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Research Article

Removal of auramine-O dye from aqueous solutions using two natural adsorbents and process optimization using RSM

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ABSTRACT

The purpose of this study is to evaluate the applicability of two bio-adsorbents, tea waste and calcined eggshell, for the removal of auramine-O dye from aqueous solutions using the adsorption process. A comparison between the characterization of FTIR, XRD, and SEM for adsorbents before and after adsorption confirmed the adsorption of auramine-O on them. Effective parameters including initial solution pH, initial auramine-O concentration, adsorbent dosage, contact time, and temperature were optimized using response surface methodology with a central composite design. The highest removal efficiency was obtained under optimal conditions, predicting removal efficiencies of 97.68% for tea waste and 97.50% for calcined eggshell. Experimental results showed removal efficiencies of 96.50% and 96.20% for tea waste and calcined eggshell, respectively. The optimal pH close to a neutral medium and temperatures close to room temperature reduce the cost of scaling up the process. Adsorption isotherms, thermodynamics, and kinetics were also studied, revealing that the adsorption process follows the Langmuir isotherm, is thermodynamically exothermic and spontaneous and is kinetically a function of the pseudo-second-order model.

The adsorption amount of auramine-O on tea waste can vary depending on the type of tea and its processing method. Incomplete decomposition of calcium carbonate to calcium oxide during the calcination of eggshell and the prevention of the formation of a porous structure can affect adsorption efficiency.

Although the optimal conditions for achieving maximum adsorption are specific to these adsorbents and adsorbate, independent research can be conducted to industrialize the adsorption process.

Among the adsorbents in literature to remove auramine-O, NaX nanozeolites and multiwall carbon nanotubes have a higher removal efficiency than tea waste and calcined eggshell. However, they are not natural, abundant, are expensive and their preparation is difficult and costly. Therefore, the adsorbents used in this work are more suitable for removing auramine-O.

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1. Introduction

The expansion of various industries, such as dyeing and textiles has led to severe environmental crises. The introduction of pollutants, such as organic dyes, into water sources has made it challenging for humans and other living organisms to access clean water, poising significant health risks. The presence of organic dyes in wastewater can cause incurable diseases like cancer and genetic mutations, highlighting the importance of their removal from water and wastewater [1,2,3,4,5].

Various methods have been employed to eliminate dye pollutants from effluents, including membrane separation [6,7], flocculation and coagulation [8], photocatalytic degradation [9,10], ozonation [11,12], and adsorption [13,14,15]. Adsorption in particular, is a highly useful and effective process for removing dye pollutants due to its simplicity, cost-effectiveness, availability of effective adsorbents, high pollutant removal efficiency, and eco-friendliness without producing additional pollutants [16,17,18].

Auramine-O (AO) is a low-cost and stable cationic basic dye widely used in industries like dyeing and textiles. It dissolves in water and ethanol, and its proven toxicity and carcinogenicity to humans necessitate its removal from water sources to protect human health and the environment [19,20,21]. The chemical structure of auramine-O is depicted in Figure 1. Given its cationic nature, conducting an isoelectric point pH test for each adsorbent after preparation is essential to identify the pH range where electrostatic attraction between the dye and adsorbent surface is strongest, resulting in higher adsorption efficiency.

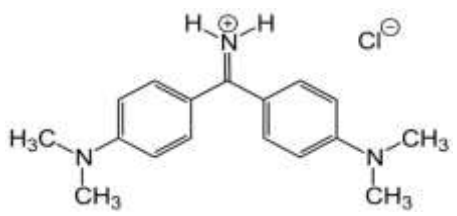

Fig. 1. Chemical structure of auramine-O.

The removal of AO from aqueous systems has been successfully achieved through the adsorption method using various adsorbents such as Psidium guava leaves [22], sesame leaf [23], peanut shells [24], multiwall carbon nanotubes [25], activated carbon loaded with ZnS:Cu nanoparticles [26], macroporous adsorption resins [27], surfactant-modified activated carbon [28], NaX nanozeolites [29], sodium dodecyl sulfate (SDS) functionalized magnetite nanoparticles [30], and Pyracantha Coccinea biosorbent [31].

Research on the adsorption of auramine-O using different adsorbents has shown that NaX nanozeolites and multiwall carbon nanotubes exhibit high removal efficiency. However, these adsorbents are not natural, abundant, and are expensive to prepare. Therefore, in this study, two natural, environmentally friendly, inexpensive, and readily available waste materials were utilized as adsorbents. Their preparation for the adsorption process is simple, cost-effective, and they demonstrate very high adsorption efficiency.

The adsorbents or their derivatives used in this study have been effective in removing various pollutants, such as, fluoride [32], methylene blue [33], Zidovudine ARV drug and phenol [34] using tea waste, as well as, phosphorus [35], phosphates [36], and carbon dioxide [37] using calcined eggshell.

In this study, tea waste and calcined eggshell were used as two effective bio-adsorbents to remove AO from aqueous solutions. The parameters affecting adsorption including initial solution pH, initial AO concentration, adsorbent dosage, contact time, and temperature were optimized using Response Surface Methodology (RSM) with Central Composite Design (CCD). The highest removal efficiency was achieved under optimal conditions. Additionally, adsorption isotherms, thermodynamic and kinetic studies were conducted.

2. Materials and methods

2.1. Materials and equipment

Hydrochloric acid (HCl) and sodium hydroxide (NaOH) were purchased from Merck. Auramine-O ($C_{17}H_{22}ClN_3$, MW=303.83 g/mol, λ_{max}=431 nm) was obtained from Sigma-Aldrich. It was of analytical grade and used without further purification.

The following equipment was utilized: SEM model VEGA3– MIRA II – MIRA III from Czech Republic, XRD model STADIP from Germany, FTIR model IRAffinity-1 from Japan, UV-Vis spectrometer model Dynamica-HALO-DB-20.

2.2. Preparation of the adsorbents

To prepare tea waste as the adsorbent, tea waste from Green Hammer tea in Sri Lanka was washed with distilled water at boiling temperature until the color was completely removed. It was then dried in an oven at 80°C for 6 hours, ground with an electric mill, and passed through a 500 micron sieve. To prepare calcined eggshell adsorbent, eggshells from home consumption were washed, dried in an oven at 100°C for 24 hours, broken into small pieces, heated in a furnace at 800°C for 2 hours, crushed, and passed through a 500-micron sieve. The prepared adsorbents were stored in sealed glass bottles until use.

2.3. Drawing the calibration curve

A stock solution of auramine-O dye with a concentration of 1000 mg/L was prepared. The maximum wavelength was determined using a UV-Vis spectrometer (Dynamica Halo, DB20 model) at 431 nm (Figure 2). Standard solutions with concentrations ranging from 1 to 10 mg/L were prepared and their absorbance was measured to draw the calibration curve. The linearity of the curve within the measured range is shown in Figure 3.

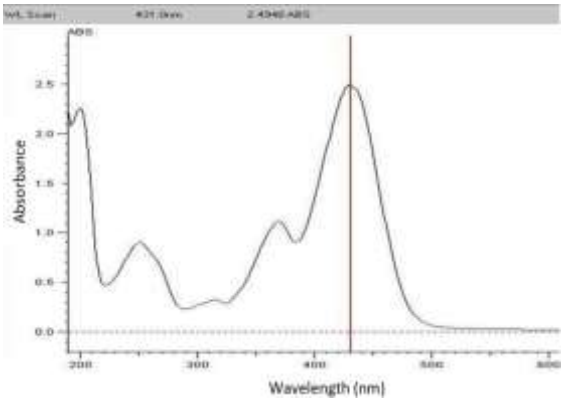

Fig. 2. Scanning wavelength for auramine-O (λ_{max} = 431 nm).

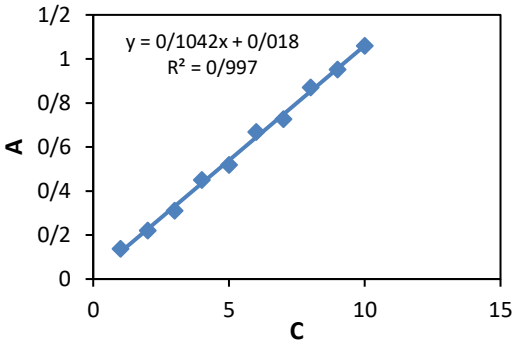

Fig. 3. Calibration curve for auramine-O dye at λ_{max} = 431 nm.

2.4. The determination of Zero point pH

Hydroxide and hydronium ions can affect the adsorption process by altering the surface charge of the adsorbent. There is a pH level at which the surface of the adsorbent becomes electrically neutral, known as the zero point pH or isoelectric point. Below the isoelectric point, the adsorbent carries a positive electrical charge, while above it, the surface charge is negative.

To determine the zero point pH, 15 ml of distilled water was added to 6 separate 100 ml Erlenmeyer flasks. The initial pH of each flask was adjusted within the range of 2-12 using 1 mg L^{-1} hydrochloric acid and sodium hydroxide solutions. Then, 0.1 g of the prepared adsorbent (either tea waste or calcined eggshell) was added to each flask and placed on a shaker for 1 hour. Finally, the final pH values were measured.

According to Figure 4a and b, the zero point pH values for tea waste and calcined eggshell were

found to be 5.5 and 11.3, respectively. At pH levels below and above the isoelectric point, the adsorbent carries positive and negative electrical charges, respectively.

The high isoelectric pH of calcined eggshells can be explained as follows:

Eggshells primarily consist of calcium carbonate, which is transformed into calcium oxide through calcination at 800°C (reaction R1). When this basic oxide partially dissolves in water, it generates hydroxide ions, leading to an increase in the solution's pH (reaction R). [38].

$$CaCO_3 \xrightarrow{\Delta} CaO + CO_2 \quad \text{(R1)}$$

$$CaO + H_2O \rightarrow Ca(OH)_2 \rightarrow Ca^{2+} + 2OH^- \quad \text{(R2)}$$

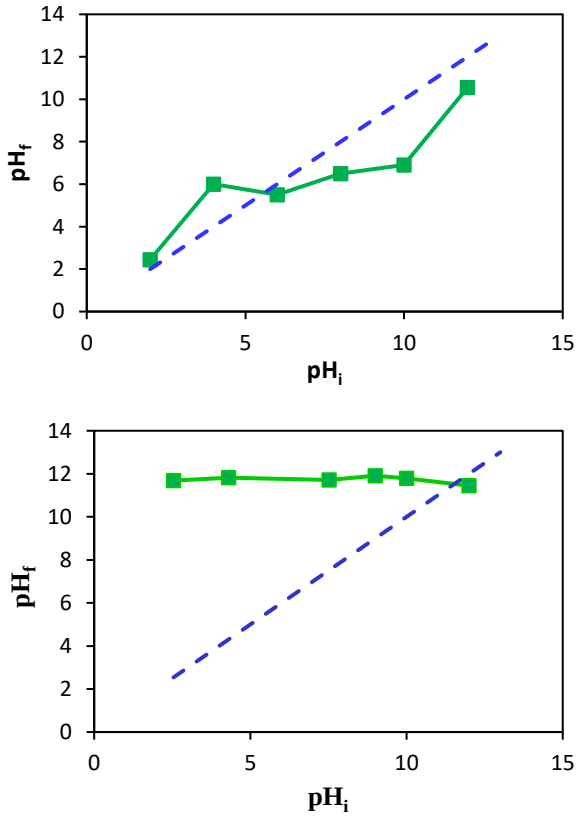

Fig. 4. Diagram of zero point pH values for (a) tea waste and (b) calcined eggshell.

2.5. The removal of auramine-O dye on tea waste and calcined eggshell adsorbents

To remove auramine-O, 50 ml of the dye solution with a concentration of 1-9 mg L^{-1} was taken and poured into an Erlenmeyer flask. Distilled deionized water was used in all adsorption experiments. The pH of the solution was adjusted using 1 mg L^{-1} hydrochloric acid and sodium hydroxide within a pH range of 3-11. Then, a specific amount (0.03 to 0.15 g) of the adsorbent was added and placed on a stirrer for a duration of 15 to 75 minutes at a temperature ranging from 15 to 75°C.

Afterwards, the sample was transferred into a test tube and centrifuged. The concentration of the solution was measured using a UV-Vis spectrophotometer with a maximum wavelength of 431 nm. The removal efficiency (%R) and the adsorption capacity of auramine-O dye were calculated using equations (1) and (2), respectively.

$$\%R = \frac{C_i - C_f}{C_i} \times 100 \quad (1)$$

$$q_e = \frac{v}{m}(C_i - C_f) \quad (2)$$

Here, C_i (mg L^{-1}) is the adsorbate initial concentration, C_f (mg L^{-1}) is the final concentrations, v (L) is the volume of the solution, and m (g) is the mass of the adsorbent.

3. Results and Discussion

3.1. FTIR analysis

The FTIR spectrum of auramine-O dye in Figure 5 contains the following signals: the signals around 3000, 1600, and 1570 cm^{-1} belong to the C-H, C=N, and C=C stretching vibrations, respectively. The peak at about 1370 cm^{-1} is related to the C-H bending vibration. The double signals around 1170 and 1200 cm^{-1} belong to the C-N aliphatic and aromatic vibrations. The peak at 800 cm^{-1} is also attributed to the C-H vibration. As seen in Figures 5 and 6, the peaks introduced in auramine-O structure are observed in the FTIR spectrum of both adsorbents after adsorption, indicating the presence of auramine-O molecules on the surface of both adsorbents. Additionally, the structure of tea waste is much more complex than that of calcined eggshell, making it difficult to distinguish important peaks. However, the structure of calcined eggshell

has a wide peak in the region 3000-3600 cm^{-1}, which is related to the OH strain functional group in $Ca(OH)_2$ and can be seen by interfering with auramine-O peak in its structure after adsorption. Furthermore, the absence of the 870 cm^{-1} peak indicates that there is no calcite in the structure of the adsorbent.

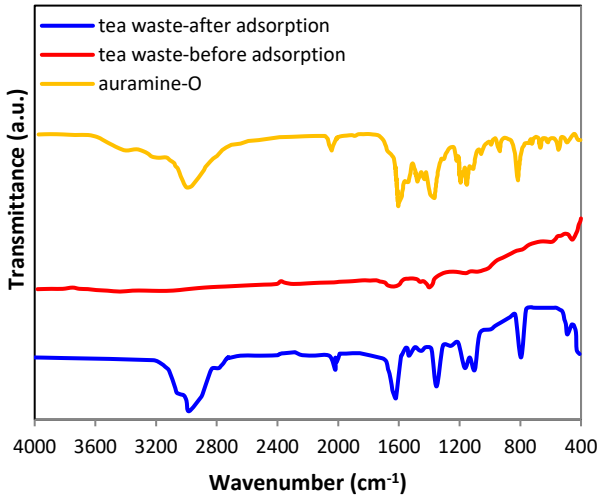

Fig. 5. FTIR spectra of auramine-O, tea waste before and after adsorption of auramine-O on it.

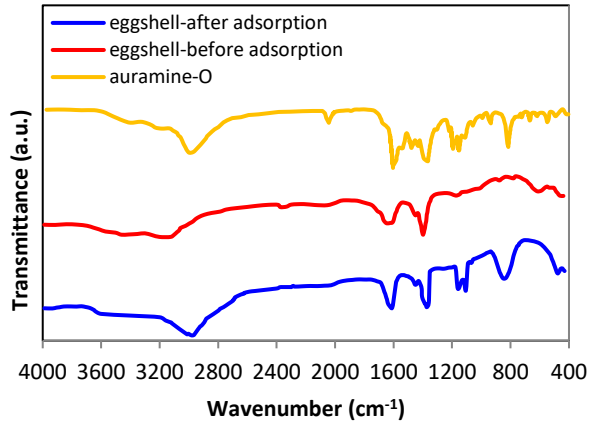

Fig. 6. FTIR spectra of auramine-O, calcined eggshell before and after adsorption of auramine-O on it.

3.2. X-ray diffraction pattern

Figures 7 and 8 display the X-ray diffraction (XRD) patterns of tea waste and calcined eggshell adsorbents before and after the adsorption of auramine-O, respectively. While the peak positions remained unchanged for both adsorbents, a reduction in intensity before and after adsorption suggests that auramine-O was adsorbed onto them. The consistent peak positions indicate that the structure of the adsorbents remained unchanged, while the decrease in intensity suggests the adsorption of other molecules, specifically auramine-O dye molecules [39,40].

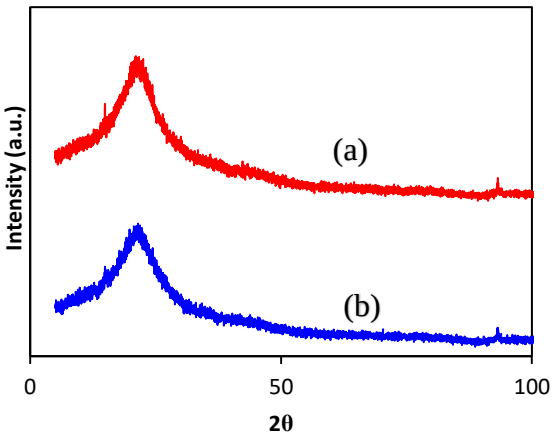

Fig. 7. XRD spectrum of tea waste adsorbent (a) before and (b) after adsorption of auramine-O.

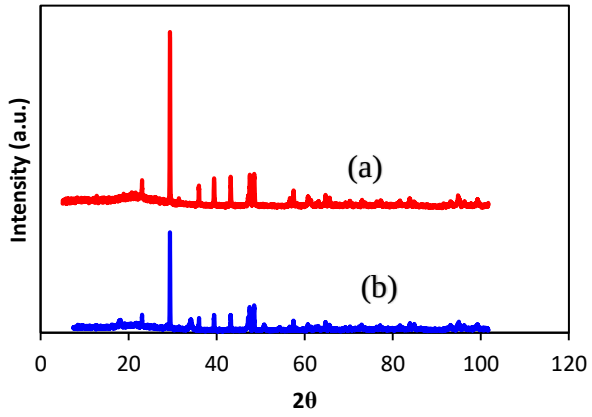

Fig. 8. XRD spectrum of calcined eggshell adsorbent (a) before and (b) after adsorption of auramine-O.

3.3. Scanning Electron Microscope (SEM) images

The morphology of two natural adsorbents, tea waste and calcined eggshell was examined using scanning electron microscope (SEM) analysis. Figures 9 and 10 depict SEM images of tea waste and calcined eggshell before and after the adsorption of auramine-O. It is evident that the surface density of both adsorbents increased significantly after the adsorption of auramine-O, indicating that the pores and surfaces of both adsorbents were filled with

auramine-O molecules confirming the adsorption of dye molecules.

3.4. Investigating the performance of tea waste and calcined eggshell adsorbents to remove auramine-O dye from aqueous solutions

Following the preparation of two natural adsorbents, tea waste and calcined eggshell, they were utilized to remove auramine-O dye from aqueous solutions. Subsequently, effective parameters were optimized using DOE software, specifically the central composite design and the response surface method.

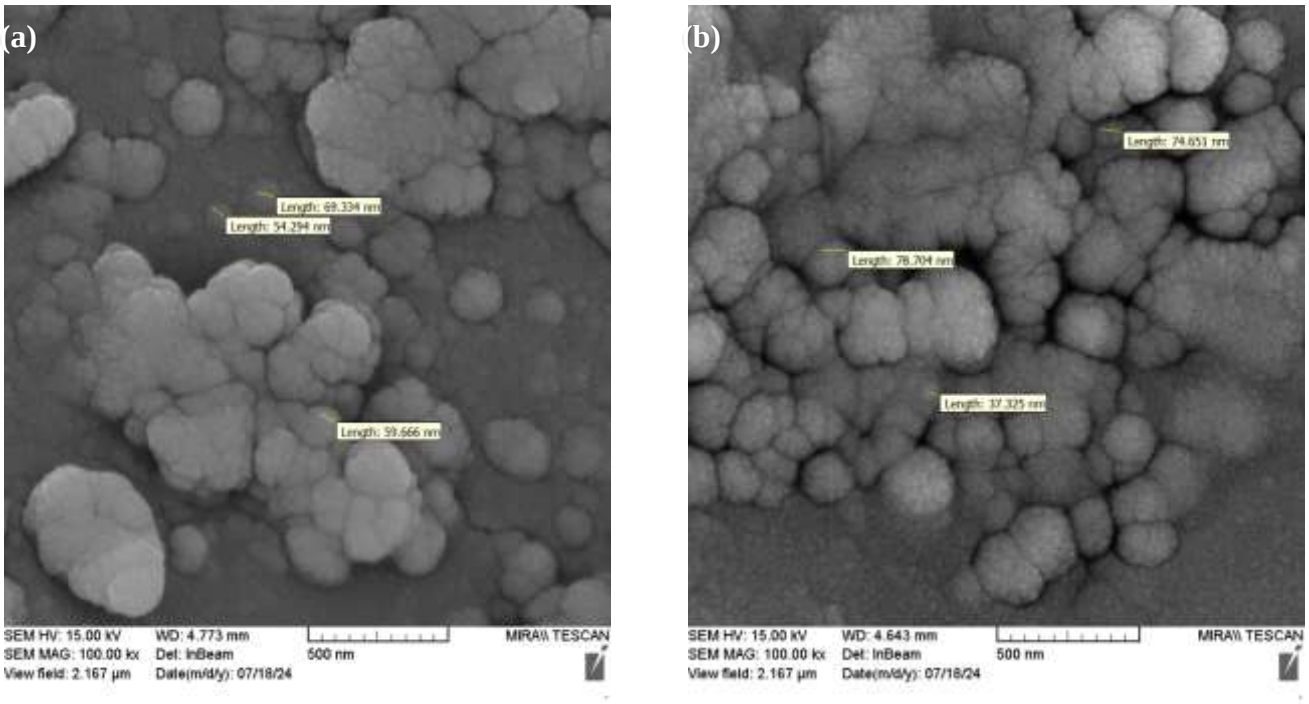

Fig. 9. SEM images of tea waste a) before and b) after adsorption of auramine-O.

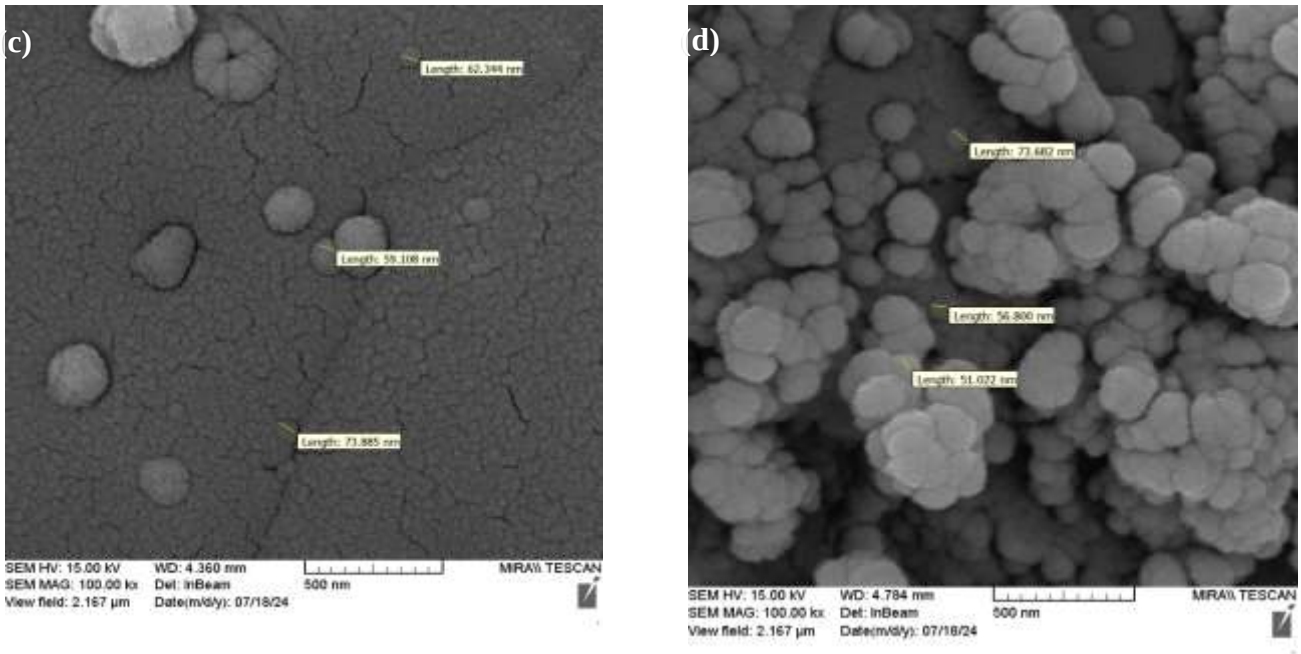

Fig. 10. SEM images of calcined eggshell c) before and d) after adsorption of auramine-O.

3.5. Design of experiments analysis

Design of Experiments (DOE) software was used to investigate parameters affecting the removal of auramine-O dye from aqueous solutions, including the initial pH of the solution, adsorbent dose, initial dye concentration, temperature, and contact time. Based on the RSM, five levels ranging from low level (-2) to high level (+2) were selected by the central composite model. The effective parameter ranges are shown in Table 1.

Table 1. Selected levels for parameters affecting dye removal by DOE software

Parameter	Component	Unit	Applied Level				
			(-2)	(-1)	(0)	(+1)	(+2)
A	pH	-	3	5	7	9	11
B	ads. dose	g	0.03	0.06	0.09	0.12	0.15
C	dye conc.	ppm	1	3	5	7	9
D	Temp.	°C	15	25	35	45	55
E	Time	min	15	30	45	60	75

Tables S1 and S2 in the Supplementary file display the 50 experiments proposed by DOE based on the factors influencing the removal process and their optimization. Following this, adsorption tests were conducted in section 2.5. The removal percentage for each solution was calculated using equation 1 and the results were submitted to DOE to determine the best model with the most suitable equation. Variance analysis tables, regression coefficients, normal charts, actual values versus responses, standard residual values versus responses, and residual values versus predicted values were examined to verify the accuracy and validation of the proposed model.

During this stage, the responses were fitted using four models: linear, factorial, quadratic, and cubic models. The quadratic model was chosen as the best-fitting model for both adsorbents (tea waste and calcined eggshell) due to the highest regression coefficients and p-probability less than 0.05 (Tables S3 and S4 in the Supplementary file).

The results regarding the separate and mutual effects of the effective parameters on the removal process of auramine-O dye on tea waste and calcined eggshell based on the quadratic model are presented in Tables S5 and S6 in the Supplementary file, respectively. For tea waste adsorbent, all data have a P-probability value of less than 0.05 except for DE (mutual effect of temperature and contact time), indicating a completely random and valid relationship between the factors except for DE. Similarly, for calcined eggshell adsorbent, the relationship between all factors is entirely random and valid.

The normal plots depicted in Figures 11a and 12a show that all responses for both adsorbents are located on or near the fitting line, and the distribution of residual values is normal. Therefore, the model proposed by the software is valid. In addition, in Figures 11b and 12b, all responses for both adsorbents are centered on the fitted line, confirming that the actual values align well with the predicted values, making the quadratic model suitable for describing the data.

Figures 11c and 12c illustrate the standard deviation of the actual values from the predicted values and the distribution of the designed experiments. In these figures, all responses for both adsorbents fall within the range of -3.6 to +3.6, indicating a strong match between the model and the responses.

The distribution of residual versus predicted values is shown in Figures 11d and 12d. According to these figures, all data for both adsorbents fall within the confidence range of -3.6 to +3.6, showing a random distribution around the zero line. This confirms that the quadratic model effectively describes the resulting responses.

After confirming the compatibility and validity of the quadratic model with the experimental responses, DOE software provides a coded and actual numbers equation to predict the removal efficiency of dye on each of the two adsorbents. Equations S7 and S8 in the Supplementary file show

the coded equations for the removal of auramine-O dye on tea waste and calcined eggshell adsorbents, respectively. Additionally, Equations S9 and S10 in the Supplementary file provide the actual numbers equations for both adsorbents.

Through the use of DOE, the parameters affecting the removal of auramine-O dye on the adsorbents were optimized to achieve the highest response value (i.e., the highest removal efficiency). The optimal conditions in Table 2 show that the maximum removal efficiencies were predicted to be 97.68% and 97.50% for tea waste and calcined eggshell, respectively. These values were experimentally measured at 96.50% and 96.20%, respectively. The close agreement between the actual and predicted values of removal efficiency for both adsorbents demonstrates the effectiveness of the model.

In addition, adsorption experiments with auramine-O in the prepared wastewater samples were conducted using tea waste and calcined eggshell adsorbents. The results showed a removal efficiency ranging from 95.1% to 96.5%, which is not significantly different from the results shown in Table 2, confirming the obtained results.

Optimal conditions for the adsorption processes were determined to be a pH close to neutral (pH 5 and 6.5) and temperatures near room temperature (20 and 25ºC), reducing the overall cost of industrializing the process.

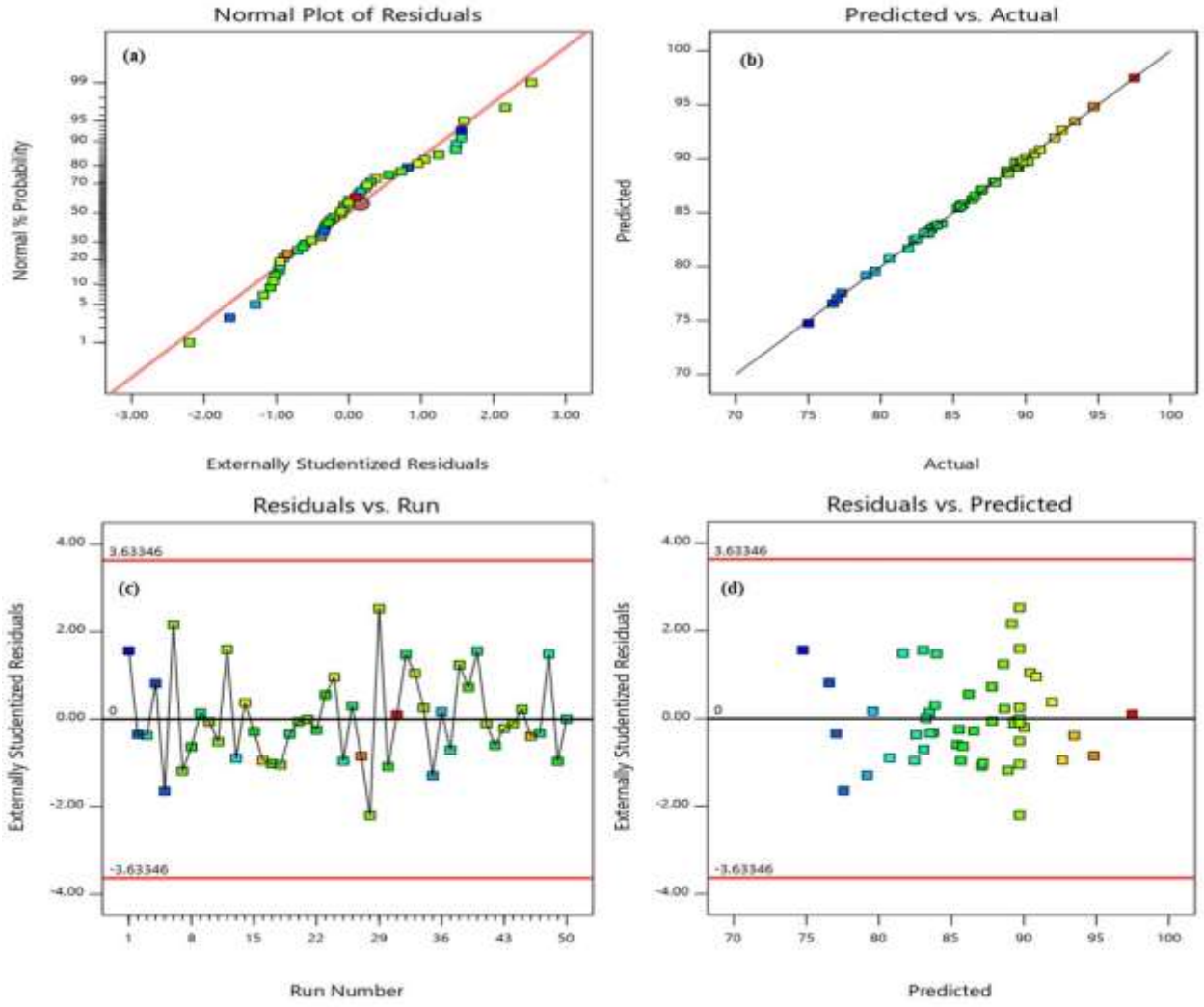

Fig. 11. a) Normal chart of residuals, b) Plot of actual versus predicted values, c) Plot of studentized residual values versus responses, and d) The graph of residual versus predicted values for tea waste adsorbent.

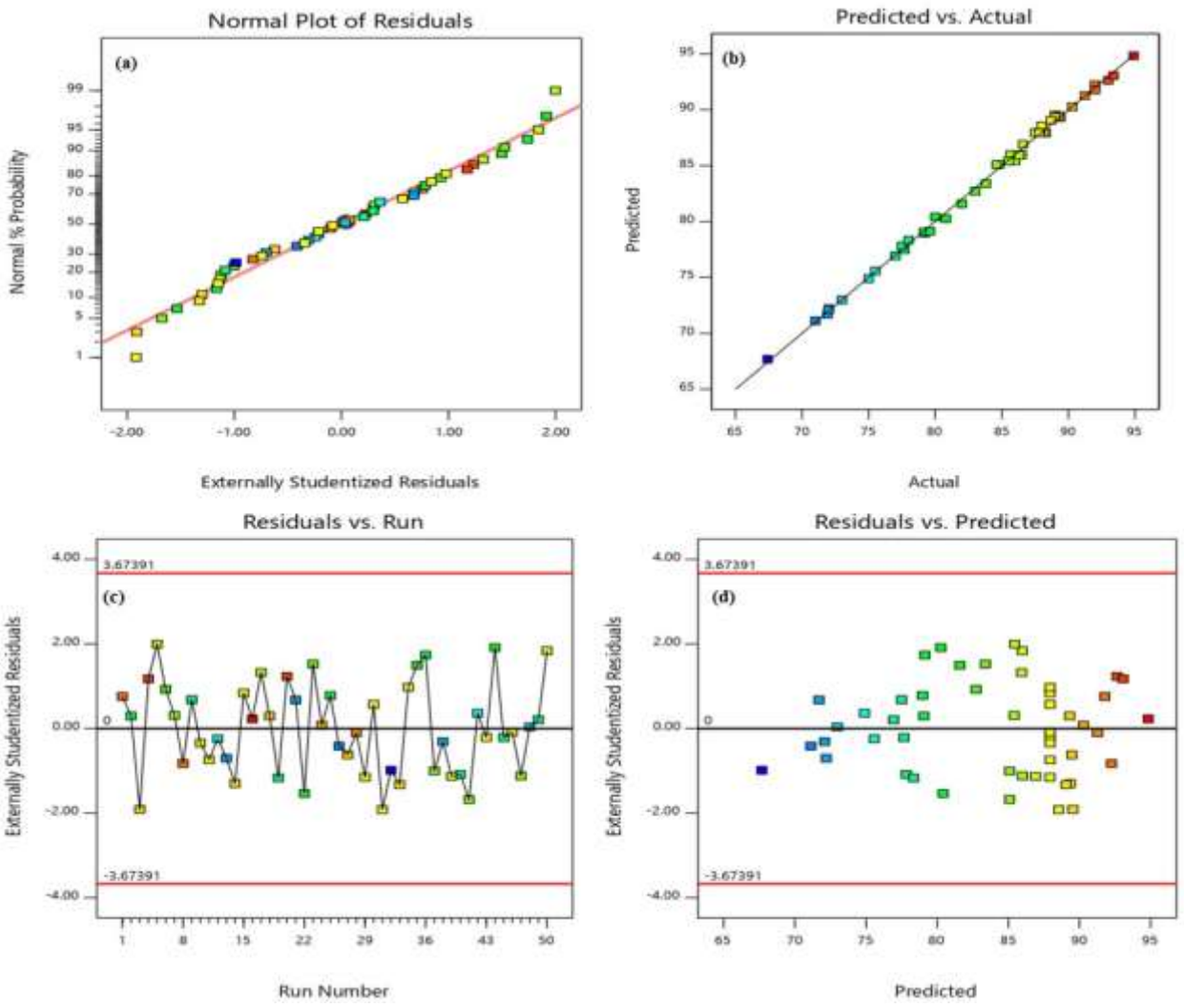

Fig. 12. a) Normal chart of residuals, b) Plot of actual versus predicted values, c) Plot of studentized residual values versus responses, and d) The graph of residual versus predicted values for calcined eggshell adsorbent.

Table 2. Optimal values for removal of auramine-O dye on tea waste and calcined eggshell adsorbents

adsorbent	pH	ads. dose (g)	dye conc. (ppm)	Temp. (°C)	Time (min.)	%R (experimental value)	%R (predicted value)	ΔR%
tea waste	5	0.10	1	25	30	96.50	97.68	1.18
calcined eggshell	6.5	0.085	2	25	65	96.20	97.50	1.3

3.6. Investigating the effective factors on the adsorption process of auramine-O on tea waste and calcined eggshell adsorbents

3.6.1. The effect of pH

The most significant factor influencing adsorption efficiency is pH. The effect of pH is determined by the change in surface charge of the adsorbent at the zero point pH (pH_{pzc}). At a pH higher than the pH_{pzc}, OH^- ions accumulate on the adsorbent's surface, making it more negatively charged and increasing removal efficiency due to electrostatic attraction between the cationic auramine-O dye and the negatively charged surface. The opposite is true at a pH lower than the pH_{pzc}. Auramine-O dye adsorption experiments were carried out on tea waste and calcined eggshell adsorbents at pH levels of 3, 5, 7, 9, and 11. Based on Figure 4, the zero point pH for tea waste and calcined eggshell adsorbents is 5.5 and 11.3, respectively. Therefore, it is reasonable to observe maximum auramine-O adsorption at pH

6.5 in Figures 13A and 14A for both adsorbents. Considering all parameters and their reciprocal effects, the optimal pH levels are 5 and 6.5 for tea waste and calcined eggshell, respectively.

3.6.2. The effect of adsorbent dosage

To assess the effect of adsorbent dosage, values of 0.03, 0.06, 0.09, 0.12, and 0.15 g were chosen. Figure 13B illustrates that for tea waste, removal efficiency increases with increasing adsorbent dosage up to 0.10 g and then decreases. Conversely, in the case of calcined eggshell (Figure 14B), efficiency decreases as adsorbent dosage increases. Optimal dosages were found to be 0.10 and 0.085 for tea waste and calcined eggshell, respectively.

3.6.3. The effect of initial dye concentration

To study the effect of the initial concentration of auramine-O dye on removal efficiency, concentrations of 1, 3, 5, 7, and 9 ppm were tested. Figures 13C and 14C indicate that removal efficiency decreases from 1 and 2 ppm onwards for tea waste and calcined eggshell, respectively.

3.6.4. The effect of temperature

Exploring the effect of temperature on the removal efficiency of auramine-O dye using tea waste and calcined eggshell, temperatures of 15, 25, 35, 45, and 55°C were studied. Optimal temperatures were found to be 15 and 25°C for tea waste and calcined eggshell, respectively. Figures 13D and 14D show that removal efficiency decreases with increasing temperature for both adsorbents. This can be attributed to the negative enthalpy and entropy values of the adsorption process, making it more spontaneous at lower temperatures. The reciprocal effects reveal the optimal temperature to be 25°C for both adsorbents.

3.6.5. The effect of contact time

To investigate the effect of time, 5 values were considered: 15, 30, 45, 60, and 75 minutes. In Figure 13E, for tea waste, efficiency increases with time until 45 minutes, then decreases. The highest adsorption occurs in the first 15 minutes when most active sites are occupied. For calcined eggshell, shown in Figure 14E, removal efficiency increases with time, likely due to more opportunities for adsorption. Based on the reciprocal effects, the software determined the most suitable times for tea waste and calcined eggshell adsorbents as 30 and 65 minutes, respectively.

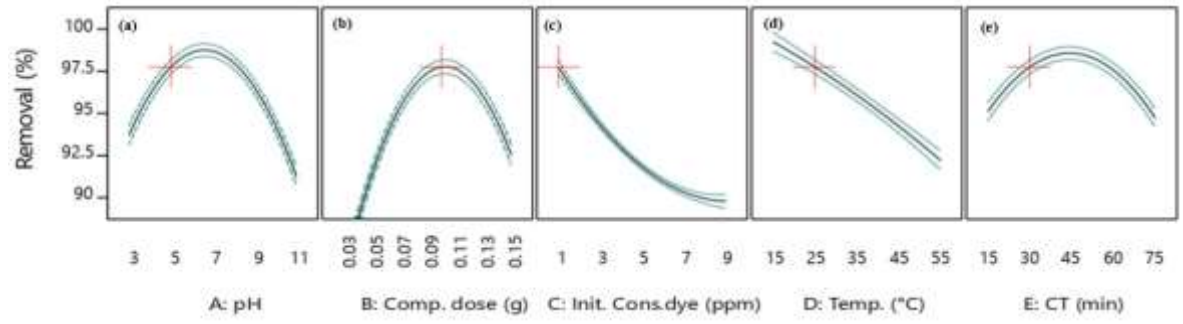

Fig. 13. The effect of important parameters on %R of auramine-O dye on tea waste adsorbent.

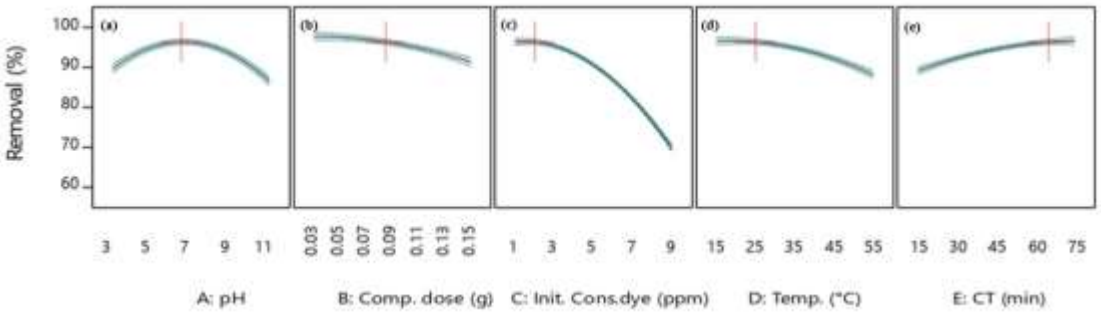

Fig. 14. The effect of important parameters on %R of auramine-O dye on calcined eggshell adsorbent.

3.6.6. The reciprocal effects of parameters

To investigate the effect of different variables on the efficiency of auramine-O removal and validate two-dimensional plots, three-dimensional (3D) response surface plots were created against two independent variables holding other variables constant at their central level. Figures 15(a-d) and 16(a-d) present the most significant 3D response surface plots for the adsorption of auramine-O dye on tea waste and calcined eggshell adsorbents, respectively. The closer the color of the graph moves towards red, the higher the adsorption percentage. The mutual effect between adsorbent dosage and dye concentration, as shown in Figures 15a and 16a, reveals that the maximum adsorption of auramine-O dye on both adsorbents was achieved by increasing the adsorbent dosage and decreasing the dye concentration. Furthermore, Figures 15b, c and 16b, c show that the adsorption processes for both adsorbents are significantly influenced by the pH of the dye solution. In addition, for both adsorbents, lower dye concentration and temperature result in higher adsorption efficiency. Moreover, the interaction between temperature and contact time in Figures 15d and 16d confirms that lower temperature and longer contact time enhance adsorption efficiency.

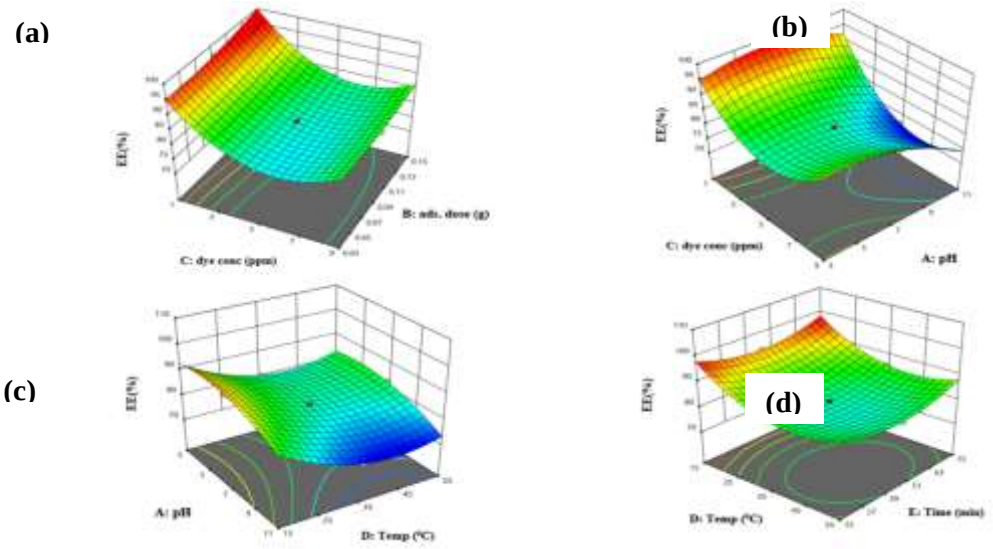

Fig. 15. Response surface diagrams for some variables on tea waste adsorbent.

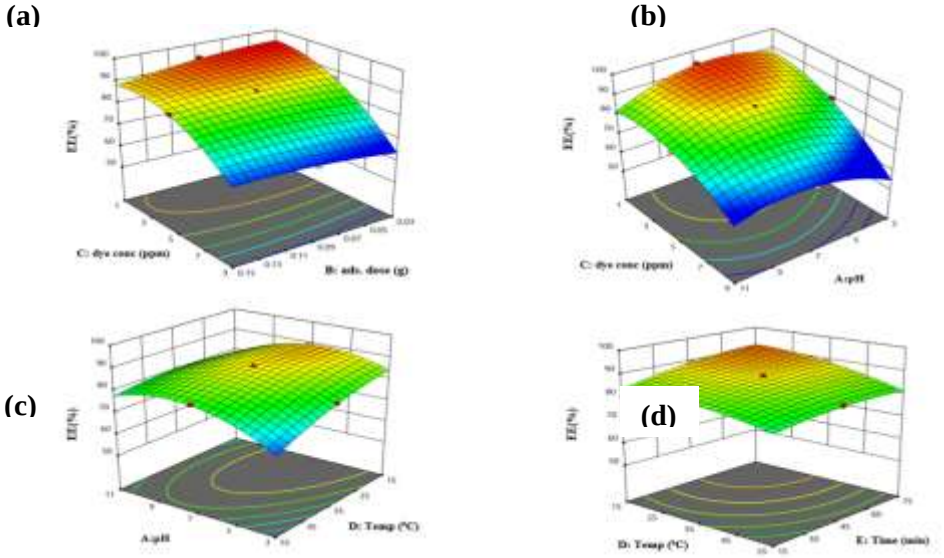

Fig. 16. Response surface diagrams for some variables on calcined eggshell adsorbent.

3.7. Adsorption isotherms

In the adsorption studies, adsorption isotherms are used to fit experimental results with theoretical models. One of the most important isotherms is the Langmuir isotherm whose linear form is represented as the equation (3).

Langmuir equation: $\frac{1}{q_e} = \frac{1}{q_m} + \left(\frac{1}{q_m K_L}\right)\frac{1}{C_e}$ (3)

where C_e (mg L^{-1}) is the equilibrium concentration of adsorbate in solution, q_e (mg g^{-1}) is the amount of adsorbate per unit mass of adsorbent, K_L (Lmg^{-1}) is the Langmuir isotherm constant, and q_m (mg g^{-1}) is the maximum adsorption capacity of the Langmuir isotherm. In the Langmuir isotherm, the plot of $1/q_e$ versus $1/C_e$ results in a straight line where K_L is determined by the slope ($\frac{1}{q_m K_L}$) and q_m is determined by the intercept ($1/q_m$) (Figure 17(a)).

The second important isotherm is the Freundlich isotherm which is written in its linear form as equation (4).

Freundlich equation: $log\, q_e = log\, K_F + \frac{1}{n} log C_e$ (4)

where K_F ($mg^{-11/n}\, L^{1/n}\, g^{-1}$) is the Freundlich constant and n is the intensity of the adsorption. For the Freundlich isotherm, the plot of log q_e versus log C_e gives a straight line where n is determined by the slope (1/n) and K_F is determined by the intercept ($logK_F$) (Figure 17(b)).

Temkin isotherm is another isotherm that is its linear form as equation (5).

Temkin equation: $q_e = B_1 \ln C_e + B_1 \ln K_T$ (5)

where $B_1 = \frac{RT}{b}$ is a constant that depends on the adsorption heat, and K_T (L g^{-1}) is the Temkin isotherm constant. In the Temkin isotherm, the plot of q_e versus $\ln C_e$ results in a straight line where B_1 is determined by the slope (B_1) and K_T is determined by the intercept ($B_1 \ln K_T$) (Figure 17(c)) [41,42].

The results relevant to parameters of the adsorption isotherms are shown in Tables 3 and 4. The results indicate that the experimental data have better agreement with the Langmuir isotherm than the Freundlich isotherm but the conformity with the Temkin isotherm is weaker for both adsorbents.

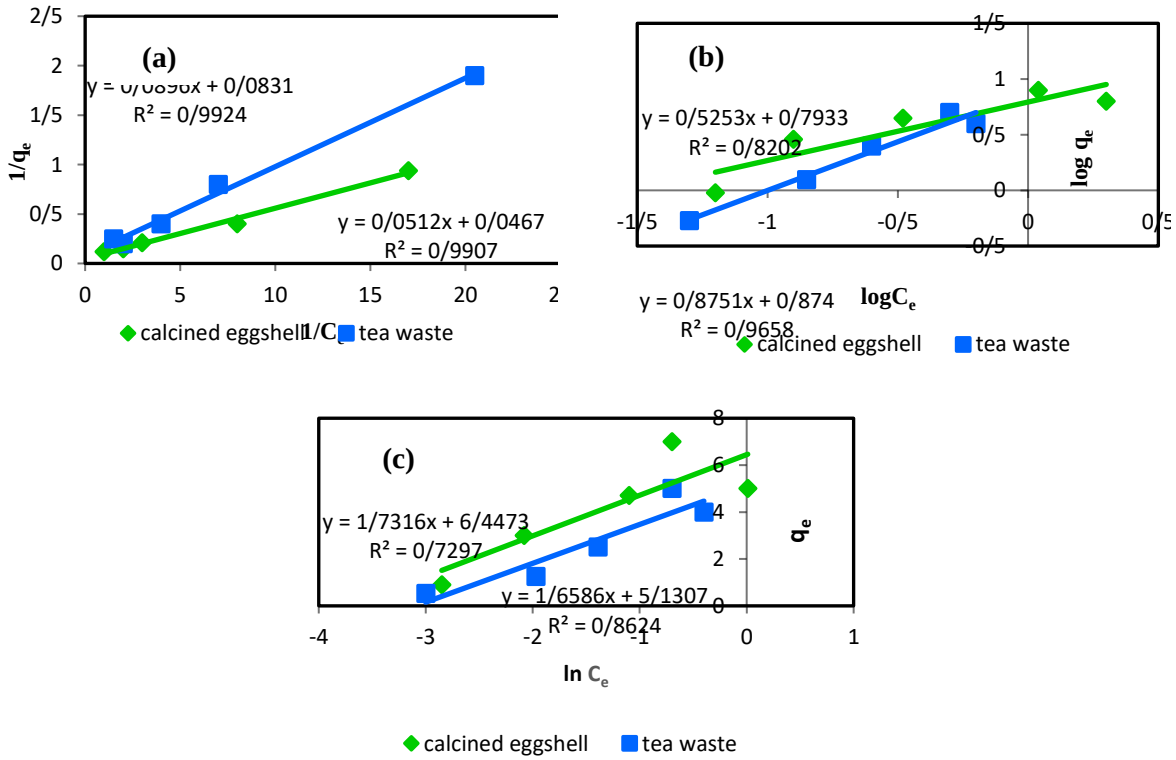

Fig. 17. Adsorption isotherms for the adsorption of auramine-O dye on tea waste and calcined eggshell adsorbents: (a) Langmuir isotherm, (b) Freundlich isotherm and (c) Temkin isotherm.

Table 3. Parameters related to the adsorption isotherms for adsorption of auramine-O dye on tea waste adsorbent.

Langmuir			Freundlich			Temkin		
K_L (Lmg^{-1})	q_m ($mg\ g^{-1}$)	R^2	K_F ($mg^{1-(1/n)}L^{1/n}g^{-1}$)	n	R^2	$K_T(Lg^{-1})$	B_1	R^2
0.93	12.03	0.9924	7.48	1.14	0.9658	164	1.65	0.8624

Table 4. Parameters related to adsorption isotherms for adsorption of auramine-O dye on calcined eggshell adsorbent.

Langmuir			Freundlich			Temkin		
K_L (Lmg^{-1})	q_m ($mg\ g^{-1}$)	R^2	K_F ($mg^{1-(1/n)}L^{1/n}g^{-1}$)	n	R^2	$K_T(Lg^{-1})$	B_1	R^2
0.91	21.40	0.9907	6.21	1.9	0.8202	601.8	1.73	0.7297

The essential characteristic of the Langmuir isotherm is represented by using an equilibrium and dimensionless parameter, the separation factor (R_L), according to Equation (6) [43]:

$$R_L = \frac{1}{1+K_L C_i} \quad (6)$$

where C_i is the initial concentration of the dye in the solution and K_L is the Langmuir constant.

This parameter is used to study the ability of the Langmuir isotherm. Information about this equilibrium parameter is shown as follows: R_L>1: Unfavorable, R_L=1: Linear, 0<R_L<1: Favorable, R_L=0: Irreversible.

The values of this parameter against the initial concentration of auramine-O dye in the solution are shown in Figure 18. The separation factor values range from 0 to 1, indicating that adsorption based on the Langmuir isotherm is favorable.

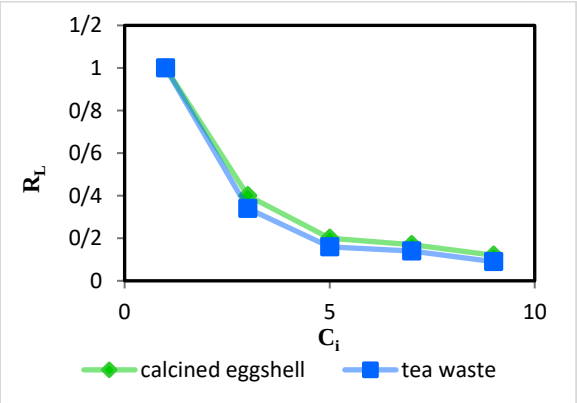

Fig. 18. Separation factor for the adsorption of auramine-O dye on tea waste and calcined eggshell adsorbents.

3.8. Adsorption thermodynamic

Investigations of a reaction typically begin with thermodynamic studies predicting the feasibility or infeasibility of a reaction. The change of standard Gibbs free energy (ΔG^0) for the adsorption process at a given temperature can be determined using the thermodynamic equilibrium constant, was shown in equation (7) [44]:

$$\Delta G^0 = -RT\ln K_0 \quad (7)$$

$$K_0 = \frac{q_e}{C_e} \quad (8)$$

where T is the absolute temperature, R (kJ mol^{-1} K^{-1}) is the gas constant and K_0 (L mol^{-1}) is the thermodynamic equilibrium constant. The standard enthalpy (ΔH^0) and standard entropy (ΔS^0) can be calculated using the equation (9) [31]:

$$\ln K_0 = \frac{-\Delta H^0}{R}\left(\frac{1}{T}\right) + \frac{\Delta S^0}{R} \quad (9)$$

The standard enthalpy and standard entropy can be determined from the plot of lnK_0 versus $\frac{1}{T}$ (Figure 19). The slope of this line is $\frac{-\Delta H^0}{R}$ and the intercept is $\frac{\Delta S^0}{R}$, from which ΔH^0 and ΔS^0 can be calculated. Tables 5 and 6 show that, based on the negative values of these parameters, the reaction is exothermic and favors adsorption. With negative values for standard enthalpy and standard entropy, it can be concluded that the adsorption processes of auramine-O dye on tea waste and calcined eggshell adsorbents are exothermic and associated with reduced disorder. Additionally, the negative values of ΔG^0 suggest that both adsorption processes under the experimental conditions are spontaneous, and as the temperature increases, the tendency for adsorption decreases.

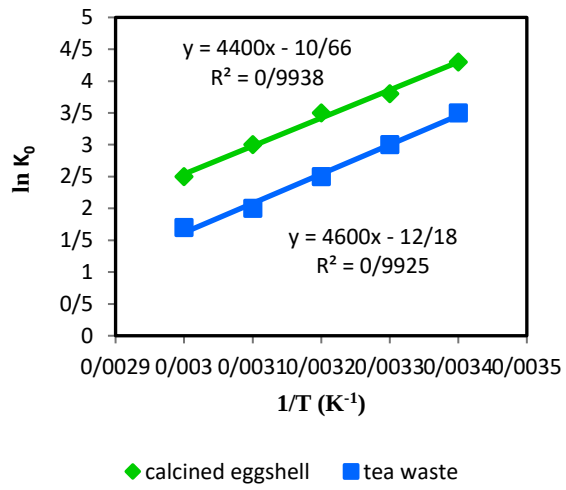

Fig. 19. ln K_0 vs. 1/T plot for the thermodynamic quantities for the adsorption of auramine-O dye on tea waste and calcined eggshell adsorbents.

Table 5. Thermodynamic quantities for the adsorption of auramine-O dye on tea waste adsorbent.

T / K	ΔG^0 (kJ mol^{-1})	ΔH^0 (kJ mol^{-1})	ΔS^0 (J mol^{-1} K^{-1})
288	-9.03	-38.2	-101.3
298	-8.01		
308	-7.00		
318	-5.99		
328	-4.97		

Table 6. Thermodynamic quantities for the adsorption of auramine-O dye on calcined eggshell adsorbents.

T / K	ΔG^0 (kJ mol^{-1})	ΔH^0 (kJ mol^{-1})	ΔS^0 (J mol^{-1} K^{-1})
288	-11.08	-36.6	-88.6
298	-10.20		
308	-9.31		
318	-8.43		
328	-7.54		

3.9. Adsorption kinetic

After conducting the thermodynamic study, the investigation of a reaction is followed by a kinetic study. This involves examining the effect of contact time on the adsorption percentage. The adsorption kinetics model is based on the pseudo-first order reaction by Lagergren (equation (10)) [45]:

$$\ln(q_e - q_t) = lnq_e - k_1 t \qquad (10)$$

where k_1(min^{-1}) is the Lagergren rate constant, q_t and q_e are the amounts of auramine-O adsorbed per unit mass of adsorbent at time t and equilibrium, respectively. In the experiments, a plot of $\ln(q_e - q_t)$ versus t shown in Figure 20(a) is not a straight line, indicating that the adsorption processes of auramine-O dye on tea waste and calcined eggshell adsorbents are not following the pseudo-first order. The pseudo-second order mechanism by Ho et al is presented (equation (11)) [32]:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \qquad (11)$$

where k_2 is the rate constant for the pseudo-second order equation, from the plot of t/q_t versus t (min). The slope of this straight line is $\frac{1}{q_e}$ and the intercept is $\frac{1}{k_2 q_e^2}$ from which k_2 and q_e can be calculated (Figure 20(b)). According to the plots, the adsorption processes of auramine-O dye on tea waste and calcined eggshell adsorbents follow the pseudo-second order with regression values of 0.9992 and 0.9997 (very close to unity), respectively. In addition, the kinetic quantities including k_2 and q_e are shown in Tables 7 and 8.

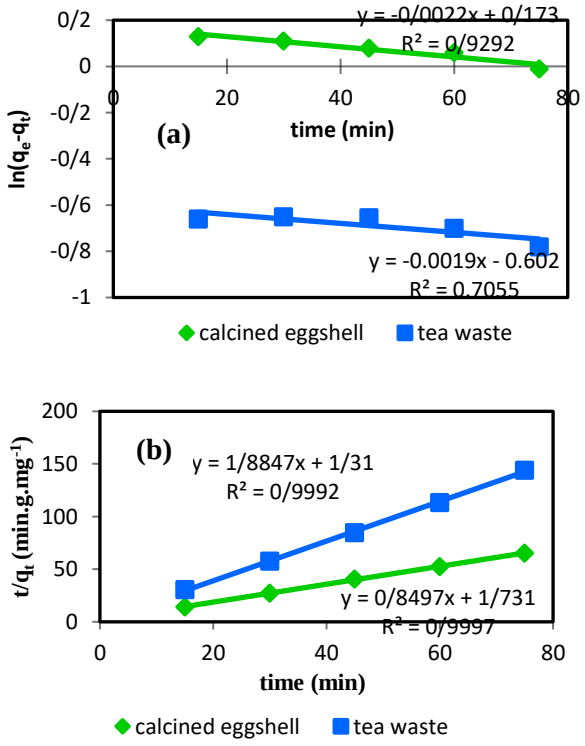

Fig. 20. Adsorption kinetics of auramine-O dye on tea waste and calcined eggshell adsorbents by (a) pseudo-first order and (b) pseudo-second order models.

Table 7. Kinetic quantities (pseudo-first order model) for adsorption of auramine-O dye on tea waste and calcined eggshell adsorbents.

adsorbent	$k_1(min^{-1})$	$q_e(mg/g)$	R^2
tea waste	0.0019	0.55	0.7055
calcined eggshell	0.0022	1.19	0.9292

Table 8. Kinetic quantities (pseudo-second order model) for adsorption of auramine-O dye on tea waste and calcined eggshell adsorbents.

adsorbent	$k_2(g\ mg^{-1}\ min^{-1})$	$q_e(mg/g)$	R^2
tea waste	2.72	0.53	0.9992
calcined eggshell	0.41	1.18	0.9997

3.10. The suggested mechanism for the adsorption process

Eggshell calcined at 800°C exhibits better adsorption properties due to the conversion of calcium carbonate to calcium oxide and surface defects (reaction R1). This process produces active sites for Ca^{2+} and OH^-, making them available for adsorption (reaction R2). The significant portion of hydroxide ions remaining on the adsorbent surface creates an electrostatic attraction with the cationic dye and, thus, effective adsorption [**Error! Bookmark not defined.**]. At basic pH levels, the excess hydroxide ions in the solution compete strongly with the dye molecules for adsorption on the adsorbent and reduce dye adsorption. At low pH, H^+ ions in the solution compete for the active sites of the adsorbent against the cationic dye [46]. Therefore, the most suitable pH for dye adsorption is in a near neutral environment.

3.11. Adsorbents reusing study

The recovery experiments were carried out to evaluate the feasibility of reusing the adsorbents containing tea waste and calcined eggshell for adsorption of auramine-O. Initially, the considered adsorbents were separately mixed in 100 ml of 0.1 mol/L NaOH solution, and after stirring for one hour, they were centrifuged and the resulting adsorbents were dried in an oven at 100°C [**Error! Bookmark not defined.**]. Then, the adsorption experiments of auramine-O were conducted on each of the regenerated adsorbents under the optimal conditions. The recovery of each adsorbent was repeated in 4 recovery cycles and the results were represented in Figure 21.

Figure 21 shows that tea waste and calcined eggshell still have 69.7% and 78.5% of the ability to adsorb auramine-O after 4 recovery cycles. Therefore, although the adsorption sites were not completely freed from dye during multiple regeneration, the acceptable reusability of the considered adsorbents suggests that they can be considered as valuable adsorbents for the removal of auramine-O from aqueous solutions.

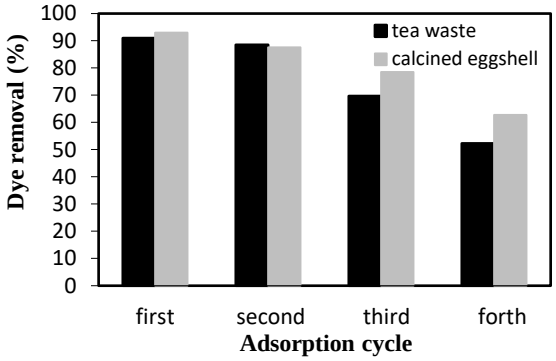

Fig. 21. Recovery and reusability of adsorbent using 0.1 mol/L NaOH solution in four recovery cycles.

3.12. The comparison of the removal efficiency in many adsorbents

The maximum removal efficiency represents the highest percentage of pollutant removal on the adsorbent, serving as a metric for comparing the effectiveness of different adsorbents. Table 9 compares various adsorbents for removing auramine-O dye from aqueous solutions. NaX nanozeolites and multiwall carbon nanotubes exhibit higher removal efficiencies compared to tea waste and calcined eggshell. However, these adsorbents are not natural and abundant, are expensive and their preparation method is difficult and costly. Therefore, the adsorbents utilized in this study are considered more appropriate for removing auramine-O from aqueous solutions.

Table 9. Comparison of the removal efficiency in many adsorbents for adsorption of auramine-O dye from aqueous solutions

adsorbent	removal efficiency (%)	Reference
NaX nanozeolites	99.61	[26]
multiwall carbon nanotubes	97	[22]
psidium guava	92	[19]
leaves	87.1	[20]
sesame leaf		
peanut shell	83.81	[21]
sodium dodecyl sulfate (SDS) functionalized magnetite nanoparticles	74	[27]
tea waste	96.50	current work
calcined eggshell	96.20	current work

4. Conclusion

The applicability of two bio-adsorbents, tea waste and calcined eggshell, for the removal of auramine-O pollutant dye from aqueous solutions using the adsorption process was studied. Characterization and comparison of FTIR, XRD, and SEM for tea [1]waste and calcined eggshell after and before adsorption confirmed the adsorption of auramine-O on both adsorbents. Response surface methodology (RSM) with central composite design (CCD) was used to optimize the parameters affecting the adsorption such as initial solution pH, initial auramine-O concentration, adsorbent dosage, contact time, and temperature. The optimized conditions were found as follows: For tea waste pH 5, initial auramine-O concentration 1 mg L^{-1}, adsorbent dosage 0.10 g, contact time 30 min, temperature 25°C. For calcined eggshell pH 6.5, initial auramine-O concentration 2 mg L^{-1}, adsorbent dosage 0.085 g, contact time 65 min, temperature 25°C. Based on the confirmed quadratic model, the highest removal efficiency was predicted to be 97.68% for tea waste and 97.50% for calcined eggshell. The obtained experimental values at 96.50% and 96.20% were consistent with the theoretical data. The optimal pH close to neutral medium (pH: 5 and 6.5) and temperatures close to room temperature (20 and 25°C) reduce the cost of the scaling up. In addition, the results show that for both adsorbents, the adsorption process follows the Langmuir isotherm as opposed to the Freundlich and Temkin isotherms. Thermodynamic studies showed that the adsorption processes of auramine-O dye on both adsorbents are exothermic and spontaneous, and these processes are associated with reduced disorder. Finally, kinetics studies indicated that both adsorption processes follow the pseudo-second-order model.

Acknowledgements

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