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Research Article

Isotherm and kinetic modeling of the adsorption of methylene blue, a cationic dye, on pumice

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ABSTRACT

The removal of dyes from aqueous solution with cheap and abundant adsorbents is becoming increasingly important for the solution of a serious environmental problem such as wastewater treatment. In this study, isotherm and kinetic modeling of the adsorption of methylene blue on pumice, a porous and glassy volcanic rock resistant to physical and chemical factors, was aimed. For this, the compatibility of the experimental data with the isotherm and kinetic models was examined, and information about the efficiency, effectiveness and rate of adsorption was tried to be obtained. Experimental data have been applied to isotherm models such as Langmuir, Freundlich, Temkin, Brenuer-Emmet-Teller (BET), Dubinin- Radushkevich, and Harkins-Jura, and kinetic models such as pseudo-first order, pseudo-second order, and intraparticle diffusion. While the order of fit for the isotherm models was determined as Freundlich > Harkins-Jura > Dubinin- Radushkevich based on the results of the regression analysis, the highest fit was obtained with the Freundlich equation (R^2:0.993). Thus, the adsorption intensity (n) of methylene blue on pumice was calculated as 1.14 and the adsorption capacity (k) of pumice was calculated as 6.43. On the other hand, the order of fit of the experimental data to the kinetic models was determined as pseudo-second order > pseudo-first order > intra particle diffusion according to the regression coefficients. However, the highest consistency among the kinetic models was obtained with the pseudo-second order kinetic model(R^2:1.000).

Keywords: Adsorption, pumice, methylene blue, ısotherm models, adsorption kinetics.

Katyonik bir boya olarak, metilen mavisinin pomza üzerine adsorpsiyonunun izoterm ve kinetik modellemesi

ÖZ

Boyaların sulu çözeltiden ucuz ve bol bulunan adsorbentlerle giderimi, atık su arıtımı gibi ciddi bir çevre sorununun çözümü için giderek önem kazanmaktadır. Bu çalışmada metilen mavisinin fiziksel ve kimyasal etkenlere dayanıklı, gözenekli ve camsı bir volkanik kayaç olan pomza üzerine adsorpsiyonunun izoterm ve kinetik modellemesi amaçlanmıştır. Bunun için deneysel verilerin izoterm ve kinetik modellere uyumu incelenerek, adsorpsiyon verimi, etkinliği ve hızı hakkında bilgiler edinilmeye çalışmıştır. Deneysel veriler, Langmuir, Freundlich, Temkin, Brenuer- Emmet-Teller (BET), Dubinin- Radushkevich ve Harkins-Jura gibi izoterm modellerine ait denklemlere ve yalancı birinci derece, yalancı ikinci derece ve parçacık içi difüzyon gibi hız denklemlerine uygulanmış hem izoterm hem de kinetik modellere ait parametreler hesaplanmıştır. İzoterm modellerine uyum sırası, regresyon analiz sonuçları esas alınarak, Freundlich>Harkins-Jura > Dubinin- Radushkevich şeklinde belirlenmiş olmakla birlikte, en yüksek uyum, Freundlich denklemiyle elde edilmiştir(R^2:0,993). Böylece Metilen mavisinin pomza üzerindeki adsorpsiyon şiddeti (n), 1,14 ve pomzanın adsorpsiyon kapasitesi (k) ise 6,43 olarak hesaplanmıştır. Diğer yandan deneysel verilerin kinetik modellere uyum sıralaması da yine regresyon katsayılarına göre yalancı ikinci derece>yalancı birinci derece> parçacık içi difüzyon olarak belirlenmiştir. Ancak kinetik modeller arasında en yüksek uyum yalancı ikinci mertebe kinetik modelle elde edilmiştir(R^2:1,000).

Anahtar Kelimeler: Adsorpsiyon, pomza, metilen mavisi, izoterm modelleri, adsorpsiyon kinetiği.

1.INTRODUCTION

Industrial wastewater is one of the most significant environmental pollutants, and textile production, which is a major industry in every nation, is a major source of colored wastewater due to the usage of dyes.[1-3] However, colored wastewater is produced not only by the textile industry, but also by various industries such as paint, cosmetics, medicine, leather, food, health, and paper.[4] Moreover, colored wastewaters not only change the color of water bodies in terms of aesthetics, but also affect photosynthesis and the aquatic ecosystem by preventing light transmission, and may cause the extinction of many aquatic organisms.[5] Methylene blue (MB) is extensively used in many areas such as paper dyeing, temporary hair dye, cotton, and wool dyeing, and the environmental impact of its discharge can cause health problems such as eye burns and permanent eye injuries in humans and animals.[6,7] It has also been reported to cause vomiting, drowsiness, cyanosis, jaundice, tissue necrosis, increased heart rate, shock, and tetraplegia in humans.[7,8] For this reason, it is extremely critical to remove methylene blue and similar dyestuffs from domestic and industrial wastewater, primarily due to health and other environmental reasons.[9] Complete removal of methylene blue is difficult because it has a stable aromatic structure that consists of chromophores and polar groups, and so many methods have been proposed for effective removal up to date.[10] Adsorption[11-16], flocculation[17], membrane filtration[18], electrolysis, biological processes, oxidation[19,20], precipitation, reverse osmosis[21], and photocatalytic degradation[22] have been used to remove dyes from industrial wastewater. Adsorption, however, has been found to be superior to other strategies in terms of initial cost, ease of design, and efficiency.[23-25] Various adsorbents such as perlite[26,27], bentonite[28], silica gel[29], fly ash[30], lignite[30], peat[31], silica[32], metal ions, and organic substances from aqueous solutions for the removal of dyes have been widely used. Pumice, which is a volcanic rock with low density and porous structure, which is found in many parts of the world and does not require calcination, which is a high-energy pre-stage, has a high specific surface area due to its microporous structure and has a great potential for usage as an adsorbent.[5] Today, natural pumice is gaining popularity with its porous structure and large surface area for the removal of pollutants from wastewater. Since most of the internal pores of pumice, especially the micro and meso pores are not connected to each other, it may exhibit low permeability, and the fact that it is a relatively hard material due to its silica content makes it a potential adsorbent. [33-35]

Although there are many studies on the adsorption of a cationic dye such as methylene blue from an aqueous solution onto many other natural materials, those related to the use of pumice are extremely limited. Therefore, this study aims to analyze the isotherm and kinetic modeling of the adsorption of methylene blue onto pumice as a representative cationic dye sample as well as to elucidate its adsorption mechanism.

2. MATERIALS AND METHODS

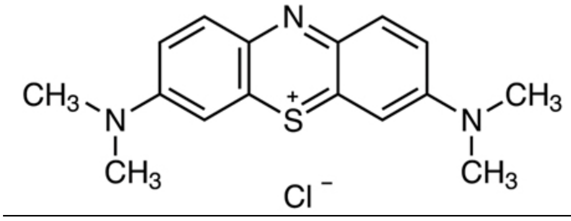
In this study, granulated pumice from BlokBims Co., Çankırı, Turkey was used as an adsorbent and its chemical composition is given in Table 1.

Table 1. Chemical composition of pumice (wt%).

Component	%	Component	%
SiO_2	73.35	TiO_2	0.08
Al_2O_3	12.88	MnO	0.05
CaO	0.77	Cr_2O_3	0.01
MgO	0.08	SrO	0.01
Fe_2O_3	1.10	SO_3	0.44
K_2O	4.40	P_2O_5	0.01
Na_2O	3.82	LOI*	3.88

For use in adsorption experiments, granulated pumice was dried, ground and then sieved using sieves in keeping with ASTM standard, and finally 180-250 µm size fraction was taken and stored. The chemical structure and properties of methylene blue, a cationic dye selected as an adsorbate, are given in Table 2. Cationic dye, methylene blue, analytical purity, and purchased from Merck Co.

Table 2. Chemical structure and properties of methylene blue.

Chemical structure	Chemical formula	CI	λ_{max} (nm	MW (g/mol)
	$C_{16}H_{18}N_3SCl$	52015	666	319.85

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2.1. Adsorption experiments

For the adsorption experiments, methylene blue stock solution at 500 mg/L was prepared and diluted to the desired concentrations. Dye solutions prepared at various concentrations (10-80 mg/L) were taken into 100 mL round bottom flasks and 0.2 g pumice was added to them at a solid/liquid ratio of 0.2 g/100 mL. Afterwards, the balloons placed in the thermostatic shaker were mixed at room temperature (25°C), 150 rpm stirring speed and natural pH (pH:8.0) for different times. The samples taken from the suspensions were centrifuged at 500 rpm for five minutes at the end of the adsorption period in order to separate the phases from one another. The equilibrium concentration (C_e) of methylene blue in the aqueous phase was identified by UV spectrophotometer (Shimadzu 1201 UV-Vis) at 666 nm, the wavelength at which the dye solution gives maximum absorbance.

Equation (1) was used for the calculation of the amount of dye adsorbed (mg/g) by pumice after adsorption.

$$q = \frac{(C_0 - C_e)V}{m} \tag{1}$$

where C_o and C_e are the initial and equilibrium dye concentrations (mg/L), V is the total volume (L), and m is the mass of pumice (g).

3. RESULTS AND DISCUSSION

3.1. Adsorption isotherms

Equilibrium adsorption isotherms can provide very critical information about adsorption efficiency, effectiveness, adsorbent capacity and orientation of adsorbate at the interface, and therefore isotherm analysis results are widely used in the design of adsorption systems. The experimental isotherm drawn using the data obtained in this study is shown in Figure 1.

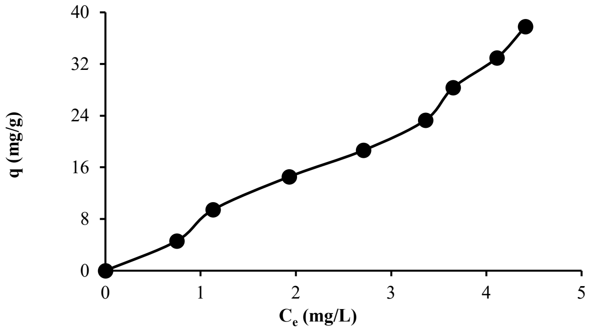

Figure 1. Experimental adsorption isotherm for the adsorption of methylene blue at the pumice aqueous solution interface at 25°C.

It can be detected from Figure 1 that the variation of adsorption with equilibrium dye concentration exhibits high adsorption efficiency at low equilibrium dye concentrations, partially low at medium equilibrium dye concentrations, and quite high at high equilibrium dye concentrations. This behaviour can be explained by the predominance of different adsorption mechanisms. Accordingly, it can be said that adsorption occurs with ion exchange at low equilibrium dye concentrations, ion pairing at medium equilibrium dye concentrations, and pi-pi stacking interactions at high equilibrium dye concentrations.

For modelling experimental equilibrium adsorption data, the data were applied to isotherm models such as Freundlich, Langmuir, Dubinin-Raduskhevic (D-R), BET, Harkins-Jura and Temkin. Mathematical equations of these models and Freundlich's empirical equation are given in Table 3 along with the calculated isotherm parameters and regression coefficients.

Although the adsorption isotherm model of Langmuir is generally suitable in order to describe chemical adsorption, it can be easily extended to explain the behaviour of adsorption systems where physical adsorption occurs through relatively strong interactions. The Langmuir model is based on the assumption that monolayer coating, no lateral interaction between adsorbed species, and that the surface is homogeneous in both energy and morphology.[36]

The Freundlich isotherm is an experimental equation that takes into account that adsorption can be multi layered and that the adsorbent surface can also be heterogeneous both morphologically and energetically.
According to the Temkin isotherm model, heats of adsorption linearly decrease with increasing adsorbent surface coverage up to their maximal binding energies for adsorption, and is characterized by a regular distribution of binding energies.

The Dubinin-Radushkevich isotherm (D-R isotherm) model is often used to estimate adsorption energies, especially in porous adsorbents, taking into account the characteristic porosity of the adsorbent and a heterogeneous surface.

As can be seen from Table 3, the highest fit of the experimental information received from methylene blue's adsorption on pumice at 25°C was obtained with the Freundlich equation. The coefficients related to the calculated adsorbent capacity (k) and adsorbent intensity (n) show that pumice is an effective adsorbent.

In the study of Koochakzadeh et al., the adsorption of crystal violet, a cationic dye, on pumice stone investigated and it was found to be compatible with the Freundlich isotherm. Furthermore, the coefficients related to the adsorbent capacity (k) and adsorbent density (n) were found to be 6.86 and 3.24, respectively.

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It was reported in the study of Akbal (2005), the adsorption experiments indicated that pumice powder was effective in removing basic dyes such as methylene blue and crystal violet from aqueous solution and compatible with the Freundlich isotherm.

Table 3. Linear equations, related parameters and regression coefficients of the investigated adsorption isotherm models for the adsorption of methylene blue on pumice at 25°C.

Isotherm model	Equation	Parameters and R^2	
Langmuir	$\frac{C_e}{q_e} = \frac{1}{q_m.K} + \frac{C_e}{q_m}$	q_m	-188.68
		K	-0.0356
		R^2	0.235
Freundlich	$lnq_e = lnk + nlnC_e$	n	1.139
		k	6.425
		R^2	0.993
Temkin	$q_e = \frac{RT}{b_T} lna_T + \frac{RT}{b_T} lnC_e$	b_T	0.448
		a_T	1.523
		R^2	0.887
BET	$\frac{C_e}{q(1-C_e)} = \frac{1}{q_m k} + \frac{(k-1)C_e}{q_m k}$	q_m	5.178
		k	0.855
		R^2	0.006
Dubinin- Radushkevich	$lnq_e = lnQ_{DR} - K\varepsilon^2$ $\varepsilon = RTln\left[1 + \frac{1}{C_e}\right]$	K	4.23×10^{-7}
		Q_{DR}	28.47
		R^2	0.931
Harkins-Jura	$\frac{1}{q_e^2} = \frac{B}{A} - \left(\frac{1}{A}\right) logC_e$	B	0.642
		A	57.80
		R^2	0.960

3.2. Adsorption kinetics

Information about the rate and mechanism of adsorption can be obtained by examining the adsorption kinetics. For this reason, in this study, for different initial dye concentrations, the variation of the amount of adsorbed dye with the adsorption period was examined, and the findings are given in Figure 2.

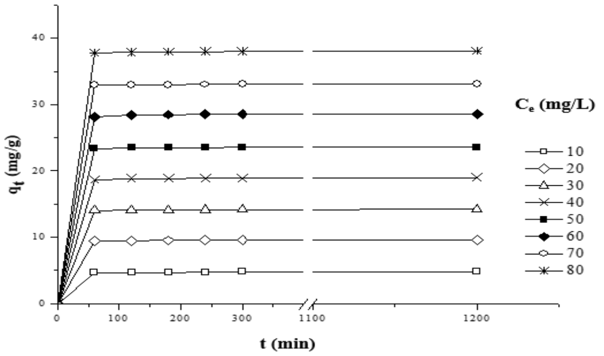

Figure 2. Variation of adsorbed dye amount with adsorption time for different initial concentrations (25°C).

Generally, two different mechanisms are proposed for adsorption on the solid surface, namely fast binding of the adsorbate on the adsorbent surface and relatively slow intraparticle diffusion.

The mathematical equation of the pseudo-first-order kinetic model by Lagergren is as follows:

$$\ln(q_e - q_t) = \ln q_e - k_1 t$$

In this formula, q_t is the quantity of substance adsorbed per gram of adsorbent at any given time t, q_e is the amount of substance adsorbed per gram of adsorbent at equilibrium (mg/g), k_1 is the rate constant (min^{-1}), and t is the adsorption time. The rate constant k_1 is calculated from the slope of the graph of ln (q_e-q_t) against t, and the theoretical q_e value is calculated from the shift of the curve.[39]

The pseudo-second-order kinetic model equation is as follows:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \qquad (3)$$

where, q_t is the amount of substance adsorbed per gram of adsorbent at any time t (mg/g), q_e is the amount of substance adsorbed per gram of adsorbent at equilibrium (mg/g), k_2 is the rate constant (g/mg. min). The rate constant k_2 and theoretical q_e values are calculated from the slope and slip point of the graph of t/q_t against t.[40]
The intra-particle diffusion can be explained in three steps.[41]

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(i) Transport of adsorbate by particle diffusion from the bulk solution to the outer surface or film layer of the adsorbent.

(ii) Transport of the adsorbate from the adsorbent surface to the pores is known as internal diffusion.

(iii) Adsorption on pore surfaces.

The slowest rate-limiting step determines the average adsorption rate. In adsorption systems where intra particle diffusion is the rate limiting phase, the Weber and Morris intra particle diffusion approach is used and the rate constants for intra particle diffusion (k_i) are obtained using the following equation.[42-44]

$$q_t = k_i t^{1/2} + c \tag{4}$$

where k_i and c are the intraparticle diffusion rate constant (mg/gmin$^{1/2}$) and a constant, respectively, and k_i is the slope of the linear portions of the plot of q_t versus $t^{1/2}$.

The kinetic curves plotted at different concentrations at 25°C, considering three different kinetic models, are given in Figure 3, Figure 4, Figure 5 and the calculated model coefficients, together with the regression coefficients, are shown in Table 4.

Table 4 shows that the pseudo-second-order model has highest fit for the experimental adsorption data of methylene blue by pumice. Accordingly, it can be asserted that the dye and the active groups on the pumice surface interacted quite effectively.

For methylene blue adsorption on pumice, Sharafi et al. and Soltanian et al. reported that the kinetic data showed a high fit to the pseudo-second-order kinetic model. In addition, Table 5 provides a concise comparison of results from this study with results from other studies.

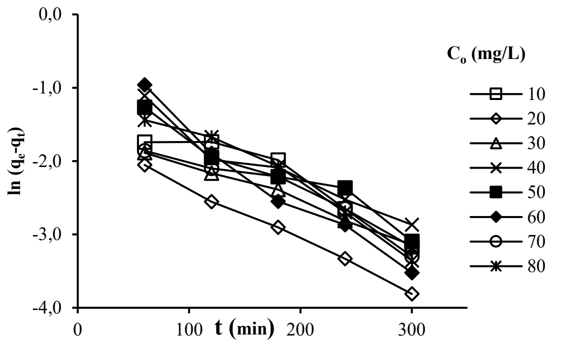

Figure 3. Pseudo-first order kinetic plots for the adsorption of pumice on dye at 25^{0}C.

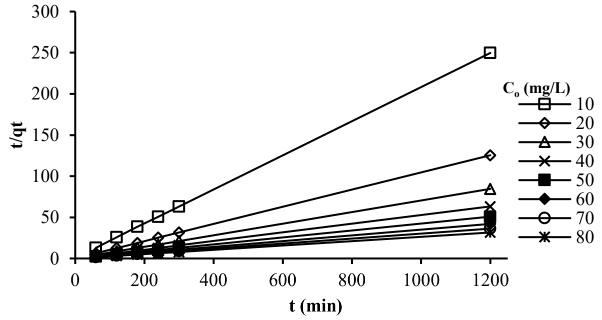

Figure 4. Pseudo-second-order kinetic plots for the adsorption of pumice on dye at 25^{0}C.

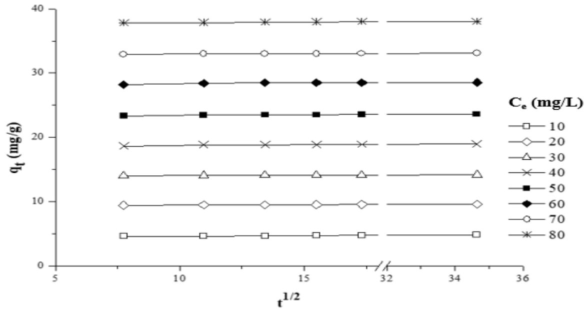

Figure 5. The intraparticle diffusion model plots for the dsorption of pumice on the dye at 25^{0}C.

Table 4. Kinetic parameters and regression coefficients obtained from the application of data on the adsorption of cationic dye on pumice to kinetic models (25^{0}C).

C_i	Pseudo-first order		Pseudo-second-order				Intraparticle diffusion		
mg L^{-1}	k_1 min^{-1} (10^{-3})	R^2	k_2 gmg^{-1}min^{-1}	$q_{e,exp}$ mg g^{-1}	$q_{e,cal}$ mg g^{-1}	R^2	k_i mg min$^{-1/2}$ g^{-1}	C	R^2
10	6.27	0.890	0.0431	5.18	4.81	1.000	0.0013	4.594	0.779
20	7.16	0.997	0.0109	10.43	9.57	1.000	0.0009	9.437	0.993
30	5.27	0.989	0.0050	15.80	14.20	1.000	0.0011	14.023	0.917
40	6.76	0.934	0.0028	21.02	18.98	1.000	0.0023	18.652	0.967
50	6.79	0.943	0.0018	26.36	23.64	1.000	0.0020	23.330	0.980
60	10.16	0.973	0.0012	31.43	28.57	1.000	0.0028	28.209	0.958
70	5.72	0.915	0.0005	26.56	43.44	1.000	0.0011	32.922	0.868
80	8.10	0.964	0.0007	41.98	38.02	1.000	0.0018	37.777	0.938

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Table 5. Comparison of the results obtained from this study with those obtained from other studies.

Adsorbent	**Adsorbate**	**q_{max}(mg/g)**	**Reference**
Chitosan/Fe_3O_4	Methylene Blue	149.20	[46]
Chitosan/ Fe_3O_4/GO	Methylene Blue	180.83	[47]
Modified pumice stone	Methylene Blue	15.87	[5]
Agar/κ-carrageenan	Methylene Blue	242.30	[48]
Modified pumice	Congo Red	23.32	[49]
$CaCO_3$/chitin aerogel	Methylene Blue	266.40	[50]
Activated carbon		53.00	
Fe3O4/bentonite	Methylene Blue	181.82	[51]
Pumice powder	Remazol Red RB	38.90	[52]
Raw pumice	Remazol Black B	5.26	[35]
Pumice	Methylene Blue	2.80	[53]
Pumice-nZVI		4.27	
Mesoporous silica	Methylene Blue	5.58	[54]
Zeolite	Methylene Blue	3.79	[55]
Pumice	Methylene Blue	37.79	**This study**

4. CONCLUSION

The results obtained in this research, in which the adsorption of methylene blue, a cationic dyestuff, on pumice was investigated, are as follows:

- An hour is sufficient to reach the adsorption equilibrium and this is a very short time.

- It was observed that the isotherms reflecting the relationship between the equilibrium dye concentration and the amount of dye adsorbed at constant temperature were partially similar to the Type IV isotherm, that is, methylene blue was adsorbed on pumice in a multilayered manner.

- Although the shape similarity to the Type IV isotherm was observed, the experimental data were found to be strongly in accord with the Freundlich isotherm model.

- It was determined that the experimental data showed a high fit to the pseudo-second-order kinetic model and it was concluded that the interactions between the dye and the active groups were quite effective.

Conflict of interest

I hereby certify that I have no financial or other relationships with any institution, business, or individual.

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