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Adsorption of Inorganic and Organic Waste of Chemistry Laboratory by Using Eggshell-based CaO

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Abstract

Laboratory activities are closely related to using corrosive, acidic, and toxic chemicals in producing inorganic and organic waste, such as heavy metals, resulting from chemical reactions, and organic waste originating from ethanol solvents. Adsorption can be an appropriate waste handling step to reduce metal levels in waste. CaO can be used as an adsorbent through several mechanisms. One involves the basic nature of CaO, which forms ionic bonds with acidic compounds in the environment, so these molecules or ions can be adsorbed on the CaO surface. The aims are to characterize CaO and to determine the amount of adsorption power of the CaO catalyst on metals (Pb and Cd) and organic waste (ethanol) originating from the chemistry laboratory. The SSA measurement showed that the waste tested contained the highest levels of Cd and Pb metals, namely 16.82 ppm and 1.52 ppm. The adsorption results using CaO showed that the absorption capacity for Cd and Pb metal was up to 99%, while for ethanol, it was around 20%. It showed that CaO can be used as an adsorbent for laboratory waste.

Keywords: Eggshells, CaO, Heavy metal, Cd, Pb

INTRODUCTION

The high egg production in Indonesia makes eggshells very potential to be utilized more optimally (Nurhasanah et al., 2023). Chicken eggshell waste is valuable because it is widely used in everyday life. Therefore, chicken eggshell waste is very abundant, and there has been no utilization of the waste (Tumbel, et.al., 2020: 293).

The eggshell is the outermost part of the egg, which protects the components of the egg from physical, chemical, and microbiological damage. Eggshells each have 10,000-20,000 pores, therefore, it is estimated that they can absorb a solute and can function as an adsorbent (Sunardi et al., 2020). Eggshells have only been considered as waste and have not been maximally utilized. Eggshells are equivalent to 11% of the total egg weight and contain 91%-94% calcium carbonate ($CaCO_3$ 1% calcium phosphate and organic matter. CaCO3 can be converted into CaO through the calcination process (Firnanelty et al., 2023) so it is expected that eggshells can be used as a source of CaO which has a high level of purity so that it has great potential as the main ingredient of the CaO catalyst (Oko & Feri, 2019).

One alternative that is starting to be developed as a good adsorbent material is catalysts (Chen et al, 2018). This type of catalyst has been the research focus in dealing with waste. Porous materials and large surface areas are possessed by materials capable of functioning as catalysts and adsorbents at the same time (Koloay et al., 2024). The use of catalysts as adsorbents has advantages due to their good adsorption ability to various types of organic and inorganic compounds, and they have a fast adsorption ability and can be regenerated, so that they can significantly reduce production costs (Lamuru et al., 2023).

Research has proven that CaO can efficiently absorb organic pollutants from water in time and shows that the regeneration process of CaO can be done by heating at a certain temperature (Yani et al., 2023). CaO as an adsorbent for Congo red dye and the maximum adsorption capacity reached 238.66 mg/g at the optimum time of 30 minutes (Amri et al., 2017).

The adsorption method has the potential to overcome various types of waste. The chemistry laboratory is a place to conduct practicum activities using solid, liquid and gas chemicals. Laboratory activities are very close to the use of chemicals that are corrosive, acidic and toxic and in their implementation produce inorganic waste in the form of heavy metals from chemical reactions and organic waste derived from solvents such as methanol, ethanol, chloroform and other coloring substances that are mixed into waste (Ruspita, 2024).

Chemical waste is any substance or chemical that is no longer needed and is disposed of as a result of human activities, especially in laboratories or other activities involving the use of chemicals that can have potential hazards to the environment and human health (Sekewael et al., 2013).

Chemistry laboratory liquid waste contains various kinds of waste both from inorganic waste such as Cu metal (Tahya et al., 2023), Pb, Cd and organic waste such as alcohol or ethanol (Wijayanti et al., 2023). Waste generated from both practicum and research activities is accommodated in an IPAL (Waste Water Treatment Installation) container. However, before being put into the WWTP, initial treatment is needed to reduce the concentration of the waste.

The initial analysis of Pb and Cd metal waste content and ethanol obtained results that exceeded the threshold and did not meet SNI. This has also been done research which states that using chemical laboratory waste test samples obtained initial concentrations for Pb metal of 3,491 ppm and Cd metal of 2,790 ppm (Ruspita, 2024). Based on SNI 6989.8 of 2009, the maximum limit of Pb and Cd metals in the environment is 0.03 ppm and 0.01 ppm. So that these results have exceeded the threshold set by SNI (Ruspita, 2024).

METODOLOGI

Materials and Instrumentals

X-Ray Diffraction Shimadzu maxima XRD-7000, Fourier Transform Infrared (FTIR) nicole iS10, Scanning Electron Microscopy (SEM), Atomic Absorption Spectrophotometer (SSA), UV-Vis Spectrophotometer Varian 50 Conc, furnace, oven, analytical balance, sieve shaker, centrifuge, hotplate magnetic stirrer, shaker, beaker, Erlenmeyer, volumetric flask, stative and clamp, burette, pestle and mortar, stirring rod and funnel.

Chicken eggshell, ethanol, benzoic acid, methanol, ammonium, universal indicator, plastic wrap, ordinary filter paper,Whatman no. 42 filter paper, aluminum foil, distilled water.

Methods

1. Preparation of Eggshell Waste

Eggshell waste was obtained from the Gowa Broiler Farm area, which will be used as a catalyst in the adsorption of laboratory wastewater.

2. Preparation of CaO catalyst

Eggshells were cleaned of as much as 1 kg of mucus membranes and dirt. Next, it was washed using clean water. After that, it was dried at 105°C for 2 hours using an oven. Next, the eggshells were pulverized into powder. After that, the eggshells were calcined at 900°C for 5 hours using a furnace to obtain calcium oxide (CaO). Furthermore, the solid powder obtained was crushed, ground, sieved with a size of 100 mesh, 150 mesh, 200 mesh and stored in a desiccator to keep it dry (Yani et al., 2023).

3. Characterization of CaO Catalyst from Eggshells

CaO analysis using XRD and SEM EDX instruments.

4. Initial concentration analysis of Laboratory Liquid Waste

At this stage, testing is carried out by taking laboratory waste samples, which are then analyzed for Cd and Pb metal levels using SSA instruments and analyzing ethanol content.

5. Adsorption of inorganic waste (Cd and Pb metals) and organic waste (ethanol) using CaO catalyst

A total of 1 gram of CaO catalyst with each particle size of 100 mesh, 150 mesh, and 200 mesh was put into 3 different Erlenmeyer flasks containing 100 ml of laboratory liquid waste, then placed on a magnetic stirrer hotplate for 60 minutes with a rotation speed of 200 rpm. The sample was then filtered. Then the filtrate was analyzed to determine the levels of Cd and Pb metals using an Atomic Absorption Spectrophotometer (AAS) (Oko & Feri, 2019). Organic waste is also tested for ethanol content using an ethanol meter.

RESULTS AND DISCUSSION

Calcium Carbonate ($CaCO_3$) from Eggshells

Eggshells have a high content of primary and secondary metabolites. Eggshell is a by-product of egg consumption that contains 97% CaCO3, 3% phosphorus, 3% magnesium, sodium, potassium, zinc, manganese, iron, and copper (Hasibuan et al, 2021) shown in Figure 2a. Eggshells containing calcium carbonate (CaCO3) as the main constituent have the potential to become calcium oxide (CaO) nanoparticles. Figure 1 shows the results of XRD analysis of CaCO3. The CaCO3 crystal phase is aragonite formed from the most saturated conditions and requires high temperature and pressure. This phase is synthesized at temperatures of more than 600 ºC (Putranto & Khaeruman, 2020).

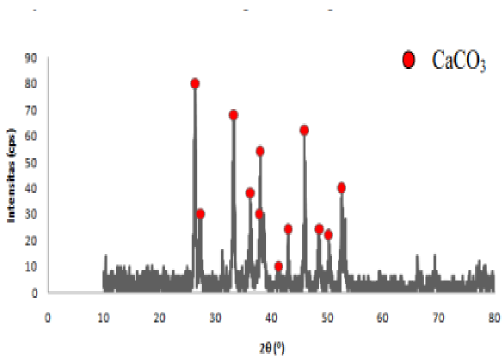

Figure 1. Difractogram of CaCO3

In general, aragonite forms are found in nature as biominerals in ale-ale shells (Suci & Dala Ngapa, 2020), coral reefs, tutut shells (Firnanelty et al., 2017) and several other examples. Aragonite forms isostructures with carbonates of divalent cations such as Ba, Sr, and Pb (Vironika et al., 2022).

Calcination of $CaCO_3$ to CaO

Eggshell powder in $CaCO_3$ is calcined at 900 ºC for 5 hours. The choice of temperature in the calcination process is very important. Low temperatures can cause the resulting CaO compound to change back to $CaCO_3$, and the resulting CO_2 decomposition will be quite low. The calcination process aims to convert $CaCO_3$ compounds into CaO. This condition causes all organic components of eggshells to burn out into CO_2 and H2O (Khalid et al., 2022). Calcination can also remove organic compounds and impurities that interfere with the hydroxyapatite formation process (Charlena et al., 2022). Hus, at the end of the calcination process, the eggshell is expected to turn into CaO and cause the mass of the sample to decrease. The calcination results showed a color change from yellowish to white as shown in Figure 2b. The reaction that occurs is as follows:

$$CaCO_{3(s)} \rightleftharpoons CaO_{(s)} + CO_{2(g)}$$

The main compound that makes up almost all parts of the eggshell is $CaCO_3$, so that calcination at high temperatures can convert it into CaO, where $CaCO_3$ is decomposed into CaO due to the influence of high temperatures. From the observation, it can be concluded that the best calcination temperature to produce CaO is 900ºC (Vironika et al., 2022).

Calcium Oxide (CaO) is a white crystalline solid that can be synthesized from eggshells using calcination. In addition, CaO can be referred to as quicklime, which is made from the thermal decomposition of limestone or shells containing CaCO3 as the main constituent, with the potential to become CaO nanoparticles. CaO can also be chemically synthesized in many industries in the form of toxic waste remediation materials, as well as bactericides (Zahara et al., 2020).

(a) (b)

Figure 2. Chicken Egg Shell Calcination (a) $CaCO_3$; (b) CaO

The presence of the CaO peak in Figure 3 is supported by several peaks corresponding to the Joint Committee on Powder Diffraction Standards (JCPDS) database. The peak = 32.220o is shown at an angle of 2θ: 18.00°; 28.72°; 29.42°; 34.12°; 39.42°43.04°; 47.14°; 48.46°; 50.86°; 54.40º; 59.48° ,62.68˚; 64.32˚.

Another phase in CaO from chicken eggshell is $Ca(OH)_2$, which is in accordance with JCPDS standard data. The Ca(OH)2 phase at angles 18.029º, 28.669º, and 34.138º corresponds to JCPDS no. 84-1263 (Gago & Dala Ngapa, 2021).

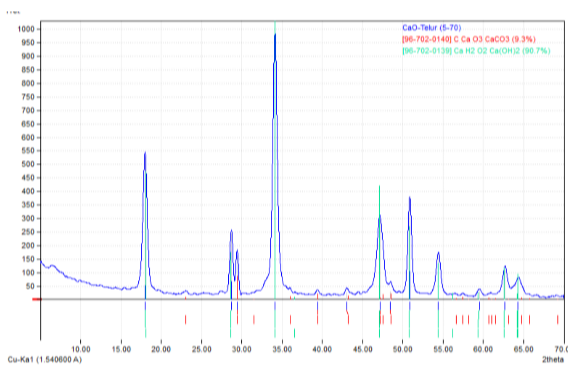

Figure 3. Difractogram of CaO

Analysis Results Using SEM-EDX Instrument

SEM-EDX analysis shown in Figure 4 aims to see the surface morphology of the CaO catalyst and see the chemical components on the catalyst. The particle size of CaO compound in the image with 2000 times magnification is around 10 µm. The calcium oxide particles are clumped together. This clumping of particles shows the polycrystalline character of CaO nanoparticles (Sunardi et al., 2020).

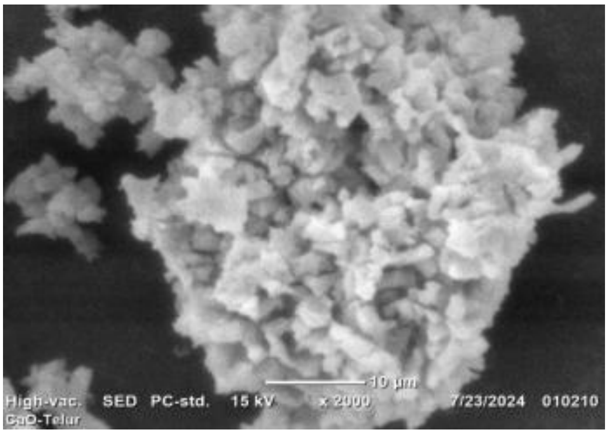

Figure 4. SEM Morphology Image of CaO at 2000x magnification

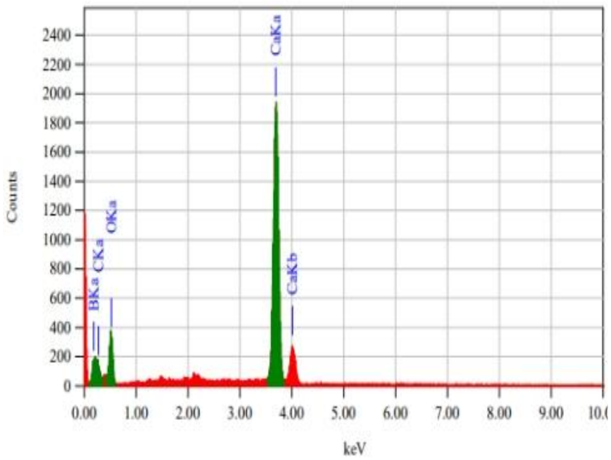

Figure 5. Spectrum EDX of CaO

Table 1. CaO content test results

Element	Series	Mass (%)	Sigma
O	K-series	4.83	0.13
C	K-series	1.45	0.07
Ca	Kseries	73.43	0.74

Figure 5. shows intense peaks indicating the presence of Ca, C and O. The constituent elements of calcium oxide are shown in Table 1, namely O (4.83%), C (1.45%), and Ca (73.43%). The presence of Ca and O elements with dominant percentages indicates that the calcination of CaCO3 has been successful using a temperature of 900°C (Yani et al, 2023).

Analysis Results of Inorganic Waste (Cd and Pb Metals) and Organic Waste (Ethanol) Levels

The levels of Cd metal detected in Table 2 show significant variation between samples. Sample E had the highest Cd level of 16.82 ppm, followed by sample D with 14.9 ppm. Meanwhile, samples A, B, and C had lower Cd levels of 2.87 ppm, 0.88 ppm, and 1.70 ppm, respectively. The high Cd levels in samples D and E indicate a potentially serious pollution risk, as Cd is a highly toxic heavy metal even at low concentrations. According to WHO, the maximum limit of Cd in drinking water is 0.003 ppm, so the levels found in these laboratory effluents far exceed the safe threshold. Cd metal is one of the heavy metals often found in chemical laboratory waste, especially from the use of certain chemicals such as catalysts, electrolyte solutions, or reagents. The high levels of Cd in samples D and E could be due to laboratory activities involving large amounts of Cd-based compounds or waste that is not properly treated before disposal (Hidayah, 2021).

Table 2. Cd metal content in laboratory waste

No	Effluent sample	Cd content (ppm)
1.	A	2.87
2.	B	0.88
3.	C	1.70
4.	D	14.90
5.	E	16.82

Description
A: storage duration of 1 year
B: storage duration of 2 years
C: storage duration of 3 years
D: storage duration of 4 years
E: storage duration of 5 years

Pb Level Analysis Results

Table 3. shows the levels of Pb metal in the four laboratory effluent samples with more uniform concentrations than Cd. Sample A had the highest Pb level of 1.52 ppm, followed by sample D at 1.20 ppm and samples B and C at 1.00 ppm each. Pb is a very harmful heavy metal, especially to the human nervous system, and can cause long-term effects such as decreased cognitive function and kidney damage. Pb levels were highest in sample A with a storage duration of 1 year.

The presence of Pb in laboratory waste can come from Pb-based chemicals, such as dyes, analytical reagents, or standard solutions widely used in practicum and research activities. Pb metal is often found due to the use of certain chemicals that contain heavy metals as the main ingredient (Wijayanti et al., 2023).

Table 3. Pb metal levels in laboratory waste

No	Effluent sample	Pb content (ppm)
1.	A	1.52
2.	B	1.00
3.	C	1.00
4.	D	1.20

Description
A: storage duration of 1 year
B: storage duration of 2 years
C: storage duration of 3 years
D: storage duration of 4 years

Ethanol Content Analysis Results

Ethanol is an organic compound that is soluble in water and has volatile properties. High ethanol content in effluent can affect water and soil quality if the effluent is directly discharged into the environment without treatment. High concentrations of ethanol in wastewater effluents can lead to increased BOD and COD levels in waters, which in turn can reduce dissolved oxygen levels and disrupt aquatic life (Yani et al., 2023).

In addition, ethanol, as a volatile organic compound (VOC), can potentially increase air pollution if released in vapor form. Long-term exposure to ethanol vapor can have an impact on human health, especially in laboratory workers, such as irritation of the respiratory tract and neurotoxic effects at high concentrations.

A preliminary analysis of laboratory waste, which showed an ethanol content of 45%, indicates that this waste has a significant proportion of volatile organic compounds. The high ethanol content often comes from using ethanol in various laboratory processes, such as solvents widely used in extraction processes or as reagents in chemical reactions. Ethanol, a volatile organic compound, has potential environmental and health impacts that must be taken seriously, especially if the waste is not treated correctly.

In the context of chemical laboratories, high ethanol content is often found in organic waste. Laboratory waste containing 40-50% ethanol generally comes from washing processes or organic chemical reactions. Handling of this waste needs to be in line with environmental standards, such as pretreatment using distillation techniques or adsorption processes, to minimize the impact on the environment (Sriyanto et al., 2024).

Hence, an environmentally friendly waste management system is essential for organic compounds like ethanol. From the results of his research, combining physical (distillation) and biological (anaerobic digestion) treatment can reduce the ethanol concentration to below the safe threshold, so that the waste can be disposed of without significant risk to the environment (Sulistyanti et al., 2018).

Adsorption Results of Inorganic Waste (Cd and Pb Metals) and Organic Waste (Ethanol) using CaO Catalysts

Table 4 shows the adsorption results of cadmium (Cd) levels in laboratory waste measured after the adsorption process using three mesh sizes, namely 100 (sample code A), 150 (sample code B), and 200 mesh (sample code C). After the adsorption process, the Cd metal absorption efficiency varied based on the mesh size used. The Cd absorption efficiency ranged from 50.66% to 99.80%. Overall, the Cd adsorption process was very effective, especially for samples with higher initial concentrations, although a decrease in efficiency occurred at finer mesh sizes. This indicates that eggshell-based CaO samples have the potential to be used as inorganic waste adsorbents in the laboratory.

The results of lead metal (Pb) analysis showed that the initial concentration of Pb in samples A to D was 1.52 ppm, 1.00 ppm, 1.00 ppm, and 1.20 ppm, respectively. The results of Pb absorption efficiency ranged from 9.26% to 99.08%. Based on the observations, it shows that high initial Pb concentrations showed high overall uptake at all mesh sizes. Pb adsorption using CaO is quite good but not as effective as Cd adsorption and the results tend to be more fluctuating, especially at finer mesh sizes..

The adsorption process was also carried out on laboratory organic waste as shown in Table 5.

Table 4. Adsorption results of Cd and Pb Inorganic Waste

Mesh Size	Sample	Initial Concentration of Cd	Absorption efficiency of Cd	Initial Concentration of Pb	Absorption efficiency of Pb
100	A1	2.87	99.65	1.52	85.38
	B1	0.88	61.47	1	68.52
	C1	1.7	98.34	1	99.08
	D1	14.9	99.80	1.2	87.66
	E1	16.82	99.76	-	
150	A2	2.87	99.02	1.52	89.28
	B2	0.88	98.56	1	38.89
	C2	1.7	94.09	1	11.12
	D2	14.9	97.26	1.2	35.19
	E2	16.82	99.56	-	-
200	A3	2.87	83.84	1.52	89.14
	B3	0.88	50.66	1	9.26
	C3	1.7	97.42	1	88.89
	D3	14.9	99.52	1.2	27.47
	E3	16.82	99.51	-	-

Table 5. Ethanol Absorption Efficiency

Sample	Absorption efficiency (%)
A	5.92
B	15.55
C	17.03

The initial ethanol concentration was found to be 45% for the CaO sample at 200 mesh size, the ethanol sorption efficiency reached a maximum value of 17.03%. Although the ethanol sorption efficiency is lower than that of heavy metals, the results show that the finer the particle size, the higher the ethanol sorption efficiency.

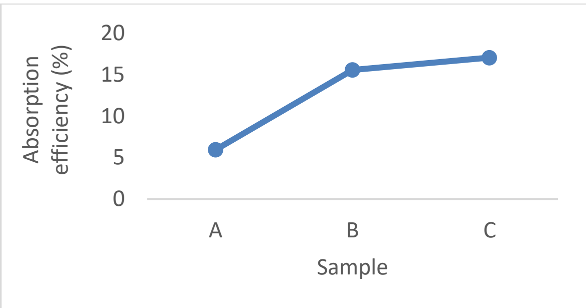

Figure 6. Relationship graph of ethanol absorption efficiency to sample particle size

This shows that the particle size relationship greatly affects the adsorption power of organic waste as shown in Figure 6.

CONCLUSION

Calcium Oxide (CaO) was obtained from the calcination of eggshell calcium carbonate (CaCO3) at 900 °C. The resulting CaO is able to become an adsorbent as well as a catalyst used in the sorption reaction of inorganic (Cd and Pb) and organic (ethanol) wastes in laboratory waste.

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