Scientific paper

Biosorption of Methylene Blue by *Chaetophora Elegans* Algae: Kinetics, Equilibrium and Thermodynamic Studies

Mouhiaddine Mohamed El Jamal[1,*] and Mohamed Chaker Ncibi[2]

[1] *Lebanese University, Faculty of Sciences (I), Chemistry Department, El Hadath, Lebanon.*

[2] *University of Antilles and Guyane, Faculty of Exact and Natural Sciences, COVACHIMM Lab., Pointe à Pitre, 97157 Guadeloupe, France*

* *Corresponding author: E-mail: mjamal@ul.edu.lb*

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Abstract

Adsorptive removal capacities of renewable and highly available *Chaetophora elegans* algae have been investigated in this study. To assess the use of this soft water algae to remove organic pollutants from aqueous solution, Methylene blue (MB) dye was used as a model molecule. The effect of dye concentrations, pH, adsorbent mass, temperature, and particle size have been evaluated. The algal biomass showed quite interesting adsorption capacity under optimized operating conditions (333 mg of dye per gram of biomass at 30 °C). Pseudo-first and pseudo-second order kinetic models were applied to the adsorption dynamic data. Pseudo second order model was well in line with the experimental data, therefore suggesting a probable chemically-based adsorption process. Several isotherm models were investigated to monitor the adsorption behavior. The Langmuir-Freundlich isotherm model fitted the experimental data best. The adsorption thermodynamic parameters $\Delta G°$, $\Delta H°$ and $\Delta S°$ were calculated. The maximum uptake is independent of temperature. From the values of the thermodynamic parameters, we concluded that the adsorption is exothermic, more ordered and spontaneous.

Keywords: Chaetophora algae, dye, adsorption, isotherms, kinetics, thermodynamics

1. Introduction

Water pollution mostly comes from wastewater which contains industrial and environmental pollutants. Dye manufacturing causes serious problems in wastewater. Dyes affect biodegradation, light penetration and photosynthesis. Minor releases of colorants impact the aesthetics and cause health disorders in organisms exposed to them producing imbalances in these ecosystems. Furthermore, some of these dyes pass to drinking water and damage human life. For example methylene blue usually used to dye cotton and wool, can cause serious health problems such as vomiting, hard breathing, and mental disorder.[1] Possible treatments such as filtration, oxidation, sedimentation and adsorption for cotton textile wastes and their associated advantages and disadvantages are presented in the literature.[2] Adsorption techniques for wastewater treatment have become more popular due to their efficiency. Activated carbon is mainly used to remove dyes from aqueous solutions, but it is very expensive.[3] Many reviews have appeared in literature about the low cost adsorbents used.[3–5] To remove dyes from aqueous solution, the adsorbent can be from agricultural waste such as sugarcane bagasse pith,[6] dates stones,[7] industrial waste products including fly,[8] natural inorganic materials such as desert sand,[9] bentonite,[10] gypsum,[11] and kaolinite.[12]

Many researchers studied the ability of marine and soft water algae to remove toxic dyestuff from water including *Cystoseira barbatula* brown algae,[13] *Spirogyra*,[14] *Pithophora*,[15] and *Enteromorpha*.[16] The ability of algae to remove dyes is due to the functional groups present in the principal organic constituents of algae such as polyphenols, polysaccharides, and alginic acid.[17]

In this work, a batch-based adsorption study on *Chaetophora elegans* algae is undertaken. The effect of pH, initial concentration and temperature were tested in order to optimize the adsorption process. Besides, a comparison between linear and non-linear regression method has

been made to predict the best adsorption kinetics and to obtain a more realistic set of dynamic parameters.

2. Experimental

2. 1. Materials

The soft water alga *Chaetophora elegans* were collected from the pond in Ammik's reserve in Bekaa valley (Lebanon) in June 2010. The material was first washed several times with tap water to remove dirt and small aquatic organisms. Afterward, they were washed several times with distilled water. The material was dried at room temperature then rubbed by hands to obtain it as fine powder. The obtained powder was dried at 100 °C for 12 h. Other samples are dried at higher temperature to find the best preparation conditions for algae. The dried powder was characterized by FTIR (spectrophotometer Thermo, Nicolet IR 200, dilution with KBr) to know the functional groups that might intervene in the adsorption process. XRD analysis was carried out with D8 Focus Bruker (CuKα 1.54 K at 50 KV).

Methylene blue (chemical structure in Figure 1) was used in this study as a model molecule for organic pollutants in general and basic dyes in specific. This dye was also chosen because of its extensive use in sorption studies, which would enhance the forthcoming comparative section.

Hence, accurately weighted quantities of dyes were dissolved in ultra pure water (with conductivity of 18 μS) to prepare the stock solutions. The solutions were prepared by diluting the stock solutions to give the appropriate concentrations.

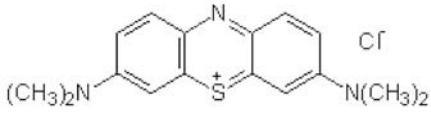

Figure 1: Chemical structure of Methylene Blue

2. 2. Batch Mode Adsorption Studies

All adsorption experiments were carried out by agitating the required amount of algae with 50 mL of dye solution of a desired concentration at constant temperature in a water bath shaker at 215 rpm.

Several operating conditions affecting the dye uptake were studied in order to optimize the overall adsorption process. After a predetermined time interval, a small volume of the solution was extracted, centrifuged and analyzed for residual dye concentration via the spectrophotometric technique. The concentration of dye was determined by double beam UV– Visible, Specord 200, Analytical Jena spectrophotomer. Blank samples were run under similar experimental conditions but in the absence of adsorbent and did not show a significant loss of dye on the container walls.

The amount of dye adsorbed per unit weight of algae at equilibrium; q_e (mg g^{-1}) or at time t; q_t (mg g^{-1}) were calculated according to the following equations:

$$q_e = (C_o - C_e) \times \frac{V}{m} \quad (1) \quad and \quad q_t = (C_o - C_t) \times \frac{V}{m} \quad (1)$$

C_o and C_e: The initial and equilibrium concentrations of dye (mg L^{-1}), respectively. V is the volume of the dye solution (L) and m is the amount of the adsorbent used (g). To determine the percentage of dye removal equation (3) is used:

$$\% \, removed = \frac{(C_o - C_e)}{C_o} \times 100 \quad (2)$$

3. Results and Discussions

3. 1. IR and XR Diffraction Analysis

The infrared spectrum of pure algae, shown in Figure 2, displays a number of absorption peaks, indicating its complex nature. Several bands were found at 3350 cm^{-1} (–OH or $–NH_2$), 2915 cm^{-1} (–CH or COOH), 1620 cm^{-1} (>C=O), 1424 cm^{-1} (aromatic ring) and 1060 cm^{-1} (C–O or >S=O). Quite similar spectra were found in the literature from other algae such as *Cystoseira barbatula,*[13] and *Cystoseira baccata.*[17] MB shows several intense bands at 3425 cm^{-1} (T%: 31), 1600 cm^{-1} (T%: 4), 1350 cm^{-1} (T%: 10). The comparison of the IR spectrum of algae before and after adsorption of MB did not show a clear difference, but a deep look shows the appearance of two small peaks at 1600 cm^{-1} and 1350 cm^{-1} attributed to aromatic ring and C–N aromatic tertiary amine respectively, both are present in MB. A similar difference was also observed with crystal violet.[18]

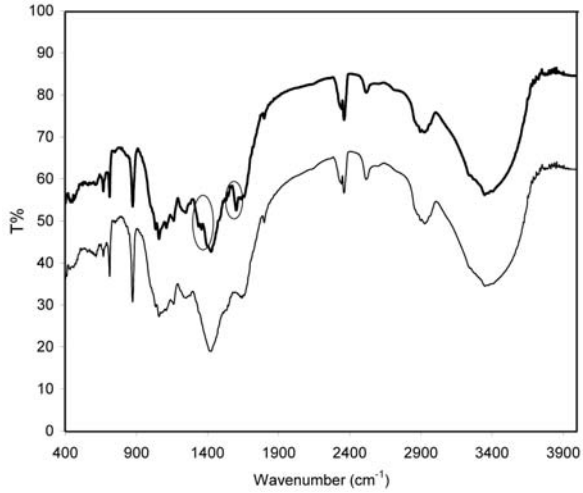

Figure 2: FT-IR spectrum of raw algae (—) before adsorption, (—) after adsorption.

The XR diffraction spectrum of MB shows three intense peaks at 2θ: 9.18° (88.5 %), 11.26° (63 %) and 25.57° (100 %). The XR diffraction spectra of algae before and after adsorption of MB did not shows the apparition of any of the intense peaks of MB (Figure 3).

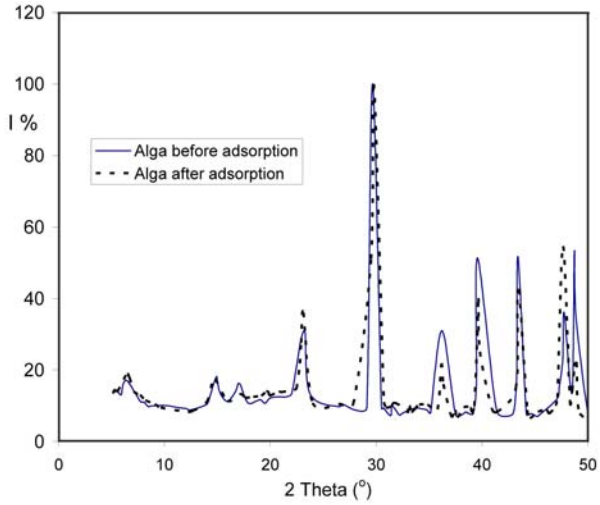

Figure 3: XR diffraction Spectra of algae before and after adsorption of MB.

3. 2. Effect of Thermal Activation

The algae was dried at different temperatures ranging from 100 to 200 °C (higher temperatures tend to completely carbonize the algae and entirely change its structure and composition). The drying time was 12 h. As shown in Figure 4, the thermal treatment did not influence the MB sorption capacity of the green algae for temperatures between 100 and 150 °C, regardless of the initial dye concentration.

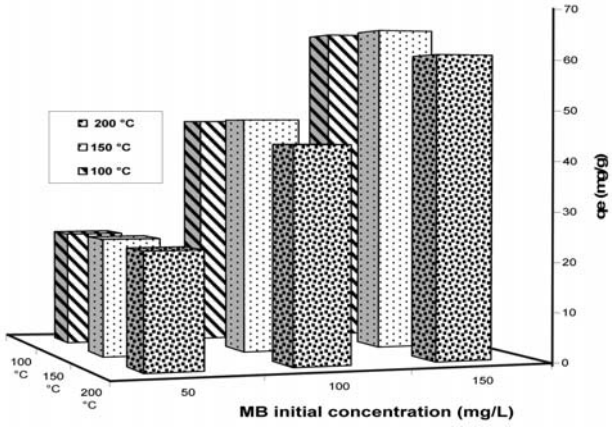

Figure 4: Effect of drying temperature on the adsorption of MB onto Chaetophora biomass (V = 50 mL, temperature = 30 °C and pH_o = 6)

At 200 °C, the biomass revealed two behaviors: (i) for lower MB concentration (i.e. 50 mg L^{-1}), there is no significant difference between the dye removal values at different thermal treatments. ii)as the MB initial concentration increased, the biomass treated at 200 °C showed a slight decrease in the sorption capacity. Such behavior could be linked to heat-related structural destruction and/or changes in the surface chemistry of the algae. A couple of optical microscope observations revealed that the dried powder algae at 100 and 150 °C are very similar (slim, narrow with transparent membranes). However, the shape and color of the algal biomass treated at 200 °C became different. There is apparition of tiny dark particles (*Cf.* Figure 5).

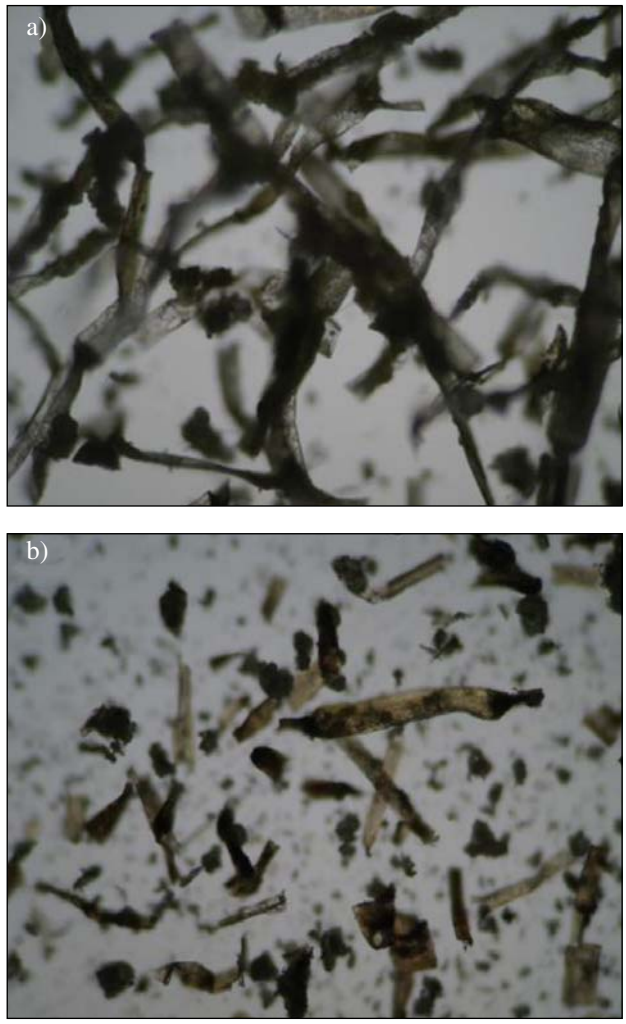

Figure 5: Effect of the drying temperature on the shape of the algal biomass. (a) Thermal treatment at 100 °C and (b) treatment at 200 °C [amplification X100]

Hence, in order to avoid unnecessary energy loss related to the heating process, the following experiments will be done with algal biomass dried at 100 °C.

3. 3. Effect of Initial pH

The interaction between a dye and biosorbent is mainly affected by ionisation states of the functional

groups on both dye molecule and biosorbent surface.[19] For the case of MB, the pH effect on the dye ionization is considered negligible, since MB molecules tend to remain positively charged. Experimentally, the visible spectrum of MB does not change in function of pH according to the Beer's law, with a constant K=A/[MB] corresponding to 0.184 L mg^{-1} at λ_{max} of 656 nm.

The effect of pH was studied for the pH range of 2 to 10 at a temperature of 30 °C. As shown in Figure 6, the tendency depicted with respect to the pH evolution was: (i) in the pH range 2 to 4.5, the $[MB]_f$ decreased (i.e. uptake of dye increased). Then, (ii) above pH 5, the dye removal remained constant. The increase in MB removal is mainly attributed to the electrostatic interactions between adsorbent and MB molecules, which is enhanced considering the negatively-charged biomass surface (i.e. gradual deprotonation as the pH increases) on one hand and the positively charged cationic dye on the other.[20,21]

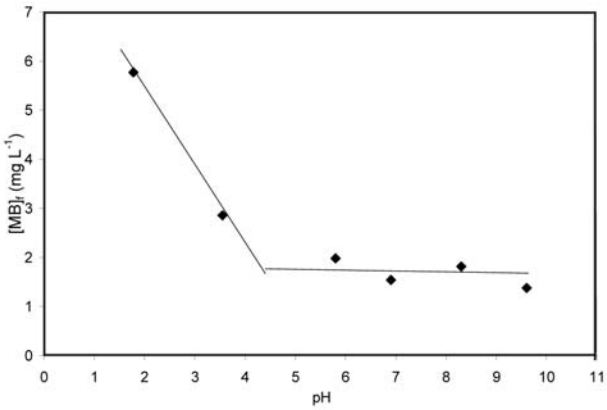

Figure 6: Variation of the MB final concentration as a function of pH (50 mL of 40 mg L^{-1} of MB at 30 °C)

3. 4. Effect of Adsorbent Dosage

The effect of adsorbent dosage on MB adsorption is shown in Figure 7. The results show that as the adsorbent dosage increases, as the number of adsorption sites increases. As for the sorption capacity (calculated for Eq. 1) the values increased from 25.9 mg g^{-1} using 0.2 g of algae to 202 mg g^{-1} using 0.025 g of biomass. Similar results were obtained for dye removal using the Brown algae *Cystoseira barbatula,*[13] and marine *Posidonia oceanica* fibers.[16]

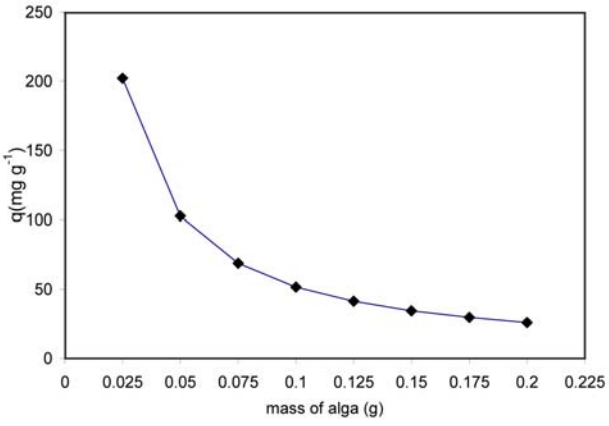

Figure 7: Variation of the MB uptake as a function of *Chaetophora* quantity (50 mL of 100 mg L^{-1} of MB, Temperature = 30 °C and pH_o = 6)

3. 5. Effect of Contact Time under Several Initial Dye Concentrations

Results of the effect of exposure time for MB adsorption onto soft water C*haetophora* algae is shown in Figure 8.

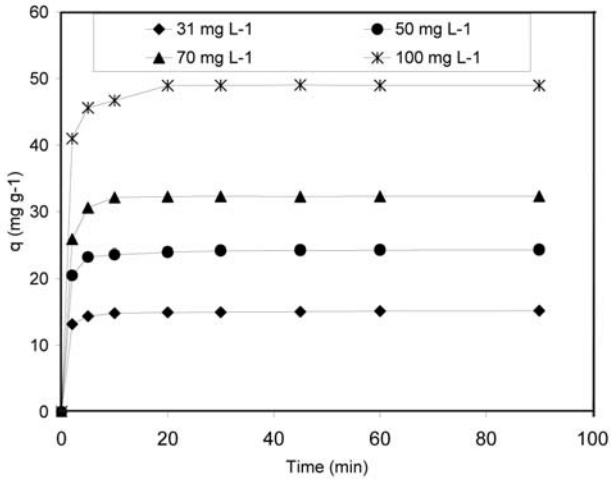

Figure 8: Effect of contact time on MB adsorption under several initial concentrations (V = 50 mL, mass of algae = 0.1g, Temperature = 30 °C and pH_o = 6)

It can be observed that, the rate of adsorption was very fast in the first 3 minutes, then gradually slowed down from 3 to 15 minutes until equilibrium is reached. The results show that the equilibrium states were attained at almost 15 to 20 minutes within the experimental concentration range. Furthermore, raising the dye concentration from 30 to 100 mg L^{-1} allows the fibers to increase their sorption capacities (Figure 8).

Hence, although 30 minutes seem to be sufficient to reach equilibrium for all studied MB concentrations, the isotherm experiments were left for 4 h to ensure a full saturation.

3. 6. Kinetic Modeling Analysis

Different kinetic models were used to predict the mechanism involved in the sorption process. In the present study, a comparison between linear and non-linear regression methods has been used to predict the best sorption kinetic model, and also to obtain reliable kinetic parameters. The kinetic parameters were evaluated by linear

and non-linear regression analysis by using Excel and OriginPro® 7.0.

Lagergren first order equations are[22]:

$$\text{Linear form: } \log(q_e - q_t) = \log q_e - \frac{k_I}{2.3}t \quad (3)$$

$$\text{Non Linear form: } q_t = q_e(1-e^{-K_I t}) \quad (4)$$

The first order rate constant k_I can be obtained from the slope of plot between $\log(q_e - q)$ versus time t.

The pseudo-second order model can be expressed as[23,24]:

$$\text{Linear form: } \frac{t}{q_t} = \frac{1}{k_{II} \times q_e^2} + \frac{t}{q_e} \quad (5)$$

$$\text{Non linear form: } q_t = \frac{q_e^2 \times k_{II} \times t}{1 + k_{II} \times q_e \times t} \quad (6)$$

Where t is the contact time (min), q_e (mg g^{-1}) and q_t (mg g^{-1}) are the amount of dye adsorbed at equilibrium and at any time, t. A plot between t/q_t versus t gives the value of the constants k_{II} (g mg^{-1} min^{-1}) and q_e (mg g^{-1}).

The dynamic sorption behavior of MB onto *Chaetophora elagans*' surface under several initial dye concentrations was monitored and modeled using both linear and non linear regression analyses (Table 1 and 2). Thus, considering the linear modeling approach, the deduced χ^2 and the R^2 values clearly showed that the pseudo-second order model is the most suitable one to fit the experimental data. Using the non linear regression analysis, the pseudo-second order model still fit the kinetic data well. However, the Lagergren pseudo-first model showed a clear and significant improvement in complying with the experimental data. For instance, the R^2 values, for a MB concentration of 30 mg L^{-1}, increased from 65% with a linear analysis up to 99.8% using a non linear regression analysis. Thus, it is clear that when the pseudo-first order equation is applied to fit kinetic data in its linear form, a serious error occurs when fitting the data.

3. 7. Isotherm Modeling Analysis

The analysis of the sorption equilibrium data is quite important to accurately monitor the adsorption behavior and exploit the isotherm data for design purposes. Several isotherm models are used in the literature to find the relationship between q_e and C_e. The experimental data related to the adsorption of MB molecules onto the algal biomass at different temperatures were fitted using Langmuir,[25] Freundlich,[26] Temkin,[27] and combined Langmuir-Freundlich equations.[28] In this study, the theoretically predicted isotherm data were determined using a non-linear regression analysis.

(i) The Langmuir model: $$q_e = \frac{q_{max} \times b \times C_e}{1 + b \times C_e} \quad (7)$$

with q_{max} in mg g^{-1} and b in L mg^{-1}.

(ii) The Freundlich model: $$q_e = k \times C_e^{\,n} \quad (8)$$

(iii) Temkin isotherm:

$$q_e = \frac{RT}{b}(\ln K_T + \ln C_e) = A + B \times \ln C_e \quad (9)$$

Table 1. Pseudo first and pseudo second order adsorption kinetic parameters and error estimation deduced from linear regression analysis at different initial dye concentrations

	Pseudo-first order (Linear)				Pseudo-second order (Linear)		
$[MB]_o$ (mg L^{-1})	q_e exp. (mg g^{-1})	q_e calc. (mg g^{-1})	k_I (min^{-1})	R^2	q_e calc. (mg g^{-1})	k_{II} (g $mg^{-1}min^{-1}$)	R^2
30	15.2	2	0.1	0.65	15.7	0.234	1
40	15.3	6.7	0.187	0.91	15.38	0.176	1
50	24.3	3	0.175	0.75	24.3	0.27	1
70	32.29	17.3	0.4	0.96	32.47	0.125	1
100	48.99	22.4	0.28	0.96	49.5	0.064	1

Table 2. Pseudo first and pseudo second order adsorption kinetic parameters and error estimation deduced from non linear regression analysis at different initial dye concentrations

	Pseudo-first order (Non linear)					Pseudo-second order (Non linear)			
$[MB]_o$ (mgL^{-1})	q_e exp. (mg g^{-1})	q_e calc. (mg g^{-1})	k_I (min^{-1})	R^2	χ^2	q_e (mg g^{-1})	k_{II}	R^2 (g mg^{-1} min^{-1})	χ^2
30	15.2	14.98	1.04	0.998	0.053	15.3	0.21	1	0.006
40	15.3	15.13	0.55	0.73	0.069	15.4	0.149	0.97	0.007
50	24.3	24.17	0.97	0.999	0.06	24.77	0.125	0.996	0.265
70	32.29	32.15	0.8	0.999	0.137	32.84	0.063	0.998	0.236
100	48.99	48.3	0.92	0.995	1.42	49.4	0.0486	1	0.137

Where B is a factor related to the heat of adsorption and K_T is Temkin equilibrium constant (L mg^{-1}).

(iv) <u>*Combined Langmiur-Freundlich*</u>:

$$q_e = \frac{q_{max} \times b \times C_e^n}{1 + b \times C_e^n} \quad (10)$$

The experimental data related to the adsorption of MB molecules onto the algal biomass at different temperatures are shown in Figure 9.

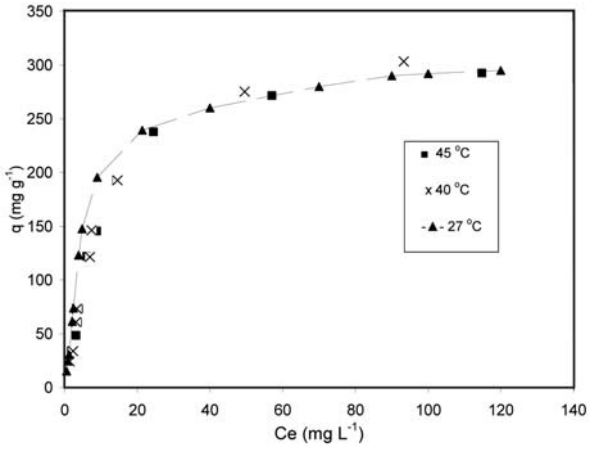

Figure 9: Isotherm adsorption of MB at several temperatures (pH_o = 6, V = 50 mL and mass of algae = 0.1 g)

The calculated isotherm constants by non linear methods are shown in table 3. As seen, the best fitting equation is the Langmuir- Freundlich model for all three temperatures based on the highest R^2 and the lowest χ^2. The Langmuir model is also suitable to describe the present sorption process. The high value of q_e (Langmuir's Q_{max} = 333 mg g^{-1} at 30 °C) makes the *Chaetophora elegans* algae a very promising and highly performing biomaterial for dye removal from aqueous solutions (Table 4).

Table 4: Adsorption capacities for bioadsorbents.

Adsorbents	Adsorption capacity (mg/g)	Reference
Green algae Ulva lactuca	40.2	[20]
Enteromorpha spp.	274	[21]
Algae Sargassum muticum seaweed	279.2	[29]
Brown algae Cystoseira barbatula	38.61	[13]
Posidonia oceanica (L.) fibres	5.56	[16]
Activated carbon (groundnut shell)	165	[30]
Activated carbon (Bamboo dust)	102	[31]
Caulerpa lentillifera	417	[32]

3. 8. Thermodynamic Analysis

The effect of temperature (found in the literature) on the biosorption parameters and the thermodynamic parameters presents different and opposite behaviors.[9,17,31–35] The thermodynamic parameters are calculated according to the equations below:

$$\ln K = \frac{-\Delta H^o}{R} \times \frac{1}{T} + \frac{\Delta S^o}{R} \quad (11)$$

$$\Delta G^o = \Delta H^o - T \times \Delta S^o \quad (12)$$

$$\text{With } K = \frac{[MB]_{ads}}{[MB]_{final}} = \frac{C_o - C_e}{C_e} \quad (13)$$

For an initial MB concentration of 100 mg L^{-1}, the Van't Hoff equation is $\ln K = 1861/T - 2.43$ (Figure 10).

Table 3. Isotherm modeling parameters related to the biosorption of MB onto *Chaetophora elegans (non-linear approach)*

	Models	45 °C	40 °C	30 °C
Langmuir	χ^2	155	131.5	140
	R^2	0.985	0.99	0.987
	Q_{max} (mg g^{-1})	333.77	347.3	333.4
	b	0.08	0.079	0.116
Freundlich	χ^2	1258	966	639.4
	R^2	0.883	0.92	0.91
	K	54.38	50.5	53.15
	n	0.38	0.41	0.52
Temkin	χ^2	422	146	270
	R^2	0.96	0.987	0.96
	A	–0.42	–10.79	29.9
	B	65.7	71.6	68.5
Langmuir–Freundlich	χ^2	46.6	77	67.76
	R^2	0.996	0.995	0.995
	Q_{max} (mg g^{-1})	299.1	315.6	299
	b	0.052	0.058	0.088
	n	1.34	1.25	1.27

As can be seen from Table 5, the adsorption is exothermic, a more ordered and spontaneous process. The same behavior of adsorption is observed with the adsorption of MB onto desert sand.[8]

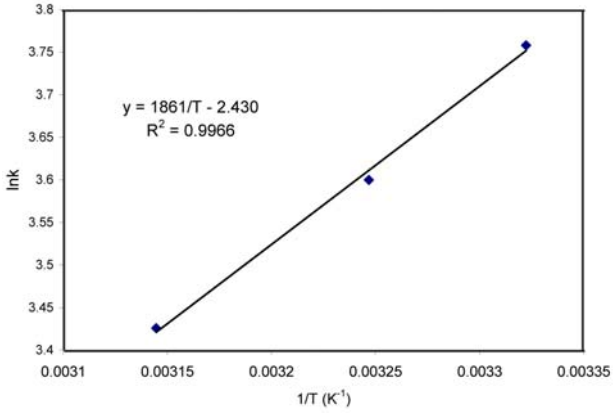

Figure 10: Plots of ln K versus 1/T for the adsorption of MB. (50 mL of 100 mg L^{-1} of MB, pH_o = 6, and mass of algae = 0.1 g)

Table 5: Thermodynamic parameters of MB onto *Chaetophora elegans* algae (T = 298, 308, 318 K).

[MB] (mg L^{-1})	$-\Delta H°$ (kj mol^{-1})	$-\Delta S°$ (kj mol^{-1} K^{-1})	$-\Delta G°$ (kj mol^{-1})		
			298 K	308 K	318 K
100	15. 45	0.02	9.49	9.29	9.09

4. Conclusions

The adsorption of MB onto *Chaetophora elegans* algae was investigated in order to assess the possibility of using this renewable and low cost biomass in removing organic molecules from aqueous media. The effects of several operating conditions were studied. It was shown that the sorption process is dependent on contact time (until equilibrium), the adsorbent dose, the pH and the initial dye concentration. However, the temperature variations do not influence the sorption capacity between 30 and 45 °C. As for the modeling section, experimental data of MB adsorption were best followed by the combined Langmuir–Freundlich model for isotherms and pseudo-second order for kinetics. The thermodynamic analysis revealed that the present adsorption process is exothermic and spontaneous.

Thus, the high adsorption capacity (Langmuir's Q_{max} = 333 mg g^{-1} at 30 °C) and the fast uptake (equilibrium within the first 20 minutes) of *Chaetophora elegans* makes this algal biomass are very promising biomaterial for dye removal from aqueous media and eventually for the treatment of industrial effluent containing basic dyes.

5. Acknowledgment

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Povzetek

Adsorptivne lastnosti široko dostopne in obnovljive alge *Chaetophora elegans* smo proučevali na primeru adsorpcije barvila metilen modro kot modelne molekule. Spreminjali smo koncentracijo barvila, pH, maso in granulacijo adsorbenta ter temperaturo. Ugotovili smo, da največjo kapaciteto adsorpcije (333 mg barvila na g biomase) dosežemo pri 30 °C. Eksperimentalne podatke smo analizirali s kinetičnim modelom psevdo prvega in psevdo drugega reda. Izkazalo se je, da psevdo drugi red bolje opiše eksperimentalne podatke, kar kaže na možnost kemijske sorbpcije. Nadalje smo proces adsorpcije opisali z različnimi adsorpcijskimi izotermami, od katerih se je Langmuir-Freundlichova izoterma izkazala kot najustreznejša. Določili smo tudi termodinamske parametre, $\Delta G°$, $\Delta H°$ in $\Delta S°$, ki pričakovane kažejo na to, da je proučevana adsorpcija spontan in eksotermen proces, ki ga spremlja znižanje entropije.