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The equilibrium, kinetic and thermodynamic studies on the adsorptive removal of alizarin dye utilizing dead blue-green algal biomass

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Abstract

The removal of the anthraquinone dye Alizarin from synthetic aqueous solutions, by adsorption onto dead blue-green algal biomass, *Oscillatoria* sp., is detailed in this communication, as well as the kinetics, adsorption isotherms, and thermodynamics of the process. Using the optimized parameters from batch adsorption investigations, many adsorption equilibrium isotherms (Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich isotherm) and kinetic models (pseudo-first-order, pseudo-second-order, and intraparticle) were applied. Under the optimized conditions of 1 pH, 0.4 g/L adsorbent dose, 318 K temperature, and 120 minutes of contact time, the Langmuir isotherm analysis revealed the maximum monolayer adsorption capacity to be 80.64 mg/g. In a statistical comparison of regression results, it was determined that the Pseudo-second-order kinetic model and the Langmuir adsorption isotherm provided the best fits (R^2). The variations in the thermodynamic parameters indicate that adsorption is an endothermic, random, and spontaneous process in this study. With eluent containing 0.1 M HCl, up to 70% of the algal biomass used as the adsorbent material could be recovered, and the same biomass could be reused five times without appreciably decreasing its adsorption ability. Moreover, compared to other adsorbents, the *Oscillatoria* sp. algae utilized in our work showed a high capacity for Alizarin dye adsorption. This study implies that the biomass of *Oscillatoria* sp. could be a beneficial biomaterial for treating synthetic aqueous solutions containing alizarin dye.

Keywords: Alizarin, blue-green algal biomass, *Oscillatoria* sp. adsorption

1. Introduction

Due to the fact that dye chemicals are used extensively in the colouring process across a wide range of product categories, the industrial sector places a high value on the production of these chemicals. On the other hand, one of the most significant factors that leads to the issue of water pollution is the discharge of industrial effluents that contain dyes [1]. It is common knowledge that dyes can cause toxicity, cancer, and genetic mutations in humans [2]. In addition, dyes have the potential to cause discoloration in natural waters, which may have an adverse effect on the process of photosynthesis that is carried out by algae and other submerged plants [3]. Because of this, the removal of dyes from industrial effluents prior to their discharge into natural waters is a task that needs to be given the utmost concern.

Although numerous researchers have explored various treatment approaches, such as physical, chemical, and biological ones, to deal with effluents containing dyes, only a small number are actually used by the relevant industries due to limitations and unsuitability in terms of efficiency and economy [4]. When compared to competing methods for wastewater treatment, adsorption has been shown to be superior [5-6] because to its lower cost, simpler design, easier operation, and less susceptibility to hazardous substances. Additional studies using biomass-based adsorbents for dye removal have also demonstrated the method's benefits [7-8]. Alizarin is a type of anthraquinone dye that is used to colour fabrics. It is one of the most long-lasting dyes, which can't be broken down completely by chemical, physical, or biological processes. So, researchers are always looking for ways to clean up wastewaters that contain alizarin dye. There have been findings indicating that mango seeds and mustard husk are effective at removing alizarin dye [9-10].

Organic contaminants such as dyes could also be degraded by biomaterials such as algae [11]. There has recently been a surge of interest in the use of algae to remove coloured wastewater.

As a result, *Oscillatoria* sp., a locally accessible blue-green alga, is used as a bio adsorbent material in this communication. Previous research has shown that *Oscillatoria* sp. has a high adsorption capacity for dyes such as Basic blue 41, Methylene blue, and Methyl Orange [12-15]. In this study, we present our findings by using dead *Oscillatoria* sp. as a bioadsorbent to remove alizarin dye from synthetic aqueous solutions.

So, the primary goal of this communication is to estimate adsorption isotherms, kinetic parameters, and thermodynamic parameters for alizarin dye adsorption onto dead blue-green algal biomass *Oscillatoria* sp. Furthermore, reuse experiments of the bioadsorbent are reported, as well as a safe mode of disposal. In addition, the adsorption capacity for alizarin dye achieved using this test biomass was compared to a few different adsorbents.

2. Materials and Methods

2.1 Bioadsorbent and adsorbate preparation method

For the purpose of this study, an *Oscillatoria* species was selected to serve as the bioadsorbent. It is classified as a species of filamentous cyan bacterium. Before being dried on filter paper, the test biomass, collected from local ponds and rivers, was first washed in water that had been through a double distillation process to remove any debris or impurities. After being exposed to the Sun for two days, the biomass was then heated to 343 K for 24 hours in order to remove any trace of moisture. They were crushed even further with a mortar and pestle before being sieved through a 100 µm mesh. Vacuum desiccators were used to keep the powdered non-viable adsorbent dry.

SD Fine Chem. Ltd., India supplied this study's alizarin dye adsorbate. This commercially pure adsorbate can be utilized directly. It has the chemical name 1, 2-dihydroxyanthraquinone, the molecular formula $C_{14}H_8O_4$ and molecular weight of 240.21 g/mol. Artificial (synthetic) aqueous solutions were developed in the lab by mixing dyes with distilled water.

2.2 Adsorption experiments methodology followed

Various batch adsorption studies were carried out in 100 ml of dye solution with algal biomass at a constant temperature of 318 K and agitation speed of 200 rpm. Preliminary experiments were carried out to investigate the effect of initial dye concentration, pH, temperature, and contact time. In each experiment, the procedure of the test was performed under the condition that one parameter was changed at a time while the other parameter was fixed. The dye content in the solution before and after desorption was measured by UV/Vis spectrophotometer (model UV-Vis 119 Systronics India Ltd.) at λ_{max} of 567 nm. The pH readings were taken using a pH meter (PERFIT, India).

Adsorption isotherms were determined using a set of 100 ml of alizarin solutions with different initial concentrations (50, 100, 150 and 200 mg/L) at a fixed bioadsorbent dosage of 0.4 g/L. The contents were agitated for 120 min. of contact time to reach its maximal adsorption capacity levels. Similarly, the kinetic experiments were also carried out at 318K temperature, under optimized conditions at different dye initial concentrations. The dye solution was at pre-set time interval and filtered through a whatman filter paper to collect the supernatant. The amount of dye adsorbed by the adsorbent is determined using the equation:

$$q_e = (C_o - C_e)\, V/M \qquad (1)$$

Where q_e is the adsorption capacity of algae (mg/g), C_o and C_e are the initial and the equilibrium concentration of dye (mg/L), V is the volume of reaction mixture (L) and M is the mass of adsorbent used (g).

2.3 Data Analysis

Various models employed for the analysis of adsorption isotherm and kinetics for the adsorption of alizarin dye onto dried *Oscillatoria* sp. including their mathematical expressions are listed in Table 1.

Table 1: Various Models and Mathematical equations applied in this study

Parameter	Model	Mathematical equations and number	References
Adsorption Isotherm	Langmuir	$\frac{1}{q_e} = \frac{1}{Q_0} + \frac{1}{bQ_0C_e}$ (2) Where Qo and qe are the maximum and equilibrium adsorption capacity, Ce is equilibrium dye concentration and b is the Langmuir constant	[16]
	Freundlich	$\ln q_e = \ln K_F + \frac{1}{n}\ln C_e$ (3) Where K_F is the Freundlich constant and n is the intensity of adsorption.	[17]
	Temkin	$q_e = \frac{RT}{b_T}\ln(A_T C_e)$ (4) Where R is gas constant, T absolute temperature, b_T constant associated with the heat of adsorption and A_T constant associated with the heat of adsorption.	[18]
	D-R Isotherm	$\ln q_e = \ln q_m - \beta\varepsilon^2$ (5) $\varepsilon = RT\ln(1+\frac{1}{C_e})$ (6) Where q_m is maximum adsorption capacity, β constant related to adsorption energy and ε Polanyi potential	[19]
Adsorption Kinetic	Pseudo-First Order	$\log(q_e - q_t) = \log q_e - \frac{k_{1,ads}}{2.303}t$ (7) Where q_t is the amount of dye adsorbed at equilibrium time t and k_1 pseudo-first order rate constant.	[20]
	Pseudo-Second Order	$\frac{t}{q} = \frac{1}{k_{2,ads}q_e^2} + \frac{1}{q_e}t$ (8) Where q_e is the amount of the dye adsorbed at equilibrium, q amount of the dye adsorbed at time t and k_2 rate constant of second order adsorption	[21]

	Intra- Particle Diffusion	$q_t= k_{id}\, t^{1/2}$ (9) Where q_t is the amount of dye adsorbed per unit mass of adsorbent at time t and k_{id} intra-particle diffusion rate constant	[22]

2.4 Thermodynamic study

Using the following equations, the thermodynamic analysis is done to find the changes in standard free energy (ΔG°, kJ/mol), enthalpy (ΔH°, kJ/mol) and entropy (ΔS°, kJ/mol/K) at three different temperatures (298, 308, 318 K).

$$\Delta G^{\circ} = -RTln(b) \qquad (10)$$

$$\ln\left(\frac{b_2}{b_1}\right) = \frac{\Delta H_o}{R}\left(\frac{1}{T_2}-\frac{1}{T_1}\right) \qquad (11)$$

$$\Delta G^{\circ} = \Delta H^{\circ} - T\Delta S^{\circ} \qquad (12)$$

Where b_1 and b_2 are Langmuir constants at different temperatures and other terms have their usual meanings. The negative value ΔG° reflects the feasibility of adsorption and vice-versa, whereas positive and negative ΔH° values show the endothermic or exothermic nature of adsorption respectively. Similarly, positive or negative ΔS° gives an idea about the increase or decrease in randomness.

2.5 Regeneration study

To reduce secondary pollution, five cycles of repeated adsorption-desorption tests were performed using an acid, a base, and a chelating agent for Alizarin-dyed alga. Under optimal conditions, algal biomass was mixed with 1g L^{-1}dye solution. To determine the amount of dye that had been adsorbed, the remaining dye concentration in the solution was measured. After filtering, the adsorbent was vacuum-dried at 70 °C for 24 h. The dye-loaded adsorbent was then subjected to 50 ml of HCl (acid), NaOH (base), and EDTA (Ethylene diamine tetra acetic acid), a chelating agent, in a 100 ml conical flask under equilibrium conditions for five cycles using the same adsorbents. The UV-Vis Spectrophotometer measured the desorbed dye concentration. The desorption ratio is 100 times the dye desorbed/adsorbed ratio.

3. Results and discussion

3.1 Equilibrium modeling of adsorption

The equilibrium adsorption isotherms are of fundamental importance in the design of adsorption systems. The study of isotherm is helpful in determining the adsorption capacities of different adsorbents for the removal of synthetic dyes. In the present study the Langmuir, Freundlich, Temkin and Dubinin-Radushkevich adsorption isotherms at three different temperatures (298, 308 and 318K) were used to fit to the experimental data and their linear regression were used to find out the fit model among them. Values of resulting parameters and regression coefficients (R^2) are listed in Table 2.

Langmuir plots for Alizarin dye as given in Figure1, is found to be linear over the whole concentration range studied, and suggest the presence of a homogeneous surface, equivalent adsorption energies and no interaction between adsorbed species [23]. Values of Langmuir constants b and q_e were calculated from slope and intercept respectively from the plot $1/q_e$ *vs* $1/C_e$ (figure 1) and it has been observed that the values of both the constants increase with increase in temperature. Langmuir constant related with monolayer adsorption capacity were found to be 80.64mg/g at 318K for Alizarin dye. Similarly, constant related with adsorption energy varied from 0.056 to 0.069L/mg for Alizarin indicating higher value at higher temperatures. Linearity of each plot was evaluated in terms of linear regression coefficients (R^2) which were found to vary between 0.990-0.996 for alizarin dye. The values for the dimensionless constant (separation factor) for the equilibrium parameter, R_L, was calculated using the Langmuir constant, b, as follows.

$$R_L = 1/(1 + bC_o) \qquad (13)$$

R_L values was found to be less than unity and greater than zero at all temperatures, confirming the favorable adsorption process.

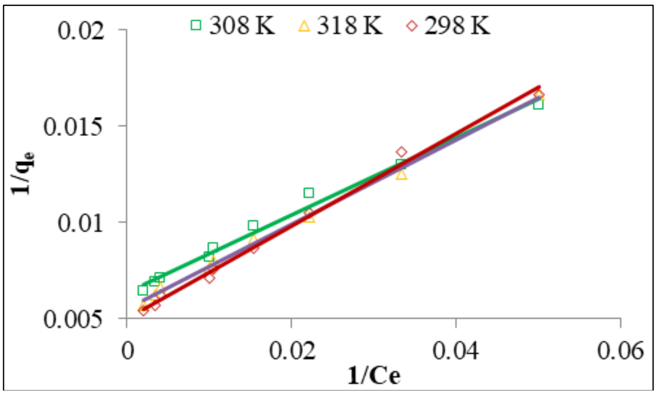

Fig 1: Langmuir plots for the adsorption of Alizarin dye onto dead *Oscillatoria* sp.

Similarly, adsorption data for the test dye was also analyzed by Freundlich plots at three different temperatures and the Figure 2 shows that the plots exhibit slight deviation from linearity thus allowing limited multilayer adsorption [24]. The values of Freundlich constants n and K_F were calculated from slope and intercept respectively and these values along with correlation coefficients are included in Table 2. Freundlich heterogeneity factor n was high indicating high affinity between the solute molecules and the adsorbents. On the contrary, values of Freundlich constants K_F (an indicative of adsorption capacity) was found to vary from 2.360-34.320mg/g and the values of coefficient correlation ranged from 0.967-0.964 for this dye.

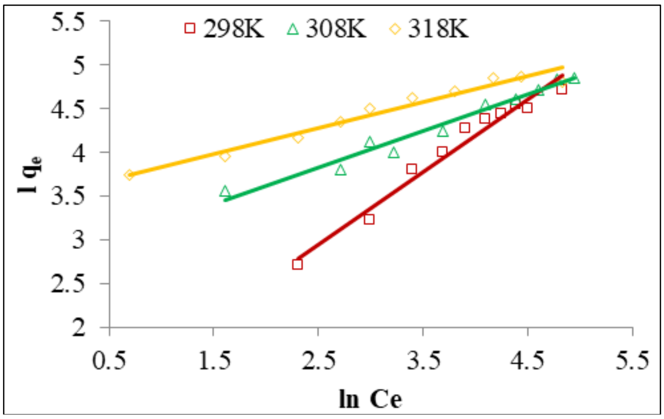

Fig 2: Freundlich plots for the adsorption of Alizarin dye onto dead *Oscillatoria* sp.

The Temkin model takes into account the interaction between adsorbents and adsorbate to be adsorbed and is based on the assumption that the free energy of adsorption is a function of surface coverage. A plot of qe versus ln C_e enables the determination of the isotherm constants A_T and b_T from the intercept and the slope, respectively (figure 3). The results also fitted the Temkin model (Table 2), which suggested a reduction in heat of adsorption along with coverage due to adsorbent-adsorbate interaction [25].

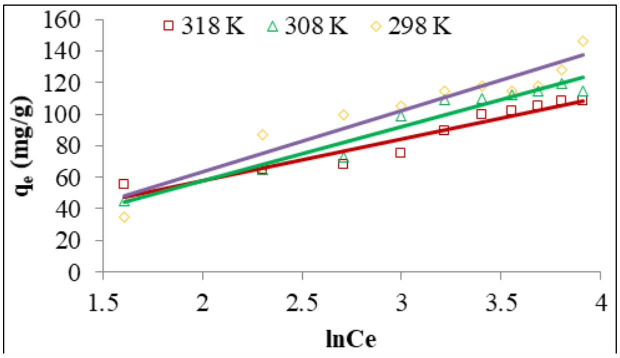

Fig 3: Temkin plots for the adsorption of Alizarin dye onto dead *Oscillatoria* sp.

The Dubinin and Radushkevich (D-R) isotherm was chosen to calculate the porosity apparent free energy (figure 4). This isotherm does not assume a homogeneous surface or constant adsorption potential but is related to the porous structure of the adsorbent. The calculated value of E, the mean free energy of adsorption is found to be less than 8kJ/mol, which indicates physical adsorption [26].

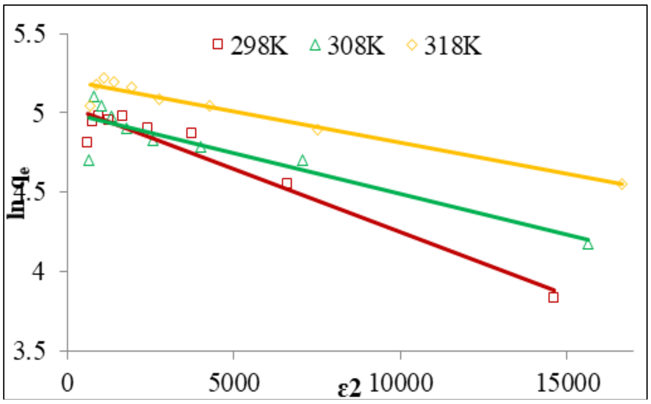

Fig 4: D-R plots for the adsorption of Alizarin dye onto dead *Oscillatoria* sp.

The correlation coefficient shows that the Langmuir model provides a significantly better fit to the experimental data compared to other models studied ($R^2 \geq 0.99$). This proved that a monolayer of dye molecules had formed on the adsorbent's surface, indicating its homogeneous appearance. Also, the adsorption capacity values obtained by the Langmuir isotherm is much higher having a value of 80.64 mg/g at 318K. Iqbal *et al.* and Fu *et al.*, using activated charcoal and activated clay, respectively, reported similar results of isotherms for the adsorption of Alizarin Red S dye [27-28].

Table 2: Isotherm parameters for the adsorption of Alizarin dye onto dead blue-green algal biomass (*Oscillatoria* sp.)

Isotherm Parameters	**Alizarin**		
	298 K	**308 K**	**318K**
Langmuir Isotherm			
b (L mg^{-1})	0.056	0.060	0.069
q_e (mg g^{-1})	52.08	64.51	80.64
R^2	0.990	0.993	0.996
Dimensionless Separation Factor			
R_L	0.079	0.075	0.065
Freundlich Isotherm			
N	1.197	2.398	3.367
K_F (mg g^{-1})	2.36	16.19	34.32
R^2	0.967	0.968	0.964
Temkin Isotherm			
A_T	0.803	0.869	0.948
b_T	64.01	74.91	100.45
R^2	0.909	0.946	0.927
D-R isotherm			
q_m (mg g^{-1})	63.50	79.33	91.14
E (kJ mol^{-1})	0.79	1.00	1.11
R^2	0.944	0.819	0.929

3.2 Thermodynamics of adsorption

Adsorption is temperature dependent process that is associated with three thermodynamic parameters namely change in enthalpy of adsorption (ΔH), change in entropy (ΔS), and change in Gibb's free energy (ΔG). Table 3. represents the values of ΔG°, ΔH°, and ΔS° for adsorption of Alizarin onto *Oscillatoria* sp. The negative value of ΔG° confirms the thermodynamic feasibility and spontaneity of the process, while the increase in its absolute values with temperature (-23.58 to -25.71 for Alizarin) points to an increase in such a tendency for the dyes. On the other hand, the positive value of ΔH° confirms the endothermic nature of dyes adsorption, and the values are also found to be less than 25KJ/mol (8.134 Alizarin) at 318K, which also approves the physical nature of adsorption [39]. The positive value of ΔS° reflects the increased randomness at the alga-solution interface during the adsorption process. These results are quantitatively similar to those obtained by other researchers for the removal of dyes by different adsorbents. Kousha *et al.* have reported the negative value of ΔG° and the positive value of ΔH°, and ΔS° for the removal of Acid Orange II dye by brown macro alga *Stoechospermum marginatum* [30].

Table 3: Thermodynamic Parameters estimated for the adsorption of Alizarin dye onto dead *Oscillatoria* sp

Test Dye	Thermodynamic parameters	Temperature 298 K	Temperature 308 K	Temperature 318 K
Alizarin	ΔG°	-23.58	-24.54	-25.71
	ΔS°	0.1064	0.1066	0.1070
	ΔH°*	8.134		

*ΔH° Measured between 298 and 318K

3.3 Kinetic modeling

Kinetic models are widely used for interpreting the dynamic interaction between adsorbent and adsorbate. Also, it is employed to ascertain the potential rate-controlling steps concerned in the process of adsorption of dye molecules onto the adsorbent. The pseudo-first-order, pseudo-second-order rate equation, and Intra-particle diffusion kinetic models at two different concentrations (100 mg/L and 200 mg/L) were tested to fit the experimental data.

The kinetic data from the study of contact time and concentration, are re-plotted according to pseudo-first-order equation. The plot is given in Figure 5 for the Alizarin dye. The value of maximum adsorption q_e (calculated) and pseudo-first order rate constant K_1 for the test dye was calculated from intercept and slope respectively and are given in Table 4, along with R^2 of the respective plot. The result in Table 4 depicts the values of R^2 are low (0.919) for Alizarin and the q_e (calculated) values do not agree well with the q_e (experimental) values.

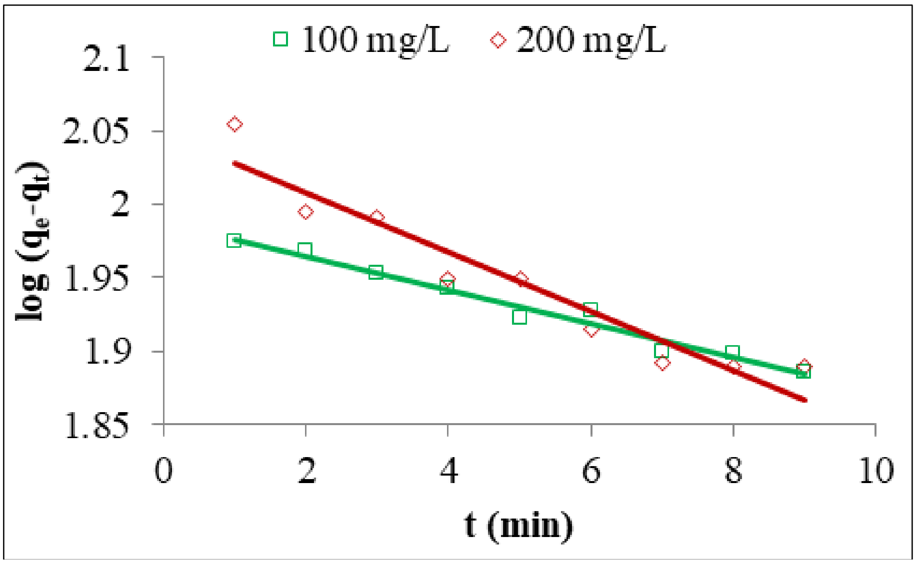

Fig 5: Pseudo-first-order kinetic modeling of adsorption of Alizarin dye onto blue-green algal biomass *Oscillatoria* sp. (adsorbent dose: 0.4g/L, temperature: 318K)

Similarly, the plot of pseudo-second-order model is also drawn in the same way using second-order equation and the plot is shown in Figure 6. The value of maximum equilibrium adsorption (q_e) and rate constant (K_2) for test dye is calculated from the intercept and slope of plot respectively and is given in Table 4 along with the correlation coefficient of the plots. The plot t/q versus t (Figure 6) gives a straight line and the values of correlation coefficient R^2 for this model is relatively high and the adsorption capacities calculated are also close to those determined by experiments.

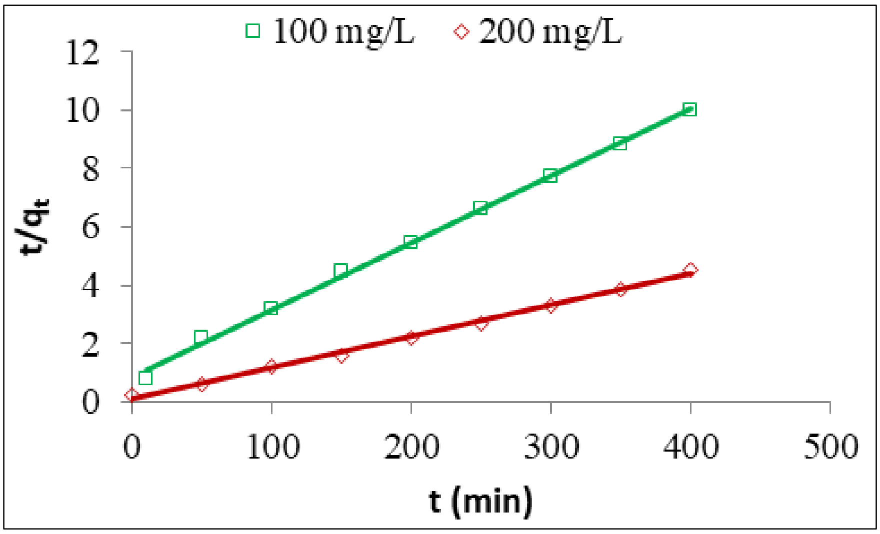

Fig 6: Pseudo-second-order kinetic modeling of adsorption of Alizarin dye onto algal biomass *Oscillatoria* sp. (adsorbent dose: 0.4g/L, temperature: 318K)

The intra-particle diffusion model was used to assess the contribution of the diffusion of dyes within the algal biomass in the whole adsorption process. Figure 7 displays the plot of q_t versus $t^{0.5}$ and the slope of the linear portion was defined as rate parameter K_W. Table 4 shows that the correlation coefficient value (R^2= 0.912 for Alizarin) obtained at 200 mg/L was low. The perusal of the graphs indicated that it was not linear and did not pass through the origin for the Alizarin dye and so is not the only rate-limiting step as stated earlier by Gupta *et al.* [31].

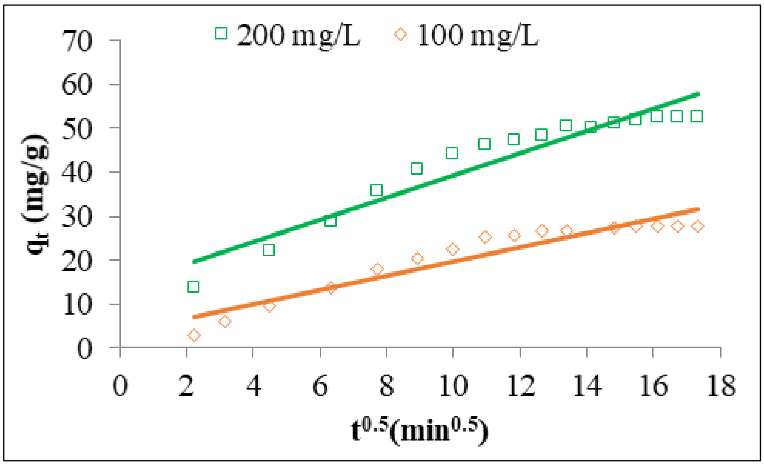

Fig 7: Intra-particle diffusion plot for the adsorption of Alizarin dye onto algal biomass *Oscillatoria* sp. (adsorbent dose: 0.4g/L, temperature: 318K)

Therefore, it can be concluded that as the q_e (calculated) value was closer to q_e (Experimental), and the R^2 value was high and close to unity for the Pseudo-second order adsorption model, thus, it is more suitable to describe the adsorption kinetics of dyes over algal biomass.

Table 4: Kinetic parameters estimated by various models for blue-green algal biomass *Oscillatoria* sp. at two different concentrations of dye

Dye	Initial dye conc. (mgL^{-1})	q_e (exp.) (mg g^{-1})	First-order model			Second-order model			Intra-particle model	
			K_1(x10^{-3} min^{-1})	q_e (mg g^{-1})	R^2	K_2 x 10^{-3} (g mg^{-1} min^{-1})	q_e (mg g^{-1})	R^2	K_W (mg g^{-1} min$^{0.5}$)	R^2
Alizarin	100	22.59	25.33	97.05	0.969	0.563	23.95	0.998	1.625	0.902
	200	85.75	46.06	110.40	0.919	1.030	84.47	0.995	2.532	0.912

3.4 Bioadsorbent Regeneration studies

Regeneration of adsorbent for repeated reuse is of crucial importance in the industrial practice for dye removal from wastewater. For this purpose, the algal biomass was eluted using HCl, NaOH and EDTA, the adsorption-desorption cycles of the dye were repeated five times using the same adsorbent. The effect of various reagents used for desorption studies shows that HCl is a better reagent for desorption. Hydrochloric acid showed the maximum desorption efficiency for Alizarin. Stirk and Staden found that acids are more effective for desorbing as compared to other eluents [32]. The adsorption capacity of the tested alga did not noticeably change (Only a maximum of 20-25% change was observed) during repeated adsorption-desorption operations. The desorption efficiency decreases with an increase in the number of cycles due to the decrease in the adsorption capacity as shown in Figure 8. Hu and Shipley also concluded similar results [33]. Thus, the reuse of biomass is an important feature for its possible utilization in the continuous system in industrial processes.

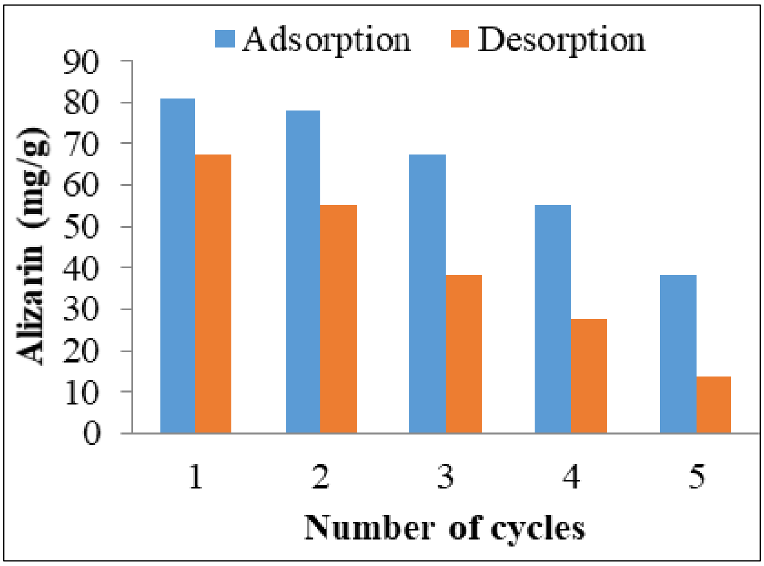

Fig 8: Adsorption/Desorption cycles of Alizarin dye onto dead *Oscillatoria* sp. using HCl

3.5 Disposal of the used bioadsorbent

For the ultimate disposal, the used biomass was sterilized by microwave irradiation for 10 minutes followed by encapsulation in concrete very deep pits. This was done in order to reduce the process's impact on the environment.

3.6 Comparison with other adsorbents

A comparison has been made between the adsorbent used in the present study (i.e. *Oscillatoria* sp.) and previously used adsorbents for the removal of Alizarin dye (Table 5). The comparison showed that the algae used in the present study as an adsorbent showed better results in terms of adsorption capacity.

Table 5: Comparison of the maximum adsorption of various adsorbates onto various adsorbents

Adsorbents	Adsorbate	Q_0 (mg/g)	References
Mustard husk	Alizarin Red S	6.08	[10]
Activated charcoal	Alizarin	8.97	[27]
Modified clay	Alizarin Red S	32.7	[28]
Activated carbon	Alizarin	108.69	[34]
Cobalt and Copper ferricyanides	Alizarin Red S	6.42 and 50.51	[35]
Saccharum spontaneum	Alizarin yellow	3.424	[36]
Chara sp.	Alizarin	76.92	[37]
Vaucheria sp.	Alizarin	83.33	[38]
Oscillatoria sp.	Alizarin	80.64	This study

4. Conclusions

At optimal conditions of pH 1, contact period 120 min, dosage 0.4 g/L, and temperature 318 K, this study reports the maximum adsorption capacity of *Oscillatoria* sp. for Alizarin dye to be 80.64 mg/g. By comparing various adsorption isotherms models to the experimental equilibrium data for alizarin dye, the Langmuir isotherm was found to provide the greatest fit. After observing the data, it became clear that the process exhibited pseudo-second-order kinetics rather than pseudo-first-order dynamics. The adsorption process is feasible, spontaneous, and endothermic between 298 K and 318 K, as shown by a thermodynamic analysis of equilibrium data. The adsorbent was subjected to five cycles of adsorption and desorption with only a minor loss of adsorption capacity after each use. Hence, the results of this work support the use of *Oscillatoria* sp. blue-green algal biomass as a bioadsorbent for the sustainable and effective removal of Alizarin dye from synthetic aqueous solutions.

5. Acknowledgement

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6. Conflict of interest

The author declares no conflict of interest.

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