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Biosorption of Methylene blue from aqueous solutions by using blue green algae *Oscillatoria sp.*: Kinetic and equilibrium studies

Surabhi Sagar[1] and Arshi Rastogi[1]*

1. Department of Chemistry, KLDAV (P.G.) College, Roorkee, Uttarakhand, 247 667, **INDIA**

Email: arshirastogi@gmail.com

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ABSTRACT

This study is aimed at investigating the sorption of Methylene blue (MB) dye from aqueous solutions using a blue green alga Oscillatoria sp. as an adsorbent. The impact of several influential parameters such as pH, contact time, temperature, initial dye concentration, biosorbent particle size and its dose on the adsorption capacity of Oscillatoria sp. was evaluated. The maximum adsorption capacity of this biomass was found to be 129.58 mg/g at an initial dye concentration of 100 mg L^{-1}, pH 7, biosorbent size 100 µm and dosage 5 g L^{-1} at a contact time 60min respectively. Results were analyzed by Langmuir and Freundlich models of adsorption. Dye adsorption equilibrium data fitted well in Langmuir isotherm than the Freundlich isotherm which showed monolayer adsorption. The rate of adsorption followed the second order kinetic model and the thermodynamic studies showed that the adsorption of methylene blue dye was favorable, spontaneous and endothermic in nature. The FT-IR analysis showed the involvement of different functional groups. This study indicates that this algal biomass could be used as an effective and efficient biosorbent for the removal of Methylene blue dye.

Keywords: Biosorption, Methylene blue, *Oscillatoria sp*., Kinetics.

INTRODUCTION

Water pollution by dyes is a widespread problem particularly where textile industries discharge large quantity of dye effluents from dyeing process. Discharge of these colored effluents without discoloration may cause serious problems in the receiving environments. Water contamination by visible colored pollutants has become a major concern for the environment due to extensive usage of dye and dyestuff [1-2]. Dyes are widely used in many industries other than textile industries, such as rubber, paper, plastic, cosmetic etc. There are more than 10,000 commercially available dyes with over 7×10^5 tons of dyestuff being produced annually across the world [3]. Producers and consumers of dyes are only aiming at the stability and fastness of dyes and hence are leading to the production of dyestuffs which are more difficult to degrade after use. 10-15% of the dye is lost during the dyeing process and is released as effluent [4]. Advanced water treatment methods are suggested for the re-use of industrial wastewater. Wastewater containing dyes represent water pollution that is not only harmful for mankind but also for aquatic life. So, it is vital to treat these polluted waste waters efficiently as well as effectively.

Since dyes are highly soluble in water their removal from water by membrane filtration, activated sludge process and convectional coagulation is difficult. These processes require high economical cost, considerably high energy and high operational costs. Hence an alternative method is sought. No single treatment is adequate for removing impurity from wastewater [5]. Among many different technologies for treatment of water, the sorption process is one of the effective, inexpensive and most importantly eco-friendly technique for the removal of color from wastewaters [6-8]. Biomaterials that are available in good quantities have a great potential to be used as low cost adsorbents, as they represent unused resources which are widely available and also ecofriendly nature [9-10]. Nowadays, there are number of low cost, commercially available adsorbents which had been used for dye removal [11-13].

Methylene blue (MB), the dye used in the present study, is most commonly used for dyeing cotton, wood and silk. It causes eye burns which may be responsible for permanent injury to the eyes of human and animals. On inhaling, it gives rise to rapid or difficult breathing while ingestion through the mouth produces a burning sensation and may cause nausea, vomiting, profuse sweating and mental confusion [14]. Therefore, the treatment of effluent containing such dye is of interest due to its harmful impacts on receiving waters. In recent times, there has been increased interest in the use of algae for dye removal by adsorption from aqueous solutions because of their natural availability and higher removal efficiency. There have been reports on MB adsorption by various marine as well as fresh water algae [15-19] but there are no reports on blue green algae *Oscillatoria sp.* Although, Balakrishnan et al have reported the ability to use the diazo dye, C.I. Acid Black as nitrogen source by the marine cyanobacterium *Oscillatoria curviceps* [20]. Earlier, our lab has worked on blue green algae *Nostoc sp.* for the adsorption studies of metal ions like lead (II) and chromium (VI) [21-22].

Therefore, for this study, *Oscillatoria sp.* which is unbranched filamentous alga, occurring singly or in tangled mats, found in watering-troughs or water streams, roadside ditches, drains and sewers, was selected for biosorption studies. Thus, the aim of this study was (i) To find the potential use of this alga for sorption of MB from aqueous solution; (ii) To evaluate the effect of adsorbent dose, pH value, temperature, initial dye concentration and contact time on the adsorption in batch system; (iii) To investigate sorption kinetic models, equilibrium studies and thermodynamic parameters.

MATERIALS AND METHODS

Chemicals: Analytical grade reagent like Methylene blue was used to prepare standard solutions for the adsorption studies. HCl and NaOH solutions (AR grade) were used to adjust pH and the buffer solutions used to calibrate pH meter were of E. Merck., Germany.

Equipments: The pH measurements were made using a pH meter (PERFIT) and the aqueous solution was analyzed using a UV-Vis spectrophotometer -119 (Systronics India Ltd.). Infrared spectra were recorded using KBr pellets on a Thermo Nicolet FTIR (Germany) within 4000-400 cm^{-1}. To examine the morphological characteristics of the alga before and after adsorption of MB dye, samples were viewed using a LEO 435 VF (Leo Electron Microscopy Ltd, England) at acceleration voltage of 20 KV.

Biosorbent preparation: The material upon whose surface the adsorption takes place is called biosorbent. For this study *Oscillatoria* sp. *was* used as a low cost biosorbent. The collected biosorbent was washed with distilled water several times to remove all the undesired materials. The biosorbent was then kept on a filter paper to reduce water content. It was Sun dried for two days followed by oven drying at 70^0C for 24 h. Subsequently, it was ground on an agate stone pestle mortar and sieved, to select the particles of 100, 200 and 300 *µm* mesh sizes.

Adsorbate: The basic dye, Methylene blue (C.I. 52015, S.D. Fine Chemicals, 85% dye content, chemical formula $C_{16}H_{18}N_3SCl$, FW 319.86, nature basic blue, and λ_{max} 665 nm) has been used in this study. The

chemical structure of MB is shown in figure 1. This dye was chosen for study because of its known strong adsorption onto solids.

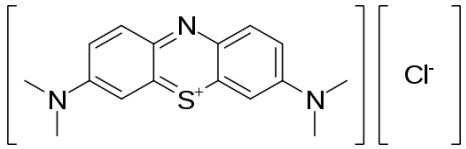

Fig 1. Chemical structure of Methylene blue dye (MB)

Batch adsorption studies: Biosorption experiments were performed in batch mode to find the effect of various parameters such as pH, biosorbent dosage, initial dye concentration, contact time and temperature. The stock solution of MB (1g L^{-1}) was prepared in distilled water. It was then diluted to prepare working solutions of chosen concentrations. To investigate the effect of pH on sorption capacity, the initial pH of solutions was adjusted to desired value in range of 3-10 by addition of 0.1M HCl or 0.1M NaOH according to the conditions. To investigate the effect of adsorbent dose on dye removal, different amount of biosorbent in range of 1-10 g L^{-1} was used. The mixture was stirred using magnetic stirrer at room temperature. The adsorbent in the sample were separated by centrifugation and the concentration of dye at any time was determined in the supernatant solutions. All the experiments were carried in duplicate and the result expressed as the mean values.

The amount of dye adsorbed on adsorbent at equilibrium was calculated based on mass balance equation given as:

$$q_e = (C_o - C_e)\, V / M \text{ ----------(1)}$$

Where q_e is the adsorption capacity of algae (mg g^{-1}), C_o and C_e are the initial and the equilibrium concentration of dye (mg L^{-1}), V is the volume of reaction mixture (L) and M is the mass of adsorbent used (g).

RESULTS AND DISCUSSION

Characterization of the biosorbent: The Scanning electron micrographs were used to study the surface texture and morphology of biosorbent. The micrographs of before and after adsorption of MB on *Oscillatoria sp.* are shown in figure 2. The images exhibit rough uneven tangled and cave like surface morphology.

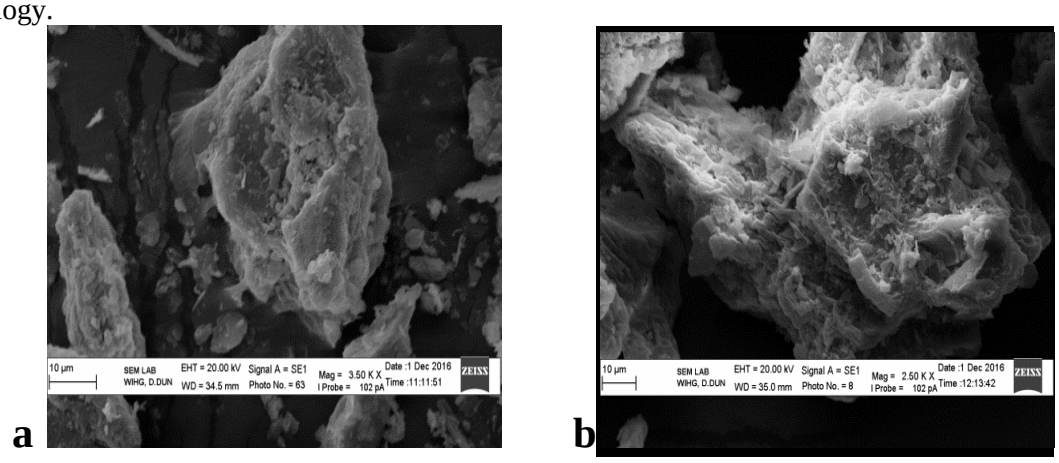

Fig 2. SEM photograph of (a)*Oscillatoria sp* before adsorption (b) after adsorption of MB dye.

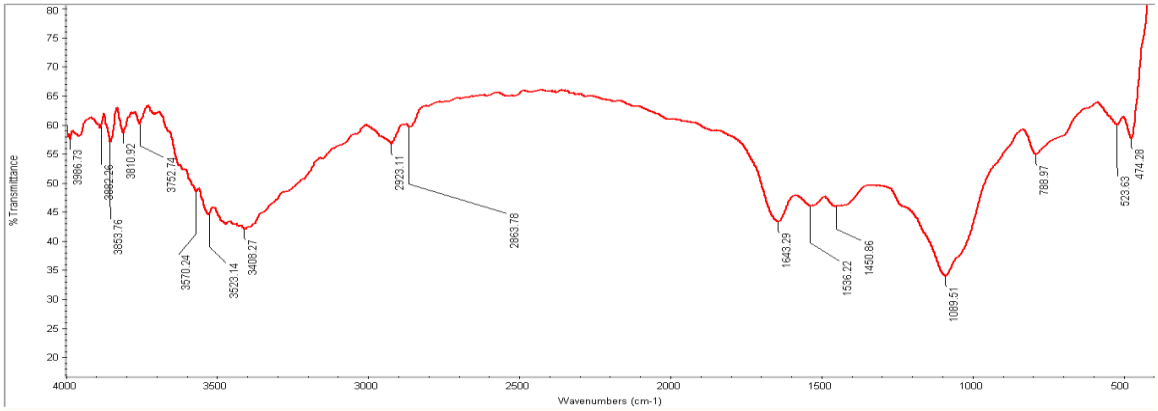

Fig 3. FTIR spectra of adsorbent before biosorption

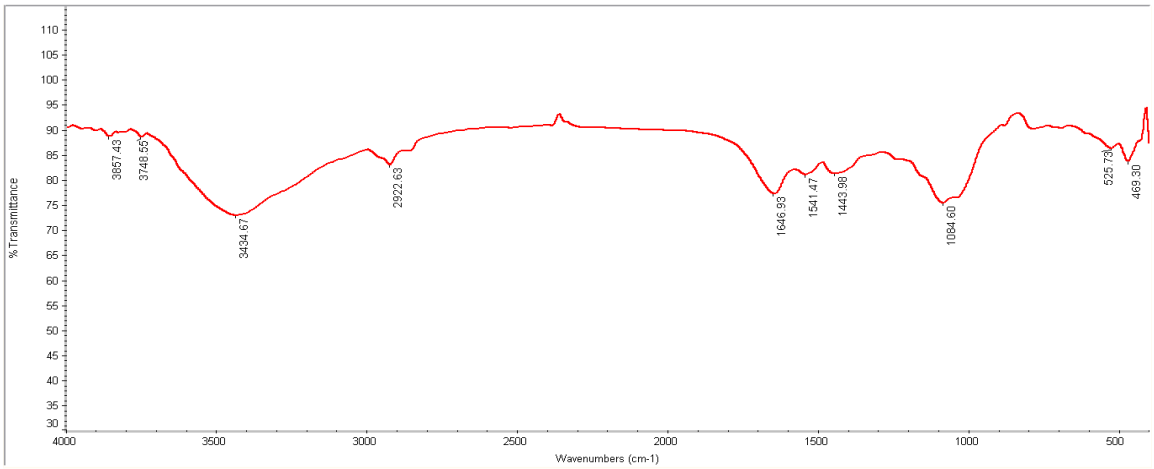

Fig 4. FTIR spectra of adsorbent after biosorption of Methylene blue

FTIR technique was used to study the surface binding mechanism of *Oscillatoria sp.* and to identify the functional groups present on the surface of biomass that are responsible for the sorption of Methylene blue dye. The FTIR spectra for this biomass before and after adsorption of dye are shown in figure 3 and figure 4 respectively. Spectrum of unloaded algae showed major bands at 3408, 2923, 1643, 1536, 1450, 1089, 523 and 474 cm^{-1}. The strong band at 3408 cm^{-1} could be assigned to O-H groups of glucose and –NH groups of proteins, whereas absorption peak at 2923 cm^{-1} is due to carboxylic/phenolic stretching bands [**23**]. Other major bands could be attributed as follows: 1643 cm^{-1} (C=O Chelate stretching of carboxylic group), 1536 cm^{-1} (amide band of protein peptide), 1450 cm^{-1} (asymmetric bending of the CH_3 of the acetyl moiety), 1089 cm^{-1} (-CN stretching), and some bands in the finger print region (523 cm^{-1} and 474 cm^{-1}) can be attributed to the phosphate groups. After the adsorption process, the peaks at 2923 ,1643, 1536 ,1450, 523and 474 cm^{-1} shifted to 2922, 1646, 1541, 1443, 1084, 526 and 469 cm^{-1} respectively that shows, there was a binding process taking place on the surface of the alga and confirms the presence of carboxylic, amino, amide and hydroxyl functional group on the algal cell wall.

Effect of initial pH: pH of dye solution has been found to be one of the important factors that affect the process of adsorption as it influences the degree of ionization of the adsorptive molecule and the surface properties of adsorbent [19]. The effect of pH on MB adsorption capacity of *Oscillatoria sp* was investigated for the values between 3 and 10 as shown in figure 5. The equilibrium adsorption capacity was low at pH 3 and increased up to 7, and remains constant for higher pH ranging from 7-10.

The algal cell wall contains different functional groups such as carboxyl, hydroxyl and other charged groups. When MB molecules are dissolved in water it releases cations which get attracted towards the surface of biosorbent. Thus at lower pH the biosorbent surface gets positively charged and thus decreases the adsorption of dye cations. At higher pH the biosorbent surface gets negatively charged which enhances the electrostatic attraction of dye cations. Similar trend was observed for MB uptake by *Trichoderma viride* [24]

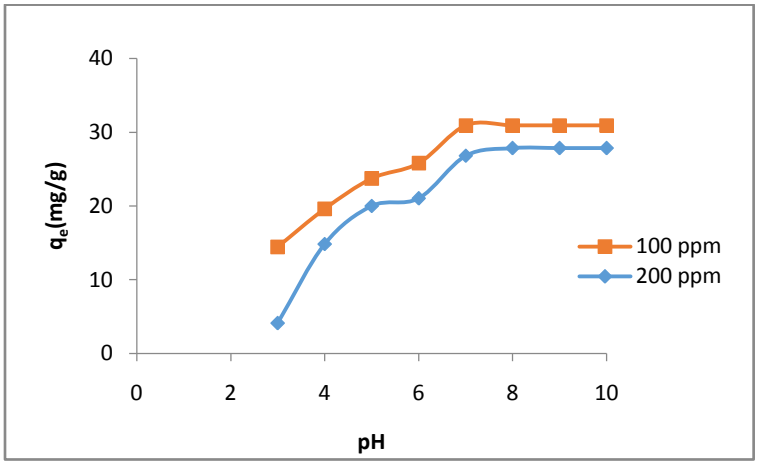

Fig 5. Effect of pH on biosorption of Methylene blue

Effect of contact time: Contact time is one of the most important parameters for the assessment of practical application of adsorption process. 10 ml of the working solution with 100 mg L^{-1} and 200 mg L^{-1} concentration MB were taken with initial pH of 7 and 5mg L^{-1} of biosorbent. A graph was plotted with q_e versus contact time at two different concentrations of dye. It was observed that the adsorption increased with increase in time, reached a constant value at which no further dye was removed from the solution. The maximum adsorption took place within first 60 min as shown in figure 6. The fast sorption at initial stage may be due to the presence of large number of negative charged surface active sites responsible for adsorption of MB cations in algal biomass. Similar trends were observed by adsorption of MB by *Lantana camara* [25]

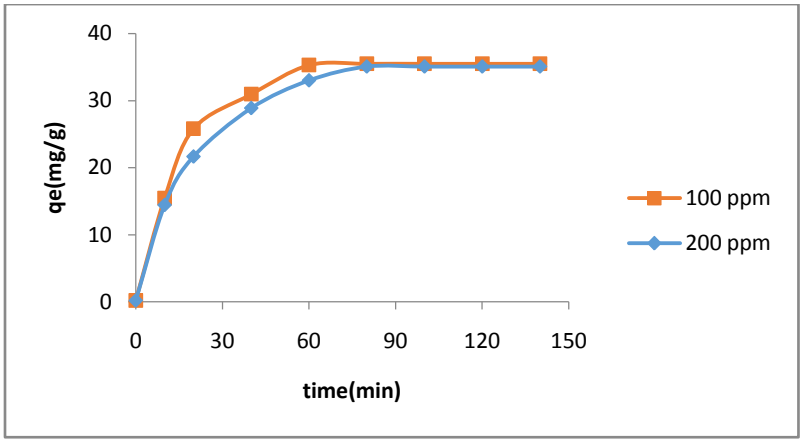

Fig 6. Effect of contact time on the biosorption of Methylene blue at two different concentrations, 100 and 200mg L^{-1}.

Effect of biosorbent dose: The effect of biosorbent dose on the removal of dye was evaluated at different values form 1-10 g L^{-1} suspended in 10 ml of the working solution having 100 and 200 mg L^{-1} dye concentration. Other parameters i.e. pH, temperature and contact time were kept constant at 7, 318 K and

60 min. respectively. The absorbance of all the solutions was then measured. A graph was plotted with q_e versus adsorbent dose (fig 7). As shown in the figure, the adsorption capacity of the adsorbent increased with an increase in biosorbent dose and reached to a maximum value at 5mg L^{-1}. Further increasing the dose did not show any increase in the adsorption value. This is a result of increasing particle interaction and aggregation that leads to reduction of total biosorbent surface area [26]. Hence the optimum adsorption dose was found to be 5mg L^{-1}

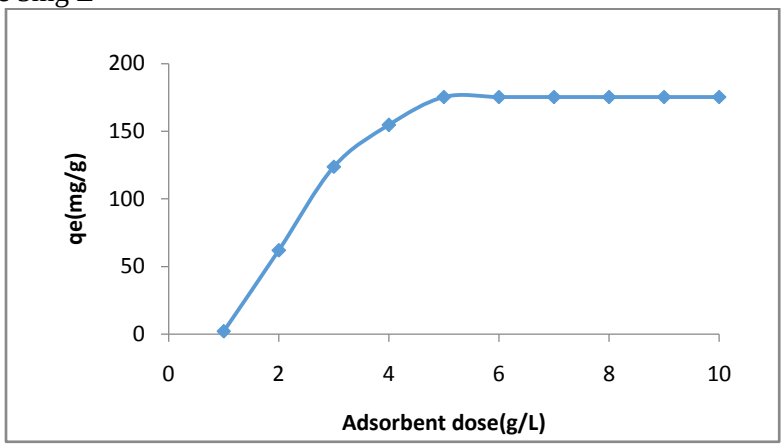

Fig 7. Effect of adsorbent dose on the biosorption of Methylene blue

Effect of particle size: The biosorbent particle size influences the external surface of a sorbent thus impacting on the interaction of the solution [27]. A variation in biosorbent size can affect the availability and accessibility of the reactive sites present on the surface [28]. Breaking up of bigger particles into smaller particles may open more and more blocked channels for adsorbent. The adsorbtion of Methylene blue by *Oscillatoria sp* was investigated at three particle sizes, 100, 200, 300 µm meshes. From (fig 8) it is clear that as the size of particle increases the adsorbtion capacity of adsorbate decreases. Hence particle size 100 µm mesh was used for the further experiments.

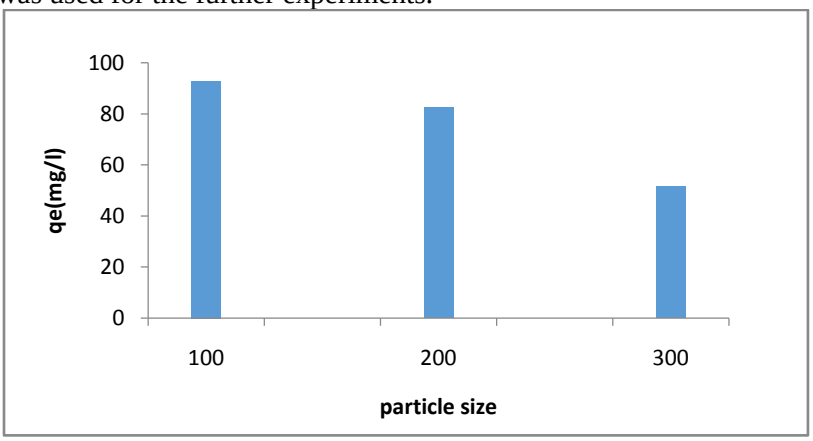

Fig 8. Effect of particle size on biosorption of Methylene blue

Effect of temperature: The effect of temperature on dye biosorption was investigated by taking three different temperatures (298, 308 and 318 K). With an increase in temperature there was an increase in adsorption. This shows that adsorption is endothermic. This phenomenon can be understood as chemical reaction between adsorbate molecules and the surface activity of adsorbent. Moreover, high temperature favors diffusion of dye molecules in the internal porous structure of adsorbent. Similar increase in sorption capacity with temperature was reported for removal Malachite Green by *Caulerpa racemosa* var. cylindracea [29].

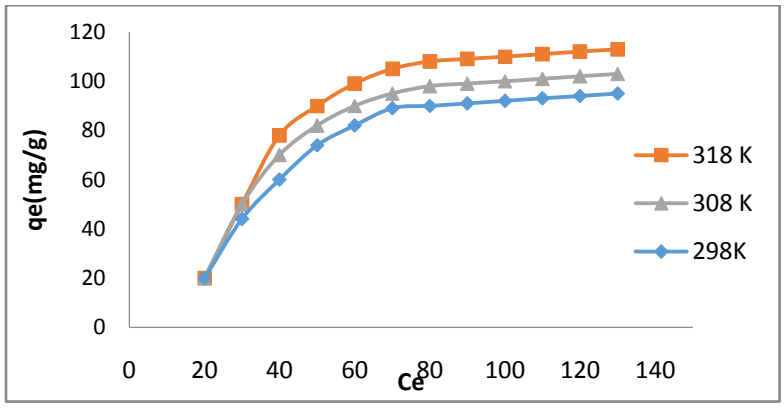

Fig 9. Adsorption isotherms at three different temperature

Adsorption Isotherm: The Langmuir and Freundlich models were employed to study the adsorption isotherms of dye. Langmuir isotherm shows that dye is adsorbed on a uniform surface with a finite number of adsorption sites and energy of sorption is constant. Monolayer sorption is expressed as:

$$\frac{1}{q_e} = \frac{1}{Q_0} + \frac{1}{bQ_0C_e} \quad \text{.....(2)}$$

where q_e is the amount adsorbed (mg/g), C_e is the equilibrium dye concentration of adsorbate (mg/L), Q_0 is the Langmuir constant related to maximum monolayer adsorption capacity (mg g^{-1}) and b is the constant related to free energy. The plot of 1/ q_e versus 1/ C_e is shown in figure. 10, to calculate constants given as in Table 1.

The Freundlich model can be expressed as the equation

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

The plot between ln qe versus ln C_e was drawn as given in fig 11 to calculate the value of K_F and 1/n given in table 1. The value of regression coefficient obtained from these models was used to find the best fit isotherm which was observed to be Langmuir model.

Table 1- Langmuir and Freundlich parameters for the adsorption of Methylene blue on *Oscillatoria* sp

Langmuir Constants			Freundlich Constants		
b(L/mg)	q_e(mg/g)	R^2	n	K_F(mg/g)	R^2
0.1196	129.58	0.999	3.33	3.115	0.933

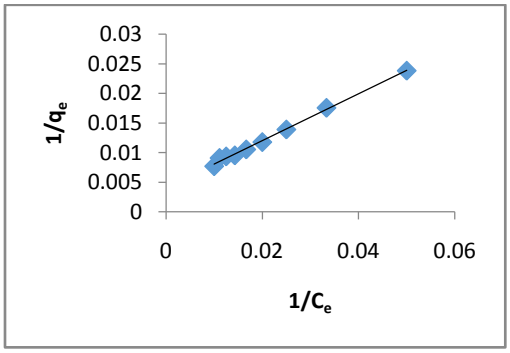

Fig 10. Langmuir fitting for the adsorption of MB

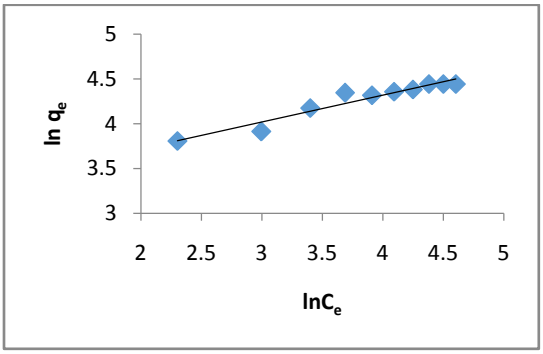

Fig 11. Freundlich Isotherm for the adsorption of MB

Kinetic study: The mathematical equation of pseudo-first and second-order kinetic models are given below.

The linear form of the pseudo-first order rate expression of Lagergren can be expressed as

$$\log (q_e - q_t) = \log q_e - \frac{k_{1,ads}}{2.303} t \quad \text{----------(4)}$$

where q_t (mg/g) is the amount of dye adsorbed by biomass at equilibrium time t, k_1 is the pseudo-first order rate constant (min^{-1}). The graph of log ($q_e - q_t$) versus log $q_e$is shown in figure 12 and the kinetic parameters are given in table 2.

The pseudo-second order model can be expressed in its linear form by the equation

$$\frac{t}{q} = \frac{1}{k_{2,ads} q_e^2} + \frac{1}{q_e} t \quad \text{.....(5)}$$

where k_2 (g mg^{-1} min^{-1}) is the rate constant of second order adsorption. The graph t/q versus t is shown in figure 13, and the kinetic parameters in table 2. The value of correlation coefficient for pseudo-second order adsorption model was found to be relatively high to that of pseudo-first order thus concluding second order kinetics for adsorption of dye on *Oscillatoria sp.*

Table 2: Kinetic parameters for the adsorption of Methylene blue on *Oscillatoria sp*

Initial concentration (mg/L)	First order kinetics			Second order kinetics		
100	K_1 (x 10^{-3} min^{-1})	q_e cal. (mg/g)	R^2	K_2 (x 10^{-3} g/mg/min)	q_e cal. (mg/g)	R^2
	11.515	216.77	0.965	0.140	90.90	0.994

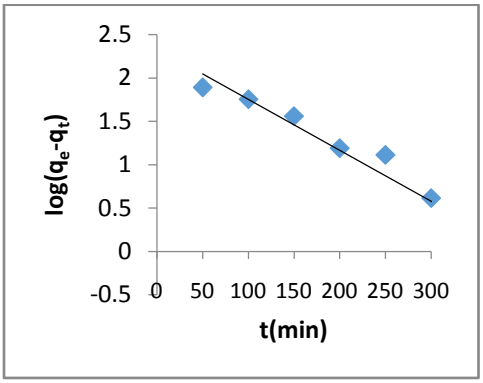

Fig 12. First-order kinetic modeling of MB

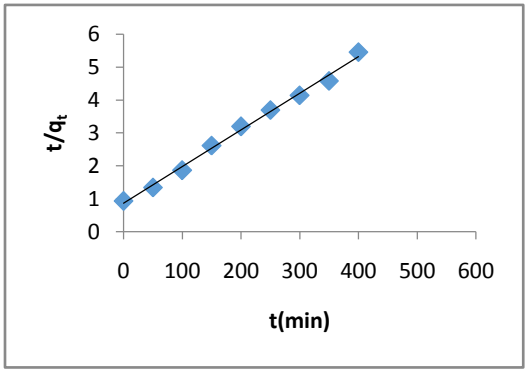

Fig13. Second-order kinetic modeling of MB

Thermodynamic study: Various thermodynamic parameters i.e. standard free energy changes (ΔG^0, kJ/mol), enthalpy changes (ΔH^0, kJ mol^{-1}) and entropy changes (ΔS^0, J mol^{-1} K^{-1}) can be deduced that are associated with adsorption mechanism. These parameters were calculated using following equations:

$$\Delta G^o = - R T \ln (b) \quad \text{----------(6)}$$

$$\ln (b_2 / b_1) = - \frac{\Delta H^0}{R} \left(\frac{1}{T_2} - \frac{1}{T_1} \right) \quad \text{---------- (7)}$$

$$\Delta G^o = \Delta H^o - T \Delta S^o \quad \text{----------(8)}$$

The value of all the three parameters is given in table 3. The enthalpy change ΔH^0 is positive as there is an increase in adsorption with an increase in temperature (endothermic). Spontaneity of the reaction can be depicted by the negative value of ΔG^0 and the positive value of ΔS^0 reflects the randomness of algal-solution interface and the affinity of *Oscillatoria* sp. towards dye molecules.

Table 3: Thermodynamic parameters for adsorption process of dye on *Oscillatoria* sp.

Temperature(K)	ΔG^0(kJ/mol)	ΔS^0(kJ/mol/K)	*ΔH^0(kJ/mol)
298	-20.2625	0.07751	2.83548
308	-21.037	0.077508	
318	-21.181	0.075523	

*Measured between 298 and 318K

APPLICATIONS

Dyeing effluents are harmful not only to humans but also to aquatic life. Hence present study can be useful in the removal of Methylene blue dye from wastewater using abundantly available blue green alga *Oscillatoria sp* as low cost adsorbent.

CONCLUSIONS

This study reports on the usage of *Oscillatoria* sp. as an adsorbent for the removal of Methylene blue from aqueous solution. The amount of MB dye adsorbed was found to be dependent on pH, adsorbent concentration, initial dye concentration, and contact time. The results showed that biosorption was a quick process and equilibrium was reached within 60 min, and the adsorption capacity decreased on increasing the pH. The biosorption followed the pseudo-second order kinetics. The negative value of free energy proved it to be thermodynamically feasible and spontaneous process, while the positive value of enthalpy change suggested the endothermic nature of the process. Langmuir isotherm was found to be the best fit. Conclusively, the present study showed that *Oscillatoria sp* algal biomass has a potential as an adsorbent for the removal of MB from aqueous solutions.

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AUTHORS' ADDRESSES

1. **Ms. Surabhi Sagar**
Research Scholar
Department of Chemistry
KLDAV PG College, Roorkee-247667, Uttarakhand, India
Email: surabhi726@gmail.com.
Mob: +91-9634874344
2. **Dr. Arshi Rastogi**
Assistant Professor, Department of Chemistry
KLDAV PG College, Roorkee-247667, Uttarakhand, India
Email: arshirastogi@gmail.com.
Mob: +91-9412912391