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Synthesis and Characterization of an Adsorbent from Chicken Eggshell and Its Application for Methyl Orange

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ABSTRACT. Eggshell is a part that protects egg components from various external threats, including chemical, physical, and biological factors. This study aimed to obtain an adsorbent from chicken eggshells using physical and chemical activation techniques. The quality of the eggshell-derived adsorbent was assessed and compared with the Indonesian National Standard (SNI) 1995, with a focus on parameters such as ash content, moisture content, and iodine absorption. The adsorbent obtained was applied to a solution of methyl orange, and Fourier transform infrared spectroscopy (FTIR) analysis was conducted before and after the adsorption process. Furthermore, the study stages include adsorbent preparation, activation, and adsorption. The adsorbent weight for this process was 17 grams, with time intervals of 15, 30, 45, 60, and 75 minutes. The results showed that both physically activated as well as physical and chemical activated adsorbents met the SNI 1995 standard, based on the parameters of water content (1.73% and 2.86%), ash content (8.18% and 6.745), and iodine absorption (761.40 mg/g and 774.09 mg/g). FTIR analysis confirmed the presence of a hydrocarbon and an OH group in the structure before and after adsorption. The optimum methyl orange adsorption capacity was achieved at an adsorbent mass of 17 grams and a contact time of 60 minutes, resulting in removal efficiencies of 20.38% for physical activation and 24.11% for physical and chemical activation.

INTRODUCTION

The ever-increasing global consumption of hen eggs is leading to a proliferation of a large quantity of eggshells (Abatan *et al*., 2020). Eggshells, primarily considered as waste, are often repurposed for poultry feed or organic fertilizer. Only a few small-scale industries use eggshell waste as raw material for handicrafts. In 2019, Indonesia produced 4.753.382.23 tons of eggs from both laying and free-range chickens (Ginting, 2008). Since 10% of eggs consist of shells, the approximate yearly waste generated was 475.338.223 tons (BPS, 2024). Calcium carbonate, the main constituent of eggshell, possesses polar adsorption properties due to its inherent polarity and hexagonal lattice structure, which is punctuated with ions like H^+ and Na (Khirunnisa, 2008).

Among the many low-cost adsorbents available, chicken eggshells have demonstrated considerable potential for dye removal (Haqiqi and Hikmawati, 2019). Eggshell-derived materials can effectively adsorb various dyes, including methylene blue (cationic), bromophenol blue (anionic), and methyl orange (anionic), due to their distinct surface properties (Salman *et al*., 2012). In recent decades, wastewater effluents containing toxic synthetic dyes have become a critical issue due to the rapid development of industries and economies worldwide, posing a significant threat to human health and safety (Yaseen and Scholz, 2019). Over 7×10^5 ton of dyestuffs are produced worldwide annually, and 5 – 20% of dyes are discharged as industrial effluents (Yagub *et al*., 2012).

Dyes are chemical substances that provide color to materials through molecular binding. These compounds are typically ionic and aromatic, featuring aryl rings with delocalized electron systems that contribute to their

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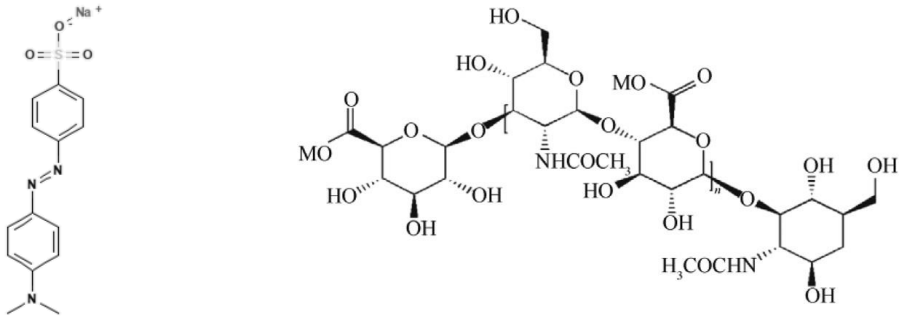
stability and reactivity (Belay and Hayelom, 2014). Methyl orange (MO), an azo dye widely utilized in the textile industry, has the molecular formula $C_{14}H_{14}N_3NaO_3S$, and chicken egg powder with its chemical structure shown in Figure 1. In addition to its function as a dye, methyl orange serves as an acid-base indicator. During azo dye degradation, the breakdown of aromatic rings yields aromatic amines, which are known to be mutagenic to both humans and the environment. Methyl orange exhibits high resistance to light, heat, and oxidizing agents, making it difficult to decompose, thus posing a considerable challenge in wastewater treatment (Jain and Sikarwar, 2008). As a result, effective remediation strategies such as adsorption and biodegradation are necessary to minimize its environmental impact (Mauliddawati and Purnomo, 2014).



(a) (b)

Figure 1. Chemical structure of (a) methyl orange and (b) chicken egg powder.

Previous research by Narwati *et al*. (2021) indicates that chicken eggshells are promising adsorbents. In this study, chicken eggshells are processed to serve as adsorbents, then physically and chemically activated to reduce the concentration of the methyl orange dye. The examination considered variations in contact time. The adsorbent obtained from this process was then characterized for its water content, ash content, and iodine adsorption, in comparison with SNI 06-3730-1995 as the benchmark. To identify the adsorbent structure, Fourier transform infrared spectroscopy (FTIR) is used to obtain spectra before and after adsorption, and the adsorbent capacity is calculated.

RESEARCH METHODS

Adsorbent Preparation

The adsorbent was prepared from 2 kg of chicken eggshells that were thoroughly washed, had their membranes removed, then ground and sieved through a 100-mesh screen (Endecotts Ltd., UK). The resulting powder was rinsed with distilled water (Merck, Germany) until the pH reached neutrality, then oven-dried at 100 °C for 1 h in a laboratory oven (Memmert GmbH, Germany). The dried material was stored in a desiccator (Duran Group, Germany) prior to activation.

Adsorbent Synthesis from Chicken Eggshells

Activation was carried out using two methods: (1) physical activation by reheating the adsorbent at 100 °C and (2) combined physical-chemical activation by immersing the physically activated powder in 0.05 M H_2SO_4. This activation aimed to expand the surface area and create active sites to enhance adsorption efficiency. After activation, the adsorbent yield was 49.91%, and its quality was evaluated in accordance with the SNI 06-3730-1995 standard for activated carbon.

Preparation of Methyl Orange Solution

Five samples of methyl orange solution were prepared in the laboratory, where each sample was made to have a concentration of 20 ppm and a total volume of 50 mL. The preparation process began by carefully weighing 1.0 mg of methyl orange powder using an analytical balance to ensure accuracy, then dissolving it in a small portion of distilled water while stirring until fully homogeneous. Afterward, the solution was transferred into a 50 mL volumetric flask and diluted with distilled water up to the calibration mark to achieve the desired final concentration. This procedure was repeated for each of the five samples in order to obtain solutions with the same concentration and volume, ensuring consistency and reliability for further analysis.

Effect of Contact Time of Physically Activated Adsorbent on Methyl Orange Solution

Eggshell, treated with both physical and chemical activation, served as the adsorbent sample. The optimal adsorbent mass, determined in the prior procedure, was then added to each sample. Activation process including heating the sifted shells in an oven at a temperature of 100 °C for 15 minutes, followed by storage in a desiccator. For combined physical and chemical activation, the physically activated eggshell was immersed in a 0.05 M H_2SO_4 solution and stirred for 2 hours.

Effect of Contact Time of Physically and Chemically Activated Adsorbent on Methyl Orange Solution

Eggshell, treated with both physical and chemical activation, served as the adsorbent sample. Furthermore, a total of five methyl orange solution samples were prepared, each with a concentration of 20 ppm and a volume of 50 mL. The optimal adsorbent mass, determined in the prior procedure, was then added to each sample. Contact time was then varied, starting from 15, 30, 45, 60, and 75 minutes. Stirring was conducted at a speed of 130 rpm, followed by solution separation using filter paper. Subsequently, the resulting solution was analyzed with a UV-Vis Spectrophotometer at λ = 464 nm.

The solution obtained was analyzed using UV-Vis spectrophotometry at the maximum wavelength. Subsequently, the absorbance values were incorporated into the methyl orange standard curve, using the linear regression equation obtained, to determine the actual concentration in the filtrate. The standard curve obtained from UV-Vis spectrophotometry followed the linear regression equation y = 0.0813x + 0.0125, where y represents absorbance and x denotes the actual concentration of methyl orange (Equation 1). This equation was used to accurately quantify the remaining dye concentration after the adsorption process.

$$Adsorption\ (\%) = \frac{(C_o - C_t)}{C_o} \times 100\% \quad (1)$$

Where C_o (mg/L) is the initial concentration of the solute before adsorption, C_t (mg/L) is the concentration of the solute after adsorption at a specific time, and *adsorption* (%) is the percentage of the solute adsorbed by the adsorbent.

Characterization of Eggshell Adsorbent

The sample in powder form was then tested for its water content, ash content, and iodine absorption, based on the SNI 06-3730-1995 standard. The calculations were using Equations 2, 3, and 4.

$$\text{Water content} = \left[\frac{(a-b)}{a}\right] \text{x } 100 \quad (2)$$

In this procedure, the variable a represents the initial mass of the sample, which was carefully measured and recorded before reheating. The variable b, on the other hand, corresponds to the mass of the same sample after it was reheated in an oven at 105 °C for 1 hour. This heating step was performed to remove residual moisture and volatile components, ensuring that the recorded mass after reheating more accurately reflects the sample's dry weight.

$$\text{ash content} = \left[\frac{\text{Ash Weight}}{\text{Sample Weight}}\right] \text{x } 100 \quad (3)$$

The ash weight was determined after the entire sample was fully converted to ash, achieved by ashing in a muffle furnace. During this step, the sample was heated to 800 °C for 2 hours in the furnace, ensuring that all organic matter and volatile components were thoroughly combusted and removed. The remaining residue, consisting solely of inorganic minerals, was then cooled in a desiccator to avoid moisture absorption before being accurately weighed to obtain the final ash weight.

$$Iodium\ adsorption = \frac{\left[V - \left(\frac{T \cdot C_1}{C_2}\right)\right] \times 12.69 \times fp}{W} \quad (4)$$

In this calculation, V represents the volume of the filtrate obtained from the titration process, measured in milliliters (mL). The variable T represents the volume of sodium thiosulfate solution ($Na_2S_2O_3$) used during the titration, expressed in milliliters. The parameter C_1 denotes the normality (concentration) of the sodium thiosulfate solution, while C_2 corresponds to the normality of the iodine (I_2) solution involved in the reaction. The term fp stands for the dilution factor, which accounts for any dilution steps carried out prior to titration, and W indicates the weight of the sample under analysis, expressed in grams. Additionally, the constant 12.69 is used as a conversion factor, representing the amount of iodine (in milligrams) equivalent to 1 mL of 0.1 N sodium thiosulfate

solution. This relationship allows the iodine content of the sample to be expressed in milligrams of iodine per gram of sample, providing a standardized basis for further data interpretation.

RESULTS AND DISCUSSION

Quality of Adsorbent with SNI Standard

The SNI 06-3730-1995 standard serves as a critical benchmark for evaluating the quality of activated carbon, ensuring that products meet minimum requirements for key parameters, such as water content, ash content, and adsorption capacity (Table 1). By comparing actual data from physically and chemically activated adsorbents to these standards, it becomes clear whether the produced materials are suitable for industrial and environmental applications. For instance, both activation methods in the table yield water and ash contents that are significantly below the SNI maximum limits, which are essential for maintaining the structural integrity and adsorption efficiency of the activated carbon.

Table 1. Quality of adsorbent with SNI 06-3730-1995 standard for activated carbon.

Parameter	Physically Activation	Physically and Chemically Activation	SNI (06-3730-1995)
Water Content	1.73%	2.86%	Max 15%
Ash Content	8.18%	6.74%	Max 10%
Absorption of Iodine	761.40 mg/g	774.09 mg/g	Min 750 mg/g

The moisture content of an adsorbent is one of the critical parameters influencing its performance. A lower moisture content generally indicates better adsorbent quality, as excessive water can occupy and block the internal pores of the material, thereby hindering the accessibility of active sites for adsorption. According to the Indonesian National Standard (SNI 06-3730-1995), the maximum allowable moisture content for activated carbon is 15%. In this study, the moisture content values obtained for the physically activated adsorbent (1.73%) and the physically and chemically activated adsorbent (2.86%) were both significantly lower than the maximum standard. This result suggests that both activation methods successfully produced adsorbents with sufficiently low water content, thus maintaining pore availability and ensuring effective adsorption performance.

Ash content is another important factor that determines an adsorbent's efficiency. Excessive ash not only contributes to the non-carbonized inorganic fraction but can also accumulate in the pores, thereby reducing the effective surface area and lowering the adsorption capacity. The SNI 06-3730-1995 sets a maximum ash content limit of 10% for activated carbon. The results obtained in this study demonstrate that both adsorbents, with ash contents of 8.18% (physical activation) and 6.74% (physical and chemical activation), meet the standard requirements. Furthermore, the relatively lower ash content observed in the physically and chemically activated adsorbent suggests that chemical activation may enhance the removal of residual inorganic matter, contributing to a purer carbon matrix with improved structural characteristics.

The iodine adsorption number is widely recognized as a key indicator of the microporous structure and adsorption capacity of activated carbon. A higher iodine value indicates a greater surface area and an enhanced ability to adsorb small-molecular-weight solutes. The minimum requirement of adsorption capacity specified by SNI 06-3730-1995 is 750 mg/g. In this study, both adsorbents exceeded this threshold, with 761.40 mg/g for the physically activated sample and 774.09 mg/g for the physically and chemically activated sample. These results confirm not only compliance with the national standard but also highlight the superior adsorption potential of the chemically assisted activation process, which may introduce additional functional groups and enhance pore development.

Identification of Functional Groups on Adsorbent

In this study, the identification of adsorbent before and after adsorption, based on physical and physicochemical activation, was conducted using an FTIR spectrophotometer (Table 2). The following shows the IR spectra of the adsorbent subjected to physical activation prior to the adsorption process. Analysis of the physically and physicochemically activated eggshell adsorbent, both before and after adsorption, showed the presence of a hydrocarbon and an OH group. However, upon reviewing the four spectra provided, it appears that the eggshell-derived adsorbent subjected to physical and chemical activation did not exhibit significant changes in its functional groups.

Table 2. Functional group analysis based on IR spectra.

Physically Activation		
Before adsorption (cm^{-1})	**After adsorption (cm^{-1})**	**Interpretation**
3459.0	-	O–H
2325.9	2512.2	CO_3^{2-}
1692.2	1625.1	C=O
1481.1	1401.5	Ca–CO
790.2	715.6	Ca–CO
-	872.2	Ca–CO
Physically and Chemically Activation		
Before adsorption (cm^{-1})	**After adsorption (cm^{-1})**	**Interpretation**
-	3533.5	O–H
2124.6	2124.6	CO_3^{2-}
1796.6	1796.6	C=O
1647.5	1625.1	C=O
1401.5	1401.5	CO_3^{2-}
-	1133.1	SO_3H
715.6	715.6	Ca–CO
872.2	872.2	Ca–CO

Based on Table 2 and shown in Figure 2, the FTIR spectra of the physically activated eggshell adsorbent before adsorption exhibited prominent absorption bands at 790.2 cm^{-1} and 1481.1 cm^{-1}, which are characteristic of Ca–CO vibrations associated with carbonate groups in the calcite structure. Following adsorption, these bands shifted to 715.6 cm^{-1} and 1401.5 cm^{-1}. These results remain within the range reported by Zulfikar *et al.* (2013), who stated that there are indications of modifications in the carbonate environment that are likely caused by the interaction between the adsorbent surface and the adsorbate molecules. Such shifts in band positions suggest structural rearrangements or partial substitution of carbonate ions at the surface, which reflects the active role of Ca–CO functional groups in the adsorption mechanism. In addition, the broad absorption band observed at 3459.0 cm^{-1} is attributed to O–H stretching vibrations, indicating the presence of hydroxyl groups and physically adsorbed water molecules. These hydroxyl groups are often involved in hydrogen bonding interactions with adsorbates, thereby contributing to adsorption performance. However, excessive water vapor occupying the pore structure can also restrict access to adsorption sites, highlighting the dual role of O–H groups as both facilitators and potential inhibitors of adsorption, depending on their abundance.

For the physicochemically activated eggshell-derived adsorbent, the FTIR spectra revealed similar characteristic bands of O–H, CO_3^{2-}, and C=O functional groups, consistent with the carbonate-based composition of eggshell-derived adsorbents. Notably, an additional absorption band at 1133.1 cm^{-1} emerged after adsorption, assigned to sulfonic acid (SO_3H) groups. This new feature is attributed to the chemical activation process, which introduces oxygen- and sulfur-containing functionalities on the adsorbent surface. The incorporation of sulfonic acid groups is particularly important, as these groups act as strong acidic sites that can promote stronger interactions with polar adsorbates through hydrogen bonding and electrostatic attraction. The persistence of carbonate-related bands at 715.6 cm^{-1} and 872.2 cm^{-1}, together with the C=O stretching vibrations near 1625 – 1796 cm^{-1}, confirms that the fundamental calcite framework of the eggshell remains intact despite activation and adsorption. This stability is advantageous because it ensures the mechanical and chemical resilience of the adsorbent while simultaneously enabling functional modification of its surface.

The interaction between methyl orange (MO), an anionic dye, and the eggshell adsorbent is primarily governed by electrostatic attraction, hydrogen bonding, and van der Waals forces. Since methyl orange carries a negative charge, its adsorption efficiency depends strongly on the adsorbent's surface charge. Eggshell, primarily composed of calcium carbonate ($CaCO_3$), exhibits a limited number of positively charged sites under neutral to slightly acidic conditions, restricting its ability to attract the negatively charged dye molecules. Studies have shown that at lower pH levels, the surface of eggshell adsorbents can become more positively charged, enhancing electrostatic interactions with anionic dyes (Al Nasir and Mohammed, 2023). However, in the present study, the relatively low adsorption percentage suggests that the activation process did not sufficiently enhance the adsorbent's surface charge to maximize electrostatic attraction. The FTIR analysis confirmed the presence of hydroxyl (–OH) and carbonate (CO_3^{2-}) functional groups, which can interact with methyl orange through hydrogen

bonding and dipole interactions. However, the absence of significant changes in functional groups before and after adsorption suggests that chemical activation did not significantly alter the adsorbent's surface properties, thereby improving MO removal efficiency.

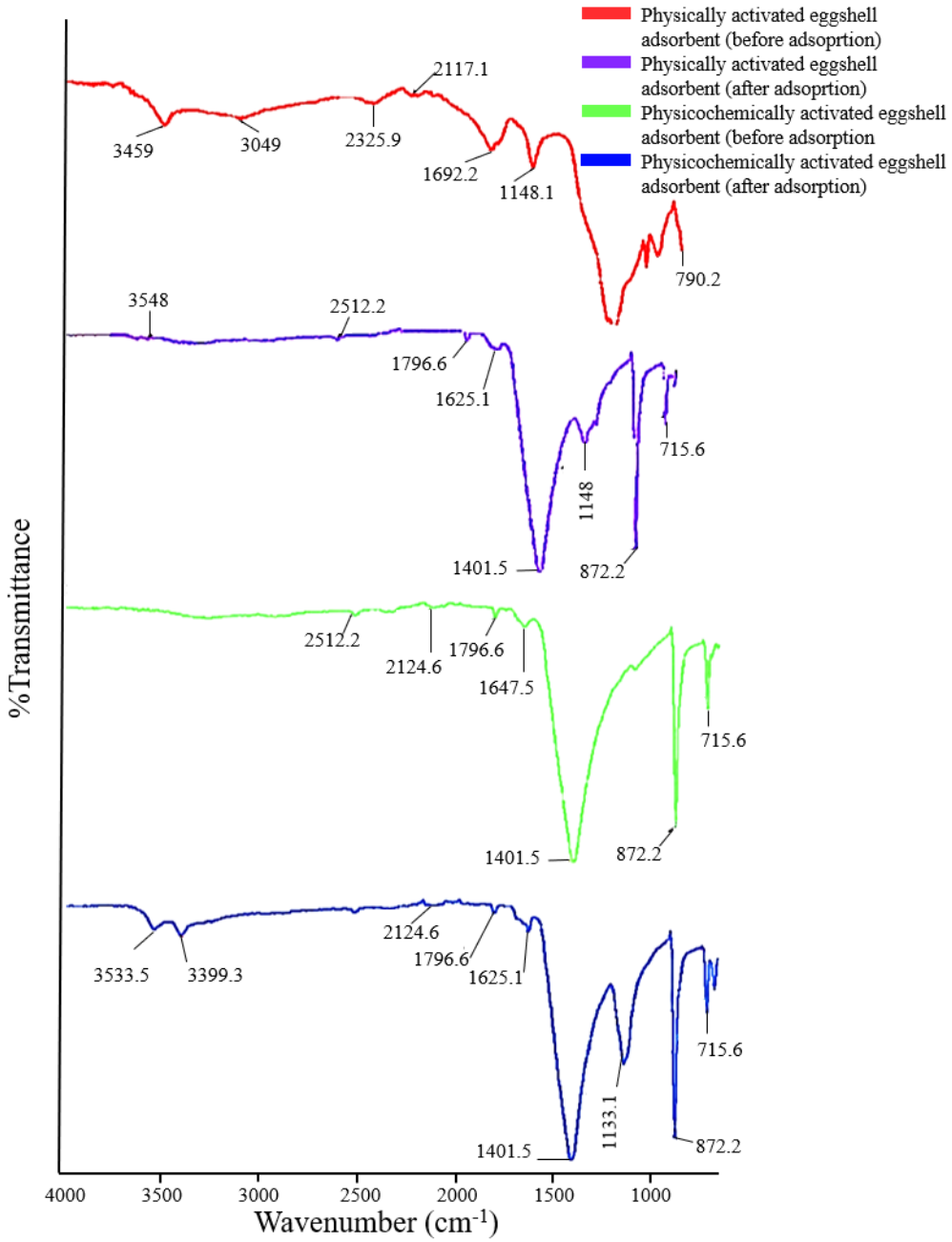


Figure 2. Spectra of adsorbent from eggshell: physically activated eggshell adsorbent before (red line) - after adsorption (purple line), and physicochemically activated eggshell adsorbent before (green line) - after adsorption (blue line).

Effect of Contact Time of Physically Activated Adsorbent on Methyl Orange Solution and Physically and Chemically Activated Adsorbent

In this study, the physicochemically activated eggshell adsorbent reached a maximum adsorption efficiency of 24.11% at 60 minutes (Figure 3), which is slightly higher than the 20.6% removal of methyl orange from a 20 ppm solution reported by Bhaumik *et al*. (2012). The observed improvement may be associated with differences in activation method, as chemical activation can introduce additional functional groups and enhance pore development, thereby increasing the number of active adsorption sites. Nevertheless, both results highlight the potential of eggshell-based materials as effective and low-cost adsorbents, and the variations demonstrate how

activation strategies and experimental conditions can influence adsorption performance. This comparison underscores that the present study complements earlier findings by providing further evidence of the versatility of eggshell waste as an adsorbent material.

The decrease in adsorption efficiency for the physicochemically activated adsorbent at 75 minutes may be attributed to several factors. One possible explanation is saturation of active sites, in which prolonged contact time leads to equilibrium between adsorption and desorption. Additionally, chemical activation may have altered the eggshell surface structure, making it more susceptible to desorption at extended contact times. The introduction of acidic treatment in the physicochemical activation process could have weakened the adsorbent's structural integrity, leading to partial release of previously adsorbed methyl orange molecules. Furthermore, prolonged stirring could contribute to mechanical desorption, thereby reducing overall adsorption efficiency. In contrast, the physically activated adsorbent, which primarily relies on surface area and porosity without significant chemical modifications, demonstrated a more stable adsorption performance, retaining 19.28% adsorption at 75 minutes. This suggests that while physicochemical activation initially enhances adsorption efficiency, its long-term stability may be affected by weaker binding interactions and increased desorption tendencies at extended contact times.

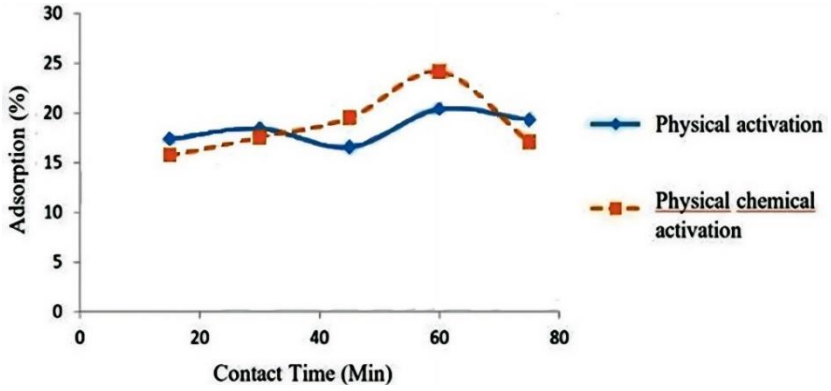


Figure 3. Percent adsorption of physically- and physicochemically activated eggshell adsorbent in various contact times.

In physicochemical activation, the mass increased over the contact time range from 15 to 60 minutes, with the highest adsorption percentage of 24.11% observed at 60 minutes. However, compared to the physically activated adsorbent, the adsorption percentage at 15– 30 minutes was lower. This discrepancy can be attributed to the rapid saturation of adsorption sites when physicochemical activation is applied within this short time frame.

Additionally, desorption may have contributed to reduced adsorption efficiency at extended contact times. According to collision theory, the reaction rate depends on the number of molecular collisions per unit time, and adsorption reaches equilibrium when the rates of adsorption and desorption are balanced (Yusof and Malek, 2009). Beyond the optimal contact time, prolonged stirring and interactions between the dye and the adsorbent surface may induce desorption, thereby decreasing adsorption efficiency (Handayani, 2005).

Overall, the low adsorption efficiency of eggshell adsorbent for methyl orange (24.11%) can be attributed to its limited surface charge enhancement, restricted porosity, and the presence of competing desorption mechanisms at extended contact times. Further modifications, such as surface functionalization with cationic surfactants or pH optimization, may enhance adsorption capacity and make eggshell-derived adsorbents more effective for dye removal applications.

CONCLUSION

The results showed that the physically activated adsorbent had a water content of 1.73%, an ash content of 8.18%, and an iodine adsorption capacity of 761.40 mg/g, while the physicochemically activated adsorbent exhibited 2.86%, 6.74%, and 774.09 mg/g, respectively, all meeting SNI 06-3730-1995 standards. FTIR analysis confirmed preservation of the carbonate structure, with chemical activation introducing SO_3H groups as additional active sites. Minimal changes in functional groups after adsorption indicate that methyl orange removal was mainly governed by electrostatic interactions and hydrogen bonding. Physicochemical activation showed better performance, achieving 24.11% adsorption efficiency compared to 20.38% for physical activation under optimum conditions (60 minutes, 17 g). Longer contact time reduced performance due to desorption. This study highlights

eggshell waste as a low-cost adsorbent for sustainable wastewater treatment. Future work should focus on optimizing activation conditions, pore analysis, real wastewater application, and adsorbent regeneration.

CONFLICT OF INTEREST

There is no conflict of interest in this article.

AUTHOR CONTRIBUTION

ESY: Conceptualization, Methodology, Supervision, and Writing Review and Editing; MM: Investigation, Data Analysis, and Writing Original Draft; EA: Methodology, Validation, and Visualization; WTR: Data Analysis, Software, and Visualization; IO: Investigation and Resources; MDM: Data Collection and Data Curation.

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