Kinetic Studies on Biosorption of Acid Blue 15 Using Nuisance Weed *Salvinia minima*

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Aquatic plant *Salvinia minima* biomass used as a sorbent for the removal of acid blue 15 from an aqueous solution was investigated. Experiments were performed by varying biosorbent dosage from 0.2 to 1.0 g/L, pH ranged from 2 to 7 and initial dye concentration from 30 to 110 mg/L at batch mode. The kinetic data were analyzed with Pseudo first-order and Pseudo second-order kinetic modeling for various initial dye concentration at different time. Langmuir and Freundlich adsorption isotherm models were used for analysis equilibrium data. Biosorption mechanism was analyzed by intraparticle diffusion model. The surface morphology of the *S. minima* biomass was analyzed by scanning electron microscope. Functional group present onto the *S. minima* biomass was analyzed by the Fourier transform infrared spectra.

KEYWORD

Biosorption, Acid blue 15, *Salvinia minima*, SEM, FTIR.

INTRODUCTION

Industries, like textile, pulp, paper, cosmetic, food, pharmaceutical and plastics need dyes for their production (Chen *et al.,* 2003). The dye effluents released from these forms a major problem because they are biologically non-degradable. The extensive use of dyes often poses pollution problems in the form of coloured wastewater. This coloured wastewater is characterized by persistent colour, high COD and biotoxicity (Fu and Viraraghavan, 2001). Among several chemical and physical methods, adsorption process is one of the most effective method, which can be successfully employed for coloured effluents. Adsorption on activated carbon is an effective process, but it is too expensive. So it is replaced by eco-friendly sorbents, such as microorganisms, like fungal, bacterial and algal biomass as a potential biosorbent (Aksu and Tezer, 2005). The disposal of the microbial adsorbent can be solved by recycling after regenerating using sodium hydroxide and organic solvents (Inbaraj *et al.,* 2002). Since, cultivation, harvesting and maintenance of pure culture are the problem in microorganism used as a sorbent.

In recent years, low cost adsorbents (Gupta *et al.,* 2003), such as quaternised rice husk (Low and Lee, 1997), barley husk (Robinson *et al.,* 2002), orange peel (Sivaraj *et al.,* 2001). But these low cost adsorbents have low adsorption capacities (Gupta *et al.,* 2003; McKay et al., 1999; Low *et al.,* 1995). Hence economical, easily available and effective adsorbents are still needed. Among the various sorbents used for biosorption, *S. minima* is an inexpensive, readily available biomass. It is a free floating aquatic fern native to Central and South America (Olguin *et al.,* 2002). It is a significant nuisance weed in southern aquatic and systems (Jacono and Richerson, 2008). It invades a variety of aquatic habitats with salinity levels as high as 4-7 ppt. *S. minima* experiences exponential growth that allows it to completely impede traffic, block sunlight, decrease oxygen levels and degrade the habitat for native species of wildlife.
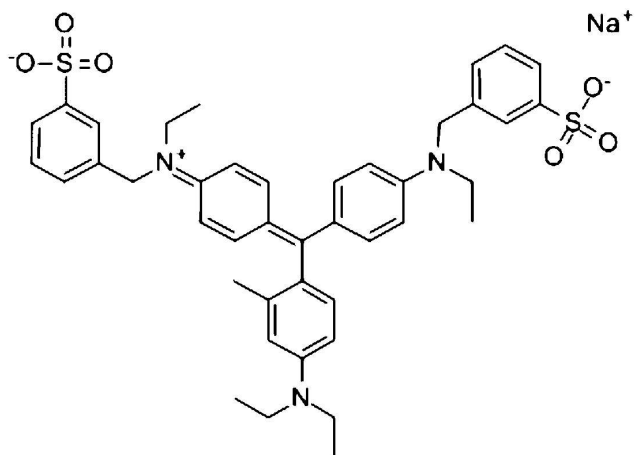
Figure 1. *Chemical structure of dye acid blue 15*

Generally *S.minima* is capable of high growth rates and is tolerant to high strength organic wastewater (Olguin *et al.*, 2002). It biomass growth reached 1.02 kg/m and causes reductions in native plant abundance (Walley, 2007). However, no research attention has been focused on utilization of *S. minima* for dye removal, but it has been shown to effectively bind lead (Estrella – Gomez *et al.*, 2009). The major objective of this study was to investigate the potential of abundantly available weed *S. minima* biomass as biosorbent for the removal of acid blue 15 from textile effluents. It was used to remove acid blue 15 dye from an aqueous solution under different pH, sorbent dosage and initial dye concentration. The sorption mechanism was investigated using different kinetic models.

EXPERIMENTAL STUDIES

Biomass preparation

The aquatic plant used in this study was collected from Osudu Lake, Pondichery. The collected plant species was identified as *S. minima* by the Centre for Advanced Studies in Botany, University of Madras, Chennai. The *S. minima* plant was washed with deionized water to remove dirt particles and dried in hot air oven at 50 °C. The dried sample was crushed and passed through 70 mesh and retained in 80 mesh size. The sieved biomass was used for all the experiments.

Dye

Textile dye acid blue 15 (C.I. No. 42645, molecular formula $C_{42}H_{46}N_3NaO_6S_2$ and molecular weight 775.95) was obtained from Balaji Chemicals Ltd., Chennai. It is widely used in dyeing the textile fabrics. The acid blue 15 chemical structure is given in figure 1. The stock solution was prepared by dissolving 1 g acid blue 15 dye powder in 100 mL of distilled water to obtain a concentration of 10,000 ppm. The further dilutions were made from the stock solution.

Study of biosorbent dosage

In batch experiments, the sorbent dosage was varied from 0.2 to 1.0 g using a fixed volume (100 mL) of 50 mg/L initial dye concentration. Different dosages of dried *S.minima* were added in 100 mL dye solution and kept in a rotary shaker at 180 rpm at room temperature. Samples were withdrawn at different time intervals and centrifuged at 10,000 rpm for 10 min and the absorbance of the supernatant was determined at maximum wavelength of λ_{max} at 564 nm using UV spectrophotometer (Hitachi U3210,Japan). Results were reported on the basis of the dye uptake capacity.

Study of initial pH

The dye solution with the concentration of 50 mg/L was equally dispersed into 6 conical flasks (100 mL in each flask). The pH of the dye solution was varied from 2 to 7 by using dilute HCl and dilute NaOH solution. The optimum dosage of sorbent of 0.2 g/L were added into the each flask. These flasks were kept in a rotary shaker and absorbance values were determined.

Study of initial dye concentration

The dye solution with different concentrations ranging from 30 to 110 mg/L was dispersed into 5 conical flasks. The optimum dosage of sorbent was added into each flask and adjusted to optimum pH and kept in a rotary shaker at room temperature. The absorbance values were determined using UV spectrophotometer.
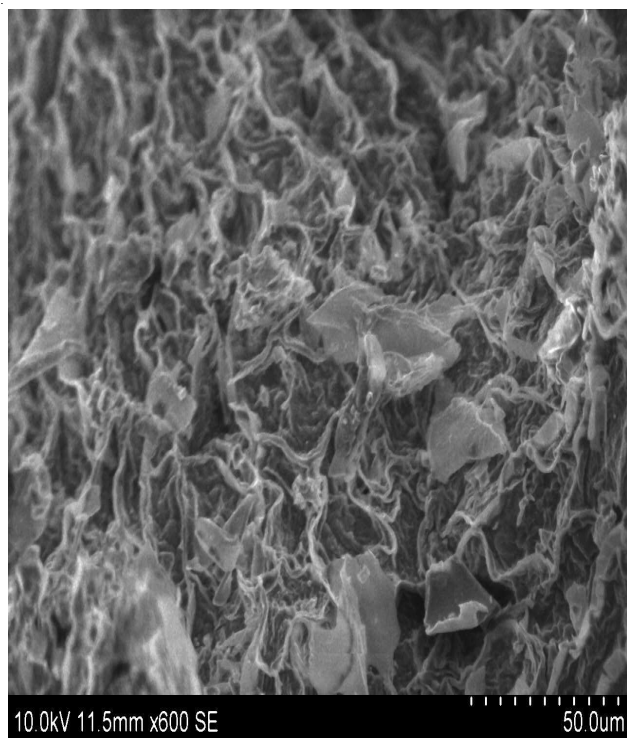
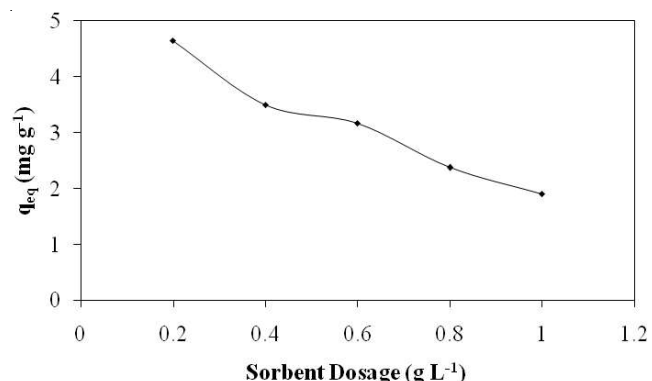**Figure 2.** *SEM image for* S. minima *plant biomass (without dye)*

Figure 3. *Effect of biosorbent dosage on biosorption of acid blue 15 dye using* S. minima *(initial dye concentration: 50 mg/L, agitation: 150 rpm)*

RESULT AND DISCUSSION

SEM imaging

Scanning electron microscopy (SEM) (JEOL JSM- 6360) is a technology, which has been increasingly used to examine the surface morphology of the biomass. The surface morphology of the *S. minima* biosorbent was examined by the scanning electron micrograph. Figure 2 shows the SEM image of the *S. minima* biomass. The rough and porous surface was found to be more in *S. minima* (Figure 2). This surface property should be considered as an important factor for providing more surface area on the biosorbent (Renganathan and Gautam, 2008). The increase in the porous surface area leads to increase in the uptake capacity of the dye (Saravanan *et al.,* 2012).

Effect of biosorbent dosage

The effect of biosorbent dosage was monitored to attain the maximum dye uptake capacity. The biosorbent dosage was varied from 0.2 to 1.0 g at a fixed initial dye concentration of 50 mg/L in 100 mL. Equilibrium uptake capacity was found to be decreased with increase in biosorbent dosage. The maximum dye uptake capacity (q_{eq}) using 0.2 g was found to be more when compared to all other dosages studied (Figure 3). So 0.2 g of biosorbent dosage was taken as optimum value for the corresponding experiments. The decrease in the equilibrium uptake was due to the amount of dye adsorbed onto unit weight of biosorbent dosage (Saranya *et al.,* 2011). A similar trend were previously reported for the removal of reactive red 120 using *Hydrilla verticilata* (Naveen *et al.,* 2011) and uptake of reactive red 198 dye using *Acalypha indica* (Praveena *et al.,* 2012).

Effect of pH

The effect of initial dye solution pH on the uptake of acid blue 15 dye was examined over a range of pH values from 2 to 7 and the results are presented in figure 4. As elucidated in figure 4, the maximum uptake capacity of the dye was found to be at a solution pH value of 2. The dye uptake capacity was found to be decreased with increase in the pH ranging from pH 2 to 7. Based on these results obtained, an optimum pH value 2 was selected for future experiments for the removal of acid blue 15 dye. The maximum dye uptake capacity was found to be 51.45 mg/g at the pH value of
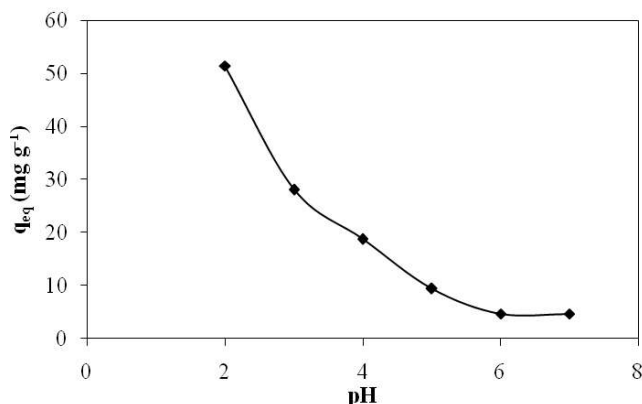
Figure 4. *Effect of pH on the biosorption of acid blue 15 using* S. minima *(initial dye concentration: 50 mg/L; sorbent dosage: 0.2 g in 100 mL; agitation: 150 rpm)*
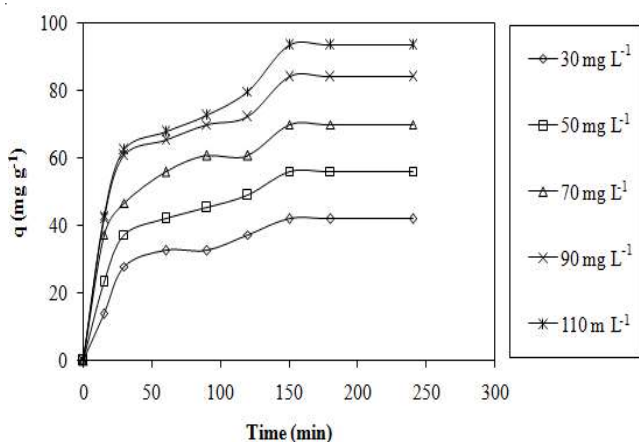

Figure 5. *Effect of initial dye concentration on biosorption of acid blue 15 using* S. minima *(sorbent dosage: 0.2 g in 100 mL; pH: 2; agitation: 150 rpm)*

2. This may be due to the protonation of biosorbent material occurring at the lower pH (Saravana *et al.,* 2012). The protonated biosorbent have more affinity towards the anionic dyes (Renganathan and Gautam, 2008).

Effect of initial dye concentration

The effect of initial dye concentration on biosorption of acid blue 15 dye is represented in figure 5. Five conical flask containing 100 mL of dye solution with varying dye concentration from 30 to 110 mg/L at fixed biosorbent dosage of 0.2 g and pH value of 2. From figure 5, it was observed that the dye uptake capacity was found to be increased linearly with contact time in the beginning, then non-linearly at slower rate and finally attained saturation called equilibrium time (Reema *et al.,* 2011). Similar trend was observed on the colour removal from synthetic dye wastewater using neem sawdust (Khattri and Singh, 1999).

The equilibrium uptake capacity was found to be more (93.52 mg/g) at 110 mg/L of initial dye concentration when compared to all other initial dye concentration studied. Increasing initial dye concentration increases the number of collisions between dye anions and biosorbent, which enhances the sorption process. Dye uptake capacity of the biosorbent was found to be increased with increases in initial dye concentration. A similar type of trend was previously reported for the removal of reactive red 2 using Nymphaea rubra (Renganathan *et al.,* 2009). This trend may be due the concentration gradient to an increase in the driving force between the dye molecules and the biomass, which leads to higher amounts of dye adsorbed as the initial dye concentration increased (Naveen Prasad *et al.,* 2008). However, in this present investigation the maximum dye uptake capacity of acid blue 15 was observed as 93.52 mg/g at initial dye concentration of 110 mg/L using the *S. minima* plant biomass. So, the biosorption capacity of the *S. minima* plant biomass was found to be high when compared to other fungal biomass, such as *Aspergillus niger* for the removal of methyl violet (25 mg/g) and basic fuchsin (25 mg/g) dye (Bhole *et al.,* 2004) and Aspergillus oryzae for the removal of procion red HE7B (12.19 mg/g) and procion violet H3R (9.92 mg/g) dye (Corso and Almeida, 2009).

Equilibrium modeling

The Langmuir and Freundlich equations are in common use to describe adsorption isotherms at constant temperature for wastewater applications. Analysis of equilibrium

Table 1. *Langmuir and Freundlich adsorption isotherm constants for the biosorption of acid blue 15 dye using* S. minima *biomass*

Langmuir isotherm			Freundlich isotherm		
$Q°$, mg/g	b, L/mg	R^2	K_F	n	R^2
333.33	0.001	0.867	3.61	1.31	0.992

data is essential to develop an equation which precisely represents the results and can be used for design purposes. Various isotherm models have been used for the equilibrium modeling of biosorption systems. The most widely used isotherm models to describe the biosorption process are Langmuir and Freundlich adsorption isotherm models (Suteu and Bilba, 2005). The Langmuir isotherm assumes monolayer adsorption and is presented by the following equation:

$$q_{eq} = \frac{Q^0 bC_{eq}}{1 + bC_{eq}} \qquad ...(1)$$

where, q_{eq} (mg/g) is dye adsorbed per unit weight of biomass, C_{eq} (mg/L) are amount of unadsorbed dye concentration in solution at equilibrium, $Q°$ is the maximum amount of dye adsorbed per unit weight of biomass required to form a complete monolayer on the surface and b is a constant related to the affinity of the binding sites (Low *et al.,* 1995) (L/mg).

The Freundlich isotherm equation is an empirical equation based on the sorption on a heterogeneous surface. It is suggesting that binding sites are not equivalent and independent. The Freundlich adsorption isotherm equation is given below:

$$q_{eq} = K_F \, C_{eq}^{\,1/n} \qquad ...(2)$$

where, K_F is biosorption capacity and n is biosorption intensity. These constants can be evaluated by plotting q_{eq} and C_{eq} in logarithmic form. The calculated isotherm constants are shown in the table 1. The best fit of equilibrium modeling was determined based on the coefficient of determination R^2. The coefficient of determination was

found to be very closer to one for Freundlich model when compared to Langmuir model studied. Freundlich model exhibited a better fit when compared to the Langmuir model studied. The Freundlich constant, n also indicates the degree of favourability of adsorption. The Freundlich constant, n should have values lying in the range of 1 to 10 for favourable adsorption (Rao and Bhole, 2001).

Kinetic modeling

The biosorption mechanism and potential rate controlling steps have been investigated by using the Pseudo-first and Pseudo-second order kinetic models. The Pseudo-first order rate expression of Lagergren (Lagergren, 1898) is :

$$\frac{dq}{dt} = k_{1,ad}(q_{eq} - q) \qquad ...(3)$$

where q (mg/g) is the amount of dye adsorbed on the biosorbent at time t (min-1) and $k_{1,ad}$ (min-1) is the rate constant for first order biosorption. The integral form of equation 3 is :

$$\log (q_{eq}-q) = \log q_{eq} - \frac{k_{1,ad}}{2.303} \qquad ...(4)$$

A linear fit of log (q_{eq}-q) vs t shows the applicability of this kinetic model. Expression for the Pseudo-second order (Hamissa and Ncibi, 2008) kinetic model is :

$$\frac{dq}{dt} = k_{2,ad}(q_{eq} - q)^2 \qquad ...(5)$$

where $k_{2,ad}$ (g min/mg) is the rate constant of the Pseudo-second order biosorption. The integrated linear form of equation 5 is :

Table 2. *Pseudo-first and second order rate constants, calculated and experimental q_{eq} values for* S. minima *biomass*

Dye concentration, mg/L	Pseudo-first order			Pseudo-second order			$q_{eq,exp}$, mg/g
	$k_{1,ad}$, min^{-1}	$q_{eq,cal}$, mg/g	R^2	$k_{2,ad}$, mg/g/min	$q_{eq,cal}$, mg/g	R^2	
30	0.01658	32.67	0.9007	0.00106	45.66	0.9822	42.09
50	0.01566	41.53	0.9188	0.00093	59.88	0.987	56.15
70	0.01589	46.73	0.8730	0.00091	76.92	0.991	70.15
90	0.01473	54.38	0.8395	0.00079	88.49	0.9895	84.15
110	0.01381	66.06	0.8850	0.00054	100	0.983	93.52

$$\frac{t}{q} = \frac{1}{k_{2,ad}q^2_{eq}} + \frac{1}{q_{eq}}t \qquad \dots(6)$$

If the experimental data fits a linear relationship with the plot of t/q vs t, the Pseudo-second order kinetic model is valid. The Pseudo-first order and Pseudo-second order kinetic models are applied to the experimental data. The plot of log (q_{eq}-q) vs t obtained the Pseudo-first order rate constant ($k_{1,ad}$) and q_{eq} values were determined from slope and intercept, respectively. The correlation coefficients of the Pseudo-first order kinetic model obtained are low (Table 2).

The theoretical q_{eq} values found from the Pseudo-first order kinetic model did not give acceptable values. It is probable, therefore, that this biosorption system is not a Pseudo-first order reaction. The Pseudo-second order rate constant ($k_{2,ad}$) and q_{eq} values were also determined from the slope and intercept of the plots t/q vs t. The values of the parameters ($k_{2,ad}$), q_{eq} calculated and q_{eq} experimental values together with coefficient of determinations are presented in table 2. As shown in the table 2, q_{eq} calculated values were found to be closer to q_{eq} experimental values in the case of second order kinetics when compared to the first order kinetics (Ho and McKay, 1999). The coefficient of determination was found to be very closer to one for Pseudo-second order rate equation when compared to Pseudo-first order rate equation. From the analysis it was observed that the kinetic data was found to be fitted very well with the Pseudo-second order rate equation.

Biosorption mechanisms

The biosorption mechanism is analyzed by fitting the experimental data in an intraparticle diffusion plot. As per the Weber and Morris (Weber and Morris, 1963), an intraparticle diffusion coefficient K_i is expressed by equation 7

$$K_i = q/t^{0.5} \qquad \dots(7)$$

Thus the K_i (mg/g min$^{-0.5}$) value can be obtained from the slope of the plot of q (mg/g) vs $t^{0.5}$ (min$^{0.5}$). The Ki values obtained for acid blue15 dye using the *S. minima* biomass was found to be more in 110 mg/L initial dye concentration when compared to all other initial dye concentration studied in the present investigation. The presence of both film and pore diffusion is confirmed by intraparticle diffusion plot. The kinetic data were further analyzed using the Boyd kinetic expression (Boyd *et al.,* 1947). The Boyd kinetic expression is presented by equations 8 and 9

$$F = 1-(6/\pi2) \exp (-Bt) \qquad \dots(8)$$

and

$$F = q/q_o \qquad \dots(9)$$

where q_o is the amount of dye biosorbed at infinite time (mg/g) and q represents the amount of dye biosorbed at any time t (min), F represents the fraction of dye biosorbed at any time t and Bt is a mathematical function of F. Substituting equation 9 in equation 8, equation 8 simplifies to :

Table 3. *Intraparticle diffusion parameter (k_i) and diffusion coefficient (d_i) for the biosorption of acid blue 15 dye using* S. minima *biomass*

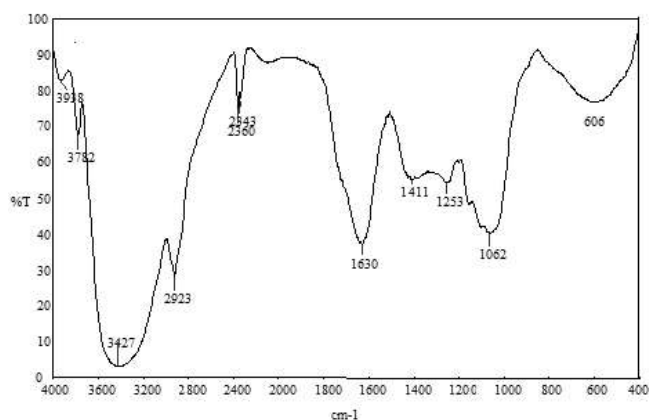| Initial dye concentration, mg/L	Intraparticle diffusion parameter, K_i mg/g min$^{-0.5}$	Diffusion coefficient, D_i, cm²/s
30	2.75	5.273E-07
50	3.56	4.940E-07
70	4.24	4.940E-07
90	5.07	4.610E-07
110	5.81	4.280E-07

Figure 6. *FTIR spectra for the biomass of* S. minima *(without dye)*

$$Bt = -0.4977 - \ln(1-F) \qquad ...(10)$$

The Bt values can be calculated at different contact times using equation 10. The calculated Bt values were plotted against time t. This plot is used to identify whether external transport or intraparticle transport control the rate of sorption (Sarkar *et al.,* 2003). It was found that the plots were linear but do not pass through the origin. From the result, it is confirming that external mass transport mainly governs the biosorption process for the studied initial dye concentration. The calculated B values were used to evaluate the effective diffusion coefficient, D_i (cm²/s) using the relation given in equation 11

$$B = \prod{}^2 D_i/r^2 \qquad ...(11)$$

where r represents the radius of the particle. The D_i values obtained using the *S. minima* biomass for different initial dye concentration is presented in table 3. The D_i values obtained at 110 mg/L initial dye concentration was found to be more when compared to all other initial dye concentrations studied in the present investigation.

Weber and Morris model was used to analyse the biosorption of acid blue 15 dye on the *S. minima* biomass as a function of initial dye concentration (Ho and McKay, 1999). It was observed that intraparticle rate constant values (K_i) were found to be increased with increase in initial dye concentration. This can be explained by the growing effect of driving force and the concentration gradient. From the plot of q (mg/g) vs $t^{0.5}$ (min$^{0.5}$) for different initial acid blue 15 dye concentration was obtained as an initial curve followed by a straight line, this indicates that more than one mode of biosorption was operating in the uptake of dye by *S. minima* biomass. The initial curve is due to boundary layer sorption while the linear portion can be attributed to intraparticle diffusion (Karthik *et al.,* 2009).

Fourier transform infrared analysis (FTIR): The FTIR study was conducted to confirm the existence of amine, carboxyl and phosphonate groups in the biomass. As presented in the figure 6, the FTIR spectroscopic (Tensor 27, Bruker optik GmbH, Germany) analysis of *S. minima* water plant biomass, 11 peaks were found, such as 3938, 3782, 3427, 2923, 2360, 2343, 1631, 1411, 1254, 1062 and 607/cm. The spectrum showed the peaks at 1631/cm indicating the carboxyl groups. Some absorption bands (P = O) stretching at 1411/cm and P-OH stretching at 1062/cm were considered to be indicative of phosphonate group (Pagenelli *et al.,* 2000). OH stretch will appear as a broad band at 3427/cm. The FTIR spectrum showed some characteristics absorption of an amine group: N-H bonding at 2343 and 2360/cm; N-H rocking near 607/cm and C-N stretching band at 1253/cm (Renganathan *et al.,* 2008).

The primary amine groups were likely to have been responsible for the biosorption of acid blue 15. As the dye solution pH decreases, the number of binding sites (positively charged amine) increases and thereby the uptake increases (Saravanan *et al.,* 2012). Therefore, it should be noted that the FTIR spectrum of the *S. minima* water plant biomass supports the presence of amine group for the biosorption of acid blue 15.

CONCLUSION

In the present study, *S. minima* water plant biomass was applied successfully for the biosorption of acid blue 15 dye from an aqueous solution. The amount of dye adsorbed was found to be varied with biosorbent dosage, initial pH and initial dye concentration. The equilibrium dye uptake capacity was found to be increased with decrease in biosorbent dosage and increase in dye solution concentration. The maximum dye uptake capacity was obtained at pH value of 2. The equilibrium data was found to be fitted very well with Freundlich adsorption isotherm model when compared to Langmuir adsorption isotherm model. The kinetics data was found to be obeyed very well with the Pseudo-second order kinetic model when compared to the first-order kinetic model. SEM and FTIR spectral analysis confirms the porous, uneven surfaces and functional group (Amine) present on the *S. minima* biomass. Biosorption mechanism clearly stated that two steps, such as boundary layer and intraparticle diffusion. The *S. minima* biomass is a low cost natural adsorbent material and may be an alternative to costly adsorbent materials.

NOMENCLATURE

b - Constant related to the affinity of the binding sites (L/mg)

C_{eq} - Unbiosorbed dye concentration in solution at equilibrium (mg/L)

$k_{1,ad}$ - Rate constant for Pseudo-first order biosorption (min^{-1})

$k_{2,ad}$ - Rate constant of the Pseudo-second order biosorption (g min/mg)

Q° - Amount of dye biosorbed per unit weight of biomass (mg/g)

q - Amount of dye biosorbed on the biosorbent at time t (mg/g)

q_o - Amount of dye biosorbed at infinite time (mg/g)

q_{eq} - Equilibrium dye uptake capacity (mg/g)

$q_{eq,cal}$ - Calculated equilibrium dye uptake capacity (mg/g)

$q_{eq,exp}$ - Experimental equilibrium dye uptake capacity (mg/g)

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