



Adsorption of Methylene Blue from Aqueous Solutions by Silk Cocoon

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ABSTRACT

This study concerns the performance of cocoons spun by silk worms as a natural adsorbent for removal of Methylene Blue (MB) from aqueous solutions. To study the adsorption process, the effect of various parameters such as contact time, adsorbent dosage, dye initial concentration, and pH of the solution were investigated. According to the experiments, the kinetic data were best described by pseudo second order model and the equilibrium data were properly fitted to Langmuir model. The maximum adsorbent capacity at ambient temperature was calculated to be 86.2 mg/g. Thermodynamic analysis showed that the process was spontaneous, endothermic with increased randomness at the solid-liquid interface. It was also observed that by manipulating the pH of the solution in acidic range, the adsorbed dye would desorb into the solution suggesting the reusability of the adsorbent. Macroscopic size of the adsorbent offered an additional advantage of ease of its separation from ease of separation from the solution.

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1. INTRODUCTION

Silkworm has been primarily cultivated for textiles since ancient era. In recent years, however, due to its desirable properties such as biocompatibility and ease of chemical modification, the application of silk is expanding from textiles to various areas like biodegradable medical scaffolds, implantable functional devices, tissue products and drug delivery [1-5].

Silk cocoon, a hierarchically structured biopolymer is produced by silk worm through a natural electrospinning-like process. Bombyx mori silk is composed of silk fibroin protein coated with sericin ($C_{30}H_{40}N_{10}O_{16}$), an adhesive protein that accounts for 25-30 wt.% of the total silk worm cocoon weight [6]. Silk fibroin with a primary structure mainly consisting of recurrent amino acid sequence (Glycine-Serine-Glycine-Alanine-Glycine-Alanine)_n is a block copolymer rich in hydrophobic β sheet-forming blocks linked by small hydrophilic linker groups [6]. Sericin consists of 18 amino acids, most of which contain hydroxyl, carboxyl and amino groups [7-9]. Sericin is more hydrophilic than fibroin.

Methylene blue ($C_{16}H_{18}N_3SCl$) is one of the most commonly used dye stuff for dyeing wool, cotton and silk. Since dye-containing effluents can cause serious environmental problems, they need to be treated before being discharged into the surroundings [10-12]. The treatment processes for removal of dye from water and wastewater can be grouped into three categories: biological (i.e. using microorganisms like bacteria, yeasts, algae and fungi), chemical (i.e. coagulation, precipitation-flocculation, oxidation, electrochemical processes), and physical (i.e. membrane-filtration, adsorption) [13]. Among these methods adsorption is considered to be more convenient from the view point of design simplicity, flexibility, ease of operation and initial costs. Moreover, additional harmful substances are not formed through adsorption process [13]. In this regard, natural organic sorbents derived from plants or animal residues are of great interest since they are abundant, environmental friendly and cost effective. Technical feasibility of various non-conventional low-cost adsorbents for MB removal from water and wastewater is addressed by Rafatullah et al.(2010) who have compiled an extensive list of adsorbents literature [14]. Since pierced and defected silk cocoons are discarded as industrial wastes, they can be regarded as

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low cost materials. Previously silk cocoon with its sericin coating had been used as sorbent for oil removal from water [15]. Recently, activated fibers from silkworm cocoon has been applied for dye removal application [16]. This study explores the performance of the degummed silk cocoon as a natural adsorbent for the removal of MB from water.

2. EXPERIMENTS

2. 1. Materials Silk cocoons were obtained from Gilan sericulture center in North of Iran. All the chemicals including MB (of Reag. Ph Eur grade) and sodium carbonate (ISO grade) were purchased from Merck.

2. 2. Adsorbent Preparation The cocoons were first cut into half and the worms were discarded. The raw cocoons were then degummed by placing them in 250 mL solution of 0.02M sodium carbonate and boiling the solution for 30 min. The cocoons were then rinsed in water for 20 min while stirring. After three rinses the cocoons were finally dried under ambient air [6].

2. 3. Adsorption Experiments For the adsorption experiments, first a stock solution of 100 ppm MB concentration was prepared and then certain volumes of the stock solution were diluted to obtain various MB concentrations. In the experimental work the effect of various parameters were studied in the batch mode. Kinetic studies of the adsorption were conducted by placing 20.5 mg of the adsorbent in 50 mL of 25 ppm MB concentration and sampling the solution at certain time intervals. The effect of adsorbent dosage was examined by placing 20-150 mg of the adsorbent in 50 mL of 25 ppm of MB solutions and the equilibrium concentrations were measured. The effect of initial concentration of MB was examined by placing 20 mg of the adsorbent in 50 mL of the 5-60 ppm of MB solutions. The initial pH values of the solutions with different concentrations of MB were around 6.3 ± 0.2 . The effect of pH on the adsorption of MB was studied by placing 16 mg of the adsorbent in 50 mL of 20 ppm of MB solution and adjusting the solution pH using 0.1 M HCl and 0.1 M NaOH solutions. To study the effect of temperature, the equilibrium experiments were carried out at 278 K, 308 K and 318 K temperatures. The dye concentration was measured spectrophotometrically. The removal percentage was defined as follows:

$$\% \text{ Removal} = 100 \times (A_0 - A) / A_0 \quad (1)$$

where A is the solution absorbance at λ_{max} (665 nm for MB) at time t, and A_0 is the absorbance of the initial

MB solution. The experiments were repeated several times and the average values were reported. The uncertainties were below 5%.

2. 4. Characterization The morphology of the cocoon structure was examined by a Stereo Scan S360 scanning electron microscope (SEM). Chemical bonds on the adsorbent were examined by Fourier Transform Infrared Spectroscopy (FT-IR, Perkin Elmer FTIR Spectrum RX I.). For concentration determination of the MB solutions, UV-Vis spectra of the solutions were recorded by a T80+UV-Vis PG Instruments spectrophotometer. The pH of the solutions was measured by a Metrohm 827 pH lab meter.

3. RESULTS AND DISCUSSION

3. 1. Morphology and Surface Chemistry Figure 1 shows the raw silk cocoon as well as the SEM images of its inner and outer surfaces at the same magnification. The cocoon morphology seems to be a highly porous structure made of continuous silk fibers. Bombyx mori cocoons have multiple layers parallel to the surface with sericin bonds between them. The relatively weak interlayer bonds form a 3D hierarchical structure in the cocoon [17]. The crystals attached to the surface of the fibers is probably calcium oxalate as reported in the previous studies [18].

FTIR spectra of the degummed cocoon before and after the MB adsorption process are shown in Figure 2. The peak at 1730 cm^{-1} can be ascribed to β sheet structure. As the silk fibroin contains amine groups, the characteristic peaks around 1620 cm^{-1} can be assigned to C=O stretching of amide I, 1525 cm^{-1} to N-H bending of amide II, 1460 cm^{-1} and 1263 cm^{-1} to C-N stretching of amide III [19, 20]. The peaks at 1620 cm^{-1} and 1263 cm^{-1} may be assigned to the amorphous structure while the peak at 1525 cm^{-1} may be ascribed to β -sheet in silkworm cocoon [16]. After adsorption of MB on the silk cocoon, the characteristics peaks are still detectable although the transmittance intensities are diminished (Figure 2b).

3. 2. Adsorption Kinetics The effect of contact time on the adsorption of MB by the silk cocoon is shown in Figure 3. It can be seen that the rate of adsorption is initially high and about 70% of dye is removed within the first 1.5 h, then it gradually reaches a plateau when the equilibrium is achieved. This can be due to the fact that all the active adsorption sites are initially available but as the adsorption process goes forward, these sites are occupied and the diffusion of dye molecules towards the adsorbent will control the adsorption rate. Interactions between dye and the adsorbent are affected by the dye molecular geometry and its charge. The porous structure of the adsorbent

provides proper accessibility for the dye molecules to diffuse into the pores. The electrostatic interaction between the cationic dye and the negative surface of the adsorbent (at $pH >$ isoelectric point) will assist the adsorption process. It should be noted that all the experiments were carried out in the static state (without stirring). To quantify the uptake rate, the kinetic data were fitted to pseudo first order, pseudo second order and intra particle diffusion models. Figure 4 shows that the data are well fitted to pseudo second order model (Equation (2)) with a regression coefficient of near unity [21].

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (2)$$

$$q_e = \left(\frac{C_0 - C_e}{m} \right) V \quad (3)$$

where, C_e , and q_e are the equilibrium adsorbate concentrations in the solution (mg/L) and solid phase (mg/g), C_0 is initial concentration of the adsorbate in the solution (mg/L), m is mass of the adsorbent (g), and V is volume of the solution (L).

K_2 is the pseudo-second-order rate constant that was calculated to be around $0.007 \text{ (g mg}^{-1}\text{h}^{-1}\text{)}$ from the intercept of the curve of t/q_t versus t (Figure 4). q_e obtained from the slope of the curve was 47.4 mg/g which was very close to the experimental value q_e (47.33 mg/g).

3. 3. Adsorption Isotherms The equilibrium data can be expressed by adsorption isotherms. Langmuir isotherm model describes a monolayer adsorption process where the adsorption surface is assumed to be homogeneous and the adsorption sites are equivalent while the Freundlich isotherm model assumes heterogeneous surface with different adsorption sites [22].

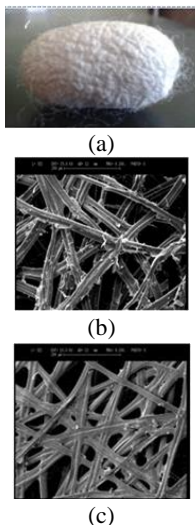


Figure 1. Silk cocoon (a), SEM images of its exterior surface (b), and interior surface (c)

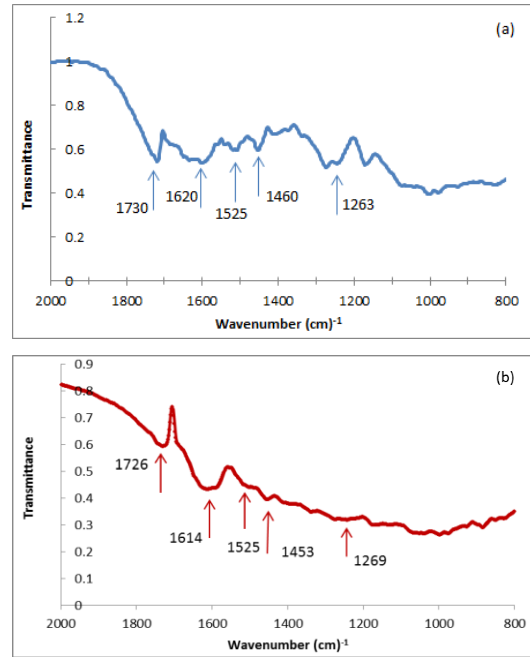


Figure 2. FTIR Spectra of the cocoon before MB adsorption (a), and after MB adsorption (b)

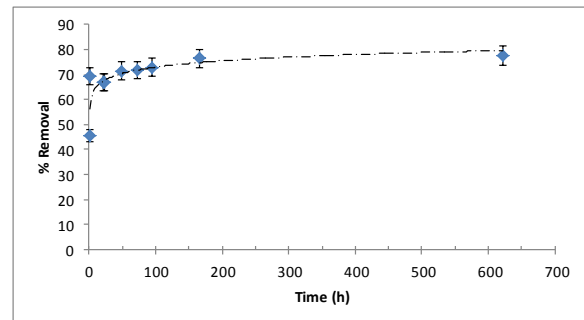


Figure 3. Variation of MB removal percent versus time

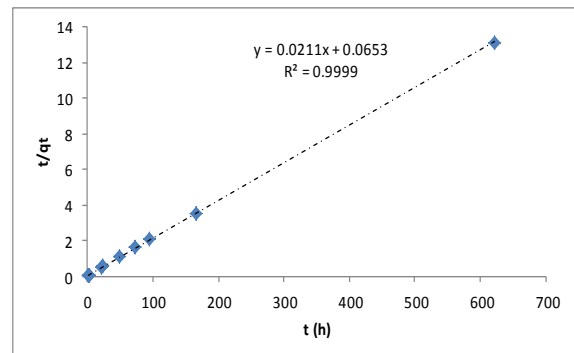


Figure 4. Kinetic data fitted to the pseudo second order model

Our experimental data were fitted to these models (Figure 5, Figure 6) and the former model showed a higher regression coefficient as depicted in Table 1.

Freundlich coefficients are $K_F ((\text{mg/g})(\text{L/mg})^{1/n})$ and n (dimensionless) which are related to adsorption capacity and adsorption intensity, respectively [23].

Heterogeneity of the adsorbent surface is reflected in $1/n$. Values of $0.1 < 1/n < 1.0$ indicate the favorable adsorption of the adsorbate on the adsorbent which in this case is calculated to be 0.86. Table 1 shows that the maximum MB adsorption capacity of the adsorbent (q_{max}) at ambient temperature is 86.2 mg/g that compared to some previous works is reasonable [14, 24-26].

3. 4. Effect of Adsorbent Dosage, Dye Initial Concentration, and Solution pH Figure 7 shows that as the weight of the adsorbent increases the removal percentage also increases up to a point where increasing the weight does not make any significant change on the removal.

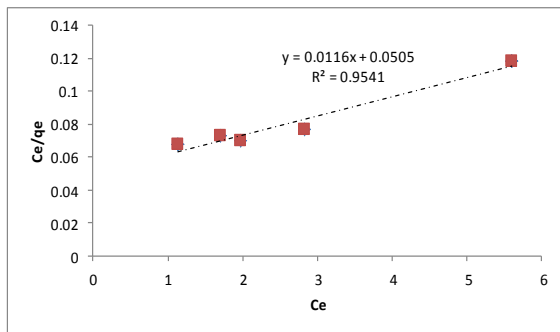


Figure 5. Equilibrium data fitted to the Langmuir model

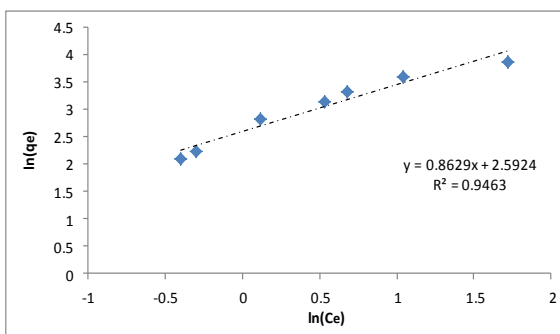


Figure 6. Equilibrium data fitted to the Freundlich model

TABLE 1. Parameters of the isotherm models

Freundlich	$1/n$	$K_F ((\text{mg/g})(\text{L/mg})^{1/n})$	R^2
$\ln(q_e) = \ln(K_F) + \frac{1}{n} \ln(C_e)$	0.86	13.4	0.9463
Langmuir	$q_{\text{max}}(\text{mg/g})$	$K_L(\text{L/mg})$	R^2
$\frac{C_e}{q_e} = \frac{C_e}{q_{\text{max}}} + \frac{1}{q_{\text{max}} K_L}$	86.2	0.23	0.9541

This can be explained by the fact that an increase in the adsorbent weight results in the abundance of the active sites and consequently enhancement of the adsorption process [22]. When the removal is completed any further increase in the adsorbent weight provides only unused excess sites.

The effect of initial concentration of MB on the removal percentage can be seen in Figure 8. At low concentrations, the dye is almost completely removed. By increasing the initial dye concentration, as the active sites afforded by the given amount of adsorbent are limited, the dye removal is imperfect [12].

Figure 9 shows the effect of solution pH on the removal of MB. Obviously at low pH, where the concentration of proton is high the dye removal is poor. This can be explained by the fact that since MB is a cationic dye, there is a competition between protons and the dye cations for adsorption on the solid surface. As the pH increases above the isoelectric point of the adsorbent (>2.1 [27]), the solid surface may gain negative charge and the electrostatic attraction enhances the adsorption process [10]. At $\text{pH} \sim 10.5$ the dye removal is accomplished and further increase in the pH is ineffective. It was observed that by lowering the solution pH the adsorbed dye was released into the solution (inset shows the applied cocoon). This might be due to the electrostatic repulsion between the adsorbed dye molecules and the solid surface.

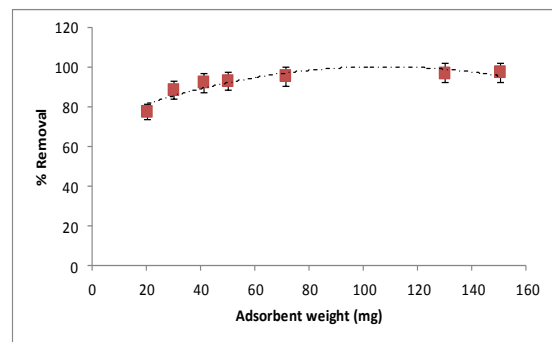


Figure 7. Effect of the adsorbent dosage on the dye removal

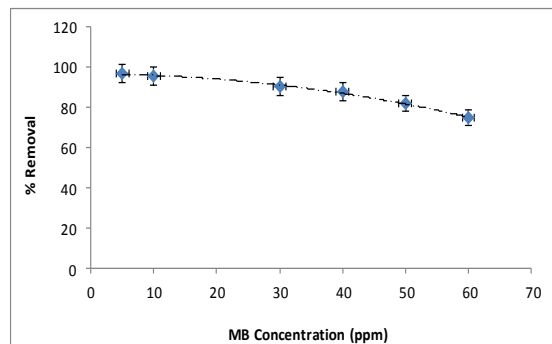


Figure 8. Effect of the initial concentration of MB on the dye removal

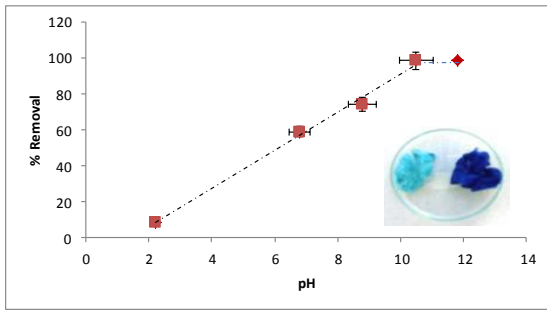


Figure 9. Effect of pH on the MB removal

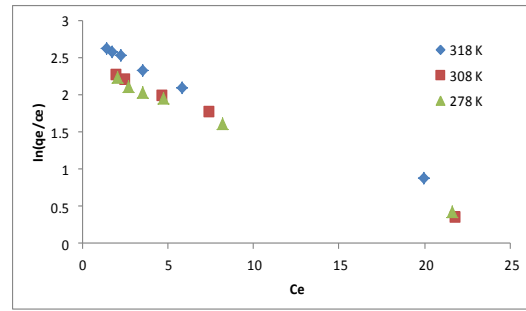


Figure 10. Effect of temperature on the adsorption of MB on the silk cocoon

3. 5. Adsorption Thermodynamics Figure 10 shows the effect of temperature on the adsorption of MB by the silk cocoon. Variation of thermodynamic equilibrium constant (K) with temperature was used to calculate the thermodynamic parameters:

$$K = \frac{a_s}{a_e} = \frac{v_s q_e}{v_e C_e} \tag{4}$$

where, a_s , is activity of the adsorbed solute, a_e the activity of the solute in the equilibrium solution, and v_s , v_e are activity coefficients of the adsorbed solute and solute in the equilibrium solution. When the concentration of the solute approaches zero, the activity coefficient approaches unity [28]:

$$\lim_{C_e \rightarrow 0} \frac{q_e}{C_e} = \frac{a_s}{a_e} = K \tag{5}$$

Therefore, by plotting $\ln(q_e/C_e)$ versus C_e and extrapolating to zero C_e , the value of K was calculated. The slope and intercept of Van't Hoff plot (Figure 11) were used to evaluate the changes of adsorption standard enthalpy (ΔH°) and standard entropy changes (ΔS°):

$$\ln(K) = -\frac{\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R} \tag{6}$$

where R is the universal gas constant (8.314 J/mol K) and T absolute temperature (K).

Besides, the following equation was used to obtain the change in Gibb's free energy:

$$\Delta G^\circ = -RT\ln(K) \tag{7}$$

From the experimental data, it is inferred that the process is endothermic as the adsorption standard enthalpy change (ΔH°) obtained from the slope of the plot in Figure 11 has a positive value (Table 2). The changes in Gibb's free energy in the applied temperature range are unremarkable and their negative values suggest that the adsorption process is spontaneous. The positive value of the standard entropy change (ΔS°) calculated from the intercept of the plot in Figure 11, can be attributed to the increased randomness at the interface of the adsorbent and solution [29].

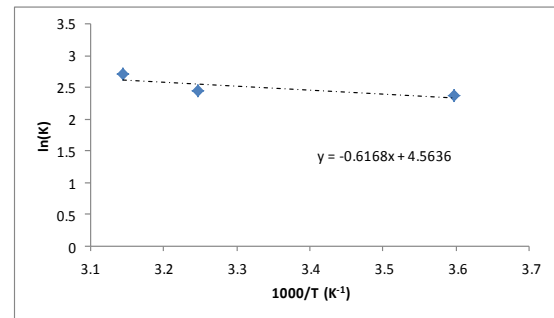


Figure 11. The Van't Hoff plot for the adsorption of MB on the silk cocoon

TABLE 2. Thermodynamic parameters of the adsorption of MB on the silk cocoon

T(K)	Ln(K)	ΔG_0 (kJ/mol)	ΔH_0 (kJ/mol)	ΔS_0 J/(mol K)
278	2.37	-5.5	5	37.9
308	2.45	-6.3		
318	2.71	-7.1		

4. CONCLUSION

Degummed silk cocoon is used as a natural adsorbent for removal of MB from aqueous solution. The experimental results show quite high adsorption capacity (86.2 mg/g) and reasonably fast kinetics.

The equilibrium data properly followed Langmuir model and the kinetic data were described by the pseudo second order model. Thermodynamic analysis showed that the process of adsorption of MB on the degummed cocoon was spontaneous, endothermic, and with increased randomness at the solid liquid interface. Direction of MB adsorption or desorption depends on the solution pH suggesting the reusability of the adsorbent. It is worth noting that the facile separation of the adsorbent from the solution due to its macroscopic size offers a reduction in the post treatment costs.

5. ACKNOWLEDGMENT

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در این تحقیق عملکرد پیله ابریشم صمغ زدایی شده که از کرم ابریشم تنیده شده، به عنوان یک جاذب طبیعی برای حذف رنگ متیلن بلو از محلول های آبی بررسی شده است. برای مطالعه فرایند جذب اثرات پارامترهای مختلف مانند زمان تماس، مقدار جاذب، غلظت رنگ اولیه، و pH محلول بررسی گردید. طبق آزمایش ها، داده های سینتیکی با مدل شبه درجه دوم به خوبی برازش شده و داده های تعادلی با مدل **Langmuir** مطابقت بیشتری دارد. ظرفیت جذب بیشینه حدود **86.2 mg/g** محاسبه شد. آنالیز ترمودینامیکی نشان داد که فرایند جذب خودبخودی، گرماگیر، و با افزایش بی نظمی در فصل مشترک مایع و جامد بوده است. همچنین، مشاهده شد که با تغییر pH در ناحیه اسیدی، رنگ جذب شده در محلول واجذب گشته و لذا استفاده مجدد از جاذب امکانپذیر می باشد. مزیت دیگر جاذب اندازه ماکروسکوپییک آنست که می توان آنرا به سهولت از محلول جدا نمود.

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