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Optimization of Volume and Mass of Eggshell in Removal of Linear Alkylbenzene Sulfonate Levels in Laundry Waste

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Abstract: Linear alkylbenzene sulfonate (LAS) is one of the most common anionic surfactants in synthetic detergent formulation. Being a component of domestic waste, LAS can potentially cause environmental damage. Detergent wastewater that contains LAS needs to be properly treated before being released into the environment. This study aims to treat the laundry waste by using thermally activated chicken eggshell adsorbent. The steps carried out in this study were the preparation of adsorbents, quality test of adsorbents produced, testing of adsorbent on used laundry waste based on reducing concentration of LAS and testing of the Langmuir adsorption equation and the Freundlich adsorption equation. Adsorption process of LAS observed variations in adsorbent mass are 4g, 8g, 12 g, and 16 g, and variations in the sample volume used in this experiment are 50 ml, 100 ml, 150 ml, and 200 ml. Stirring time is 60 minutes. The filtrate was analyzed to determine the concentration of LAS. The obtained results indicate that the optimum mass of adsorbent is 12 grams with a sample volume of 50 ml. This adsorption condition resulted in Linear Alkylbenzene Sulfonate levels of 3,376 ppm, which can reduce LAS levels by 69.86%, with an adsorption capacity of 0.2997 mg/g. Based on the result of this study, LAS adsorption follows the Langmuir isotherm equation, with a coefficient of determination (R2) greater than that of the Freundlich isotherm, which is 0.9977. This equation indicates that the adsorption process that occurs is chemisorption. The use of thermally activated chicken eggshell as an adsorbent can be an alternative, environmentally friendly waste processing technique.

Keywords: Adsorbent; Eggshell; Laundry Waste; Linear Alkylbenzene Sulfonate; Surfactant.

Introduction

Linear Alkylbenzene sulfonate (LAS) is an active compound in detergents commonly used in the laundry and household industries [1]. LAS is formed by an alkyl chain attached to a benzene ring in the para position relative to the sulfonate groups. This surfactant is composed of a mixture of homologous compounds in which the length of the alkyl chain varies from 10 to 14 carbon atoms [2]. LAS being biodegradable under aerobic conditions (having sufficient oxygen and microorganisms), so in conditions of rivers in Indonesia, which are murky, LAS cannot biodegrade. Despite being biodegradable, it takes several days and only reaches 50%, which is degraded [3]. In a previous study, biodegradation of LAS was still unable to degrade the aromatic ring. Biodegradation of LAS still remains functional group benzoic acid, hydroxyl, benzene and aliphatic groups in sufficiently large molecular weight [4][5]. This indicates that LAS need treatment before being released into the environment.

The current phenomenon, the increasing human population, has increased the demand for detergent use, resulting in increased waste from household and laundry services. This activity causes the accumulation of LAS concentrations in water [6][7][8]. LAS Toxicity Tests have also been carried out on Cere Fish, which acts as a bioindicator of water quality. The results show that the lethal toxicity value for 96 hours of LAS is 6,83 mg/L [3]. This Concentration is included in a moderately toxic substance. Extensive use causes the accumulation of LAS concentrations in water because LAS needs almost 9 days to biodegrade [8][9][10]. Severe environmental and public health consequences occur when untreated wastewater is discharged into the water and soil. In the aquatic environment, LAS breaks down the protective mucus layer of fish, which shields them from parasites and bacteria [11]. LAS and other anionic surfactants help pepticides adsorption by aquatic organisms by reducing the surface tension of water and increasing eutrophication by making pollution more soluble. For humans, the impact of LAS is disruption of the endocrine system, skin irritation and respiratory problems [11]. So, in many studies on the development of research, they observed how to reduce LAS levels before release in the environment, such as using adsorbents, such as magnetite and eggshell.

The study of LAS adsorption using magnetite was carried out in previous research, where magnetite synthesis was studied to observe the adsorption of LAS. Adsorption of LAS by magnetite synthesis is influenced by solution concentration and ph. While the optimum conditions were reached, the optimum adsorption of LAS was obtained. Increasing the concentration of the solution will increase the possibility of effective collisions, which will accelerate the reaction rate. So that mass of adsorbent becomes important in LAS adsorption [9]. Apart from magnetite, recycling waste materials as water adsorbents has gained attention, for

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example, eggshells [10][12][13][14]. The particle size of the adsorbent did not influence much at the removal of surfactant. As the mass adsorbent increases, the removal efficiency increases [13]. LAS adsorption with variations in eggshell adsorbent particle size showed optimum absorption at 120 mesh size in contact time at 90 minutes, resulting in a percent reduction of 35.67%. Apart from LAS, egg shells can also reduce BOD and COD by around 80% and 74.5%, respectively, in laundry waste [14]. The increase in concentration of LAS in wastewater indicates high concentrations of BOD and COD due to LAS, which causes eutrophication. The effectiveness of LAS absorption by eggshell waste will increase in acidic solution conditions compared to alkaline conditions; this is due to the influence of the functional groups of LAS. The adsorbent's particle size does not significantly influence the absorption process; using particle sizes of 50, 100, and 140 mesh only makes a slight difference in the % efficiency. However, for the adsorbent mass, the greater the adsorbent mass used, the greater the absorption efficiency, with variations in the use of adsorbent mass, the largest adsorption capacity is produced, namely 0.32 mg/g [13].

Based on the background above, this research aims to thermally activate eggshells to increase the efficiency of reducing LAS levels and determine the optimum conditions for eggshell adsorbent mass and variations in solution volume to increase the adsorption capacity of eggshells for LAS absorption. The resulting eggshell adsorbent will be characterized based on SNI 06-3730-1995 to improve the quality of LAS absorption by the adsorbent. Determination of LAS levels in solution was carried out using the MBAS (Methylene Blue Active Surfactant) method based on SNI 06-6989.51-2005. The development of this method will likely increase the efficiency of eggshells' LAS absorption

Research Methods

Tools and Materials

The materials used in this research were egg shells as the main adsorbent, laundry waste as the research object, linear alkylbenzene sulfonate p.a as a standard for measurement with a UV-Vis spectrophotometer, chloroform p.a (Merck), and isopropyl alcohol p.a (Merck), sulfuric acid p.a (Merck), methylene blue p.a (Merck), phenolphthalein p.a (Merck) as reagents used in the MBAS method, sodium thiosulfate solution p.a (Merck) starch p.a (Merck), iodine p.a (Merck) and potassium iodide p.a (Merck) as reagents used to determine the quality of adsorbents using gravimetric and titration methods.

The tools used are a UV-Vis spectrophotometer (Shimadzu) for analyzing LAS levels, a series of titration tools for adsorbent characterization, a 60 mesh filter, an oven, a furnace, and a series of glassware used to make eggshell adsorbent.

Method

Making Eggshell Adsorbent

Chicken egg shells are washed with water until clean, soaked in hot water, dried in the sun, and ground using a blender. After the eggshell powder is finely sieved with a 60 mesh sieve to make it homogeneous, the eggshell powder is heated in the oven for 1 hour at a temperature of 110 ºC and carbonised in the furnace at 600°C for 2 hours until completely ash in colour. The results were cooled in a desiccator for 15 minutes. Characterization of the resulting adsorbent was based on SNI 06-3730-1995, such as moisture and ash content, fixed carbon, and volatile substances, using the gravimetric method and determining the iodine value using the iodometric titration method.

Determination of adsorbent mass conditions and optimum sample volume for LAS absorption in laundry waste

Wastewater samples were put into beakers with varying volumes of 50 ml, 100 ml, 150 ml, and 200 ml. Then, activated carbon was added as an adsorbent with mass variations of 4 grams, 8 grams, 12 grams, and 16 grams. Detergent wastewater and activated carbon were stirred using a magnetic stirrer at 400 rpm for 75 minutes. The treatment results were filtered, and then the filtrate was analyzed using the MBAS method to determine the level of linear alkylbenzene sulfonate.

Determination of Linear Alkylbenzene Sulfonate Levels in Laundry Waste

Filtrate was analyzed using the MBAS method based on SNI 06-6989.51-2005. Next, the absorbance reading of the diluted $CHCl_3$ layer was carried out using a UV-Vis spectrophotometer at a wavelength of 652 nm, and the same thing was done to determine the blank. Determination of anionic surfactant levels using the MBAS method was carried out on laundry waste samples before and after processing with activated carbon so that the adsorption capacity and LAS reduction efficiency using eggshell activated carbon and the type of adsorption isotherm that occurred could be determined.

Results and Discussion

This research aims to determine the optimum sample mass and volume ratio for LAS absorption using eggshells as adsorbents. Eggshell and eggshell membrane adsorbents are widely used as biosorbents, which have the potential to reduce dyes, heavy metals, including Cd (II), Pb (II), and Cr (III) in waters, as well as metals. This is because the main content of eggshells is $CaCO_3$, which is polar, allowing interactions to form divalent bonds with metal ions between metal ions (M^{2+}) [15][16]. Apart from that, the eggshell membrane has many functional groups, one of which produces a positive charge. These amino acid groups make the eggshell able to absorb various compounds, such as surfactants and dyes [15][17][18]. Literature survey reveals that eggshell and eggshell membrane, naturally or modified, give good results as an adsorbent. This is the basis for choosing eggshell adsorbent for LAS absorption.

Characterisation of Egshell Adsorbent

The preparation of eggshell adsorbent is carried out through several stages, including washing, grinding, filtering, and activation.

The washing stage aims to remove impurities on the eggshell. Grinding and screening up to 60 mesh are carried out to homogenize and reduce the particle size so that the surface area of the adsorbent becomes larger. Smaller particle size results in higher intermolecular energy, which increases the surface area. The large surface area of the adsorbent increases the possibility of contact between the adsorbate and the adsorbent pores, which will affect the adsorption efficiency.

The egg shells are heated at 110 ^{0}C for 1 hour in the oven and then calcined at 600^{0}C. Heating at a temperature of 110 ^{0}C does not cause physical changes because it is only intended to remove impurities that are still in the eggshell, so that it will inhibit the adsorbate absorption process. When heated to a higher temperature, namely 600^{0}C, a calcination process occurs, which causes the release of carbon dioxide (CO_2) and the formation of calcium oxide (CaO), which is the main component of $CaCO_3$ [19][20]. The higher calcination temperature, the more $CaCO_3$ is decomposed into CaO, as shown in the reaction below [15]:

$$CaCO_3 \rightarrow CaO + CO_2$$

The results of the calcination process from the eggshell adsorbent reduce the water content and release CO_2 gas, reducing the mass weight of the adsorbent produced. CO_2 from the decomposition of $CaCO_3$ in eggshells can be used to activate substances and produce activated carbon [19].

After thermal activation, the eggshell adsorbent was characterized by determining its water, ash, and volatile substance content using the gravimetric method and its iodine number using iodometric titration. The results of eggshell adsorbent characterization are shown in Table 1.

Table 1. Results of determining the content of water, ash, volatile substances, pure carbon, and iodine value in eggshell adsorbents

Parameter	Research result	SNI 06-3730-1995
Moisture content	0.15%	Max. 15 %
Ash content	6.69%	Max. 10%
Volatile substances	5.49%	Max. 25%
Fixed carbon	80.79%	Min. 65%
Iod number	838.34%	Min. 750 mg/g

Based on Table 1, all test parameters meet the quality requirements for activated carbon by SNI 06-3730-1995. Testing the water content parameters shows that if the moisture content exceeds the maximum limit, the adsorption capacity will decrease due to the active carbon surface pores being covered by water molecules, which will prevent the adsorbate from entering. The research results show that the water content of 0.15% is far below the required maximum limit of 15%, meaning the quality of the activated adsorbent produced is excellent. Similar to ash content, the ash content parameter shows the content of inorganic compounds in activated carbon, so that if it exceeds the maximum requirement, it means that there are more inorganic compounds contained in activated carbon, which can cover the surface pores, consequently reducing the absorption capacity of activated carbon [21]. In this study, the ash content was below 10%, which shows the ability of the activated charcoal to be produced.

The volatile substance test parameter indicates the content of non-carbon compounds. If it exceeds the requirements, the presence of non-carbon compounds is large enough to cover the surface of the activated charcoal, causing a decrease in the adsorption ability of the activated charcoal. In this study, the content of volatile substances was below 25%, which shows that the activated charcoal produced has good adsorption capabilities. Determining the pure carbon parameter shows the amount of carbon in activated charcoal; the lower the carbon content, the more carbon reacts with water vapour to produce CO and CO_2 gas [14]. The higher the pure carbon produced, the better the activated charcoal produced. The last parameter tested was the iodine number. The high or low iodine number produced shows whether the adsorption ability of activated charcoal is good or not for adsorbing molecules. In this research, the iodine number produced was 838.34 mg/g, meaning that the activated charcoal produced had good adsorption capacity for adsorbate molecules.

Determination of LAS wavelength using a UV-Vis spectrophotometer UV-Vis

The wavelength obtained is 652.18 nm, as shown in Figure 1.

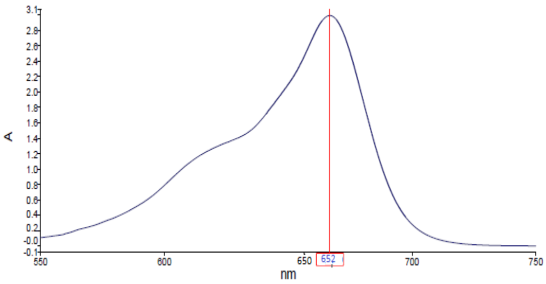

Figure 1. Maximum wavelength of the LAS standard by using a UV-Vis Spectrophotometer

Determining the LAS wavelength is needed to determine the maximum absorbance of LAS as read by the UV-Vis spectrophotometer, so that the maximum standard solution reading will minimize errors in the measurement units. The maximum LAS wavelength in previous research was 653.6 nm [15]. Wavelength shifts may occur due to differences in solvents, analysts, and instruments. After the maximum LAS wavelength is determined, a standard LAS calibration curve is created using a series of standards measured at 652,18 nm wavelength.

Preparation of Calibration Curve

A calibration curve is prepared to ensure the linear relationship between absorbance and concentration, expressed in the regression equation. The higher the concentration, the higher the absorbance, as shown in Figure 2. The LAS standard calibration curve was obtained by making LAS standard solutions with concentrations of 0, 0.4, 0.8, 1.2, 1.6, and 2 ppm and measuring at a wavelength of 652.18 nm.

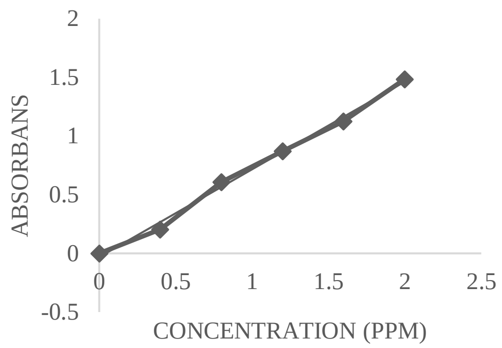

Figure 2. LAS calibration curve at a maximum wavelength of 652.18 nm using a UV-Vis spectrophotometer

Based on the measurement results, a calibration curve was obtained with the equation y = 0.7473x-0.0316. The correlation coefficient (r) = 0.9972. This data shows that the linearity of the relationship between the concentrations of standard solutions meets the acceptance limit, close to 1, so the resulting line equation can be used to determine LAS levels.

Effect of adsorbent mass and sample volume on eggshell in reducing the concentration of LAS

LAS levels before the adsorption process were measured at levels of 11,203 ppm. This amount is still above the quality standard for liquid waste, which in the Decree of the Governor of Region I of West Java No. 6 of 1999 states that the maximum level of LAS in liquid waste is 5 ppm. After adsorption was carried out using eggshell adsorbent with adsorbent mass variations of 4, 8, 12, and 16 grams and volume variations of 50, 100, 150, and 200 mL, there was a decrease in LAS levels, in several variations, the LAS levels had met the LAS quality standards in liquid waste as stated in shown in Table 2.

Table 2. Reduction of LAS levels (ppm) before and after adsorption using eggshell adsorbent with variations in adsorbent mass and sample volume

Volume	Adsorbent Mass (gram)			
Sample (ml)	4	8	12	16
Blanko	11,203			
50	6.5570	4.7360	3.376	3.9385
100	7.7010	5.2235	3.712	4.3970
150	8.0930	6.6945	4.388	4.4595
200	8.6360	6.0820	4.759	4.5200

Based on Table 2, the concentration of LAS before and after treatment by eggshell adsorbent gives different results. The higher eggshell mass gives a lower concentration of LAS in the sample. On the other hand, the highest mass of eggshell, which is 16 grams, gives the greatest LAS levels than 12 grams. It may be that the eggshell adsorbent had met the optimum condition. This is because the greater the adsorbent used, the greater the number of particles and surface area, causing the number of LAS binding sites to increase, so that the adsorption efficiency increases. This shows that eggshells have the ability as an adsorbent to absorb LAS. The efficiency of reducing LAS levels after the adsorption process using eggshells is shown in Figure 3.

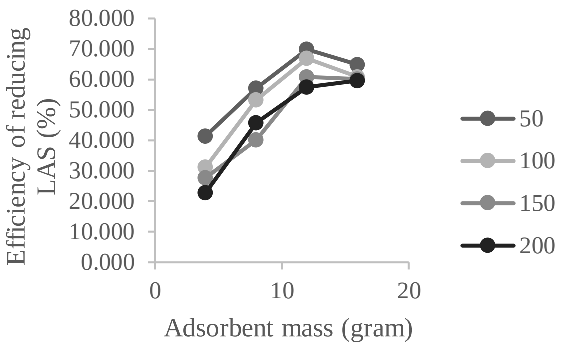

Figure 3. The efficiency of reducing LAS levels after adsorption with eggshell adsorbent, with variations in adsorbent mass and sample volume

Based on Figure 3, it can be seen that the optimum amount of LAS successfully absorbed by the eggshell adsorbent was under the condition that the adsorbent mass was 12 grams and the sample volume was 50 ml, namely 69.86%. This is because the more significant the mass of the adsorbent used, the greater the surface area of the active side carboxyl, sulfate, and amine, which will increase the opportunity for the adsorbate to bind to the surface of the adsorbent. However, there are conditions when the adsorbent is saturated, so the desorption process will occur. This desorption process occurs because the adsorption process occurs reversibly, where the adsorbate is not firmly bound to the surface of the adsorbent, so it is easily separated from the surface. The stirring factor also causes the adsorbate that has been absorbed to be rereleased from the surface of the adsorbent [14] . This can be seen in the mass condition of 16 grams, where there is a decrease in LAS adsorption. This is also supported by a study of the adsorption capacity of egg shells in Figure 4.

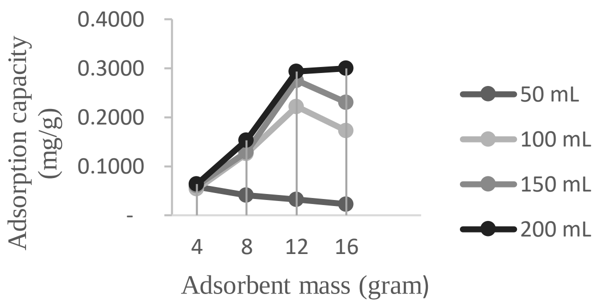

Figure 4. Effect of adsorbent mass and sample volume on eggshell adsorption capacity

Based on Figure 4. The optimum adsorption capacity is 0.2997 mg/g at an adsorbent mass of 16 grams and a sample volume of 200 ml. The tendency of the data in the figure above is that the higher the adsorbent mass produced, the greater the adsorption capacity.

Determination LAS adsorption isotherm model

This research also determined the LAS adsorption isotherm model using the Freundlich and Langmuir isotherm models. As shown in Figures 5 and 6.

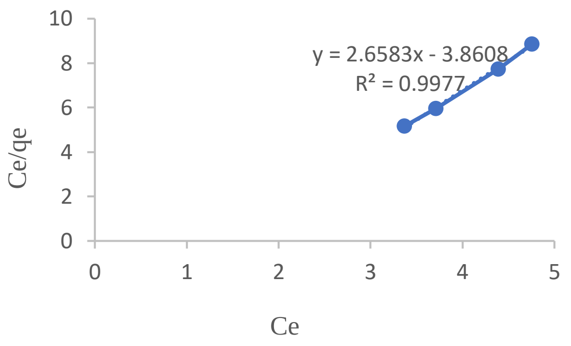

Figure 5. Langmuir Isotherm Equation

The Langmuir isotherm model gives the coefficient of determination close to 1 based on Figure 5. It means the adsorption process, which follows the Langmuir isotherm, was reversible or occurred physically.

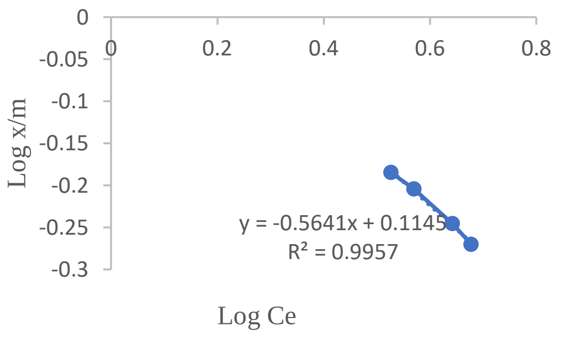

Figure 6. Freundlich Isotherm Equation

Testing of the Langmuir adsorption equation and the Freundlich adsorption equation was proven by a good linearization graph with a coefficient of determination $R^2 \geq 0.9$ (close to 1). From the figure, it can be seen that the adsorption equation for anionic surfactants by activated carbon more closely satisfies the Langmuir adsorption equation with an R^2 value of 0.9977, which is greater than the R^2 of the Freundlich isotherm equation. These results indicate that the adsorption process that occurs is chemisorption and there is a single layer of adsorbed material (LAS) on the surface of the adsorbent, which is homogeneous at constant temperature [22]. However, because the adsorption also satisfies the Freundlich equation, which is the bonds formed are physical bonds with more than one surface. The pores in activated carbon are heterogeneous, so that LAS is adsorbed on the surface of the adsorbent to form a multilayer.

Conclusion

Based on the results obtained, it can be concluded that the optimum conditions for reducing LAS (Linear Alkylbenzene Sulfonate) levels using the tested adsorbent were achieved at an adsorbent mass of 12 grams and a sample volume of 50 ml. Under these conditions, the adsorbent demonstrated its highest efficiency, successfully reducing LAS concentration by 69.86%. This significant reduction indicates that the adsorbent has a promising potential for application in wastewater treatment, particularly in removing surfactant-type organic pollutants under optimized parameters.

Author's Contribution

Inggis Pinarti: Designing the research, conducting data collection, Responsible for the entire research process, including planning, implementation, reporting and interpretation of results, and also refining manuscript contents. Fitri Noorbilqis: acted as research assistant and assisted in the revision of the writing. Sani Widyastuti Pratiwi: Contributed to research methodology, also edited the manuscript. Ratna Nurmalasari: acted as research implementer and was responsible for analysing the results.

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