ORIGINAL ARTICLE

Removal of Methylene Blue from Aqueous Solutions by Waste Paper Derived Activated Carbon

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Abstract

Background and Objectives: Activated carbon is an adsorbent which is mostly employed in order to remove dye from aqueous solutions; however, commercial activated carbon is expensive and this is considered as an obstacle for its usage. Therefore, the present study aimed to use waste paper as an inexpensive precursor to produce activated carbon.

Materials and Methods: In the present study, KOH was used for the chemical activation process; in order to prevent the precursor oxidation during the carbonization process, a completely sealed furnace was used. In order to determine the characteristics of the prepared activated carbon texture, N_2 gas adsorption and BET isotherm were used. Moreover, Langmuir, Freundlich, Temkin, and Redlich-Peterson isotherm models as well as Pseudo-first order, Pseudo-second order, and intra-particle diffusion kinetic models were used to determine the characteristics of Methylene Blue adsorption on the prepared adsorbent.

Results: According to the results, S_{BET} and V_{TOTAL} were 66.01m^2/g and 0.063cc/g, respectively. Ferudlich and pseudo-second order models were the best isotherm and kinetic models for prediction of the Methylene Blue adsorption on the produced adsorbent. Furthermore, the q_{max} constant from the Langmuir model was obtained as 68.03 mg/g.

Conclusion: In comparison to other studies, the produced activated carbon in the present study had a desirable surface area and adsorptive capacity for methylene blue adsorption in aquatic environments. Moreover, it seems that preparing activated carbon from waste newspaper is inexpensive, effective, and environment friendly.

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Introduction

One of the most important industrial pollutants, especially in textile industries, is the dyes that even at low concentrations of one parts per million (ppm) are recognizable by naked eyes.[1] The history of using the first industrial dye goes back to 1856.[2] Since then, production and usage of this matter have increased and it is estimated that 7×10^5 tons of this material are produced every year.[3] It is also estimated that about 10 % of the produced dyes are annually entered into the environment, which causes problems for both humans and the wildlife.[2] One of the mostly consumed materials in the dye industry is Methylene Blue (MB) which is used for cotton and silk dyeing.[4] Up to now, a great number of methods have been proposed in order to remove dyes from the industrial waste water, among which adsorption is the most acceptable due to its cost effectiveness and the possibility of usage in large scales.[1] The chemical structure of MB is illustrated in Figure 1.

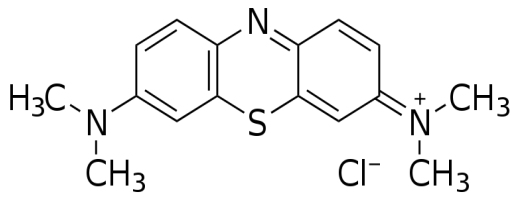

Figure 1: Chemical structure of MB

Activated Carbon (AC) is an adsorbent which is mostly employed for removing dye from aqueous solutions because of its excellent adsorption properties.[5] Therefore, AC is used in the adsorption process in order to remove different pollutants from the wastewater, especially colored contaminants.[2] For many years, petroleum, natural charcoal, and various types of wood were the major precursors for producing AC and since many of these materials are expensive and imported, they cause commercial AC to be costly.[4, 6] Therefore, recently, great efforts have been made to use inexpensive precursors for producing AC. For instance, Haimour et al. produced AC by using date kernels in 2006.[7] Moreover, Hasar succeeded to produce AC from almond husk in 2003.[8] In 2009, Rahman et al. produced AC from rice husks.[9] Also, Amri et al. managed to produce AC from used tires in 2009.[10]

Waste paper is one of the main components of urban waste.[11] In a previous study conducted by Nabizadeh et al. (2008), it was estimated that about 6.43 % of the municipal solid waste in Iran were waste paper and cardboard.[12] Recycling of this valuable material not only eliminates a significant amount of solid wastes, but also it leads to protecting timber resources, saving energy consumption, protecting the environment, and a great number of economic benefits.[11, 13] So far, a large number of studies have been conducted on reusing this valuable resource or converting it to other materials and energies. For example, in 2007, Brand et al. succeeded to prepare cellulose acetate from waste paper through chemical methods. This material can be used in industries, such as cigarettes, and manufacturing semi-permeable membranes.[13] In 2007, Chakravarty et al. produced an adsorbent and removed Zinc from an aqueous solution through chemical activation (Na_2HPO_4).[14] The present study aims to prepare AC from waste paper through KOH and the chemical activation method and use it for removing MB dye from aqueous solutions.

Materials and Methods

Materials and Apparatuses

Stock solution was prepared by dissolving require amount of MB in double distilled water. The test solutions were prepared by diluting stock solution to the desired concentrations. The concentration of MB was determined at 620 nm. The pH measurements were done using pH/Ion meter (Metrohm model-827, Swiss) and adsorption studies were carried out on DR-5000 spectrophotometer (HACH, U.S.A). All chemicals including NaOH, HCl, KOH and MB dye with the highest available purity (98%) were purchased from Merck brand (Germany, Darmstadt). All solutions were stirred on hot plate and stirrer (JENWAY, model-1000, U.K). For carbonization process, seal furnace (ATASH-1200,Tehran-Iran) was used. The Excel software (Microsoft Office Version 2010) was used for calculation of different kinetic and isotherm models.

Preparation of Activated Carbon

Chemical and physical methods were two ways of producing activated carbon, In the chemical method, in the first step, the precursor was impregnated by an appropriate amount of chemical compounds, such as H_3PO_4, $ZnCl_2$, KOH, and NaOH, and in next step carbonized at 450-900 ºC. In the thermal or physical method, on the other hand, first the precursor was carbonized and then, the obtained carbon was activated by water vapor, carbon dioxide, or air at 600-1200 ºC.[2, 15, 16] In the present study, AC was prepared in two steps. In the first stage, 10g of waste paper was ground to about 5mm particles and for the chemical activation process, they were immersed in KOH solution and stirred at 80°C in a hotplate for 2hr. The weight ratio (w/w) in the waste paper, KOH, and distilled water was selected as 1:3:4. The obtained grout was dried at 120°C for 48hr and then the dried sample was completely crushed by a mortar. In the second step; i.e., the carbonization process, the sample was placed in a completely sealed furnace and carbonized at 500°C at a constant heating rate of 17°C/min. After carbonization, the sample was cooled down to room temperature and then washed with distilled water until the pH of the filtered water was stabilized at 7.5. After washing, the obtained AC was dried at 120°C for 24hr and then filtered through the standard 100-mesh sieve. Finally, the produced AC was kept in desiccators until usage.

Textural Characterizations of the Produced Activated Carbon

Textural characteristics of the produced AC were determined by a surface analyzer apparatus (Quantachrome, NOVA 2000) through adsorption-desorption of N_2 at 77 K, which was carried out by the Institute of Nuclear Science and Technology. Moreover, the percentage of the AC production

yield was calculated using Eq. (1).

%activated carbon production yield=(weight of produced activated carbon(g))/(weight of used waste paper(g))×100 (1)

Adsorption Equilibrium Study

For adsorption equilibrium study, 50ml of different concentrations of the dye solution (30-60 mg/l) was agitated with 0.05g of the adsorbent at 100 rpm speed until the equilibrium was achieved. In order to evaluate the trend of MB adsorption by the prepared adsorbent at different times (0, 5, 15, 30, 60, 80,120, 180, 200, and 250 minutes), 4ml of the solution was taken and centrifuged using a special tube at 8000 rpm in order to separate the remaining AC. Afterwards, 2ml of the centrifuged solution was taken and the rate of adsorption was analyzed by the UV-Visible Spectrophotometer (HACH DR-5000) at the wavelength of 620nm. To decreas the experimental error in the equilibrium study, the sample was returned to the system. In order to evaluate the effect of the initial pH on MB adsorption, the equilibrium study was conducted at different pH levels (3, 4, 5.5, 7, 8 and 9). Other equilibrium studies were continued at the best pH where the maximum dye removal had occurred. The pH of the solutions was adjusted by adding 0.1N aqueous solution of NaOH and HCl. When the system reached the equilibrium concentration, the equilibrium adsorption capacity was calculated through Eq. (2).[17]

$$q_e=V(C_0-C_e)/W \quad (2)$$

In this equation, q_e (mg/g) represents the rate of the adsorbed dye per mass unit of the absorbent, C_0 (mg/l) and C_e(mg/l) are initial and equilibrium dye concentrations, respectively, and V (L) and W (g) are the volume of the dye solution and the weight of the adsorbent, respectively.

Batch Adsorption Kinetic Study

Kinetic studies were used to investigate the adsorption rate of adsorbate to adsorbent. It should be noted that the method used in the equilibrium study was applied in the adsorption kinetic study, as well. In fact, in different defined time intervals, samples were taken from the system under the study and the rate of dye concentration was determined. Then, the rate of the adsorbed dye per mass unit of the adsorbent in *t* time was calculated using Eq. (3).

$$q_t=V(C_0-C_t)/W \quad (3)$$

In this study, Pseudo-first order, Pseudo-second order, and intra-particle diffusion kinetic models were used in order to investigate the adsorption rate of MB on the AC produced from waste paper. Each of these models and their linear modes of theme are shown in Table 1.

Adsorption Isotherm

Adsorption isotherms can be used to find out the nature of and the interaction between the adsorbent and the adsorbate.[19] In order to investigate the adsorption isotherm of the produced activated carbon, Langmuir, Ferundlich, Redlich-Peterson, and Temkin isotherm models were used (Table 2).

In the equations presented in Table 2, q_{max} (mg/g) is the maximum adsorption capacity of the adsorbent, K_L (L/mg) is the Langmuir constant, n and K_F (1mg $^{g-1}$(l $mg^{-1})^n$) are the Ferundlich constants and the intensity of adsorption, respectively, Q (mg/g) is the amount of MB dye adsorbed in unit mass of the adsorbent, Q_m (mg/g) is the capacity of the intended adsorbent, k (mol^{-2} kj^2) is the constant related to the adsorbent's adsorption energy, R (8.314 J mol^{-1} K^{-1}) is the universal gas constant, T (K) is the absolute temperature, a_r, K_r, and β are Redlich-Peterson

Table 1: Kinetic models and their linear Forms[6,18]

	Model	Nonlinear form	Linear form	Equation Number
1	Pseudo-first order	$(dq_t)/dt=k_1 (q_e-q_t)$	$\ln (q_e-q_t)=\ln q_e- k_1 t$	(4)
2	Pseudo-second order	$(dq_t)/dt=k_2 (q_e-q_t)^2$	$t/q_t =1/(k_2 q_e^2)+1/q_e t$	(5)
3	Intra-particle diffusion	$q_t=k_o t^{(1/2)}+C$	-	(6)

Table 2: Isotherm models and their linear forms[4, 6, 20, 21]

	Model	Nonlinear form	Linear form	Equation Number
1	Langmuir	$q_e=(q_{max} K_L C_e)/(1+K_L C_e)$	$C_e/q_e =(1/bq_{max})+1/q_{max} C_e$	(7)
2	Freundlich	$q_e=K_F C_e^{(1/n)}$	$\log q_e=\log K_F +(1/n) \log C_e$	(8)
3	Redlich-Peterson	$q_e=(K_r C_e)/(1+a_r C_e^{\beta})$	$\ln (K_r (C_e/q_e)-1)=\beta \ln C_e+\ln a_r$	(9)
4	Temkin	$q_e=RT/b_t \ln (a_t C_e)$	$q_e=A+B \ln C_e$	(10)

constants and A ($(RT/b_t)1na_t$) and B (RT/b_t) are the constants of Temkin isotherm.

R_L is a dimensionless factor which predicts the appropriateness of the adsorption by the constants obtained from Langmuir model. This model is expressed in the form of equation

$$R_L=1/(1+bC_0) \quad (11)$$

Where b (L/mg) is the Langmuir constant. If $R_L>1$, the used adsorbent is not appropriate for adsorption oftheadsorbate. If $R_L=0$, adsorption on the adsorbent will be reverse. If $R_L=1$, the isotherm is of linear type and if $0<R_L<1$, the utilized adsorbent is appropriate.[18, 22, 23]

Error Analysis

In order to determine the most consistent isotherm and kinetic models to the experimental data, Sum of Square Error (SSE%) (Eq.12) and Chi-square test (Eq.13) were used.[24,25]

$$SSE\%=\sqrt{((\sum(q_{(e,exp)}-q_{(e,cal)}))/N)} \quad (12)$$

$$\chi^2=\sum(q_{(e,exp)}-q_{(e,cal)})^2/q_{(e,cal)} \quad (13)$$

Where $q_{e,exp}$ is the values of the measured q_e, $q_{e,cal}$ is the values of the q_e predicted by the intended model, and N is the number of $q_{e,exp}$.

Results and Discussion

Textural Characteristics of the Produced Activated Carbon

AC production yield was estimated as 37% through Eq.(1). In adsorption-desorption of N_2 at 77 K, BET isotherm was the most standard procedure used when characterizing an AC[24] and using this technique, the S_{BET} and V_{TOTAL} were estimated as 66.01m^2/g and 0.063ml/g, respectively. Also, using the t-plot isotherm, the volume of micropores (V_{mic}) was calculated as 0.015cc/g and by subtracting the V_{TOTAL} from the V_{mic}, the mesopores Volume (V_{meso}) was obtained as 0.048ml/g. In the present study, about 76 percent of the total pores volume in the produced AC was related to the V_{meso}; therefore, we can say that the produced AC is appropriate for adsorption of the adsorbate with molecular dimensions of 20nm and smaller.[16]

The Effect of pH on Removal Efficiency

In order to evaluate the effect of pH on MB dye removal, the rate of dye removal was evaluated at different pH levels (3, 4, 5.5, 7, 8 and 9) and the initial concentration of 50 mg/l. Figure 2 depicts the results of pH effect on the removal percentage of MB dye. pH is very important in the adsorption system due to its great impact on changing the

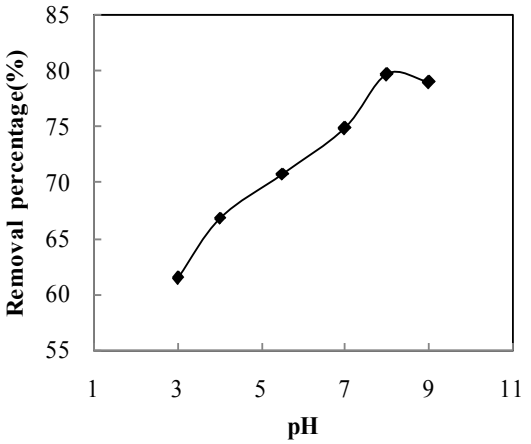

Figure 2: The effect of pH on the removal percentage of MB dye

characteristics of the adsorbate surface as well as changing the electrical charge of the absorbent surface.[20, 26] When pH increases, the concentration of OH^- ions in the desired solution is increased, as well. This causes the surface of the AC to become deprotonated and, as a result, the negative charge of the used AC surface will be amplified. Therefore, the electrostatic attractive force between the MB dye which has positive charge and the adsorbent surface increases and consequently, the rate of dye adsorption increases, as well.[25] As shown in Figure 2, as the pH of the solution increased from 3 to 9, the rate of removal also increased, which is in the same line with the results obtained by Qada et al.[3] Chen et al.[27] and Karagoz et al.[28]

The Effect of Contact Time on the Removal of MB Dye

The mechanism of transferring the adsorbate mass on the adsorbent consists of 3 stages: 1- diffusion of the adsorbate into the liquid layer around the adsorbent, 2- diffusion of the adsorbate into the adsorbent surface, and 3- surface adsorption of the adsorbate on the internal surface of the adsorbent pores. In general, diffusion of the adsorbate on the used adsorbent and ultimately the adsorption phenomena on the adsorbent are time consuming processes.[24] The effect of contact time on the removal of MB dye by the AC produced from waste paper in the desired solution is shown in Figure 3. It shows, at all the concentrations used, as the contact time between the adsorbent and the adsorbate increased, the adsorption rate increased too. According to Figure 3, the highest rate of MB removal took place in the 0-15 minute interval. In the remaining concentrations, this reduction continued up to 160 minutes with a lower slope. From this time up to 250 minutes, the system was almost constant and did not have much adsorption.

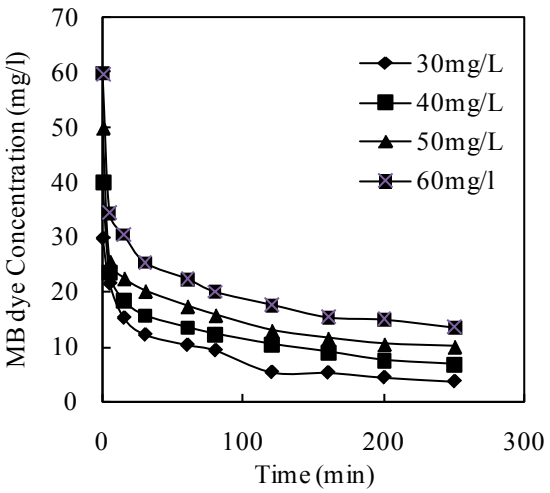

Figure 3: The effect of contact time on the MB dye concentration

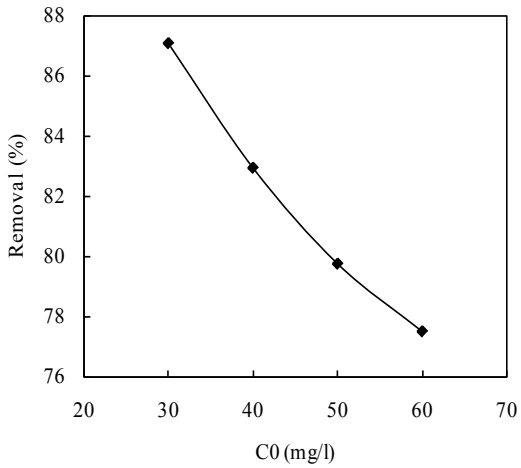

Figure 4: The effect of initial concentration on MB dye removal percentage

The Effect of Initial MB Dye Concentration on Removal Efficiency

The effect of the initial concentration of the MB dye on the adsorption efficiency of the produced AC was evaluated at different concentrations of 30, 40, 50 and 60 mg/l. As Figure 4 reveals, one of the effective factors in the rate of dye uptake was the initial dye concentration of MB. It should be noted that all the experiments were carried out at pH= 8 which was revealed as the most efficient in the test of the best pH. In the present study, with the increase of the initial concentration from 30 mg/l to 60 mg/l, the rate of dye removal was reduced from 87.1% to 77.5%. In addition, as the pollutant concentration increased in the aquatic environment, the number of the available sites on the adsorbent surface decreased.[4] In other words, with the decrease of the pollutant concentration in the aquatic environment, molecules of the adsorbate have more chance to react with the available active sites on AC and, as a result, the adsorption rate is increased. Hence, one can claim that one method to increase the percentage of dye removal is dilution of wastewater.[18]

Adsorption Isotherm of MB Dye by Produced Activated Carbon

In designing the systems which work by adsorption process, obtaining the adsorption isotherm is critical because the isotherm evaluation of the data obtained from equilibrium studies and fitting them to the related models not only predict the adsorbent behaviors in different situations, but they can also determine the maximum capacity of an adsorbent.[3, 18] In the present study, Langmuir, Freundlich, Temkin, and Redlich-Peterson isotherms were utilized in order to analyze the MB adsorption by the prepared AC. The equations and the linear forms of these models are presented in Table 2, and the results are shown in Table 3. Langmuir is the most popular and widely used isotherm model which was presented in 1916. The Langmuir model is valid for monolayer adsorption onto a completely homogenous surface with a finite number of identical sites and negligible interactions between the adsorbate molecules.[6, 21] In the Langmuir equation, q_e (mg/l) is the rate of the adsorbed dye per mass unit of the adsorbent, q_{max} (mg/g) is the maximum adsorption capacity of the adsorbent, C_e (mg/l) is the equilibrium concentration, and K_L (l/mg) is the Langmuir constant. As Table 3 shows, the maximum adsorption capacity of the produced AC (q_{max}) in the Langmuir model was obtained as 68.03 mg/g which is considered as a favorable rate compared to other similar studies (Table 4).[22, 29, 30]

Furthermore, in order to evaluate the appropriateness of the adsorbent used for MB dye removal, R_L constant was calculated.[22] As Table 3 shows, the value of R_L was found between 0-1, which confirmed the appropriateness of the used adsorbent for removing MB.

The Freundlich isotherm model is an experimental model which is used for describing a heterogeneous surface. In fact, this model assumes that the energy adsorbed on the adsorbent is non-homogeneous.[6, 17, 27] In the Freundlich model, n and K_f (1 mg g^{-1}(l mg^{-1}) ^{n}) are the isotherm constant and the intensity of the adsorption, respectively. As Table 3 depicts, MB dye adsorption on the produced AC has a high compliance with the Freundlich model (R^2=0.997). This shows that the surface of the adsorbent is heterogeneous and the adsorption of MB dye on the adsorbent is multilayer.[3, 6] Also, in this study, the value of n coefficient in the Freundlich model was found to be 2.182 which indicated a

Table 3: Isotherm coefficients obtained from isotherm models

Model	Parameter	Value
Langmuir	q_{max}	68.03
	b	0.149
	R^2	0.984
	R_L	0.14
	SSE%	1.116
	X^2	0.143
Freundlich	1/n	0.458
	n	2.182
	K_f	13.93
	R^2	0.9974
	SSE%	0.414
	X^2	0.017
Redlich-Peterson	β	0.617
	a_r	1.078
	K_r	22
	R^2	0.9952
	SSE%	0.908
	X^2	0.127
	A	3.591
Temkin	B	16.03
	R^2	0.982
	SSE%	1.026
	X^2	0.117

high tendency toward adsorbing the MB dye onto the produced adsorbent[17] and this fact had been demonstrated by R_L in advance. The coefficient 1/n in the Freundlich model is a value between 0-1 which represents the adsorption intensity of adsorbate to adsorbent. In the present study, 1/n was revealed as 0.458 which indicated the favorable adsorption of the adsorbate.[18]

Another isotherm model used in this study was Redlich-Peterson which is a combination of Langmuir and Freundlich isotherm models and is applicable to both heterogeneous and homogeneous adsorption systems.[4,6] In the Redlich- Peterson model, a_r, K_r, and β are Redlich-Peterson constants which are determined through the trial and error method. The value of R^2 shows that this model can predict the adsorption behavior of the produced adsorbent with a small rate of error. Also, the low value of β in this model shows that the Freundlich model is more valid for prediction of MB dye adsorption onto AC; thereby, the surface of the produced adsorbent is heterogeneous.[17]

Temkin isotherm is the latest model applied in this study which is based on chemical adsorption.[17] In the Temkin model, R is the universal gas constant, T (K) is the absolute temperature, A and Bare Temkin isotherm constants. The value of R^2 obtained for this model shows that the adsorption of MB dye onto the AC probably has a chemical mechanism.

The Kinetics of Adsorption

Kinetic studies evaluated the details of the process whereby a system moves from an initial state to the final state and the time required for this transition.[31] Therefore, in designing the systems working by surface adsorption mechanisms, determination of kinetics is important.[18] The pseudo-second order kinetic model is shown in Figure 5. As Figure 5 and Table 5 show,

Table 4: Comparison of the capacity of monolayer adsorption in this study and other similar studies

Precursor	Adsorption capacity (mg/g)	S_{BET} (m^2/g)	contact time (min)	Refrence
Hazelnut shell	8.82	783	1440	[29]
Walnut shell	3.53	793	1440	[29]
sunflower oil cake	15.798	114.77	1440	[22]
Apricot stone	4.11	774	1440	[29]
Sewage sludge	114.94	-	40	[30]
Waste paper	68.03	66.01	250	Present study

Table 5: Pseudo-first order, pseudo-second order, and intra-particle diffusion kinetic model coefficients

	Pseudo-first order			Pseudo-second order			Intra-particle diffusion		
Concentration (mg/l)	K_1 (min^{-1})	q_e (mg/g)	R^2	K_2 (g mg^{-1} min^{-1})	q_e (mg/g)	R^2	K_P (mg g min^{-1})	C (mg/g)	R^2
30	0.016	18.08	0.959	0.003	26.32	0.991	1.2	9.182	0.903
40	0.016	19.16	0.936	0.004	32.54	0.995	1.131	16.62	0.946
50	0.018	22.27	0.95	0.004	39.83	0.995	1.169	23.02	0.973
60	0.015	24.92	0.942	0.003	45.9	0.996	1.517	24.6	0.949

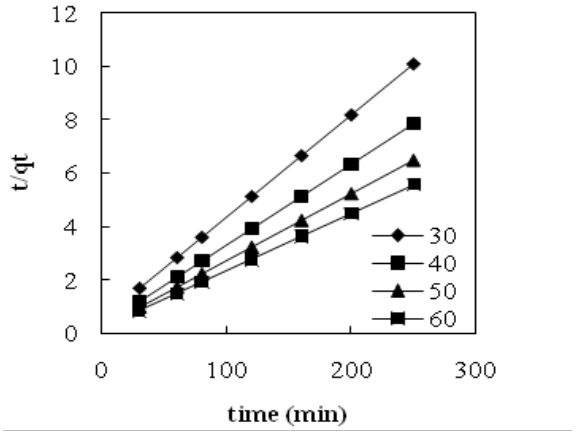

Figure 5: Pseudo-second order kinetic model plot for the MB adsorption on produced AC

pseudo-second order is best kinetic model which can describe the adsorption of MB dye by the produced AC. This finding is in line with the results obtained by Abechi et al.[32] Renugadevi et al.[29] and Altenor et al.[6] who previously studied the kinetic adsorption of MB dye by AC. It should be noted that the low value of R^2 from intra particle diffusion mole indicates that the diffusion of MB onto the produced adsorbent particles cannot be a rate limiting step in the adsorption reaction.[28]

Error Analysis

In this study, to determine the best model for predicting the adsorption behavior and investigate the fitness of different models to the obtained experimental data, the statistical analysis methods like sum of square error (SSE%) and were used in addition to the application of R^2;[24, 25] the results are presented in Table 3 and Figures 6 and 7. Given the values of R^2, SSE% and χ^2 in Tables 3 and 5 and Figures 6 and 7, it can be concluded that the Freundlich isotherm and pseudo-second-order models had the best goodness of fit.

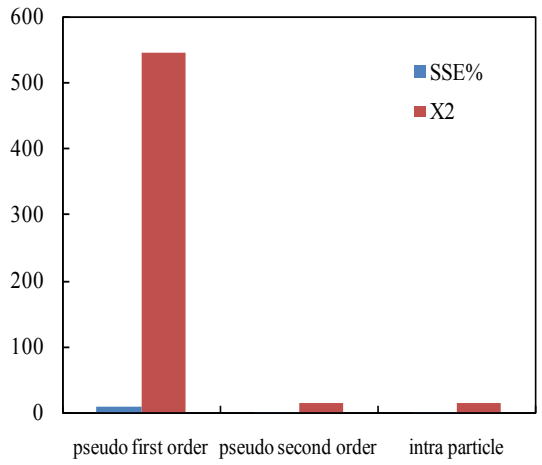

Figure 6: Comparison of different kinetic model precisions

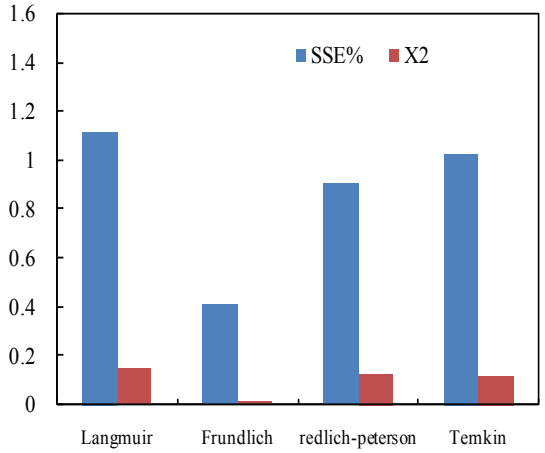

Figure 7: Comparison of different isotherm model precisions

Conclusion

Since waste paper contains a high percentage of hydrocarbon materials, it seems to be a suitable precursor for producing AC. Hence, the present study attempted to produce AC from this cheap and plentiful material and, at the same time, evaluate the ability of the produced AC for removing MB from aquatic environments. Removal of MB is pH dependent and the maximum removal was attained at pH=8. The equilibrium adsorption is practically achieved in 250 minutes. Adsorption equilibrium data follows Langmuir, Freundlich, Temkin and Redlich- Peterson isotherm models. The equilibrium data fitted very well in a Ferundlich isotherm equation. The kinetic study was performed based on pseudo-first order, pseudo-second order and intra-particle diffusion equations. The data indicate that the adsorption kinetics follow the pseudo-second order model. In comparison to other similar studies, the obtained q_{max} from the Langmuir model and S_{BET} from nitrogen adsorption and desorption tests showed that the produce adsorbent had an acceptable capacity for MB adsorption. Therefore, since waste paper constitutes a significant percentage of Iran's municipal solid wastes and according to the results of the present study, the produced AC is considered both useful and affordable.

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Conflict of Interest: None declared

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