Jurnal Pijar MIPA
https://jurnalfkip.unram.ac.id/index.php/JPM

ISSN 1907-1744 (Print)
ISSN 2460-1500 (Online)

Utilization of Chicken Egg Shells as Adsorbents for Indigosol Blue Dyes with Variations of Activators

Tuti Rustiana, Mira Aprilani*, Bella Safitri, Luthfiah Sekar Wanti

Departement of Chemistry, Sekolah Tinggi Analis Bakti Asih, Bandung, Indonesia
*e-mail: miraaprilani2014@gmail.com

Received: December 27, 2024. Accepted: January 19, 2025. Published: January 28, 2025

Abstract: The textile industry still has serious problems in managing waste disposal that causes environmental pollution, including the batik factory industry. Many studies have been conducted to overcome ecological damage by reducing the levels of dyes contained in textile industry waste. Apart from textile industry waste, household waste, especially egg shells, is very high. One alternative method for processing batik factory liquid waste is by adsorption using biosorbent, which is considered cheaper and easier to obtain. An example of a biosorbent is an eggshell. This study includes the characterization of eggshells activated by NaOH and HCl for Indigosol Blue adsorption, morphological analysis of eggshells with SEM and EDS, wavelength determination, calibration curve, and adsorption capacity. The morphology of the eggshell shows a porous surface with high levels of calcium, oxygen, and carbon, indicating that the main compound forming the eggshell is $CaCO_3$. The determination of maximum wavelength and adsorption capacity is done using the UV-Vis spectrophotometry method. The maximum wavelength is 530 nanometers, and an R2 value of 0.9596 was obtained from the regression curve equation. Characterization according to SNI eggshell before activation water content: 0.34%, ash content: 0.25%, iodine absorption: 326.552mg/gram and after activation with NaOH water content is 1.34%, ash content: 0.39%, iodine absorption: 403.015mg/gram. The optimal concentration of processed egg shells for indigosol blue adsorbent is 2% egg shells, the largest in this study as an adsorbent. It turns out that adsorption is still ongoing. The optimum type of activator for Indigosol Blue absorption is 100Mesh dry eggshell powder from NaOH activation.

Keywords: Adsorption; Eggshells; Indigosol Blue.

Introduction

The textile industry in Indonesia is growing rapidly every year, which benefits the community but hurts the environment because of the waste produced, such as dye waste from dyeing batik cloth [1]. In the batik dyeing process, only about 45% of the dye sticks to the batik cloth; the rest becomes waste during washing. Commonly used dyes are synthetic indigosol blue dyes that are toxic and can pollute non-biodegradable water [2].

The batik industry generally uses synthetic dyes because the resulting colors are more durable, long-lasting, cheap, and diverse. Naphthol black blue (NBB) and indigosol blue (IB) are commonly used synthetic dyes [2].

Indigosol blue has molecular bonds -NH and C=C, which are not easily destroyed either through chemical treatment or photolysis; therefore, if the waste containing this dye is disposed of into the environment, it will have a negative impact with specific concentrations such as a concentrated cloudy color and unpleasant odor. Thus, dye waste treatment must be done before being discharged into the environment [3].

Indigosol Blue 04-B is a synthetic dye often used in batik fabrics because this dye provides good color but this dye is difficult to decompose. This hurts the environment because it produces waste. For this reason, it is necessary to process the disposal of this dye before it is discharged into the wastewater [4], [5], [6].

Efforts to handle batik factory liquid waste are the same as those to handle textile waste in general, involving chemical, physical, and biological processing.[7] The widely used processes include absorption methods (adsorption), coagulation, sedimentation, and biological processing using activated sludge [8], [9].

Adsorption is an attraction and accumulation of gaseous or liquid substance leading to the formation of molecular (atoms or ions) layers on the surface of another solid material. The adsorbate is the substance in the adsorbed state, and the substance to be adsorbed (before it is on the surface) is the adsorption or adsorptive. The material onto which adsorption curry out is called the adsorbent [10].

The adsorbent commonly used is activated carbon; the resulting adsorption capacity is due to its high surface area and exchange capacity, but activated carbon is also expensive. The abundance and availability of agricultural by-products make it a good source of raw materials for natural adsorbents. Rice bran, a farming waste, has been reported to be a promising adsorbent for many organic and inorganic compounds. Eggshells, abundant in household waste and the food industry, offer low costs for natural adsorbent (biosorbent) [11]. Several studies have examined the potential of eggshells to adsorb different dyes such as reactive yellow 205, Crystal Violet, cationic and anionic dyes, acid-base blue 51 orange from Direct (DR80) and acid (AB25) textile dyes. Eggshells because they have pores and $CaCO_3$ content [12], [13], [14], [15].

How to Cite:
T. Rustiana, M. Aprilani, B. Safitri, and L. S. Wanti, “Utilization of Chicken Egg Shells as Adsorbents for Indigosol Blue Dyes with Variations of Activators”, *J. Pijar.MIPA*, vol. 20, no. 1, pp. 59–64, Jan. 2025. https://doi.org/10.29303/jpm.v20i1.8286

Because of this, a study has been conducted to determine the potential of eggshells to absorb Indigosol Blue batik dye. The study included activation with NaOH and HCl and determination of which activator can be used, characterization (determination of water content, ash content, and iodine absorption) Morphological studies using SEM-EDS and determination of the adsorption capacity of eggshells at various concentrations using UV-Vis spectrophotometry.

Research Methods

Tools and Materials

The tools used are equipment glass laboratory, paper filter, macerator, gravimetry apparatus, analytical balance, oven, tweezers, rack tube reaction, spoon horn, spatel, and UV-Vis spectrophotometry-devices for pounding, filtering, glass devices for making solutions, cuvettes, UV-VIS spectrophotometer. The ingredients used are eggshells, Indigosol blue solutions, NaOH, HCl, and Aquadest.

Procedure

Preparation

A 0.1 N I2 solution was prepared and standardized against Na2S2O3 and K2Cr2O7 to determine the absorption of iodine.

Prepared a 0,1 N of NaOH solution and a 0,1M HCl solution for the activating process of eggshells.

Prepared 2% indigosol blue solution with the addition of concentrated HNO_3 as a sample solution and for the manufacture of standard series solution that is 0.05%, 0.10%, 0.15%, 0.20%, 0.25%. As an Indigosol Blue waste simulation solution, it is a 0.2% Indigosol Blue solution.

Maceration of eggshells

Used chicken eggshell is washed with tap water to remove dirt in the eggshell, dried using an oven for 2 hours at a temperature of 90° C, crushed into small pieces with mortar and pestle, and then pureed with a blender, chicken eggshell powder is sifted with a sieve 100 mesh.

Adsorben activation with HCl

As many as 150 g of eggshells are activated with 150 ml of 0.1 M HCl for 48 hours, then filtered and washed with aquadest until the pH is neutral and dried in the oven until it is dry.

Adsorbent activation with NaOH

150 g NaOH eggshells contained with 0.1 M NaOH for 48 hours. The eggshell is filtered and rewashed with distilled water until the filtrate pH is neutral. The eggshell is then dried and stored in a closed container until used. The characterization of eggshells in the form of determination of ash content and water content is carried out b y gravimetry, and the determination of absorption of iodine is carried out by titrimetric.

Calibration Curve

Indigosol blue solution series with a concentration of 0.05%, 0.10%, 0.15%, 0.20%, 0.25%then measured absorbance and a calibration curve with linear regression.

Determination of the Eggshell Concentration for Indigosol Blue Absorption: Eggshells with a concentration of 0.5%, 1%, 1.5%, and 2% are contacted with 0.2% indigosol blue solution for 90 minutes. Then, the filtrate is filtered and measured for absorption at a maximum wavelength of 530 nm**.**

Result and Discussions

Characterization According to SNI Procedure

Eggshells, before activation, have a water content of 0,34%, ash content of 0,25%, and adsorption capacity toward iodine is 326,552 mg/ gram. Eggshells that NaOH activates have a water content of 1.34%, ash content of 0.39%, absorption capacity toward iodine of 403,015 mg/gram, and after activation with HCl, have a water content of 0,42%, ash content of 0.06%, absorption capacity toward iodine is 115,87 mg/gram.

The eggshell has a porous structure with an attractive surface area to connect with adsorbate, mostly calcium carbonate.

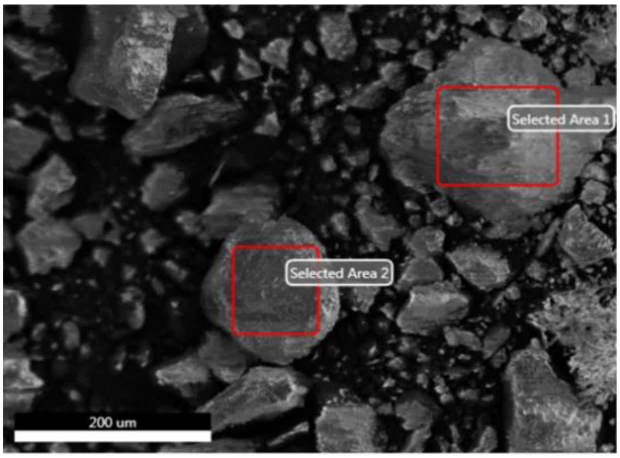

Figure 1. Morphology of Eggshells Before Activated

Figures 1, 2, and 3 present SEM images of eggshells after activation by NaOH and HCl, respectively. It can be seen that the eggshell has an irregular, rough, and porous surface with high granularity, as well as some tubular holes scattered along the surface of the adsorbent.

The eggshell morphology shows a group of coarse particles arranged irregularly and like a sponge, gathering together with microspores scattered on the sample's surface. The small grain and aggregate size can provide a higher specific surface area because the particle size must immediately respond to the surface area. The EDX spectrum shows that the particles mainly contain calcium oxide.

The pores in the eggshell play a role in the exchange of gas between the external and internal environment. This characteristic benefits the adsorption process on solids by increasing the surface area available to interact with pollution.

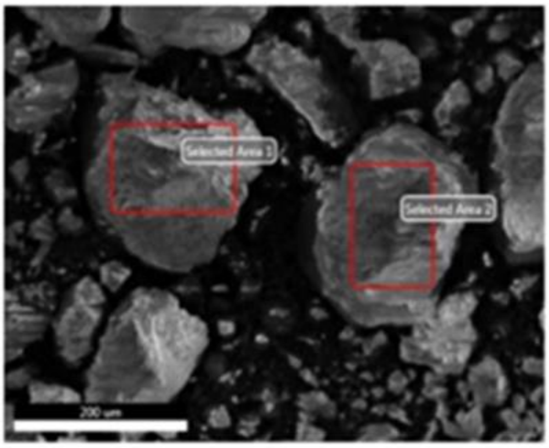

Figure 2. Morphology of Eggshells after Activated by NaOH

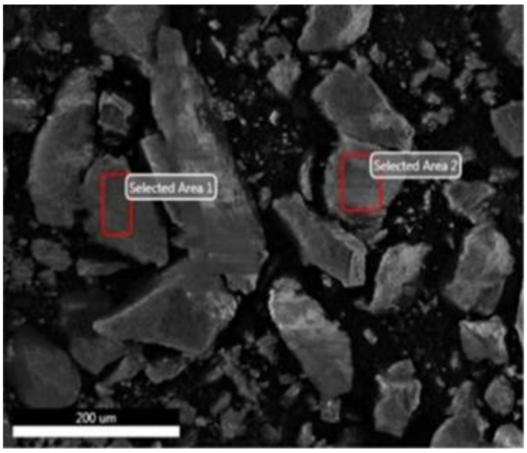

Figure 3. Morphology of Eggshells after Activated by HCl

The surface area of eggshell micropores can be enhanced by calcination to increase their surface area and activation through heat treatment. The enhanced eggshell pores can be helpful in several applications, such as adsorption, solid catalysts for biodiesel production, and more.

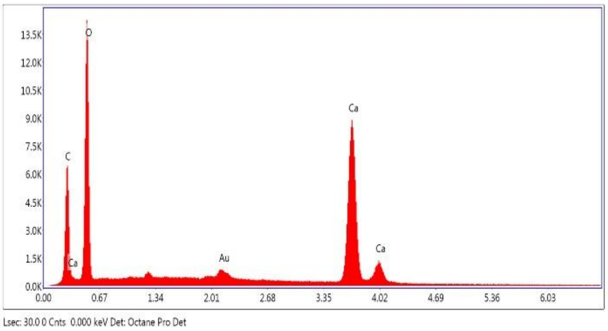

Figure 4. Data on Eggshells Constituents Before Activated

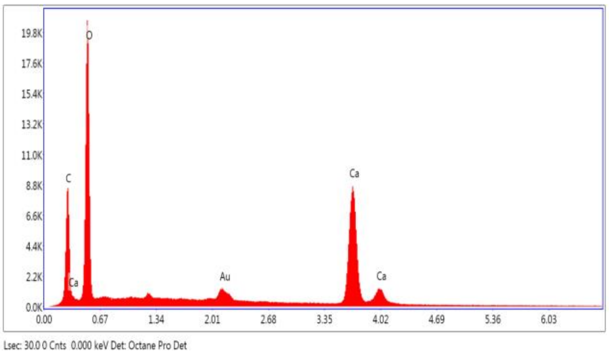

Figure 5. Data on Eggshells Constituents After Activated by NaOH

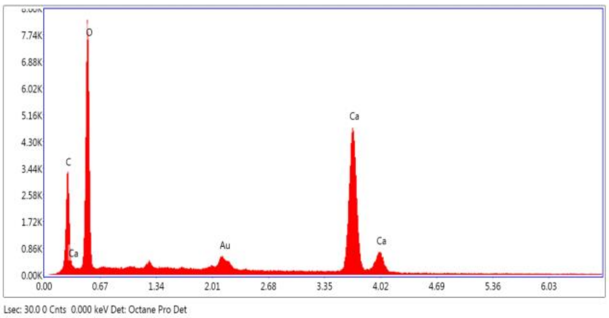

Figure 6. Data on Eggshells Constituents After Activated by HCl

Table 1. Eggshell Constituent Data Chart Before Activation

Element	weight %	Atomic %	Net Int	Error %	Kratio	Z
CK	7.81	14.93	785.36	6.17	0.0576	1.1609
OK	38.86	55.03	2381.38	9.50	0.1138	1.0997
AuM	1.69	0.20	159.01	14.25	0.0187	0.5786
CaK	52.13	29.85	3098.98	3.27	0.4783	0.9101

Table 2. Eggshell Constituent Data Chart After Activated by NaOH

Element	weight %	Atomic %	Net Int	Error %	Kratio	Z
CK	9.05	16.31	1046.67	6.09	0.0658	1.1497
OK	43.98	59.49	3507.89	9.13	0.1435	1.0889
AuM	2.72	0.3	253.27	11.92	0.0256	0.5726
CaK	44.26	23.9	3037.79	3.27	0.401	0.9004

Table 3. Eggshell Constituent Data Chart After Activated by HCl

Element	weight %	Atomic %	Net Int	Error %	Kratio	Z
CK	7.21	13.83	403.28	6.51	0.053	1.164
OK	39.35	56.66	1401.56	9.55	0.12	1.1028
AuM	2.63	0.31	123.05	14.42	0.026	0.5804
CaK	50.81	29.20	1686.88	3.40	0.4666	0.91

The graph at the top shows signal intensity (y-axis) against energy (x-axis) in units of keV. The peaks seen reflect the elements present in the analyzed area. On the graph, there are significant peaks for elements such as O (oxygen), C (carbon), and Ca (calcium), and a slight peak for Au (gold).

EDS analysis shows that eggshells before activation are composed of 38.36 % oxygen (O), 52.13 % calcium (Ca), and 7.81 % carbon (C). EDS analysis shows that eggshells activated by NaOH are composed of 43.13 % oxygen (O), 45.87 % calcium (Ca), and 9,05 % carbon (C). EDS analysis shows that eggshells activated by HCl are composed of 32,23 % oxygen (O), 52.13% calcium (Ca), and 7.21 % carbon (C). The EDS spectrum shows that the solids mainly comprise calcium carbonate ($CaCO_3$).

Carbon present in the elemental composition can make it suitable as catalyst support because carbon can make it capable of building pores on the carbon surface and allow it to be treated as a heterogeneous catalyst. The higher the amount of carbon (i.e., in unactivated eggshell at 7.81%; in HCl-activated eggshell at 6.67%, in NaOH-activated eggshell at 8.61%), the more effective the material is for adsorption. The implication is that chicken eggshells rich in carbon content can be an efficient adsorbent [12], [13], [14].

Determination of Maximum Wavelength

The maximum wavelength of Indigosol Blue was measured by measuring the Indigosol Blue solution at a concentration of 1000 ppm using a UV-Vis spectrophotometer at a wavelength of 400-800 nm. In the image above, the maximum wavelength measurement of Indigosol Blue is 530 nm. Because 530 is the maximum wavelength, it can be used for further measurements of the Indigosol Blue solution.

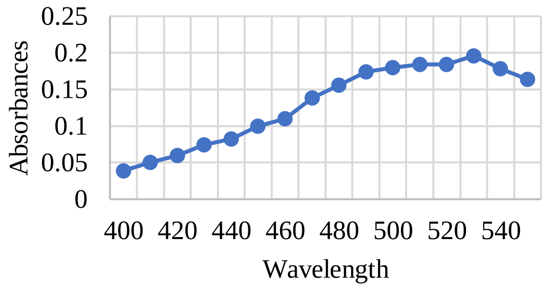

Figure 7. Maximum Wavelength graph

Determination of Calibration Curve

The equation of the line with linear regression is: Y = 1.196X + 0.0276, with R^2 = 0.9596. This calibration curve can then be calculated.

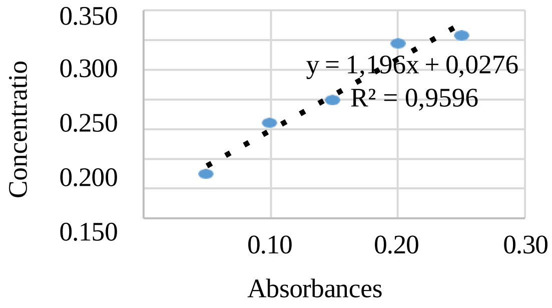

Figure 8. Determination of Curve Calibration

Table 4. Result of Adsorption Treatments

Eggshell Concentration	Adsorbent	Concentration Of Filtrate Colour After Contact With Eggshell	Average (%)	Percentage Of Indigosol Level Reduction
0%	0.294	0.223	0.223	
	0.295	0.224		
	0.294	0.223		
0.5%	0.085	0.048	0.049	78.23
	0.088	0.051		
	0.084	0.047		
1%	0.075	0.040	0.039	82.73
	0.074	0.039		
	0.072	0.037		
1.5%	0.072	0.037	0.042	80.98
	0.082	0.045		
	0.081	0.045		
2%	0.064	0.030	0.035	84.48
	0.075	0.040		
	0.068	0.034		

From Table 1 at 0% concentration, there is no decrease in Indigosol levels because the average value of filtrate color concentration is 0.223 and does not change. The percentage of reduction in Indigosol content is 0%. At a concentration of 0.50%, the average filtrate color concentration decreased to 0.049, and the percentage decrease in Indigosol levels reached 78.23%. At a concentration of 1.00%, the average filtrate color concentration dropped to 0.039, with a reduction in Indigosol of 82.73%. At a concentration of 1.50%, the average filtrate color concentration was 0.042, and the percentage of Indigosol decrease was 80.98%. At a concentration of 2.00%, the average filtrate color concentration reached 0.035, with the highest Indigosol reduction of 84.48%. The higher the eggshell concentration, the more significant the percentage reduction in Indigosol levels. This shows that eggshells have the ability as an adsorbent to absorb Indigosol dyes. A significant decrease was seen, especially at the concentration of 2.00%, where the percentage decrease reached the highest value (84.48%), but statistically, it was not the optimum concentration.

In the results of statistical processing of indigosol adsorption by eggshells that NaOH has activated, it can be seen that the degree of difference is insignificant for absorption in all eggshell concentrations (except the difference between 0.5% and 2%). This means that if the experiment is repeated several times in the same eggshell population, the results of reducing the concentration of indigosol can be higher, the same, or lower. This means that eggshells at concentrations of 1% and 1.5% did not give a statistically significant decrease in indigosol levels. But between 0.5% and 2% showed a significant difference. It is clear that 2% of eggshell levels substantially reduce indigosol concentration than the others. This also indicates that the optimum level of eggshell has not been reached to adsorb indigosol. This is due to many variations.

Eggshells have good adsorption capabilities due to their composition of calcium carbonate ($CaCO_3$), which is also proven by the results of the EDS test.

The amount of water content of the resulting charcoal is very influential on the absorption of active charcoal, if the water content obtained is significant, the adsorption power will decrease because water molecules cover the pores of the active charcoal surface and vice versa if the water content is small, the adsorption power will increase. Determination of the ash content of charcoal is done to determine the amount of mineral content contained in active charcoal. The higher the ash content, the more inorganic compounds will cover the pores of the active charcoal, which can cause a decrease in its absorption capacity [4].

Vat dyes, such as indigo dye, are widely used in the batik industry. Many steps are involved in the batik industry, such as dyeing and the final washing process, during which a large amount of indigo is rinsed and discharged into wastewater. The discharge of dye effluent into water without proper treatment of the effluent can cause environmental pollution. Many types of dyes used in the batik industry cannot be decolorized and are toxic to aquatic living organisms, including plants, animals, and microbes, causing serious long-term health effects. A large amount of effluent containing indigo dye must be treated before being discharged into the environment. Indigo dye is insoluble in water and is considered a poorly soluble. Substance that causes environmental problems, so the effluent must be effectively treated [16], [17], [18].

The initial concentration of the adsorbate can also affect the specific weight adsorption capacity of the adsorbent surface. The increase in the concentration of the absorbent material is carried out when the temperature is confirmed. Still, in some cases, the adsorption may be limited to one layer of the adsorbent solute, so the increase in the concentration of the solute may not increase the adsorption due to the coverage of the crystal surface network of the adsorbent surface. Changes in pH have an important effect on the solution adsorption process due to their impact on the solvent and solvent surface. In the case of surfaces containing polarized or charged positions, the amount of adsorption increases if the surface acquires a charge that exceeds the charge of the charged minute through the influence of the acid function; on the contrary, the amount of adsorption decreases if the surface and the adsorbed minute acquire the same charge. In the case of the fact that the pH of the solution is low, the adsorption rate of the elements can be reduced due to the competition shown by (H^+) ions for effective sites on the surface, in the case of a low solution, the adsorption rate of the elements can be reduced due to the competition of (H^+) ions (OH^-) the influence of the acid function is shown by the competition of the surface of the solvent and the solvent and the solvent on the ions. As a result of these actions negatively or positively affect the sorption process and adsorption isomerization behavior, In the capacity and quantity of substances adsorbed on the surface from one composite to another [19], [20], [21].

Conclusion

Characterization according to SNI eggshell before activation water content: 0.34%, ash content: 0.25%, iodine absorption: 326.552mg/gram and after activation with NaOH becomes water content: 1.34%, ash content: 0.39%, iodine absorption: 403.015mg/gram. The optimum type of activator for Indigosol Blue absorption is 100Mesh dry eggshell powder from NaOH activation. The optimal concentration of processed egg shells for indigosol blue adsorbent, at 2% egg shells, is the largest egg shell concentration in this study as an adsorbent; it turns out that adsorption is still ongoing.

Author's Contribution

Tuti Rustiana: Designing the research and conducting data collection. Responsible for data analysis and supervising the entire research process, including planning, implementation, reporting, and interpretation of results. Mira Aprilani (Corresponding Author): Contributed to developing the theoretical framework and research methodology. Provided critical feedback on draft articles and refined manuscript content, Supported statistical processing and validation of analysis results. Lutfiah Sekar Wanti: Acted as a research implementer and assisted in revising the manuscript. Bella Safitri: Acted as a research implementer and assisted in the revision of the writing.

Acknowledgements

The authors would like to thank the Ministry of Education, Culture, Research and Technology Directorate General of Vocational Education for providing grant funding for this research. Also, the authors would like to thank the Chemistry Laboratory, Chemistry S1 Study Program, Medical Analyst D3 Study Program of Bakti Asih Analyst College Bandung, for their invaluable support during this research. The provision of access to facilities and the generous guidance of experts were essential to complete this research. The authors are very grateful for their collaboration and assistance, without which this research would not have been possible.

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