



EQUILIBRIUM AND KINETIC MODELING OF ACID RED 88(AR88) BIOSORPTION BY ULVA RETICULATA

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ABSTRACT

Equilibrium, kinetics and thermodynamic studies on the removal of Acid Red (AR88) using *Ulva reticulata* have been investigated. In batch experiments, the parameters studied included the effect of the dye concentration, temperature, contact time, adsorbent dosage, pH. The pseudo-first-order and pseudo-second-order kinetic data were tested with experimental data. The optimum conditions were: pH 5, temperature 30°C, biomass size 0.5 mm and maximum metal uptake was 75.4 mg/g. Various thermodynamic parameters such as ΔH° , ΔG° and ΔS° were calculated indicating that the present system was spontaneous and endothermic process. The pseudo first and second order kinetic models were also applied to the experimental kinetic data obtained during Biosorption of AR88 and High correlation coefficients with low standard deviations favor the pseudo-second-order model for the present systems. The ability of *Ulva reticulata* to Biosorption AR88 in a packed column was investigated, as well. A glass column was used to conduct the experiments. At 20 cm (bed height), 5 ml/min (flow rate) and 100 mg/L (initial concentrations), *ulva reticulata* exhibited AR88 uptake of 88.65 mg/g. The Thomas model was used to predict the breakthrough curves.

Keywords: Biosorption, Acid dyes, *Ulva reticulata*, Kinetics, Thermodynamics, Packed column.

Contribution/ Originality

This study contributes in the existing literature that the selected non -living biomass is growth -independent and not subjected to toxicity limitations of the cells. In this work no costly nutrient is required for the growth of cells in the feed solutions.in this study the selected biomass behaves as an ion exchanger and hence, the process is very rapid and takes place between few minutes and few hours.in this work the primary contribution is utilizing the availability source of algae in uvari and treating dyes effluents.

1. INTRODUCTION

Pollution caused by wastewater has become a major problem in India. Pollutions from industrial effluents disturb human health and ecological systems [1]. Treatment of dyes from wastewater is still of major environmental problem, because they are difficult to remove chemicals, oxidizing agents, light, and heat and are biologically nondegradable [2]. Existing colored wastewater treatment methods involve combination of physical and chemical processes including adsorption, precipitation, sedimentation, ultrafiltration, reverse osmosis, flotation, color irradiation, oxidation, ozonation, and coagulation.

However, the use of these technologies in dyes removal are restricted due to technical and economic reasons ranging from little applicability to a wide range of dye wastewaters to high operating costs [3, 4]. The removal of synthetic dyes from aquatic systems, is extremely important from the healthiness viewpoint [5] because most of these dyes are toxic, causing allergy, skin irritation, besides most of them are mutagenic and/or carcinogenic. Therefore, industrial effluents need to be treated before being delivered to environment. Most of the industries in Erode, Tirupur, i.e. textiles, paper, plastic, leather, food, cosmetics, etc. Such extensive use of dyes often possess problems in the form of colored wastewater that require pretreatment for color prior to disposal into receiving water bodies or publicly owned treatment works.

The methods of color removal from effluents include biological treatment, coagulation, flotation, adsorption, oxidation, hyper filtration etc. [6]. Currently the research community is focused on technologies for the treatment of polluted environment that are less costly and ecofriendly. Physical and chemical methods used for the Biosorption are often cost prohibitive while biological methods are relatively cheap as well as ecofriendly. The most efficient procedure for removal of synthetic dyes from industrial effluents is the adsorption procedure, because the dyes species are transformed from the water effluent to a solid phase, diminishing the effluent to a volume to a minimum. Subsequently, adsorbent can be regenerated or kept in a dry place without direct contact with the environment. Biosorption technology is a promising alternative method to treat this kind of materials with high % removals.

The objective of this research was to evaluate the feasibility of Biosorption potential of *Ulva reticulata* on the removal of acid dyes Acid Red 88(AR88) from aqueous solutions in batch and continuous modes of operation.

2. MATERIALS AND METHODS

2.1. Biomass Preparation

The biomass *Ulva reticulata* was collected from seashore near southern coast of India (uvari, thirunelveli district). After harvesting from sea, the samples were washed with distilled water to

remove particulate materials and salts from the surface. They were then dried in an oven at 60°C for 24 hrs. [7].

2.2. Batch Experiments

Batch mode adsorption studies were carried out to investigate the effect of different parameters such as initial concentration, Temperature, pH, biomass dosage. Batch Biosorption experiments were performed in a rotary shaker at 150 rpm using 250ml Erlenmeyer flasks containing 0.5 g of *Ulva reticulata* biomass in 50 ml solution containing different dye concentrations. After 6 h, the reaction mixture was centrifuged at 2000 rpm for 10 min. The dye content in the supernatant was determined using UV-Spectrophotometric(Hitachi Japan) at respective wavelength of λ_{max} 503. The amount of dye biosorbed per unit mass was calculated from the difference between the dye quantity added to the biomass and the dye content of the supernatant using the following equation:

$$q_e = (C_0 - C_e) * V / M \quad (1)$$

Where q_e is the dye uptake (mg/g), C_0 and C_e are the initial and final dye concentrations in the solutions (mg/L), respectively; V is the solution volume (L), and M is the mass of biosorbent (g).

For kinetic experiments, samples were taken at regular time intervals and analyzed for dye concentrations. To evaluate the differences in the Biosorption rates and uptakes, the kinetics data were described with pseudo-first and pseudo second-order models. The linearized form of pseudo-first and pseudo-second order model [8] are shown below as Eqs. (2) and (3), respectively:

$$\log(q_e - q_t) = \log q_e - k_{ad}t / 2.303 \quad (2)$$

$$\frac{dq_t}{dt} = K_{ad}(q_e - q_t) \quad (3)$$

Where q_e is the amount of dye sorbed at equilibrium (mg/g), q_t the amount of dye sorbed at time t (mg/g), k_1 the first-order rate amount(1/min), and k_2 is the second order Rate constant (g/mg min).

2.3. Isotherm Models

The Langmuir and freiundlich isotherm has been used to characterization of dyes solutions. These isotherms are the following

$$\text{Langmuir; } \theta = \frac{q_e}{Q_m} = \frac{bC_e}{1 + bC_e} \quad (4)$$

$$\text{Freiundlich: } q = K_f C_f^{1/n} \quad (5)$$

Where q_{\max} is the maximum dye uptake (mg/g), b the Langmuir equilibrium constant (L/mg), K_f is the Freundlich constant (l/g), n is the Freundlich constant. Langmuir and Freundlich models were evaluated by non-linear regression using MATLAB software.

2.4. Batch Studies

The batch kinetic data were analyzed using pseudo-first order and pseudo-second order models. The linearized form of pseudo-first and pseudo-second order model has been described in Eqs. (6) and (7), respectively:

$$\log (q_e - q_t) = \log q_e - k_{ad}t / 2.303 \quad (6)$$

$$\frac{dq_t}{dt} = K_{ad}(q_e - q_t) \quad (7)$$

Where q_e is the amount of dye sorbed at equilibrium (mg/g), q_t the amount of dye sorbed at time t (mg/g), k_1 the first-order rate constant (1/min), and k_2 is the second order Rate constant (g/mg min).

2.5. Column Experiment

Fixed bed Biosorption were conducted in a glass column internal diameter 3cm and height 30 cm [9]. A known quantity of *Ulva reticulata* was packed in the column to yield the desired bed height of the sorbent. A peristaltic pump (Miclins) was used to pump the known concentration (100 mg/L) of dye solution (pH 5) through the column in the upward direction. The aliquots of dye at the outlet of the column were collected at regular time intervals. The operation of the column was stopped when the effluent dye concentration exceeded a value of 100 mg/L.

The breakthrough time (t_b , the time at which dye concentration in the effluent reached 1 mg/L) and bed exhaustion time (t_e , the time at which dye concentration in the effluent reached 100 mg/L) were used to evaluate the breakthrough curves. The slope of the breakthrough curve (dc/dt) was determined from t_b to t_e . The total quantity of dye mass biosorbed in the column (m_{ad}) is calculated from the area above the breakthrough curve (outlet dye concentration c vs. time (t)) multiplied by the flow rate. Dividing the dye mass (m_{ad}) by the sorbent mass (M) leads to the uptake capacity (q) of the algae.

Effluent volume (V_{eff}) can be calculated as follows.

$$V_{eff} = F.t_e * 60 / 1000 \quad (8)$$

F is the volumetric flow rate (mL/min).

Total amount dye sent to column (m_{total}) can be calculated as follows

$$m_{total} = C_o F t_e / 1000 \quad (9)$$

C_o is the inlet dye concentration (mg/L)

Total dye removal percent with respect to flow volume can be calculated as follows:

$$\text{Total dye removal (\%)} = m_{ad}/m_{total} * 100$$

Batch and continuous experiments were operated at 30°C.

3. RESULTS AND DISCUSSION

3.1. Batch Method

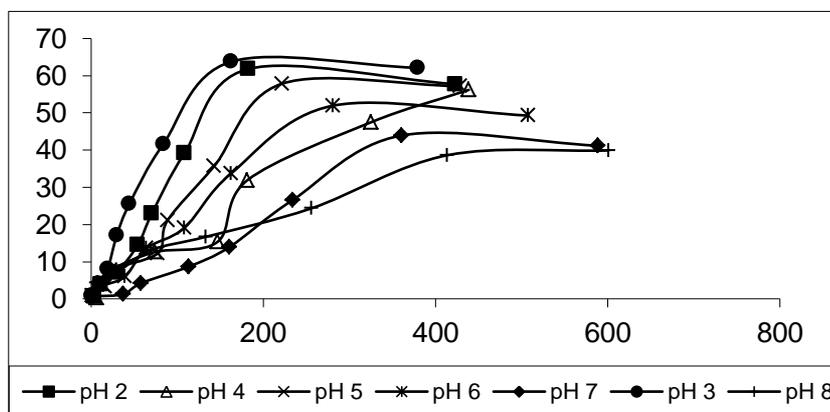
The prediction of batch sorption kinetics is necessary for the design of industrial sorption columns. The nature of the sorption process depends on physical or chemical characteristic of the biosorbent and also on the system conditions. In this study, the applicability of the pseudo-first order and pseudo second-order model has been tested for the sorption of Acid Red 88 onto *Ulva reticulata*. The best fit model was selected based on the regression coefficient R² values.

3.2. Effect of Initial Concentrations

The experimental results of acid red 88 Biosorption as function of time on *ulva reticulata* at various initial concentrations have been shown in Fig.1. The process was found to be initially very rapid, thereafter the dye uptake process tended to proceed at a slow rate. The initial rapid uptake of the dye indicates that the sorption process could be ionic in nature where the acidic (anionic) dye molecules bind to the various positively charged organic functional groups present on the surface of the biomass [8]. The result explained that increasing the dye concentrations with increase dye uptake and reached constant value and the total percent removal decreased. On changing initial AR88 concentrations from 10 to 1000 mg/L, the amount sorbed increased from 1.17 to 45.6 mg/g at pH 5. But the removal efficiency decreased from 46.6 to 14% as the AR88 concentration increases from 10 to 1000 mg/l.

This is because at lower concentration the ratio of the initial moles of dye molecules to available surface area is low and subsequently the fractional sorption becomes independent of initial concentrations.

Figure-1. Effect of initial dye concentration on uptake Capacity of *Ulva reticulata* (Time (h) vs uptake (mg/g))



However, at higher concentrations the available sites of sorption become fewer compared to the moles of dye present and hence the percentage dye removal is dependent upon the initial concentrations. It can be concluded that the rate of AR88 binding with alga biomass is more at initial stages, which gradually decrease and remains almost constant after an optimum period of 7-8 hr.

In order to obtain the rate constants and equilibrium dye uptake, the straight line plots of $\log(q_e - q_t)$ against t of Eq.2 were made for *Ulva reticulata* at different initial dye concentrations (Figure not presented). The intercept of the above plot should be equal to $\log q_e$. The rate constants, corresponding correlation coefficients for all concentrations tested have been summarized in Table 1. For AR88, correlation coefficients were found to be 0.965, but the calculated q_e is not equal to experimental q_e , suggesting the insufficiency of pseudo first order model to fit the kinetic data for the initial concentrations examined. The reason for these differences in the q_e values is that there is a time lag, possibly due to a boundary layer or external resistance controlling beginning of the sorption process (7).

Table-1. Pseudo-first order constants

C_0 (mg/L)	$(q_e)_{exp}$ (mg/g)	Pseudo-first order		
		K_1 (l/min)	q_e (mg/g)	R^2
100	12.42	0.322	9.8	0.954
300	22.6	0.336	33.5	0.924
500	33.7	0.376	43.6	0.945
700	48.9	0.355	39.8	0.966
1000	55.4	0.387	37.6	0.976

Table-2. Pseudo-first order constants'

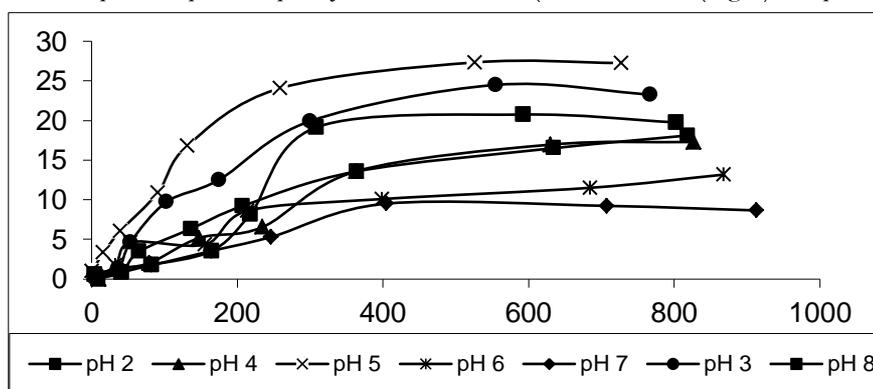
C_0 (mg/L)	$(q_e)_{exp}$ (mg/g)	Pseudo-second order		
		K_2 (g/mg min)	q_e (mg/g)	R^2
100	10.4	0.022	12.3	0.994
300	23.6	0.016	34.5	0.992
500	23.7	0.016	53.6	0.993
700	28.9	0.015	59.8	0.997
1000	35.9	0.027	67.9	0.998

The R^2 value were found to be in the range of 0.954 to 0.976 for pseudo first and 0.994 to 0.998 for second order model. Hence high correlation coefficient R^2 values suggest that the sorption data is described by pseudo second order. Contrary to other well- established models, it predicts the behavior over the whole range of studies and it is in agreement with the chemisorption mechanism being the rate controlling step.

3.3. Effect of pH

The adsorption process increases with increase in the pH. The pH value of the solution was an important controlling parameter in the adsorption process. The percentage sorption of Acid Red88 at every concentration was minimum at the initial pH 2, then increased and remained nearly constant over the initial pH ranges of 5-8 (Fig.3). The increase of temperature led to slight increase in dye uptake, this indicates that the adsorption of the AR88 by *Ulva reticulata* is endothermic in nature. When the temperature was increased, the mobility of the dye molecules increased and the retarding forces on the diffusing ions decreased, thereby increasing the sorption capacity of the adsorbent. The percentage sorption of Acid Red88 was not significantly altered when the initial pH was increased from 5 to 8. So pH 5 was chosen for the study on the effect of sorbent dosage.

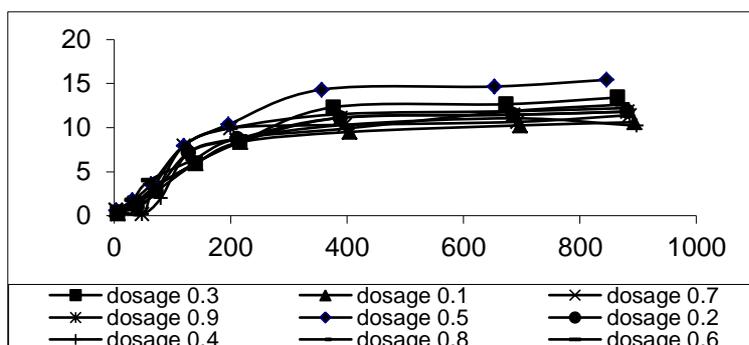
Figure-2. Effect pH on uptake capacity of *Ulva reticulata* (concentration (mg/l) vs uptake (mg/g))



3.4. Effect of Sorbent Dosage

The adsorption of dyes is seen to increase with the sorbent dosage and reached an equilibrium value after 0.5 g of sorbent dosage in (Fig.5). Higher percentage of adsorption with the increase of adsorbent concentration may be due to the availability of more surface area, after which equilibrium is achieved. However, the biosorption capacity (mg/g) decreased with increase in biosorbent dosage, due to the agglomeration of the adsorbent particles. When the dried *Ulva reticulata* was increased from 0.5 to 1.0 g the ratio of dye sorbed to biomass (mg/g) showed no significance different. So 0.5 g of dried *Ulva reticulata* biomass was chosen for the next study on the effect of contact time.

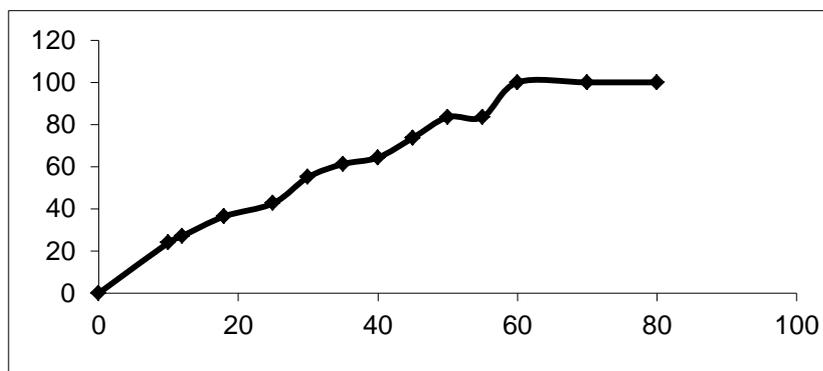
Fig-3. Effect of dosage on uptake capacity of *Ulva reticulata* (concentration (mg/l) vs uptake (mg/g))



3.5. Effect of Contact Time

Dried *Ulva reticulata* (0.5) g was shaken in 100 ml solution at concentration of 10 to 1000 mg/L at 30°C of pH 5 on a rotary shaker operating at 200 rpm. Aliquots of 2 ml solution were withdrawn at different time intervals, filtered and analyzed for the dye content using a UV Spectrometer, until no more dye was removed and the equilibrium was achieved. It can be observed that percentage uptake was increased with time and, at some point in time, reached a constant value where no more dye was removed from the solution (Fig.4.). At this point, the amount of dye being adsorbed onto the adsorbent was in at age of dynamic equilibrium with the amount of dye adsorbed from the adsorbent. The time required to attain this state of equilibrium was termed equilibrium time and the amount of dye adsorbed at the equilibrium time reflected the maximum dye adsorption capacity of the adsorbent under these particular conditions. It was observed that maximum adsorption equilibrium was attained during first 60 min. The dye concentration showed no significant difference when the contact times were longer than these. The maximum amounts of dye adsorbed were 76 and 87 mg/g for 500 and 1000 mg/L respectively).

Fig-4. Effect of contact time on *Ulva reticulata* (Time (min) vs % Removal)



3.6. Adsorption Isotherm Study

In the present study, two equilibrium models were analyzed to investigate the suitable adsorption isotherms. Table.3 shows the model constants along with correlation coefficients obtained from four isotherm models. By comparing all R^2 value it was observed that the R^2 value were found to be higher in Freundlich isotherm. In order to understand the kinetics of dye adsorption using *Ulva reticulata* as an adsorbent, pseudo first-order and second-order kinetic models were tested with the experimental data. The values were calculated and listed in Table 3. The maximum correlation coefficients were obtained in pseudo second order kinetics. On the basis of correlation coefficient values, it can be concluded that pseudo second order model may be suitable for the present systems.

3.7. Thermodynamic Properties

Based on the fundamental thermodynamic properties, it is assumed that in an isolated system, energy cannot be gained or lost and the entropy change is the only driving force. In chemical engineering aspects, both energy and entropy factors will occur spontaneously [10]. The Gibbs free energy, ΔG° , is the fundamental criterion of spontaneity. It can be calculated from

$$\Delta G^\circ = -RT \ln b \quad (10)$$

Where R is the gas constant (8.314 J/mole K) and T is the absolute temperature (K). The relationship between Gibbs free energy change, Entropy change (ΔS°) and enthalpy change (ΔH°) can be expressed as

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (11)$$

The value of ΔG° was obtained as -11.02, -11.23, -15.06 and -18.34 KJ/mol at 20, 30 and 45°C. This indicated that the magnitude of ΔG° increases with increase in temperature. The negative value of ΔG° confirmed the feasibility of the process and the spontaneous nature of sorption of AR88 onto *Ulva reticulata*. The values of ΔH° and ΔS° were positive (53.55 KJ/mol and 0.286 KJ/mol), indicating that the binding of AR88 to alga was endothermic. This enthalpy change agrees well with those reported in the literature [10, 11]. Also the ΔS° was observed positive, indicating the increasing randomness at the solid/liquid interface during the Biosorption [10].

3.8. Column Studies

Biosorption of acid red 88 by *Ulva reticulata* was presented in the form of breakthrough curves. Fig. 5 shows the breakthrough profile of Acid Red AR88 Biosorption for different bed heights (10, 15, 20 cm). In order to yield different bed heights 3.5, 6.5 and 8.5 g of biomass were added to produce 10, 15 and 20 cm respectively. The inlet concentration 100 mg/L and the flow rate (5 mL/min) were kept constant. The uptake of AR88 increased in bed height shown in (Table.4). The increase in AR88 uptake capacity with the increase in the bed height in the column was due to increase in the surface area of Biosorption [12]

Table-3. Isotherm model parameters for *Ulva reticulata* on AR88

Langmuir model				Freundlich model			
pH	$Q_{max}(mg/g)$	$b(L/mg)$	R^2	pH	$K_F(L/g)$	n	R^2
2	18	0.00245	0.969	2	0.32	1.1	0.999
3	15	0.002657	0.947	3	0.244	1.43	0.987
4	14.985	0.002786	0.945	4	0.336	1.54	0.968
5	12.92	0.0026	0.947	5	0.226	1.1	0.979
6	15.76	0.00298	0.968	6	0.327	1.34	0.999
7	16.54	0.002874	0.982	7	0.354	1.93	0.969
8	14.5	0.0065	0.969	8	0.432	1.29	0.999
Temp	$Q_{max}(mg/g)$	$bL/mg)$	R^2	Temp	$K_F(L/g)$	n	R^2
20	20	0.0032	0.945	20	0.33	1.1	0.968
30	23.43	0.0045	0.876	30	0.23	1.65	0.977
40	26.87	0.0056	0.896	40	0.54	1.94	0.987
50	29.46	0.0054	0.859	50	0.28	1.45	0.989
Dosage	$Q_{max}(mg/g)$	$b(L/mg)$	R^2	Dosage	$K_F(L/g)$	n	R^2
0.1	14.32	0.0045	0.956	0.1	0.33	1.89	0.968
0.2	14.56	0.0098	0.934	0.2	0.223	1.58	0.989
0.3	15.65	0.0076	0.965	0.3	0.334	1.47	0.994
0.4	12.54	0.0056	0.936	0.4	0.335	1.94	0.995
0.5	11.76	0.0038	0.986	0.5	0.435	1.93	0.996
0.6	16.76	0.0069	0.948	0.6	0.224	1.11	0.957
0.7	18.45	0.093	0.928	0.7	0.254	1.34	0.984
0.8	17.45	0.0078	0.917	0.8	0.228	1.65	0.995
0.9	15.2	0.0076	0.938	0.9	0.228	1.54	1

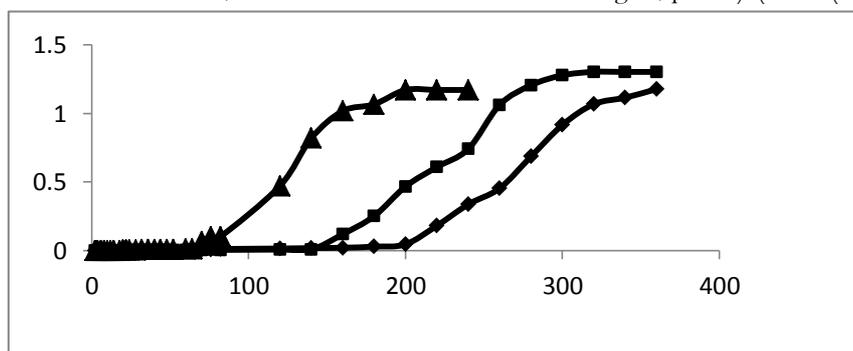
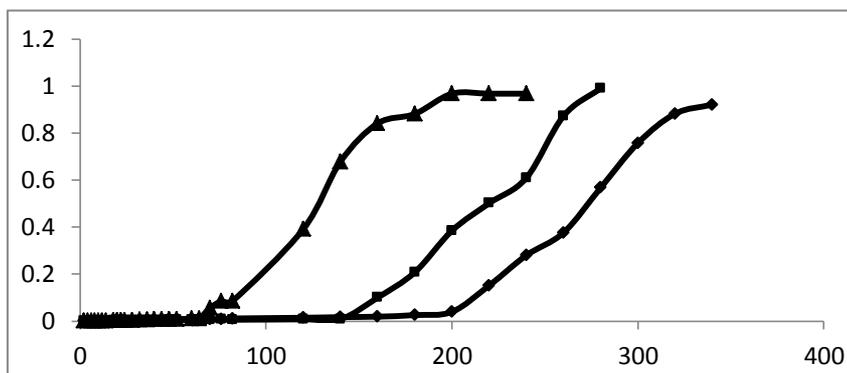
Figure-5. Breakthrough curve for AR88 Biosorption onto *Ulva reticulata* biomass at different bed heights (flow rate=5 mL/min, initial AR88 concentration=100 mg/L, pH=5). (Time (h) vs C/C_0)

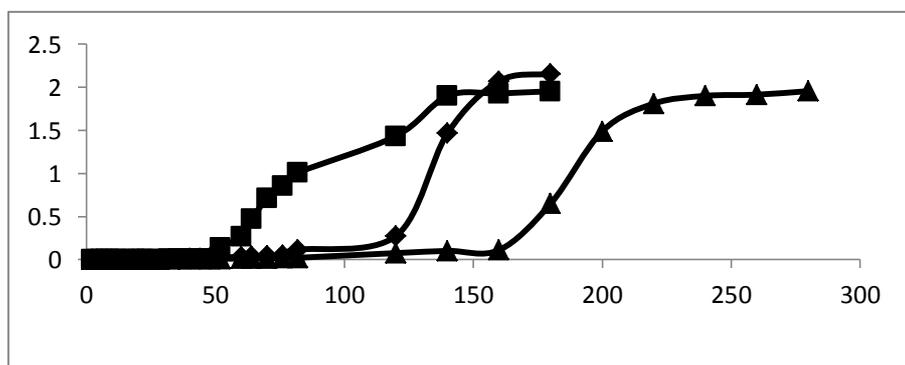
Figure 6. Shows the influence of flow rate on Biosorption of AR88 by *Ulva reticulata* by keeping initial bye concentration (100 mg/l and bed height 20 cm) constant and varying the flow rate 5 to 15 ml/min. In contrast to bed height results, the column performed well at low flow rate. Earlier breakthrough time appeared for highest flow rate, resulting in low uptake and least % removal (Table.4).This behavior may be due to insufficient time for the solute inside the column and the diffusion limitations of the solute into the pores of the sorbent at higher flow rates.

Figure-6. Breakthrough curves for AR88 onto *Ulva reticulata* biomass at different flow rates (bed height=20 cm, initial concentration=100 mg/L, pH=5). (Time (h) vs C/C₀)



The breakthrough curves obtained by changing AR88 concentration from 50 to 100 mg/l and 5 ml/min flow rate and 25 cm bed height are shown in Fig.7. At the highest AR88 concentration (100 mg/L) the *Ulva reticulata* bed saturated quickly leading to earlier breakthrough and exhaustion time. Table 4, shows the highest uptake and high percentage dye removal are obtained at the highest dye concentration. Also steep breakthrough curve was obtained for 100 mg dye/L. The driving force for biosorption is the concentration difference between the dye on the biosorbent and the dye in the solution.

Figure-7. Breakthrough curves for AR88 Biosorption onto *Ulva reticulata* biomass at different dye concentrations. (Time (h) vs C/C₀).



Thus the high driving force due to the high AR88 concentration resulted in better column performance. Comparison of experimentally determined and Thomas model predicted breakthrough curves are shown in Fig 5,6,7 and Table.4,5 summarizes the Thomas model parameters obtained at different bed heights, flow rates and initial AR88 concentrations. As bed height increased the values of Q₀ increased and the values of k_{th} decreased.

Table-4. Column data parameters

Bed height (cm)	Flow rate (mL/min)	C ₀ (mg/L)	t _b (h)	t _c (h)	Uptake (mg/g)	dc/dt (mg/L h)	V _{eff} (l)	Dyes Removal (%)
10	5	100	1.3	18.4	46.3	5.4	6.87	43.27
15	5	100	2.5	22.4	47.8	5.2	7.32	45.64
20	5	100	4.7	26.5	47.5	4.3	8.97	55.82
20	10	100	3.7	22.4	43.4	4.5	6.5	65.34
20	15	100	2.9	17.8	41.9	7.6	5.4	88.65
20	5	75	2.7	46.2	47.9	2.3	11.45	74.89
20	5	50	4.7	75.4	39.8	1.9	16.5	53.23

The bed capacity Q_0 decreased and Thomas constant k_{th} increased with increasing flow rate. In general, good fits were obtained in all cases correlation coefficients ranging from 0.923 to 0.949 for AR88.

Table-5. Thomas model parameters

Bed height (cm)	Flow rate (mL/min)	C ₀ (mg/L)	Q _{sp} (mg/g)	Q ₀ (mg/g)	K _{th} (L/mgh)	R ²
15	5	100	46.3	45.3	0.003	0.993
20	5	100	47.8	44.	0.005	0.995
25	5	100	47.5	38.9	0.002	0.992
25	10	100	43.4	38.76	0.006	0.996
25	15	100	41.9	38.9	0.007	0.997
25	5	50	47.9	42.3	0.005	0.999
25	5	75	39.8	36.6	0.005	0.990

4. CONCLUSIONS

Biosorption capacity (mg/g) increased with increase of initial dye concentration and time but decreased with increase in biosorbent dosage. It was also found that increasing temperature and decreasing pH resulted in higher dye loadings per unit weight of the sorbent (mg/g). The adsorption process follows fully the pseudo-second-order adsorption rate expression. The dye adsorption process is a feasible, nonspontaneous chemisorption process and is endothermic in nature. Column experiments were performed in a packed column, as it makes the best use of the concentration difference known to be a driving force for adsorption. This study proved that Biomass of *Ulva reticulata* can be an attractive candidate for removal of acid dyes from wastewater.

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BIOGRAPHICAL NOTES

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