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EFFECTIVENESS OF CALCIUM CARBONATE IN CHICKEN EGG SHELLS AS A COPPER ADSORBENT IN KOTAGEDE SILVER CRAFT LIQUID WASTE

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ABSTRACT

Effectiveness of Calcium Carbonate in Chicken Eggshell as a Copper Adsorbent in Kotagede Silver Craft Liquid Waste. The silver craft industry's liquid waste contains hazardous heavy metals such as Cu. Waste containing Cu is directly discharged into the environment, harming the environment and living organisms. An alternative treatment for this liquid waste is the adsorption method using activated chicken eggshells, which have a high CaCO3 content as an adsorbent. This study aims to determine the effectiveness of chicken eggshell adsorbents in reducing Cu levels in silver craft liquid waste solutions. The research employs a quasi-experimental method. The dependent variable in this study is the Cu content in the silver craft liquid waste. In contrast, the independent variables are adsorbent particle size with variations of 50 mesh, 100 mesh, and 150 mesh and adsorbent concentrations with variations of 30 g/L, 40 g/L, and 50 g/L. The statistical test used is the General Linear Model (GLM). The results showed that eggshell adsorbents could reduce Cu levels by 80.82% (from 2.671 mg/L to 0.512 mg/L), which occurred in the particle size group of 150 mesh and a concentration of 40 g/L. However, this result has yet to effectively reduce Cu levels below the standard (0.5 mg/L) stipulated by DIY Regional Regulation No. 7 of 2016. Data analysis with the GLM test showed a significant difference in Cu levels based on variations in adsorbent particle (p-value=0.000) and adsorbent concentration (pvalue=0.024). The interaction between particle size and adsorbent concentration did not show a significant difference (pvalue=0.810), indicating it did not reduce Cu levels effectively.

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INTRODUCTION

The silver craft industry is one of the industries that is still engaged in by Indonesian people in several regions, one of which is the Kotagede Silver Craft Industry in DIY. According to data from the Yogyakarta City Department of Industry, Cooperatives, and MSMEs in 2021, the total number of small and medium industries in the field of silver and metal crafts in Kotagede is 76 IKM, with sales reach in DIY, nationally and internationally. If we look at the production process, the silver craft industry can produce B3 waste in the form of liquid waste containing heavy metals, such as silver, copper, chrome, zinc, lead, nickel, etc. [1,2]

Copper (Cu) is a heavy metal that is often found in liquid silver craft waste because it is an alloy that is often used to make silver crafts stronger than pure silver. According to preliminary studies, the "X" Silver Crafts Industry in Kotagede produces liquid waste with a

Cu content of 500-600 mg/L that is directly disposed of on the ground in the production house's yard. Meanwhile, DIY Regional Regulation No. 7 of 2016, concerning Waste Water Quality Standards, states that the quality standard value for Cu content in metal plating waste water is 0.5 mg/L. ThThe preliminary study's results align with Prakoso's research, which reported that the Cu levels in liquid silver craft waste in Kotagede were 1,259 mg/L. If high levels of Cu are immediately discharged into the environment without processing, it will have a negative impact on the environment and surrounding living creatures. InIn 2021, researchers will conduct a study on the groundwater quality vulnerability in Kotagede subdistrict using the modified DRASTIC method. The study reveals that Kotagede is highly vulnerable to pollution from the silver craft industry, with a DRASTIC index of >153 (high), which covers an area of 0.522 ha, and >177 (very high), which covers an area of 130.17 ha. Adsorption is one method for processing liquid waste. Adsorption is a process when a dissolved substance (adsorbate) accumulates or adheres to the surface of a solid phase (adsorbent). In this research, a physical adsorption process occurs, resulting in a weak attractive force (the Van der Waals force). Adsorbents in the adsorption process have various types that can be selected according to needs. The effectiveness of adsorbents can be influenced by several things, such as the size and concentration of adsorbent particles. The smaller the particle size, the wider the adsorption surface, because it has more active sites. Similarly, an increase in adsorbent concentration results in an increased active surface area. By increasing the number of active sites on the adsorbent, the adsorption process that occurs will increase, so that the amount of adsorbate that is adsorbed also increases. [5]

Egg shell is an example of a material that can be used as an adsorbent. According to BPS data for 2022, the national need for chicken eggs is around 5.3 million tons. According to Sumiati et al.'s research, the shell weight of eggs ranges from 11% to 11.7 percent of the total egg weight. ^[6] So, it is estimated that chicken egg shell waste in Indonesia will reach 584,130 metric tons in 2022, based on the national demand for chicken eggs. Eggshells can be used as an adsorbent because they contain 98.41% calcium carbonate, which is the most common mineral in them. They are also in the polar or hydrophilic adsorbent group, which means they can absorb water, so they work well in watery solutions after being activated chemically or physically. Non-activated egg shells produce an iodine absorption capacity of 18.73%, while physically activated egg shells (at 600 °C for 2 hours) produce an absorption capacity of 31% (the standard 20% absorption capacity). ^[7]

This research employs varying sizes and concentrations of adsorbent particles. According to Ratnasari et al.'s research, using chicken egg shells at a powder concentration of 30 gL significantly reduced Cu levels by 69.23%, compared to powder concentrations of 20 gL and 25 gL. [8] Furthermore, previous research has shown that egg shell adsorbent has a Pb adsorption effectiveness of 98.91% (particle size 100 mesh), whereas Cd adsorption is 93.16% (particle size 200 mesh). The purpose of this study is to find out how well different sized and concentrations of chicken egg shells can remove copper from liquid waste from the Kotagede Silver Crafts Industry.

MATERIALS AND RESEARCH METHODS

The research method used in this research is quasi-experimental, with a research design using a factorial design. There were nine treatment groups that had been determined, and this experiment was carried out five times according to the Federer formula calculation with a total sample of 45. Laboratory tests were carried out to test Cu levels in wastewater solutions, using the SSA (Atomic Absorption Spectroscopy) method. Data analysis was carried out with the SPSS application using the General Linear Model (GLM) test.

The tools used in this research were a furnace, desiccator, blender, porcelain cup, analytical balance, sieve, beaker, blender, glass funnel, measuring cup, test jar, pipette, room thermometer, and timer. The materials used are chicken egg shells (CTA), silver craft waste water, distilled water, HNO3, filter paper, litmus paper, and aluminum foil.

The process of manufacturing chicken egg shell adsorbent (CTA) involves cleaning the CTA with water, removing the membrane, washing it clean, and drying it in the sun. Next, the CTA is crushed and ground into a powder using a blender. The powder is then sifted through a 50 mesh, 100 mesh, and 150 mesh sieve. Next, the CTA was placed in a porcelain cup and put into a furnace to be activated at a temperature of 600 oC for 2 hours. After activation was completed, the CTA was removed and placed in a desiccator until it reached room temperature.

To preserve silver craft waste water, the initial pH of the waste is measured with litmus paper, and then HNO3 is added until the pH is < 2. The waste water is measured again with litmus paper.

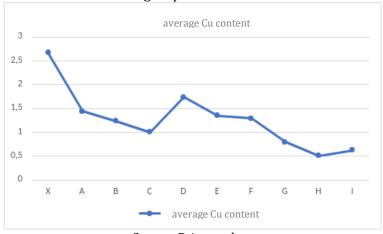
Pre-treatment We prepare silver craft waste water, a glass funnel, and an Erlenmeyer. Next, we place filter paper on the glass funnel and moisten it with distilled water. A glass funnel is placed on top of the Erlenmeyer, and the waste water is filtered through filter paper.

Dilution of silver craft waste water: take 2 ml of waste water with a pipette and put it in a beaker. Next, 498 ml of distilled water was measured and put into the same beaker as the 2 ml waste water container, then homogenized with 500 ml of the diluted waste water solution. To carry out the adsorption treatment, nine glass beakers were prepared, each containing 500 ml of liquid waste sample. Prepare egg shells in sizes 50 mesh, 100 mesh, and 150 mesh with mass variations of 15 gr, 20 gr, and 25 gr, respectively. The pH of the waste solution was measured before treatment, and then the prepared eggshell adsorbent was added to the waste solution sample. The wastewater solution samples were homogenized with a jar test at a speed of 30 rpm for 15 minutes, and then each sample was left to rest for 60 minutes. Next, the sample solution was separated from the adsorbent sediment using filter paper and a glass funnel. We measured the filtration results using the pH of the solution and treated it with HNO3 until the pH was less than 2.

RESEARCH RESULTS AND DISCUSSION

Cu Test Results

The test results come from testing Cu levels using the SSA method. In this research, liquid waste was diluted from original levels ranging from 625 mg/L to 670 mg/L and then diluted 250 times to produce an initial level of 2,671 mg/L. This study had nine treatment groups and was repeated five times, so there were 45 samples. The following is the average Cu from five repetition tests in each treatment group.



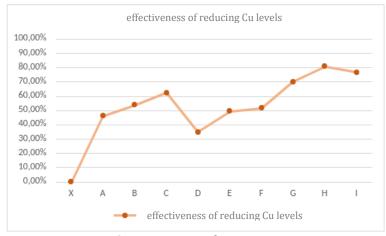
Source: Primary data
Figure 1. Average Cu content test results

Information:

X : Initial Cu levels before treatment

A : Adsorbent size 50 mesh, concentration 30 gr/L
 B : Adsorbent size 50 mesh, concentration 40 gr/L
 C : Adsorbent size 50 mesh, concentration 50 gr/L
 D : Adsorbent size 100 mesh, concentration 30 gr/L
 E : Adsorbent size 100 mesh, concentration 40 gr/L
 F : Adsorbent size 100 mesh, concentration 50 gr/L
 G : Adsorbent size 150 mesh, concentration 30 gr/L
 H : Adsorbent size 150 mesh, concentration 40 gr/L
 I : Adsorbent size 150 mesh, concentration 50 gr/L

We then calculate the average Cu content test results to see the percentage reduction in Cu levels after the liquid waste is treated. The graph below shows how effective reducing Cu levels is.



Source: Primary data
Figure 2. Effectiveness of reducing Cu levels

Prior to treatment, the initial Cu level was 2.671 mg/L. The lowest average Cu content after treatment was in group H (concentration 40 gr/L, particle size 150 mesh), with an average Cu content of 0.512 mg/L and an effective Cu reduction of 80.82%. Meanwhile, the highest average Cu content after treatment was in group D (concentration 30 gr/L, particle size 100 mesh), with an average Cu content of 1.742 mg/L and an effective Cu reduction of 34.77%. The average Cu content test results from all groups did not meet the NAB Cu for liquid metal craft waste, namely 0.5 mg/L (DIY Regional Regulation No. 7 of 2016 concerning Waste Water Quality Standards).

Table 1. General Linear Model Test Results

No.	Variable	p-value
1.	Adsorbent particle size	0,000
2.	Adsorbent concentration	0.024
3.	Particle size * concentration	0.810

The GLM test was carried out to determine the effect of the variable particle size, adsorbent concentration, and the interaction between these two variables. Variations in the particle size variable have a p-value of 0.000 (p-value <0.05), which means there is a significant difference in Cu content based on variations in particle sizes of 50 mesh, 100 mesh, and 150 mesh. Variations in the concentration variable have a p-value of 0.024 (p-value <0.05), which means

there is a significant difference in Cu levels based on variations in concentration of 30 gr/L, 40 gr/L, and 50 gr/L. Meanwhile, the interaction between the variable particle size and adsorbent concentration has a p-value of 0.810 (p-value > 0.05), which means that the interaction between particle size and CTA adsorbent concentration has no effect on reducing Cu levels.

Adsorption of Cu with Eggshell Adsorbent

This research uses physical adsorption in the presence of weak attractive forces (Van der Waals forces). In the interior of the aln the interior of the adsorbent, an attractive force and an equilibrium of forces exist in all parts, while on the surface, an inward pulling force creates an imbalance, causing Cu to experience an attractive force towards the surface of the adsorbent. ith a larger molecular weight will fall to the surface faster due to gravitational forces, thereby increasing the effectiveness of adsorptioThe electrostatic force of an ion is weaker as its radius increases. This means that the ion can't interact as strongly with water molecules, and the adsorbate moves more quickly to the surface of the adsorbent without being slowed down by the water molecules' pull. cules). [5]

Adsorbents or porous solid materials are good adsorbents because they have a large surface area and active pore volume, so there are more places for the adsorbate to stick to the adsorbent's surface. The size of the adsorbent particles affects the adsorbent's active site; the smaller the size, the more active sites are available. The adsorbent concentration affects the quantity of active sites because the more adsorbents there are, the more active sites are available. [11,12] P This research uses physical activation to enlarge the pores of the adsorbent (by breaking chemical bonds or oxidizing surface molecules). [13]

This research employs a chicken egg shell adsorbent (CTA) that has the highest concentration of CaCO3 (calcium carbonate), which belongs to the polar or hydrophilic adsorbent group and effectively absorbs water in solution. [4] CTA activated at 600 oC for 2 hours contains 94% CaCO3 and a small portion of CaO (calcium oxide). CaO is a polar component, and its presence is indicated by calcination and thite powder. [7] CTA calcination ideally occurs at 70700C–1000 1000oCoC. s study, even calcination did not occur because it was carried out at 60600 oCnd the white powder was only present at the edge of the cupcup.] The The following is the reaction between CaCO3 and CaO.

$$CaCO3(s) \rightarrow CaO(s) + CO2(g)$$

The decrease in Cu levels that occurs on the CTA surface occurs through the following reaction:^[17]

The adsorption mechanism series also features an ion exchange mechanism. Ca2+ ions from the CTA surface migrate to the solution, while Cu2+ ions from the solution migrate to the CTA surface due to their similar ionic radius and valence. This allows them to replace each other's ions, despite Cu2+ having a smaller ionic radius than Ca2+. 7] CaCO3 is composed of CO32, has a triangular structure, and forms layers perpendicular to the central c-axis. CaCa2+ ions are located between these layers to form an octahedron with a large cavity, causing the molecules on the surface to have an inward attraction. 8] Ion exchange can also occur because Ca2+ ions perpendicular to the central c-axis are replaced by Cu2+ ions. [19,20]

A CTA adsorbent can also affect the pH value of the solution. In this study, the pH of liquid waste before treatment was 3 and after treatment was between 6-7. CaCO3 is an alkaline salt, formed from a weak acid in the form of carbonic acid (H2CO3) and a strong base in the form of calcium hydroxide (Ca(OH)2). When one component of a weak acid or base, such as CaCO3, comes into contact with water, it undergoes a hydrolysis reaction. This partial hydrolysis occurs in the CO32- anion of the weak acid, leading to the decomposition of CaCO3 according to the following reaction: [18,21,22].

$$CO32-(aq) + 2H2O(1) \rightarrow H2CO3(aq) + 2OH-(aq)$$

H2CO3 will decompose based on the reaction:

$$H2CO3(aq) \rightarrow H2O(1) + CO2(g)$$

So when CaCO3 is reacted with water it produces:

$$CaCO3(s) + H2O(l) \rightarrow Ca(OH)2(aq) + CO2(g)$$

CaCO3 reacts with water to release OH- ions because it produces calcium hydroxide (Ca(OH)2), which is alkaline, increasing the amount of OH- in the water and making silver craft liquid waste alkaline. This is in line with research by Novianti et al., which states that CTA adsorbents can increase the pH value of the solution from 5 to 7.78. [22,23]

Effect of Adsorbent Particle Size Variations

We used the general linear model (GLM) test to see how changes in adsorbent particle size affected lowering Cu levels. The test came back with a p-value of 0.000, which is less than 0.05. This means that the changes in adsorbent particle size do make a difference in lowering Cu levels in liquid waste samples.

The smaller the adsorbent particle size increases the surface area of the adsorbent so that there will be more active sites or places for the adsorbate to stick to the adsorbent and increase the effectiveness of adsorption. This can be seen from the 150 mesh particle size group which has the highest reduction effectiveness, namely 80.82%. However, in this study, a particle size of 50 mesh had a higher effectiveness in reducing Cu levels than a particle size of 100 mesh. This can occur because of the contact time in this study (60 minutes)[8] has the possibility of reaching adsorption-desorption equilibrium more quickly at a particle size of 100 mesh compared to an adsorbent measuring 50 mesh, causing desorption on the adsorbent measuring 100 mesh and then Cu being released back into solution. This is in line with research by Shafirinia, et al which states that adsorbents with a size of 110-200 mesh have an optimum time of 90 minutes, while adsorbents with a size of 10-40 mesh and 50-100 mesh have an optimum time of 120 minutes.[24]This can also be caused by the activation process at a temperature of 600oC for 2 hours which is not optimal for a 100 mesh adsorbent. In the adsorption process, the adsorbent has a saturation point for adsorbing the adsorbate due to its limited surface area. The saturation point is the condition when the adsorbate has overlapped during the adsorption process and the adsorbent can no longer absorb the adsorbate because the active site on the adsorbent is full. This is related to the balance between adsorption and desorption rates, which is influenced by contact time. When the optimum contact time is reached, adsorption will have the highest effectiveness; however, if the contact time is increased, it will result in a decrease in the effectiveness of adsorption because the adsorbate is released back into the solution (desorption). Physical adsorption causes desorption because it is reversible. [18,25,26]

Effect of Varying Adsorbent Concentration

The general linear model (GLM) test was used to look at how changes in adsorbent concentration affect lowering Cu levels. The test returned a p-value of 0.024, which is less than 0.05, which means that the changes in adsorbent concentration do make a difference in lowering Cu levels in the sample. liquid waste.

The adsorbent concentration in adsorption is related to the mass of the adsorbent used. The greater the mass or concentration of the adsorbent, the more active it becomes. This research used concentrations of 30 gr/L, 40 gr/L, and 50 gr/L. The effectiveness of reducing Cu levels in this experiment shows that as the adsorbent concentration increases, the effectiveness of

reducing Cu levels in the liquid waste solution also increases. However, one treatment group performed less effectively than the group with a higher adsorbent concentration, specifically in the 150 mesh particle size group. Group H, with a concentration of 40 gr/L, reduced Cu by 80.82%, while group I, with a concentration of 50 gr/L, reduced Cu by 76.58%. This can be caused by the contact time being too long, so that the solution experiences saturation and desorption. [26,27] There is research by Nurafriyanti et al. that supports this. They found that an adsorbent concentration of 16 gr/L worked best compared to 24 gr/L, 32 gr/L, and 40 gr/L all at the same time (45 minutes), because desorption took place. [28]

The relationship between the size of the particles and the concentration of the adsorbed material

The interaction of these two variables has a p-value of 0.810 (p-vvalue > 0.05, which means that the interaction between particle size and CTA adsorbent concentration has no effect on reducing Cu levels. These results indicate that the processing of silver craft liquid waste with CTA adsorbent can be carried out by selecting one of the particle size variables or the concentration varvariable, cause each of these variables has significant results (p-value <0.05) even though only one of the variables is used. jusjust. sThis aligns with the findings of a study conducted by Ratnasariet al., which employed a CTATA adsorbent, varying only in the concentration variable, and demonstrated an effectiveness of 69.23% in reducing Cu levels. rch using CaCO3 biosorbent with variable particle size variations alone was able to reduce heavy metal levels with a reduction effectiveness of 75.37%. [29]

Empty Bed Contact Time

Empty Bed Contact Time (EBCT) is a calculation used to determine the effective contact time during the contact process of liquid waste with activated adsorbents using the adsorption method. The EBCT formula and calculation are as follows, based on the conditions used in this study: [30,31]

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EBCT = \frac{bed\ volume\ (ft^3)\ x\ 7,48\ gallons/ft^3}{flow\ rate\ (gpm)}
Bed\ volume\ (tube\ volume)
V = \pi r 2t
= 3.14\ x\ 5\ x\ 5\ x7
= 549.5\ cm3
= 0.019\ ft3
Flow\ rate
Q = 0.5\ liters/hour
= 0.002\ gpm
EBCT = \frac{0,019\ x\ 7,48}{0,002}
= 71.06\ minutes
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Treatment Advanced

This study used 250 times less silver craft liquid waste and found that Cu levels dropped the most in the adsorbent particle size group of 150 mesh and a concentration of 40 gr/L (group H). To apply this research to liquid waste without dilution, one must either carry out the current study or implement several modifications, including:

- 1. A combination of diluting the waste and increasing the adsorbent concentration is required; for example, if the original waste is diluted 50 times, an adsorbent concentration of 2000 g/L is required (based on group H).
- 2. To process large amounts of liquid waste more effectively, use pipes that contain CTA (continuous adsorption) adsorbent for draining liquid waste.
- 3. Recalculate EBCT according to the condition of the equipment used.

CONCLUSIONS AND RECOMMENDATIONS

The CTA adsorbent effectively reduces Cu levels in silver craft liquid waste. The most effective treatment group was group H (150 mesh size, concentration 40 gr/L). The CTA adsorbent can lower Cu levels by 80.82%, from 2.671 mg/L to 0.512 mg/L. However, these results are not yet good enough to lower Cu levels below the NAB of 0.5 mg/L (DIY Regional Regulation No. 7 of 2016). Significant differences in Cu levels were observed based on variations in adsorbent particle size (p-value = 0.024) and adsorbent concentration (p-value = 0.000). The interaction between the variable particle size and adsorbent concentration did not produce a significant difference (p-value = 0.810), so it had no effect on reducing Cu levels. Furthermore, it is necessary to conduct further research on other adsorption influencing factors such as pH, contact time, and activation conditions. Additionally, we need to conduct characterization tests for CTA adsorbents, such as FTIR and BET Surface Area tests, and evaluate the effectiveness of these adsorbents against heavy metals such as Ag, Rh, and Pt, as well as organic pollutants in silver craft liquid waste.

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