced impurities that cover the pores of the adsorbent [13]. Activation is carried out with HCl because it has a good adsorption capacity. After all, HCl can dissolve impurities to form more pores, and the adsorbate adsorption process is maximized [11].

3.1 FTIR Functional Group Analysis

3.1.1 FTIR characteristics of eggshell waste before and after activation

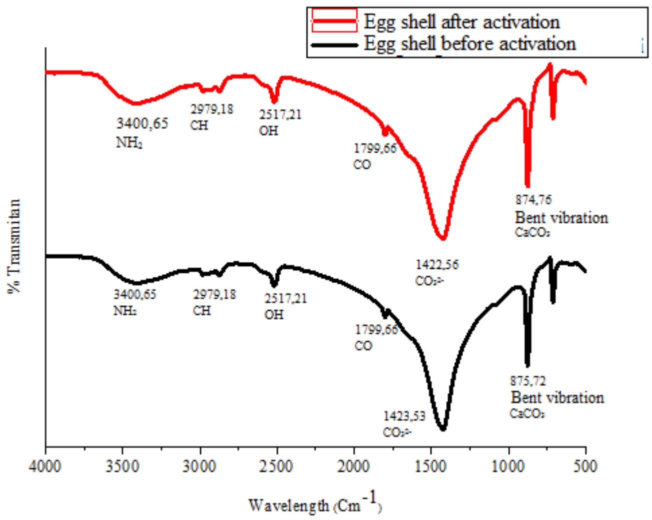

Figure 1 : FTIR Results Eggshell waste before and after activation

Based on **Figure 1**, we can see that the FTIR results show that the various functional groups contained in the eggshell before and after activation are Amino groups, CO_3^{2-}, -OH stretching, -CH, bent vibrations of calcium carbonate and -CO carbonyl. However, there is a shift in the wavenumber of the groups between before and after activation. First, the

amine functional group (stretch –NH) is shown at a wavenumber of 3400.65 cm-1, indicating the presence of amines in the eggshell [23]. This is following the fact that the eggshell contains proteins that have amine groups. This shows that the eggshell is also composed of organic substances, namely the eggshell membrane [23]. Then at wave number 2979 cm-1, it shows the stretching vibration of the alkyl -CH functional group. Next, the –OH stretching group is offered at a wavelength of 2517.21 cm-1. Then appeared the peak of CO_3^{2-} carbonate mineral, which decreased in wavenumber from 1423.53 cm-1 to 1422.56 cm-1. This indicates an increase in the mass or quantity of the CO_3^{2-} functional group. The appearance of the absorption peak of CO_3^{2-} suggests calcium carbonate $CaCO_3$ in the eggshell of broiler chickens. The CO_3^{2-} ridges reinforce the active group at wavenumbers 875.72 cm-1 and 710.8 cm-1, which indicate the bent vibration of calcium carbonate [14].

3.1.2 Characteristics of corncob waste FTIR before and after activation

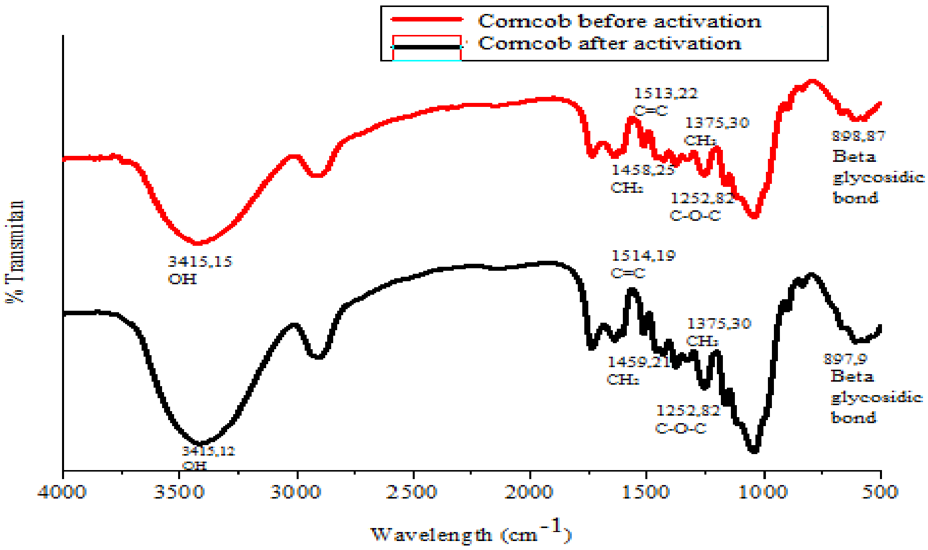

Figure 2: Results of FTIR Corncob waste before and after activation

Based on **Figure 2**, the various functional groups contained in corn cobs before and before activation can be seen. In the wavenumber area of 3415.15 cm^{-1}, a broad absorption shows the hydroxyl (OH) groups. The absorption at wave number 1458.25 cm-1 is the CH2 bending vibration functional group. The –OH group will later bind to the heavy metal Pb^{2+} [21]. The absorption at wave number 1458.25 cm-1 is the CH2 bending vibration functional group. Then the identification is the presence of the CH3 bending vibrational active group in the wavenumber region of 1375.30 cm^{-1}. At the peak of 1513.22 cm^{-1}, it indicated the presence of lignin in the corn cobs. So that the activation with strong acid still gives lignin to the adsorbent. These functional groups are found in cellulose and hemicellulose, later playing a role in the heavy metal adsorption process. The wavenumber shows the vibrational absorption of a functional group, which is inversely related to the mass or quantity of the atom that vibrates. Therefore, when the wave number decreases, this indicates that the vibration absorption of the group is reduced. That is, the mass or quantity of the group is increasing [15].

3.2 Testing the effectiveness of corncob and eggshell adsorbents

3.2.1 Variation of optimum adsorbent composition

Based on **Figure 3**, it can see that the ratio of the adsorbent composition of corn cobs and eggshells is 1:2, which is the most optimum composition ratio. The percentage of Pb adsorbed at an arrangement of 1:2 is 94.49%. In determining the design of the adsorbent used the same amount, namely 0.1 grams. At the optimum ratio of 1: 2, the plan for the eggshell adsorbent is more than the corncob adsorbent. Eggshells may contain calcium carbonate, which interacts strongly with divalent metal ions (M^{2+}). Besides that, eggshells also contain protein (amino acids) as an active compound in the adsorption process, which is more substantial with Pb metal than the –OH group in cellulose corncob. So the 1:2 composition is more effective and optimum in adsorption of heavy metal Pb. At the 0:1 composition, the eggshell adsorbent without corn cobs can absorb the adsorbent well because it has 10,000-20,000 pores that can absorb Pb well, which is 93.76%. At a composition of 1:0 corncob, the eggshell only absorbs a little Pb metal because activation using 0.1 M HCl has not been able to open the pores of the adsorbent and the adsorbent surface still covered by impurities so that the corncob adsorbent only absorbs a little, namely 0.53%. So, in this combination of adsorbents, eggshells have better adsorption ability than a corncob. The composition of 2:1 can absorb 23.80% lead metal with more corn cobs. More corn cobs will cover the surface pores of the eggshell adsorbent. Eggshell or corncob adsorbent will enter the pores on the eggshell so that the adsorption ability of Pb metal will decrease. This was also because at a 2:1 composition, the eggshell adsorbent was more petite, while those with good adsorption ability were eggshells. The composition of the 1:1 adsorbent of corncob and eggshell had the same amount of metal, resulting in higher adsorbed than 2:1 because the amount of adsorbent on corncob that covered the pores of the eggshell was less so that the ability of the adsorbent to perform better adsorption is 83.14%

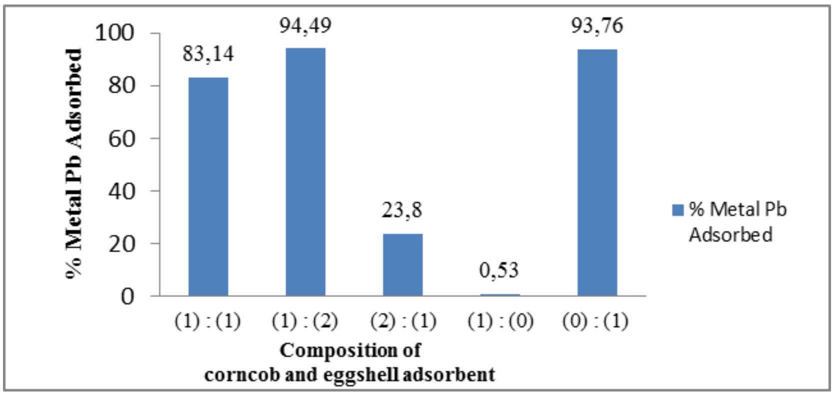

Figure 3: Determination of the optimum composition with an initial concentration of 12 ppm; the mass of the adsorbent is 0.1 grams, and the contact time is 60 minutes

3.2.2 Optimum Adsorbent Mass Variation

The increase in the weight of the adsorbent will be directly proportional to the rise in the number of particles and the surface area of the adsorbent so that the number of sides to bind metal ions increases, thereby increasing the absorption efficiency [17]. This can also be seen in the mass of the adsorbent as much as 0.05 grams to 0.2 grams. As a result, the percentage of metal adsorbed has increased. The graph (**Figure 4**) shows that the adsorbent mass of 0.2 grams is the best because it can adsorb Pb metal as much as 98.69% Pb metal is absorbed. This is because there has been a balance between the remaining Pb metal in the sample and the Pb metal absorbed by the eggshell-corncob adsorbents, and to absorb the corncob-eggshell adsorbents have maximally bound Pb metal [18]. From the graph, we can see that the adsorption percentage decreased for a mass of 0.25 grams. This is because there are overlapping events during the adsorption process caused by the density of the adsorbent particles. This density causes the surface area of the adsorbent to be smaller so that the active site of the adsorbent is reduced. In addition, at the time of

saturation, namely at mass 0.2, there will be a desorption process that is the opposite of the adsorption process, namely releasing components or impurities that have bound to the active site on the surface of the adsorbent. So that at 0.25 grams decreased.

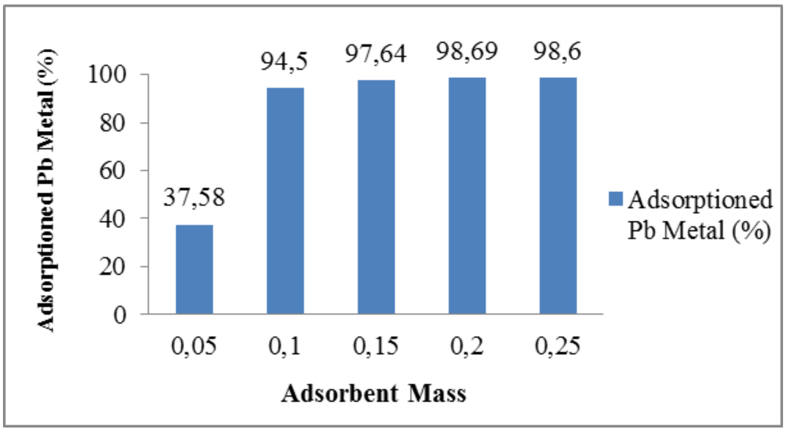

Figure 4: Determination of the optimum mass with an initial concentration of 12 ppm; optimum adsorbent composition 1:2; and a contact time of 60 minutes

3.2.3 Optimum Time Variation

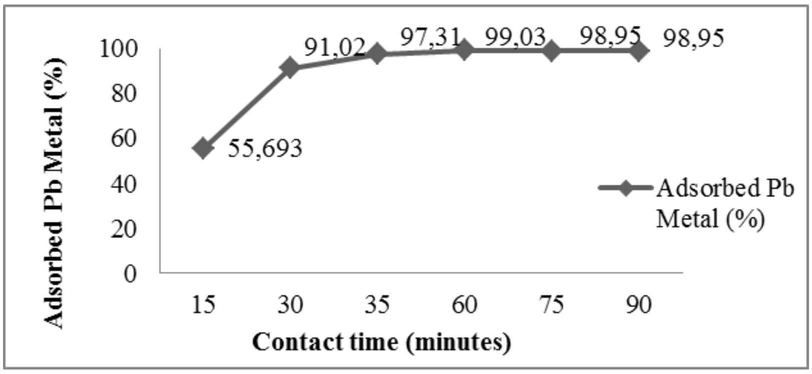

Figure 5: Determination of the optimum time with an initial concentration of 12 ppm; optimum adsorbent composition 1:2; and optimum adsorbent mass 0.2 gram

Based on the graph above, it can seem that the longer the contact time between the adsorbent and the adsorbate, the higher the absorption percentage. We can see from a contact time of 10 minutes to a contact time of 60 minutes. The percentage of metal adsorbed by the adsorbent continues to increase. The graph (**Figure 5**) shows that the contact time of 60 minutes is the best because it can adsorb Pb metal of 12.2707 mg/L with an initial concentration of 12.3897 mg/L, which means Pb metal is reduced very much, which is 99.03% metal. Pb is adsorbed, which indicates that the adsorbent is almost saturated. After the best adsorption is achieved, the addition of contact time will cause the absorption of the adsorbent because of the mechanical factor, namely stirring the adsorbent using a stirrer. So that the adsorbent is unable to bind Pb metal, releasing the metal from the adsorbent [18]. As at the time of 75 minutes and 90 minutes, the decreased adsorbent can absorb 98.95%.

3.2.4 Effect of Initial Concentration of Pb Metal

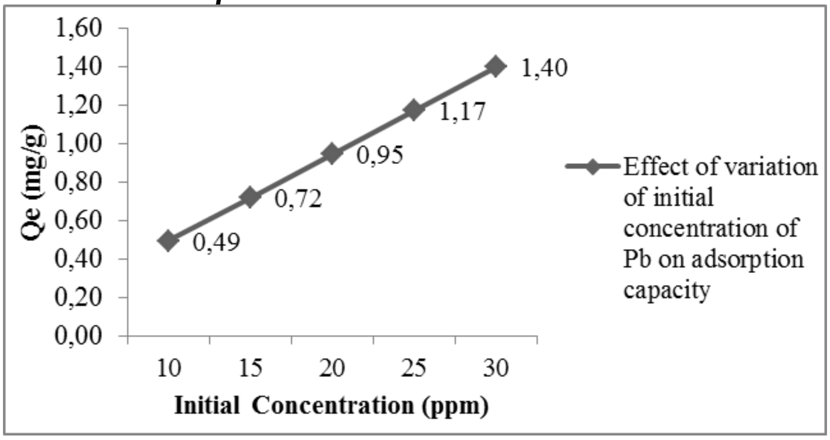

Figure 6: Determination of the effect of initial concentration variations with the optimum adsorbent composition 1:2; the optimum adsorbent mass is 0.2 grams, and the optimal contact time is 90 minutes

The largest adsorption capacity of the combined corn cobs-eggshell adsorbent at the initial concentration of heavy metal Pb of 30 ppm was 1.340 mg/g. The Pb concentration of 30 ppm is not yet optimum because a larger adsorption capacity is still possible (**Fifure 6**). After all, the adsorbent is not yet saturated. The ability of the adsorbent increases because the number of particles increases with increasing concentration. The adsorbate that is adsorping is proportional to the active group on the adsorbent. When the adsorbent is saturated, the increase in adsorbate concentration causes a decrease in adsorption percentage. However, if the active group is not saturated, the increase in concentration will increase adsorbate adsorbed. The higher the adsorbate concentration, the higher the interaction between corn cobs and eggshells with Pb metal. Then the amount of Pb metal bound to the solid surface of the adsorbent also increases [19].

3.3 Determination of Adsorption Isotherm Pattern

This study determines the pattern of adsorption isotherms. The purpose of deciding this isotherm is to determine the adsorption mechanism that occurs in the adsorbent. Determining the pattern of adsorption isotherm using the parameter can be seen in **Table 1, Table 2, Table 3,** and **Figure 7**.

Table 1: Parameters for plotting Langmuir, Freundlich, Temkin and Dubinin-Radushkevich Adsorption Pb(II) ion isotherm on corncob and eggshell adsorbent

Time (minute)	Co (mg/L)	Ce (mg/L)	Log Ce	Ln Ce	Qe (mg/g)	1/Qe	Log qe	Ln qe	Ce/qe	ε^2
15	12.389	5.490	0.740	1.703	1.725	0.580	0.237	0.545	3.182	171908
30	12.389	1.113	0.046	0.107	2.819	0.355	0.450	1.036	0.395	2523532
45	12.389	0.342	-0.466	-1.072	3.014	0.332	0.479	1.103	0.114	1.1×10^7
60	12.389	0.119	-0.924	-2.129	3.068	0.326	0.487	1.121	0.039	3.1×10^7
75	12.389	0.130	-0.886	-2.039	3.065	0.326	0.486	1.120	0.042	2.9×10^7
90	12.389	0.130	-0.886	-2.039	3.065	0.326	0.486	1.120	0.042	2.9×10^7

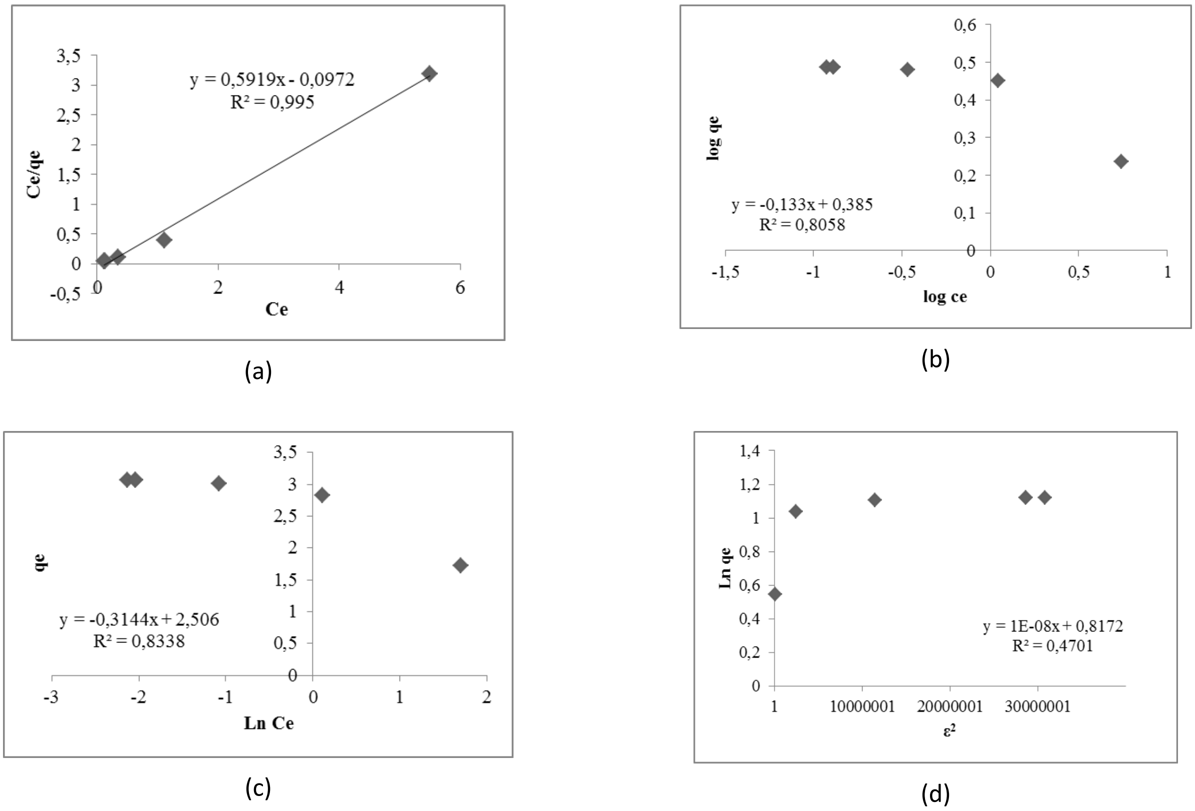

Figure 7: isotherm graph (a) Langmuir (b) Freundlich (c) Temkin (d) Dubinin-Radushkevich.

Table 2: Summary of Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich Adsorption Isotherms

No	Model	Equation	R2
1	Langmuir	$\frac{Ce}{qe} = \frac{1}{KL\ qm} + \frac{1}{qm} Ce$	0.995
2	Freundlich	Log qe = log Kf + $\frac{1}{n} \log Ce$	0.8058
3	Temkin	qc= $\frac{RT}{b} \ln A + \frac{RT}{b} \ln Ce$	0.8338
4	Dubinin-Radushkevich	Ln (qe) = Ln (qs) –(Kad ε2)	0.4701

Table 3: Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich Isotherm Comparison

Langmuir isotherm				**Freundlich isotherm**			
R^2	qm (mg/g)	K_L (L/mg)	R_L	R^2	n	K_F (L/mg)	1/n
0.995	1.6895	6.0895	0.0135	0.8058	7.5188	2.4266	0.133
Temkin isotherm				**Dubinin–Radushkevich. Isotherm**			
R^2	A(L/mg)	B	B	R^2	Qs (mg/g)	K_{ad} (mol^2/kJ^2)	E (KJ/mol)
0.8338	2894.99	7880.32	0.3144	0.4701	2.2642	$1x10^{-8}$	7.0711

This study determined the Physico-chemical parameters from four adsorption isotherm models by plotting the absorption data into the Langmuir, Freundlich, Temkin and Dubunin Radushkevich isotherms. The Langmuir adsorption model in this study had the highest regression value of 0.995, close to 1, and the Langmuir isotherm is the best and suitable. Can conclude that corn cobs and eggshells are potential biosorbents and have successfully removed

Pb^{2+} ions from the solution. These results indicate that the Langmuir isotherm model is better than the Freundlich, Temkin and Dubunin – Radushkevich model. The Langmuir isotherm model of metal adsorption of Pb^{2+} on corncob and eggshell adsorbents shows the formation of a single layer. The adsorbent is homogeneous and has the same adsorption energy for each Pb^{2+} ion adsorbed. The RL value indicates that the Langmuir isotherm is favourable for the adsorption of Pb^{2+} on corncob and eggshell adsorbents [16].

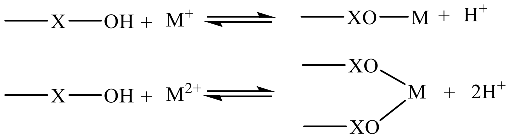

Figure 8: Alleged Ion Exchange Mechanism in Corncob Adsorbent and Pb(II) Metal Ions

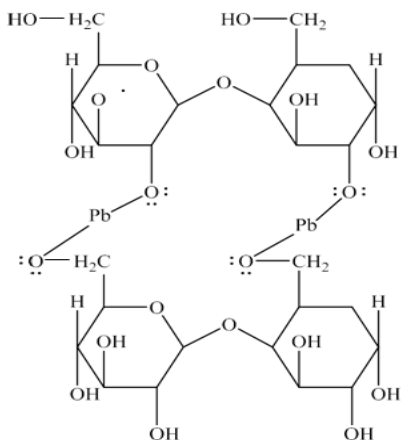

Figure 9: Formation of complex compounds between cellulose and Pb (II).

M^+ and M^{2+} are metal ions, -OH is the hydroxyl group, and X is the matrix where the –OH group is bonded (**Figure 8**). The interaction between the –OH group and metal cations are also possible by forming a coordination complex because the hydroxyl group's oxygen atom (O) has a lone pair of electrons (**Figure 9**). In contrast, metal ions have an empty d orbital. Where the lone pair of electrons will occupy the vacant orbital so that a compound or complexion will be formed [21]. In the eggshell adsorbent, the alleged mechanism of ion exchange on the adsorbent and Pb (II) metal ions can be shown in **Figure 10**.

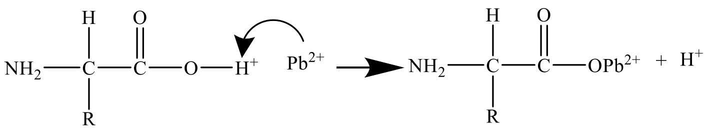

Figure 10: Alleged Ion Exchange Mechanism in Eggshell Adsorbent and Pb(II) Metal Ions.

Ion exchange occurs when the carboxylic groups (-COOH) in amino acids are deprotonated due to the presence of hydroxide ions (OH) so that the carboxylate groups become negatively charged (COO-), which is very reactive to bind to Pb^{2+} [22].

4. Conclusion

Based on the research that has been done, it can be concluded that combination corn cobs-eggshells can be used as adsorbents for Pb^{2+} ion. In the combination of corncob adsorbent and chicken eggshell, the functional groups are OH and $CaCO_3$. The process of adsorption of Pb metal by corncob-eggshell adsorbents is more dominant using the Langmuir isotherm pattern, which means that the adsorption process takes place chemically. The ratio of the optimum composition of corncob and eggshell adsorbent is 1:2, with a metal-adsorption percentage of 94.49%. The optimum

mass of the adsorbent is 0.2 grams, with an adsorption percentage of 98.68 %. The optimum time for contacting the adsorbent is 60 minutes, with adsorption percentage of 99.03%.

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