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Egg Shells as an Adsorbent for the Adsorption of Lead (Pb) and Iron (Fe) Metals

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A B S T R A C T

An eggshell is estimated to have around 10,000-20,000 pores; this condition created a chance for the eggshells to be used as an adsorbent. This research aims to activate egg shells used as an adsorbent. Egg shells were applied as an adsorbent in solutions of lead (Pb) and iron (Fe). Test parameters for adsorption capacity were carried out on the optimum mass and absorption contact time with lead (Pb) and iron (Fe) while using the eggshell waste as an adsorbent. The adsorbent quality from egg shells was tested using parameters such as ash content, water content and adsorption capacity towards methyl blue. This research aims to utilize egg shells as an adsorbent to absorb lead (Pb) and iron (Fe) content using an adsorption process. There are 3 stages of a method for this research to establish: adsorbent preparation, adsorbent activation, and adsorption process. The adsorption process was carried out with variations of the mass sample, which are 0.75 grams, 1 gram, 1.25 grams, 1.50 grams, and 2 grams and time variations when contact occurred in 20, 30, 40, 50, and 60 minutes. The outcome from this research showed that the highest adsorption capacity at the optimum mass of Pb metal was 1.5 grams at 98.914% and for Fe metal at 96.386%. The highest adsorption capacity results were influenced by Pb metal contact time in 40 minutes, which was 99.30%, and the best capacity for adsorption of Fe metal was at a contact time in 50 minutes, which was 99.82%.

1. INTRODUCTION

1.1. Research Background

Egg shells are a type of waste from households in large quantities. Even though they are considered waste, egg shells have beneficial properties when used as a waste processing material. Almost all of the eggshells of purebred chicken contain calcium carbonate. According to Ref. [1], calcium carbonate interacts strongly with several divalent metal ions (M^{2+}); the removal of metal ions in solution can be done by adsorption. The absorption process usually occurs simultaneously with dissolution on the calcium carbonate surface. This is in addition to the statement above that egg shells contain a certain active chemical compound widely known as protein (amino acids). Therefore, eggshells, a type of waste, can be used as adsorbents and support the implementation of waste minimization because they can increase the efficiency and productivity of eggshell waste using the principles of reuse and recovery [2].

The results reported by Ref. [3] stated that the effectiveness of eggshells in adsorbing heavy metals (Fe) was 99.82% at a mixing time of 60 minutes with a size of 1000 mesh. Meanwhile, the results reported by Ref. [2] show that egg shells can reduce the Pb content in liquid waste from the electroplating industry by up to 98.90%.

1.2. Literature Review

1.2.1. Egg Shell

Eggshell is the egg's outermost part, which protects the egg's components and contents from any harmful potential damage. Based on the mineral composition present, egg shells are composed of CaCO3 crystals (98.41%), MgCO3 (0.84%) and Ca3(PO4)2 (0.75%) [4]. According to [3], egg shells are household waste that has not been utilized optimally. Currently, egg shells are only used as raw material for the handicraft industry. Each eggshell has 10,000-20,000 pores properties, such as pore structure, $CaCO_3$ and mucopolysaccharide acid proteins, which can be developed into adsorbents. The most important functional groups of mucopolysaccharide acid proteins are carboxyl, amine and sulfate, which can bind heavy metal ions to form ionic bonds. In addition, eggshells are neutralization agents where all types of solutions easily experience equilibrium so that heavy metals can precipitate and be deposited in eggshell particles [11].

1.2.2. Adsorption Type

Based on the superiority of interaction, adsorption can be divided into 2 different categories: physical and chemical.

a. **Physical adsorption occurs** when the intermolecular forces are greater than the intermolecular attractive forces or the relatively weak attractive forces between the adsorbate and the adsorbent surface. This force is called the Van der Waals force so that the adsorbate can move from one surface to another surface of the adsorbent. Intermolecular forces are the attractive forces between fluid molecules and a solid surface, while intermolecular forces are the attractive forces between fluid molecules [13].

b. **Chemical adsorption** occurs due to the exchange or sharing of electrons between the adsorbate molecules and the adsorbent surface, resulting in a chemical reaction. The bond formed between the adsorbate and the adsorbent is a chemical bond, and this bond is stronger than physical adsorption.

1.2.3. Factors Affecting the Adsorption Process

In the adsorption process, there are a lot of things which can affect the rate of the adsorption process and the amount of adsorbate that can be absorbed, such as:

Contact Time

Contact time affects the amount of adsorbate that is absorbed due to differences in the ability of adsorbents to absorb different adsorbate. The equilibrium condition will be reached in no more than 150 minutes; after that time, the amount of adsorbate absorbed does not change significantly with time.

Adsorbent Mass

The ability of the adsorbent is directly proportional towards the increasing mass of the adsorbent. This happens because by increasing the amount of adsorbent, there is an increase in the active sites on the surface of the adsorbent [14]. However, under certain conditions, the absorption percentage tends to decrease because saturation of the adsorbent has occurred.

Stirring

Stirring affects adsorption because stirring aims to ensure that the adsorbent and adsorbate can make optimal contact rather than not being stirred.

Temperature

When the adsorbate molecules adhere at the surface of the adsorbent, a certain amount of energy is released so that the adsorption is classified as exothermic. If the temperature is low, the adsorption capacity increases, increasing the adsorbate.

1.2.4. Adsorption Method

There are 2 methods of adsorption: static (batch) and dynamic (column) [15].

The static method (batch) puts the solution with the desired components into a container containing the adsorbent and then stirs for a certain time. Then, it is separated by filtering or decantation. Components bound to the adsorbent are released again by dissolving the adsorbent in a certain solvent, and the volume is smaller than the volume of the initial solution.

The dynamic method (column), namely inserting the solution with the desired components into a container containing the adsorbent, then the components that have been absorbed are released again by flowing the appropriate solvent (effluent) with a smaller volume [15].

1.3. Research Objective

This research aims to determine the optimum mass, contact time and adsorption capacity of chicken egg shells under optimum conditions.

2. MATERIALS AND METHODS *(Heading 1)*

2.1. Preparation of sample

The materials used in this research were chicken egg shells, solid lead nitrate ($Pb(NO_3)_2$) and solid Iron Sulfate ($FeSO_4$), distilled water and H_2SO_4 0.05 M.

2.2. Equipment

The tools used in this research were Measuring Cups, Hotplates, Magnetic Stirrers, Glass Beakers, Glass Spatulas, Mortar, Blenders, 200 mesh sieves, Atomic Absorption Spectrometers (AAS), Volumetric Pipettes, Filter Paper, Analytical Balances, Aluminum Foil Paper, Ovens, and UV-Vis Spectrophotometers.

2.3. Egg Shell Adsorbent Preparation Method and Activation Using H_2SO_4

Chicken egg shells are washed with water. Then, soaked in hot water for 30 minutes, rinsed, and dried in the sun, theegg shells are ground using a blender. Next, sift it with a 200 mesh sieve, then heat the eggshell powder in the oven for 1 hour at 120 C; after that, it is cooled. Then, put it in a tightly closed, airtight box. Next, the adsorbent is soaked in the H_2SO_4 0.05 M solution for 2 hours, drained, filtered, and washed with distilled water until it has a pH of 7. After the pH is neutral,the adsorbent then placed in the oven for 1 hour at 110 °C.

2.4. *Adsorbent Optimum Mass Towards Adsorption of Certain Metal Solution* **($Pb(NO_3)_2$)**

A solution of ($Pb(NO_3)_2$) 50 ppm was prepared with a volume of 50 mL each for 5 samples and placed in an Erlemeer flask. Then, the adsorbent mass was added to each sample with a mass of 0.75 g, 1 g, 1.25 g, 1.5 g, and 2 adsorbents. The adsorption process was carried out by stirring at a speed of 130 rpm for 60 minutes. Then the solution was separated using filter paper. The solution obtained was then analyzed by Atomic Absorption Spectrometer (AAS).

2.5. *Adsorbent Optimum Mass Towards Adsorption of Certain Metal Solution* **($FeSO_4$)**

Prepare a solution ($FeSO_4$) 50 ppm with a volume of 50 mL each for 5 samples. Add adsorbent mass to each sample with 0.75 g, 1 g, 1.25g, 1.5 g, and 2 g adsorbent. Adsorbents of 0.75g, 1 g, 1.25g, 1.5g, and 2 g were interacted with 50 mL of Fe solution each and then put into an Erlenmeyer flask. Stir at 130 rpm for 60 minutes.

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Separated it using filter paper. The solution obtained was then analyzed by Atomic Absorption Spectrometer(AAS).

2.6. *Effect of Contact Time Towards Adsorption of Certain Metal Solution* ($FeSO_4$)

Prepare a solution ($FeSO_4$) of 50 ppm with a volume of 50 mL each. then the best adsorbent mass obtained in the previous procedure was added, then the contact time was varied starting from 20, 30, 40, 50, and 60 minutes. Stir at a speed of 130 rpm. Thenthe solution was separated using filter paper. The solution obtained was then analyzed by the Atomic Absorption Spectrometer (AAS).

2.7. *Characterization of Adsorbent Quality from Egg Shell*

2.7.1. Water Content(SNI,1995)

A total of 1 gram of adsorbent was weighed in a porcelain cup of known weight. The porcelain cup and adsorbent were placed in the oven at 110°C for 3 hours. Then cooled in adesiccator and weighed. The formula obtains water content:

$$\frac{(\text{Initial Adsorbent Mass}) - (\text{Final Adsorbent Mass})g}{(\text{Initial Adsorbent MassI})g} \times 100\%$$

2.7.2. Determination Ash Content (SNI,1995)

A 1 gram of adsorbent was weighed in aporcelain cup of known weight. The porcelain cup with the adsorbent is heated in a furnace at 600 °C for 4 hours. After the ashing is complete, the furnace lid is opened for 1 minute to complete the ashing process. Next, put it in a desiccator and thenweigh it. Ash content is calculated using the formula.

2.7.3. Adsorption Towards Methyl Blue (SNI,1995)

0.0159 grams of activated carbon were put into the Erlenmeyer. Then, 50 mL of 100 mg/L methyl blue solution was added. The mixture was stirred for 60 minutes using a magnetic stirrer, after which it was filtered. The solution was analyzed for its absorption capacity at a maximum wavelength between 630- 680 nm using a UV-Vis Spectrophotometer. A calibration curve or standard methyl blue solution is made with a concentration between 0 and 6mg/L.

2.7.4. Adsorption Percentage Towards Adsorbent Concentration

Based on [16], the adsorption percentage can be calculated using the formula:

$$\% \text{ Adsorption} = \frac{(Yi - Yf)}{Yi} \times 100\%$$

where: Yi = Initial concentration of solution (mg/L)
Yf = Final concentration of solution(mg/L)

3. RESULT AND DISCUSSION

3.1. Adsorbent Quality with SNI Standard

3.1.1. Water Content

In the tests that have been carried out, the results obtained for the physicochemical-activated eggshell adsorbent obtained a water content value of 0.9612%. The smaller the water content in the adsorbent, the better the quality of the adsorbent, and vice versa. This is because the higher the water content, the more closed the pores on the adsorbent are by the water.

3.1.2. Ash Content

Ash content affects the quality of the adsorbent. The presence of excessive ash can cause blockage of the pores in the adsorbent, causing the surface area of the adsorbent to decrease. In testing the ash content, the results obtained for the physicochemical activation adsorbent were 60.068%.

3.1.3. Adsorption Towards Methyl Blue

In testing the absorption capacity of methyl blue to determine the concentration of the methyl blue solution after the adsorption process, the solution was analyzed using a UV-Vis spectrophotometer with a wavelength of 656 nm. The results obtained were 306.693 mg/g. The absorption capacity of methyl blue can be used to determine the surface area of the adsorbent. A large absorption capacity indicates that the adsorbent has a large surface area.

3.1.4. The Optimum Mass and Contact Time for Adsorption of Pb and Fe Metals with Chicken Eggshell Adsorbent Optimum Adsorbent Mass for Pb and Fe metals

The adsorption process is influenced by several factors, one of which is the mass of the adsorbent. The adsorbent mass influences the adsorption process, both in terms of efficiency and adsorption capacity. Under operating conditions with a temperature of 30ºC a contact time of 60 minutes, and a stirring speed of 130 rpm produces the following data:

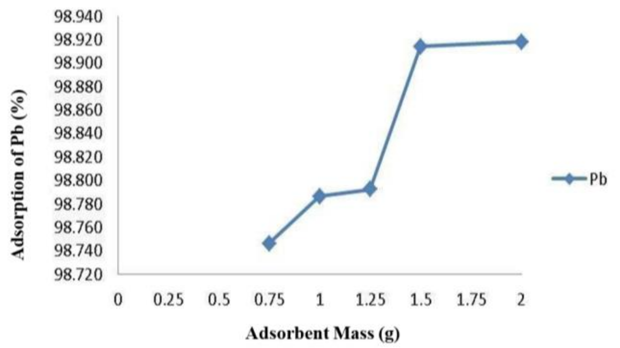

Fig 1. Graph of Metal Adsorption with Mass Variation (Pb $(NO_3)_2$)

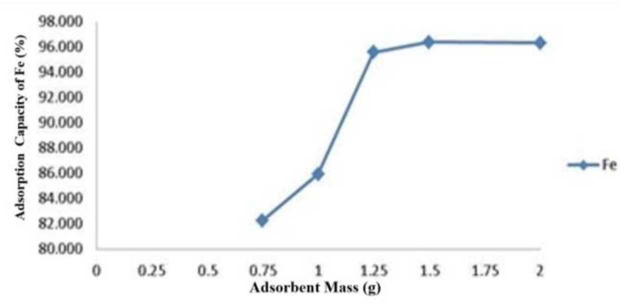

Fig 2. Graph of Metal Adsorption with Mass Variation ($FeSO_4$).

3.1.5. Effect of Contact Time Towards Pb & Fe Metals

Contact time is one factor that influences methyl orange's data shown in Figure 1; it is known that at a weight of 0.75 gams, the adsorbent obtained 98.746% and geatly increased at a mass of 1.5 gams of adsorbent amounting to 98.914%. At an adsorbent mass of 2 gams, the adsorption results obtained did not give a significant increase, namely 98.918%. No significant absorption was seen because the adsorbent mass of 1.5 gams could absorb Pb metal perfectly. The increase in adsorbent mass is proportional to the increase in the number of metal ion binding sites, and the absorption efficiency also increases [18]. In the adsorption of Pb metal using eggshell adsorbent, the best results were obtained, namely 98.914% with a mass of 1.5 grams of adsorbent. The results of this study obtained a better adsorption capacity when compared with almost similar research conducted by [19], which also used eggshell adsorbents that were activated physically and chemically to reduce Pb metal levels, with a Pb metal solution concentration of 10 ppm. The results obtained were 90% effective.

Meanwhile, mass variations of 0.75 gams to 2 gams of eggshell adsorbent are also used for the adsorption of Fe. Based on Figure 2, it is known that at a weight of 0.75 grams of adsorbent, the adsorption percentage was 82.250%, there was a very large increase in the mass of 1.25 grams of adsorbent by 95.572%, and the best results were obtained at a mass of 1.5 grams of adsorbent of 96.386%. The decrease in adsorption power occurred at an adsorbent mass of 2 games, namely 96.330% because the mass of 1.5 games was already at optimum condition. The availability of an active surface on an adsorbent is proportional to the amount of adsorbate that will be absorbed on the surface of the adsorbent in solution [17].

The Fe metal adsorption results obtained in this study were better when compared to research conducted by [20], who also used eggshell adsorbent to reduce Fe levels; the best result was 99.07% at a mass of 2.5 grams of eggshell adsorbent, concentration of Fe solution 5 ppm, with adsorbent particle size 120 mesh, stirring speed 200 rpm. This is because the optimum mass in this study was 1.5 grams (using less mass) with a solution concentration of 50 ppm, where the aim of determining the optimum adsorbent mass was to find out the minimum amount of adsorbent that could be used for the adsorption process so that the amount of adsorbent used was more efficient. and more cost-effective [17]. Fig 3. Metal Adsorption Contact Time Gaph ($Pb(NO_3)_2$)

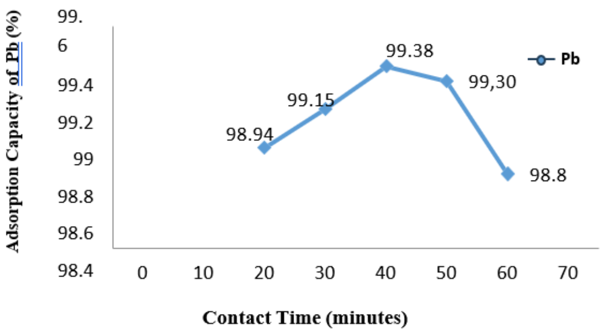

Fig 3. Metal Adsorption Contact Time Gaph ($Pb(NO_3)_2$)

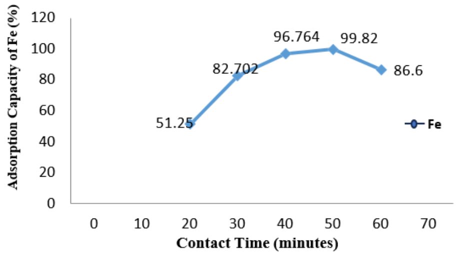

Fig 4. Metal Adsorption Contact Time Gaph (FeSO4)

Based on the picture above, it can be seen that there was an increase in the adsorption percentage at a contact time of 20 minutes to 60 minutes for eggshell adsorbents. The results shown in Figure 3 show that the Pb metal adsorption process takes place quickly at the initial contact between the adsorbent surface and a number of adsorbates. This is because there is still a lot of active surface available on the adsorbent surface.

Fast absorption is usually due to the diffusion process that occurs between the adsorbate and the adsorbent surface [21]. Fast absorption is usually due to the diffusion process that occurs between the adsorbate and the adsorbent surface [21]. In the adsorption of Pb metal, the highest adsorption percentage was obtained, namely 99.38% at a contact time of 40 minutes. However, the adsorption percentage tends to decrease at contact times exceeding 40 minutes, namely 99.30% at a contact time of 50 minutes and 98.88% at a contact time of 60 minutes. This is because the amount of adsorbent bound to the adsorbate is already in a saturated state, so if excessive adsorption time is added, it will cause a desorption or re-release process between the adsorbent and adsorbate.

When compared with almost similar research conducted by [19] shows that the eggshell adsorbent, which was activated physically and chemically to reduce Pb and Cd levels, was found to be up to 90% effective; the best adsorption results were obtained at a contact time of 15 minutes, but because the adsorbent used experienced saturation at a contact time of 15 minutes, there was a decrease in the adsorption percentage at a contact time of more than 15 minutes. The results obtained were also better than those reported by Ref. [22], where the optimum absorption time was found at 60 minutes.

4. CONCLUSION

The best adsorption performance obtained at the optimum mass for adsorbing Pb metal was 1.5 grams (optimum conditions), namely 98.914%, and the adsorption of Fe metal at a mass of 1.5 grams (optimum conditions) was 96.386%. The best capacity with variations in contact time obtained in adsorbing Pb metal using a mass of 1.5 grams was 99.30% at a contact time of 40 minutes. The best capacity obtained for adsorbing Fe metal at a contact time of 50 minutes was 99.82%.

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