



KINETIC, THERMODYNAMIC, AND EQUILIBRIUM STUDIES ON THE ADSORPTION OF ANTHRAQUINONE DYE ONTO VAUCHERIA SP. FROM SYNTHETIC SOLUTION

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Abstract : The kinetics, adsorption isotherms, and thermodynamics of the removal of the anthraquinone dye Alizarin by adsorption onto dried *Vaucheria* sp. algal biomass were investigated. Several adsorption isotherms and kinetic models were determined using the optimized parameters from the batch adsorption studies. Three different theoretical models were used to examine adsorption kinetics: the pseudo-first-order, the pseudo-second-order, and the intraparticle models. Four different models were used to determine adsorption isotherms: the Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich isotherm models (D-R). In a statistical comparison of regression values, the pseudo-second-order kinetic model and the Langmuir adsorption isotherm were found to be the best fits (R^2). The results of the Langmuir isotherm study showed that the maximum adsorption capacity was 83.35mg/g under the optimal conditions of 4 pH, 0.3 g/L adsorbent dosage, 318 K temperature, and 90 minutes of contact time. More importantly, when compared to other adsorbents, the *Vaucheria* sp. alga used in our study showed the highest adsorption capacity when it came to the Alizarin dye. Adsorption is an endothermic, random, and spontaneous process, as evidenced by the changes in thermodynamic parameters enthalpy, entropy, and Gibbs free energy. Up to 70% of the algal biomass used as the adsorbent material could be recovered using 0.1 M HCl, and the same biomass could be used five times without significantly losing adsorption capacity. Thus, this research suggests that *Vaucheria* sp. biomass may be a useful biomaterial for treating synthetic wastewater containing alizarin dye.

Key words : Alizarin; *Vaucheria* sp.; Adsorption ; Adsorption Isotherm; Kinetics

I. INTRODUCTION

Dye and dyeing materials have been used a lot in industries like cosmetics, textiles, leather, plastics, paper, food, and pharmaceuticals to meet growing consumer demand. There are a lot of adverse effects that happen when dye-containing wastewater is dumped into natural waterways. This is a big problem. In the last few years, people have paid a lot of attention to getting dyes out of wastewater. Adsorption has been the focus of previous research on dye removal from wastewater because it is effective, easy to use, cheap, and doesn't need much energy (Chamoli and Chanda 2020 ; Shinde and Patel 2021). So, in recent years, new, low-cost alternatives to activated

carbon adsorbents, have been looked into for treating large amounts of wastewater with dyes, such as *Sargassum vulgare* seaweeds and *Cassia alata* seeds (Raju and Satti Babu 2018; Nandini et al. 2018).

Biomaterials like algae could also break down organic pollutants such as dyes. In this study, Xanthophyceae algae named *Vaucheria* sp. was used as an adsorbent. This alga is widespread, and only a few studies on its use as an adsorbent material are available (Crist et al. 1994; Khataee et al. 2011). Our lab tested the ability of this algae to remove heavy metals such as copper and nickel, as well as dyes such as methylene blue and methyl orange, with very good adsorption capacity (Rastogi 2022; Rastogi 2022; Sagar and Rastogi 2019; Sagar and Rastogi 2018).

Alizarin, an anthraquinone dye chosen as a model compound for this study, is widely used as a coloring agent in the textile industry. This dye is widely regarded as the most long-lasting, stable, and resistant to degradation. This is due to the complex nature of aromatic ring structures, which provide exceptional physicochemical, thermal, and optical stability. There are only a few reports on the removal of this dye using various sorbents such as poly-2-hydroxy ethyl methacrylate (PHEMA) (Sadegh et al. 2015), iron oxides (Pirillo et al. 2009), activated clay modified by iron oxide (Fu et al 2011), mango seed (Abdus and Buhari 2014) and mustard husk (Gautam et al 2013).

In the interest of continuing research, the main aim of this investigation is to utilize *Vaucheria* sp. alga to remove alizarin dye from synthetic wastewater. Kinetic, thermodynamic and isothermal studies were performed using various theoretical models.

II. MATERIALS AND METHODS

2.1. Chemicals and Equipment used

The analytical grade chemicals used in this study were purchased from SD Fine Chemicals Ltd., India. Double distilled water was used to create an alizarin stock solution. Using a UV-Visible spectrophotometer, the dyes were determined spectroscopically (model UV-Vis 119 Systronics India Ltd.). The pH readings were taken using a pH meter (PERFIT, India). A dye solution's UV-visible absorbance spectrum was measured between 200 and 800 nm. In the current study, alizarin dye was spectrophotometrically analyzed at a maximum wavelength λ_{\max} of 567 nm.

2.2. Various Adsorption Isotherm Models Applied

Adsorption isotherm data modeling is essential for estimating and comparing the adsorbent's adsorption performance. As a result, the equilibrium data were fitted using the isotherm models Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich (D-R) (Langmuir 1918; HMF 1906; Temkin and Pyzhev 1940; Dubinin 1960). Equilibrium experiments were carried out under optimal conditions at three different temperatures (298, 308, and 318 K) with pre-weighed adsorbent doses at 200 mg/L dye concentration during the optimum contact time of 90 minutes.

2.2.1. Langmuir Isotherm

The Langmuir isotherm is based on monolayer adsorption on a surface with a finite number of adsorption sites. The Linearized Langmuir equation is:

$$\frac{1}{q_e} = \frac{1}{Q_0} + \frac{1}{bQ_0C_e} \quad (1)$$

q_e is the amount adsorbed (mg/g), C_e is the equilibrium dye concentration (mg/L), Q_0 is the Langmuir constant for maximum monolayer adsorption capacity (mg/g), and b is the free energy constant. $1/q_e$ versus $1/C_e$ was graphed to calculate these constants. From the Langmuir constant, b , the dimensionless separation factor, R_L , can be calculated as follows:

$$R_L = 1/(1 + bC_0) \quad (2)$$

The values of R_L were calculated at various temperatures, and the nature of the adsorption isotherm is determined by the criterion that if the R_L value is greater than one, it is considered linear, if the value is between zero and one, it is favorable, and if the value is equal to zero, it is considered irreversible.

2.2.2. Freundlich Isotherm

The Freundlich isotherm works under the assumption that there is a multilayer heterogeneous adsorption occurring. The following is an expression in the linear form of the Freundlich :

$$\ln q_e = \ln K_F + \frac{1}{n} C_e \quad (3)$$

where n is the adsorption intensity and K_F is a Freundlich constant ($\text{mg}^{1-1/n} \text{L}^{1/n} \text{g}^{-1}$). The intercept and slope from the plot of $\ln q_e$ versus $\ln C_e$ were used to determine the values of K_F and n .

2.2.3. Temkin isotherm

To ascertain the precise indirect adsorbate-adsorbent interactions on dye adsorption and the linear rather than logarithmic decrease in adsorption heat, Temkin isotherm was modeled. The equation below represents the Temkin model:

$$q_e = \frac{RT}{b_T} \ln(A_T C_e) \quad (4)$$

where T is the absolute temperature in Kelvin and R is the gas constant. The slope and intercept of q_e versus $\ln C_e$ were used to calculate the constants for the heat of adsorption, b_T ($\text{g kg mg}^{-1} \text{mol}^{-1}$), and A_T (L mg^{-1}).

2.2.4. Dubinin and Radushkevich (D-R) isotherm

The Dubinin and Radushkevich (D-R) isotherm was employed to calculate the apparent free energy of porosity. Instead of assuming a homogeneous surface or constant adsorption potential, this isotherm is related to the adsorbent's porous structure. The equation for the D-R isotherm can be written in linear form as follows:

$$\ln q_e = \ln q_m + \beta \varepsilon^2 \quad (5)$$

$$\varepsilon = RT \ln \left(1 + \frac{1}{C_e} \right) \quad (6)$$

where q_m is maximum adsorption capacity in mg/g , β is the constant related to adsorption energy ($\text{mol}^2 \text{kJ}^{-2}$), ε is a Polanyi potential, R is the gas constant ($8.314 \text{ Jmol}^{-1} \text{K}^{-1}$) and T is the temperature (K). From the formula given, the mean adsorption energy can be determined.

$$E = 1/\sqrt{2\beta} \quad (7)$$

The adsorption process is controlled chemically if the value of E is between 8 kJ/mole and 16 kJ/mole, and it advances physically if the value of E is less than 8 kJ/mole (Celekli et al 2012).

2.3. About thermodynamic study

Using the following equations, thermodynamic variables such as standard free energy changes (ΔG° , kJ/mol), enthalpy (ΔH° , kJ/mol), and entropy (ΔS° , kJ/mol/K) were determined at three different temperatures (298, 308, 318 K) in order to study the feasibility of the adsorption process.

$$\Delta G^\circ = -RT \ln(b) \quad (8)$$

$$\ln \left(\frac{b_2}{b_1} \right) = \frac{\Delta H^\circ}{R} \left(\frac{1}{T_2} - \frac{1}{T_1} \right) \quad (9)$$

$$\Delta G^\circ = \Delta H^\circ - T \Delta S^\circ \quad (10)$$

Where b_1 and b_2 are Langmuir constants at different temperatures and the remaining terms have their usual meanings. Positive and negative ΔH° values reveal the endothermic or exothermic nature of adsorption, respectively, while negative ΔG° values reveal the feasibility of adsorption. A positive or negative ΔS° denotes an increase or decrease in randomness, respectively.

2.4. Various Kinetic Models applied

Adsorption kinetics' fundamental idea is the pursuit of the model that most accurately captures the results of experiments. There are various models that can be used to understand the mechanism of solute adsorption on an adsorbent. Research on adsorption rate was done in order to quickly and effectively design a model. The simplest kinetic models used to test experimental data are the pseudo-first-order, pseudo-second-order, and intraparticle diffusion models (Lagergren 1898; Ho and McKay 1999; Allen et al 1989). These models are used to examine the controlling mechanisms of an adsorption process, such as chemical reaction and diffusion control.

2.4.1. Pseudo-first-order Model

The pseudo-first-order equation can be expressed in linear form as follows.

$$\log(q_e - q_t) = \log(q_e) - \frac{K_{1,ads}}{2.303} t \quad (11)$$

where k_1 is the pseudo-first-order rate constant (min^{-1}) and q_t (mg/g) is the amount of dye adsorbed by biomass at equilibrium time t (min^{-1}). The constants were determined by plotting the $\log(q_e - q_t)$ versus $\log t$ graph.

2.4.2. Pseudo-second-order Model

The pseudo-second-order equation can be expressed in linear form as follows.

$$\frac{t}{q} = \frac{1}{K_{2,ads}q_e^2} + \frac{1}{q_e}t \quad (12)$$

where q_e and q are the amount of the dye adsorbed by the algal biomass (mg/g) at equilibrium and time t , respectively, and k_2 ($\text{g mg}^{-1} \text{min}^{-1}$) is the rate constant of second-order adsorption. q_e and k_2 can be calculated from the slope and intercept of the t/q versus t plots.

2.4.3. Intraparticle diffusion

A multi-step process governs dye adsorption on algal surfaces. These are (a) "molecular diffusion," which happens from the bulk solution to a film layer around the adsorbent particle;" (b) "film diffusion," where diffusion happens from the film to the particle surface;" (c) "surface diffusion," which is movement inside the adsorbent particle;" and (d) "pore diffusion," which happens in liquid-filled pores. Adsorption uptake can involve many different kinds of interactions, such as chemisorption, physisorption, ion exchange, or complexation (Gupta et al 2014). Solutes move from a liquid to a solid during adsorption. This can happen through film diffusion, intraparticle diffusion, or both.

Following is the mathematical expression for the intraparticle diffusion rate constant.

$$q_t = k_{id}t^{1/2} \quad (13)$$

where q_t represents the amount of dye adsorbed per unit mass of adsorbent (mg/g) at time t and k_{id} represents the intra-particle diffusion rate constant ($\text{mg/g min}^{-1/2}$). If the plot of q_t versus $t^{1/2}$ is linear, the adsorption mechanism that is taking place is intra-particle diffusion. In the case where the line begins at zero, the most important rate-limiting process is intraparticle diffusion. If the line does not begin at 0, this indicates that intraparticle diffusion is not the only step that limits the rate of the reaction (Chandra et al 2015).

2.5. Studies on Adsorption, Desorption, and Reusing

In an effort to mitigate the effects of secondary pollution, experiments on desorption have been carried out. In order to evaluate the reusability of the adsorbent, alizarin was loaded onto *Vaucheria* sp. algae, and then the biomass was put through five cycles of adsorption-desorption using an acid, a base, and a chelating agent. For desorption tests, algal biomass was mixed with a dye solution at a concentration of 1 gL^{-1} under optimal conditions. Measurements of the remaining dye in the solution served to quantify the amount of dye that had been adsorbed. The adsorbent was filtered, then dried in a vacuum oven at 70 degrees Celsius for 24 hours. After loading the adsorbent with dye, it was placed in a 100 ml conical flask and treated with 50 ml of HCl (an acid), 50 ml of NaOH (a base), and 50 ml of EDTA (Ethylene diamine tetra acetic acid) (a chelating agent) at equilibrium conditions five times in a row. The process of adsorption was followed by desorption, and this sequence was repeated in order to complete one cycle. Quantitative analysis of desorbed dye was performed using a UV-Vis Spectrophotometer and the desorption percentage is calculated as given below:

$$\text{Desorption\%} = \frac{\text{mass of desorbed}}{\text{mass of adsorbed}} \times 100 \quad (14)$$

III. RESULTS AND DISCUSSION

3.1. Isotherm modeling of adsorption

Isotherms are used to model adsorption, and they relate the amounts of solute adsorbed to the solid (q_e) to the amounts of solute in the solution (C_e). Langmuir, Freundlich, Temkin, and D-R isotherms, four commonly used equilibrium models, were applied to the adsorption of alizarin dye on the alga at three temperatures, 298, 308, and 318 K. Tabulated in Table 1 are the adsorption isotherm parameters and correlation coefficient (R^2) values determined for Alizarin dye adsorption onto *Vaucheria* sp.

The linearity of the dye's Langmuir plot (shown in Figure 1) is observed across the entire concentration range investigated, indicating the presence of a surface with uniform binding sites, equal adsorption energies, and no species-specific interactions (Jalil et al 2010). Both the slope and the intercept can be used to determine the Langmuir constants b and q_e , and it has been found that the values of these constants increase with increasing temperature for Alizarin dye. This dye's linearity was evaluated using linear regression coefficients (R^2), which ranged from 0.993 to 0.998. R_L (Separation factor) is less than 1 and greater than 0 at all temperatures, confirming a favorable adsorption process.

Figure 2 shows that adsorption data analyzed by Freundlich plot at three different temperatures, allowing limited multilayer adsorption ([203]). Freundlich constants n and K_F were calculated from slope and intercept and

are listed in Table 1. Alizarin dye's Freundlich heterogeneity factor ranged from 1.74 to 2.96. Alizarin dye Freundlich constants K_F (an indicator of adsorption capacity) ranged from 5.77-29.04 mg/g, and correlation coefficients were 0.837-0.901.

The Temkin model considers the interaction between adsorbents and adsorbate and assumes that adsorption-free energy is a function of surface coverage. The results fit the Temkin model (Table 1), which suggested a reduction in heat of adsorption along with coverage (Gupta and Rastogi 2008). Alizarin dye's Temkin isotherm correlations ranged from 0.816 to 0.852. (Figure 3).

To calculate porosity apparent free energy, the D-R isotherm was used (Figure 4). This isotherm doesn't assume a homogeneous surface or constant adsorption potential, but a porous adsorbent. Mean free energy of adsorption is less than 8 kJ/mol, indicating physical adsorption (Gupta et al 2013). D-R isotherm gave the poorest fit to Alizarin dye adsorption equilibrium data ($R^2=0.894$) (Table 1).

A comparison of the relative values in Table 1 revealed that (i) maximum adsorption capacities calculated from Langmuir constants are comparable to values obtained from adsorption isotherms, whereas such values suggested by other models are much lower, and (ii) adsorption data fit well in the Langmuir equation rather than the other three models studied, as higher correlation coefficients were obtained with the former equation.

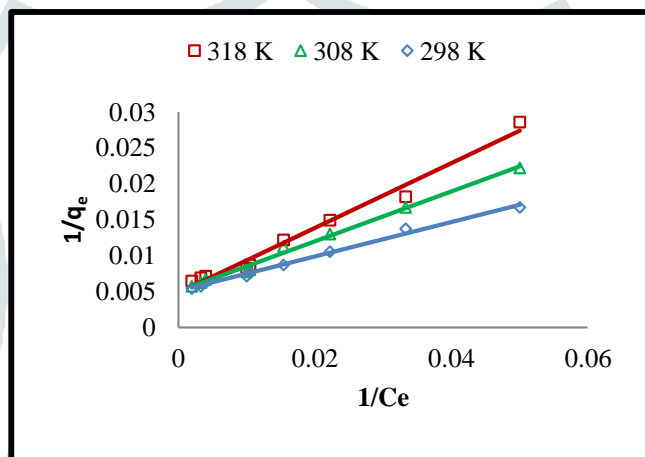


Figure 1 Langmuir Isotherm models for the adsorption of Alizarin dye by *Vaucheria sp.*

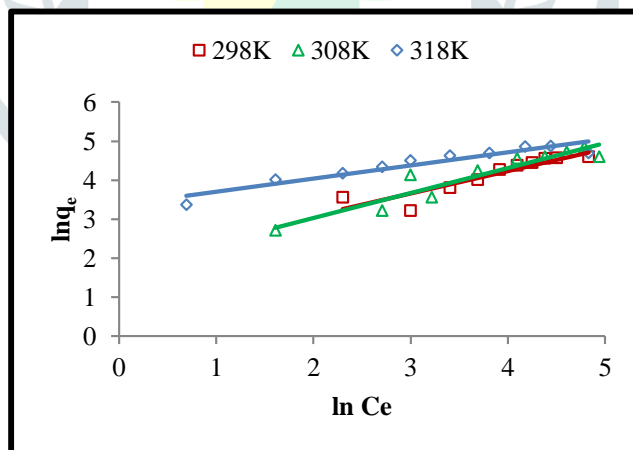


Figure 2 Freundlich Isotherm models for the adsorption of Alizarin dye by *Vaucheria sp.*

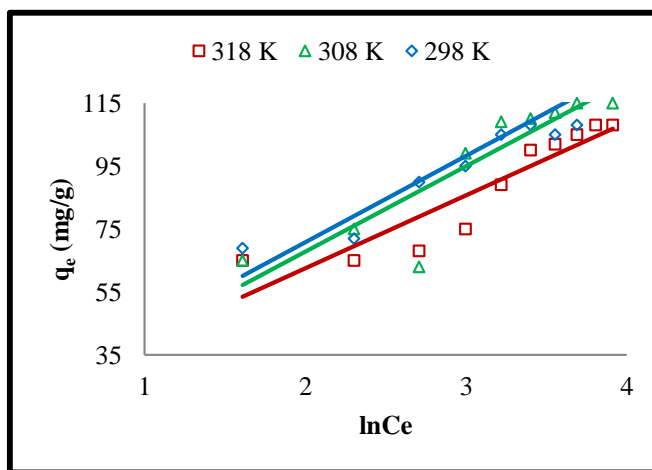


Figure 3 Temkin Isotherm models for the adsorption of Alizarin dye by *Vaucheria sp.*

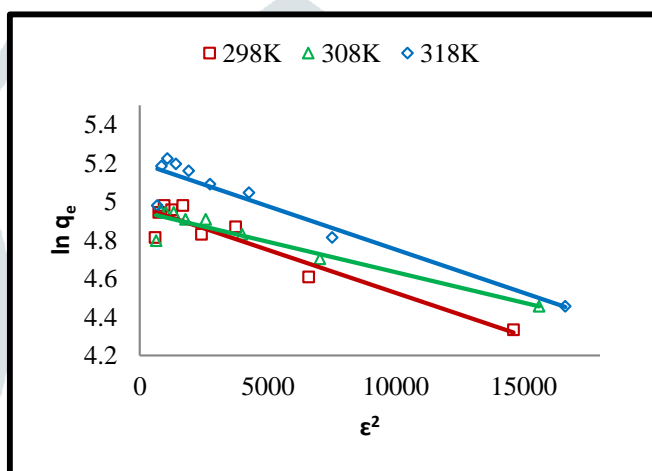


Figure 4 Dubinin-Radushkevi Isotherm models for the adsorption of Alizarin dye by *Vaucheria sp.*

Table 1. Values of various adsorption isotherm model contents for the adsorption of Alizarin dye onto algal biomass (*Vaucheria sp.*)

Isotherm Parameters	Methyl Orange		
	298 K	308 K	318 K
Langmuir Isotherm			
b (L mg⁻¹)	0.064	0.068	0.075
qe (mg g⁻¹)	64.516	74.07	83.35
R²	0.993	0.996	0.998
Dimensionless separation factor			
RL	0.072	0.068	0.062
Freundlich isotherm			
N	1.742	2.154	2.967
K_F (mg g⁻¹)	5.77	18.859	29.049
R²	0.837	0.892	0.901
Temkin isotherm			
A_T	0.816	0.821	0.862

b_T	90.290	94.49	113.95
R²	0.816	0.821	0.852
D-R isotherm			
q_m (mg g⁻¹)	66.98	76.85	86.71
E (kJ mol⁻¹)	1.118	1.29	1.0
R²	0.896	0.892	0.894

3.2. Thermodynamics of adsorption

At three different temperatures, thermodynamic parameters such as adsorption enthalpy change (ΔH), entropy change (ΔS), and Gibb's free energy change (ΔG) are calculated (298, 308 and 318 K). Table 2 shows the values of these three parameters. When the temperature was raised from 298 to 318 K, the ΔG° decreased from -23.90 to -25.93 kJ mol⁻¹, indicating that the process was feasible and spontaneous; positive ΔH° values (6.37) indicate endothermic adsorption. The disorderliness at the alga-solution interface during adsorption was described by the positive ΔS° value (Kousha et al 2012).

Table 2. Values of different thermodynamic parameters of Alizarin adsorption onto *Vaucheria* sp. Algae at various temperatures.

Biomass	Temp (K)	ΔG° (kJmol⁻¹)	ΔS° (kJ mol⁻¹ K⁻¹)	ΔH° * (kJmol⁻¹)
<i>Vaucheria</i> sp.	298	-23.907	0.1016	6.379
	308	-24.848	0.1013	
	318	-25.939	0.1016	

*measured between 298 K to 318 K

3.3. Adsorption Kinetic Studies

For interpreting the dynamic interaction between adsorbent and adsorbate, kinetic models are widely employed. In addition, it is used to determine the potential rate-regulating steps involved in the adsorption of dye molecules onto the adsorbent. Table 3 displays the outcomes of three kinetic models, including the Langergren pseudo-first-order model, the Ho second-order model, and the intra-particle diffusion model, at two different concentrations (100 and 200ppm) and optimized temperature 318K. Based on the correlation coefficient values, the optimal model was determined. The values of the kinetic constants of alizarin dye were determined using Figures 5, 6, and 7 for the above three different models respectively.

The calculated equilibrium adsorption capacity (q_e) for the pseudo-first-order kinetic model does not agree with the experimental (q_e) value (Table 3). The disparity in q_e values could be attributed to a time lag caused by external resistance controlling the adsorption process. Furthermore, the correlation coefficient values deviated slightly from unity ($R^2= 0.926$), indicating that the pseudo-first-order model was insufficient for describing the kinetics of alizarin dye adsorption onto *Vaucheria* sp., so the data was tested with the pseudo-second-order model. In addition to the calculated q_e values being closer to the experimental values, it was observed that the correlation coefficient value for the Pseudo-second order model was closer to unity ($R^2 = 0.995$).

Figure 7 depicts the plot for the intraparticle diffusion model, which revealed multi-linearity with three distinct phases. The sharply curved portion at the beginning could be attributed to the external surface adsorption or instantaneous adsorption stage. The second adsorption stage was the gradual adsorption stage, in which the intraparticle diffusion was rate-controlled, while the final linear portions could be due to the final equilibrium stage, in which the intraparticle diffusion began to slow due to extremely low solute concentrations in solution. Figure 7 also demonstrated that the straight line does not pass through the origin, implying that intraparticle diffusion was not the only rate-controlling step. The results showed that the best fit model for the adsorption of Alizarin dye onto *Vaucheria* sp. is a pseudo-second-order kinetic model.

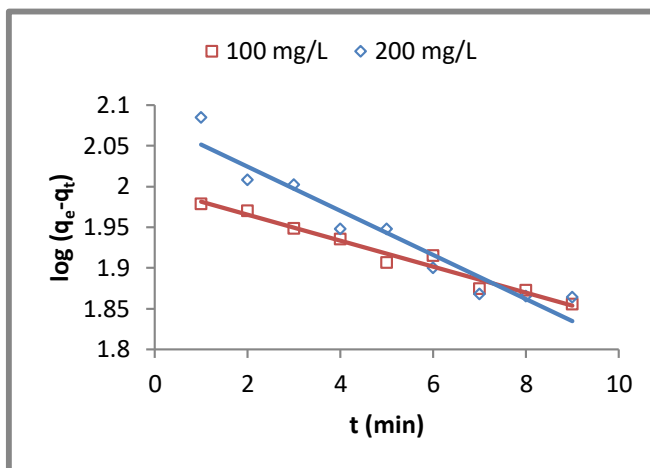


Figure 5. First order kinetic modeling of adsorption of Alizarin dye onto algal biomass *Vaucheria* sp.

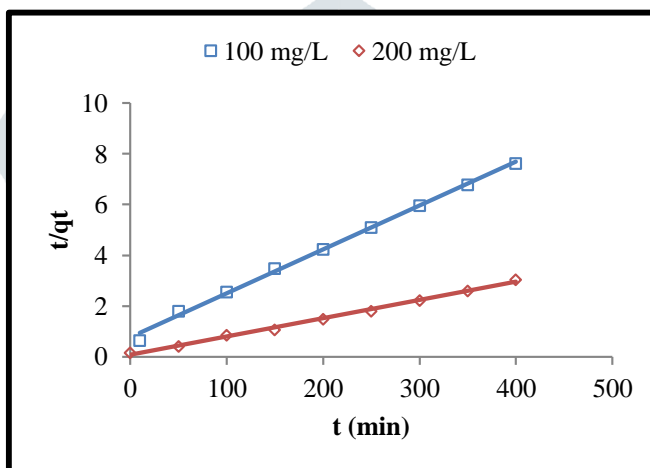


Figure 6. Second-order kinetic modeling of adsorption of Alizarin onto algal biomass *Vaucheria* sp.

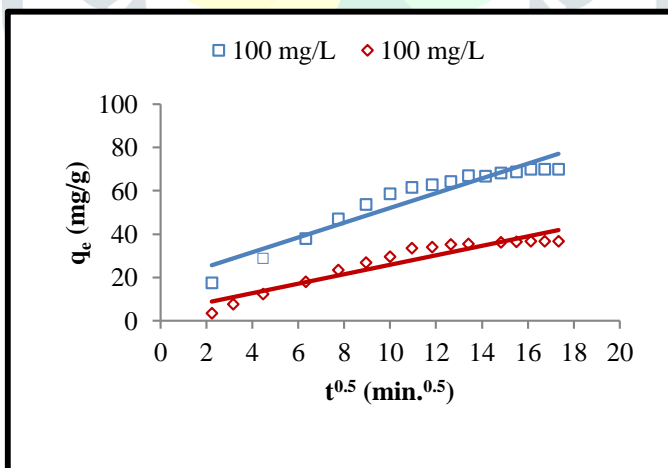


Figure 7. Intra particle diffusion plot for the adsorption of Alizarin dye onto algal biomass *Vaucheria* sp.

Table 3. Kinetic parameters estimated by pseudo-first order, pseudo-second order and intra-particle diffusion models for algal biomass *Vaucheria* sp. at two different concentrations of dyes.

Dye	Initial dye Conc. (mg L ⁻¹)	q _e (exp) (mg g ⁻¹)	First-order model			Second-order model			Intra-particle model	
			K ₁ (x10 ⁻³ min ⁻¹)	q _e (cal) (mg g ⁻¹)	R ²	K ₂ x 10 ⁻³ (g mg ⁻¹ min ⁻¹)	q _e (cal) (mg g ⁻¹)	R ²	K _w (mg g ⁻¹ min ^{0.5})	R ²

Alizarin	100	78.18	34.545	62.661	0.969	0.246	72.992	0.996	2.188	0.902
	200	80.94	63.181	75.857	0.926	1.82	85.470	0.995	3.140	0.913

3.4. Sorption-Desorption and Reuse studies

The recovery of dyes from loaded adsorbent is required for both disposal and reuse of adsorbent. The desorption efficiency is found to decrease as the number of cycles increases. Similar findings have previously been reported (Hu and Shipley 2013). New active sites were generated for each adsorption-desorption cycle, resulting in a decrease in adsorption capacity. As shown in Figure 8, hydrochloric acid had the highest desorption efficiency for Alizarin dye. Previously also it has been reported that acids are more effective at desorbing than other eluents (Stirk and Van Staden 2002). Thus, biomass reuse studies are an important feature for its potential use in continuous systems in industrial processes.

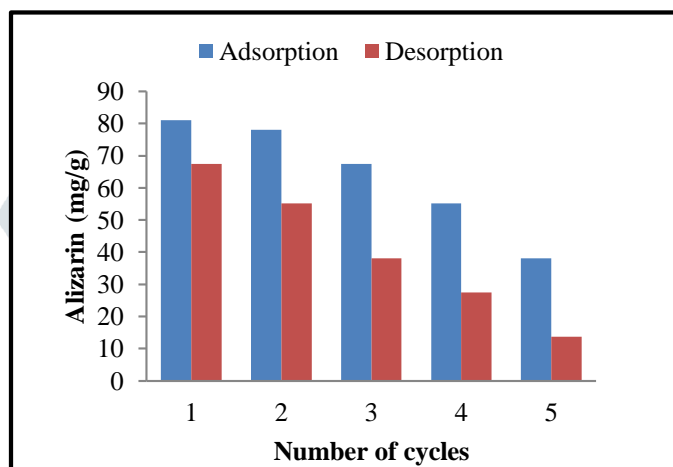


Figure 8. Adsorption/Desorption cycles of Alizarin dye on *Vaucheria* sp. using HCl

3.5. Disposal of the used biomass

The used biomass was sterilized by microwave irradiation for 10 minutes before being encapsulated in concrete very deep pits for ultimate disposal. This was done to make the process more environmentally friendly.

3.6. Comparison with other adsorbents

As shown in Table 4, adsorption capacity values were compared between the adsorbent used in this study, *Vaucheria* sp., and the adsorbents that had been used before, to remove Alizarin dye. The comparison revealed that *Vaucheria* sp algae performed better in terms of adsorption capacity.

Table 4. Comparison of the maximum adsorption of various adsorbents for Alizarin dye.

Adsorbents	Adsorbate	Q ₀ (mg/g)	References
Cobalt and Copper ferricyanides	Alizarin Red S	6.42 and 50.51	(Sharma et al 2015)
Modified clay	Alizarin Red S	32.7	(Fu et al 2011)
Mustard husk	Alizarin Red S	6.08	(Gautam at al 2013)
<i>Saccharum spontaneum</i>	Alizarin yellow	3.424	(Narayan at al 2015)
<i>Chara</i> sp.	Alizarin		(Sagar and Rastogi 2022)
<i>Vaucheria</i> sp.	Alizarin	83.33	This study

IV. CONCLUSIONS

The maximum adsorption capacity of Alizarin dye by *Vaucheria sp.* was found to be 83.33 mg/g at pH 4, contact time 900 min, dose 3g/L, and 318 K. The Langmuir isotherm was found to be the best fit with the equilibrium experimental data when compared to Freundlich, Temkin and D-R isotherm for alizarin dye. Analysis of data shows that the process followed pseudo second-order kinetics in comparison to the pseudo first-order kinetics. The thermodynamic treatment of equilibrium data shows feasible, spontaneous and endothermic nature of the adsorption process. The adsorbent was reused for five adsorption and desorption cycles with negligible decrease in their adsorption capacities. Thus, we conclude that the biomass of *Vaucheria sp.* has the potential to be used as an ecofriendly and economic adsorbent material for the removal of Alizarin dye from synthetic solutions.

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CONFLICT OF INTEREST

The author declares no conflict of interest.

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