



Dye Adsorption on the Blends of Saffron Petals Powder with Activated Carbon: Response Surface Methodology

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ABSTRACT

One of the famous dyes is methylene blue (MB) which is a symbol of hazardous dyes. In this research, methylene blue was adsorbed using the blends of saffron petals with activated carbon. Five independent variables involving pH (2-13), contact time (5-270 min), saffron petals powder dosage (0.1-10 g/l), initial concentration of methylene blue solution (20-300 ppm) and activated carbon dosage (0.1-10 g/l) for methylene blue adsorption were studied. For this purpose, pure saffron petals powder and its blends with activated carbon were used to adsorb methylene blue with various concentrations from water at the above ranges. The Central Composite Design (CCD) under Response Surface Methodology (RSM) was applied to estimate the independent variables effects on the methylene blue adsorption. The optimum conditions for the 96.5% of removal were experimentally found at pH of 10.5, initial methylene blue concentration of 85.9 ppm, saffron petals powder dosage of 7.07 g/l, contact time of 182.05 min and activated carbon dosage of 7.35 g/l while the operating conditions for the maximum removal of 95.3% were experimentally obtained at pH of 10.5, initial methylene blue concentration of 83.1 ppm, saffron petals powder dosage of 7.77 g/l, contact time of 64.7 min and activated carbon dosage of 7.77 g/l. It is concluded that saffron petals powder (as a waste) with activated carbon is able properly adsorb methylene blue.

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1. INTRODUCTION

Dyes are important hazardous substances which can be found in textile, food, pharmaceutical, paper and plastics industries [1]. The colored contamination in water causes several difficulties [2]. Dyes usually have complex aromatic molecules which make them more stable and difficult to biodegrade. Furthermore, a lot of dyes are toxic for some microorganisms and may cause direct destruction or catalytic capabilities inhibition [3]. Adsorption technology using low-cost adsorbents has been considered one of the most efficient and economic none defined methods for the treatment of polluted effluents [4]. One of the most commonly synthesis dyes that used in the textile industry is methylene blue. There are several researches on the MB adsorption by various

adsorbents such as activated carbon [5,6], rice husk [7], peanut hull [8], glass fibers [9], Indian rosewood sawdust [10], neem leaf powder [11], perlite [12], fly ash [13], yellow passion fruit peel [14], chitosan (acrylic acid)/montmorillonite super adsorbent nanocomposite [15], sand [16], silica nano-sheets derived from vermiculite [17], natural phosphate [18] and cyclodextrin polymer [19].

In this research, saffron petals (as waste of saffron plants) which are abundant and economic biomass in Iran were applied as base adsorbent for MB adsorption because saffron petals powder has functional group that useful in adsorption process. Furthermore, they were physically blended with activated carbon to consider the MB adsorption efficiency. Several parameters effect on the adsorption process was statistically and experimentally studied.

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2. MATERIALS AND METHOD

2. 1. Preparation of Adsorbent Saffron petals farmed were obtained from north-east of Iran (Ghaenat, Khorasan Razavi). The petals were then dried and grinded and passed through the standard screens (Tyler standard screen scale) [20]. The particle size of adsorbent was around 70-100 μm which remained on the screens. The saffron petals powder (as base adsorbent) was physically blended with activated carbon powder. The activated carbon powder was in mesopores and its particles diameter was about 15-25 μm that characterized by BIOCHEM (France). The specific surface area of activated carbon was at 700 m^2/g .

The colored wastewater solutions with required concentrations (20-300ppm) were prepared by dissolving methylene blue [supplied by Merck, Germany, with molar mass 319.86 g/mol (anhydrous)] in the distilled water. All of the batch tests were done in a beaker with volume of 250 cm^3 . The pH was set by 0.1 M HCl and 0.1 M NaOH and controlled by pH meter (Model: Lutron 206, Taiwan). The adsorbent with various concentrations was used for different solutions at various times and pHs. After each run, the solid phase was separated by the cellulose nitrate filter [Whatman, England (S&S 5891)]. The MB concentration in all samples before and after test was determined by using UV-Vis spectrometer (Model: SPECORD 250, Czech). A standard solution of MB was made to find maximum absorption. For this purpose, a range of wavelength was also given to UV-Vis spectrometer (400-700 nm). The device showed maximum wavelength (peak) of 664 nm. Then, several solutions with various concentrations of MB were prepared and their absorptions were found at 664 nm. A calibration chart (absorption versus MB concentrations) was provided to find each sample concentration. Furthermore, each adsorbent sample was tested by Infrared Spectrometer Transfer (FT-IR) (Model: Mattson 5000, Canada) to find the adsorbent functional groups before and after adsorption process. When a solid sample is exposed to infrared radiation, sample molecules selectively absorb radiation of specific wavelengths which causes the change of dipole moment of sample molecules.

The Scanning Electron Micrographs (SEM) (model: X-pro, Netherland) was used to see the adsorbent morphology before and after adsorption. It uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample.

2. 2. Methylene Blue Removal from Solutions and Isotherms The methylene blue removal percentage was calculated using some routine equations as mentioned in the literature [21].

According to the isotherm models, Langmuir is based on the monolayer absorption which reversibly happens on the continuous area of adsorbent while Freundlich demonstrates the heterogeneity of adsorbent surfaces [22].

2. 3. Experiments Design and Statistical Analysis

Response surface methodology (RSM) includes a set of useful statistical and mathematical methods for experiments design and mutual effects of parameters consideration on the process. The relation between a response and independent variables is shown by a polynomial equation. The graphical outputs are called levels of response and can be used for the description of unique and mutual effects of variables on the response. The goal of RSM is to optimize the surface of response that is affected by the various parameters of process [23]. Table 1 shows five independent variables in four surfaces including pH, time (min), saffron petals powder dosage (g/l), initial concentration of MB solution (ppm) and activated carbon dosage (g/l). Then, the regression analysis was done to determine the response model coefficients and standard error by applying the Design of Expert software (version: 7) [24]. The process response and five variables can be modeled by a quadratic equation. The quality of the fitness of the model equation is assessed by R^2 and adjusted R^2 . The adjusted R^2 modifies the value of R^2 for the sample amount and the number of expressions in the model.

3. RESULTS AND DISCUSSION

3. 1. FT-IR Analysis In order to recognize the characteristics of the adsorbent functional group, the infrared spectroscopy (FT-IR) was carried out.

TABLE 1. Factors and levels design surfaces for each parameter

| Variable | Surface | | | |
|------------------------------------|-----------|-------|--------|-----------|
| | $-\alpha$ | -1 | +1 | $+\alpha$ |
| pH | 2 | 4.48 | 10.52 | 13 |
| MB concentration (ppm) | 20 | 83.13 | 236.87 | 300 |
| Saffron petals powder dosage (g/l) | 0.1 | 2.33 | 7.77 | 10 |
| Contact time (min) | 5 | 64.74 | 210.26 | 270 |
| Activated carbon dosage (g/l) | 0.1 | 2.33 | 7.77 | 10 |

According to FT-IR tests, the saffron petals powder as base adsorbent contains many active functional groups [25] which are able to adsorb several ionic dyes such as MB. Figures 1(a) and 1(b) show the functional groups of saffron petals powder and the blends of saffron petals powder with activated carbon before adsorption.

As shown in these figures the peaks in the wavelength of 3427 cm^{-1} are related to -OH functional group and the spectrums from the wavelength of 3261 and 3186 cm^{-1} are related to the functional group of -C=C- . The wavelength of 2922 and 2928 cm^{-1} belongs to the functional group of -CH_3 [26]. FT-IR analysis after adsorption shows that there are some changes in the adsorption peaks. Figures 2(a) and 2(b) show functional groups change in the pure saffron petals powder and saffron petals powder blends with activated carbon after MB adsorption. As shown in these figures, the wavelength of 2854 cm^{-1} shows the aldehyde hydrogen bond in FT-IR before adsorption while it is invisible in the FT-IR after adsorption. Moreover, it seems that -OH group to be more active than C=O group in the MB adsorption. The wavelength of 1614 and 1402 cm^{-1} (before adsorption) and the wavelength of 1599 and 1402 cm^{-1} (after adsorption) belong to the benzene rings [26].

3. 2. SEM Analysis SEM analysis is a useful test for the adsorbent surface structure consideration.

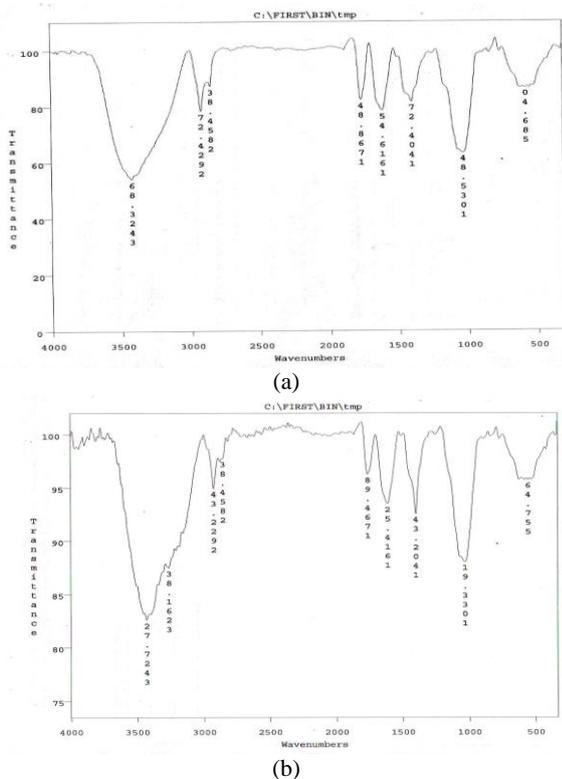


Figure 1. FT-IR spectra of (a) saffron petals powder and (b) blends of saffron petals powder with activated carbon before adsorption

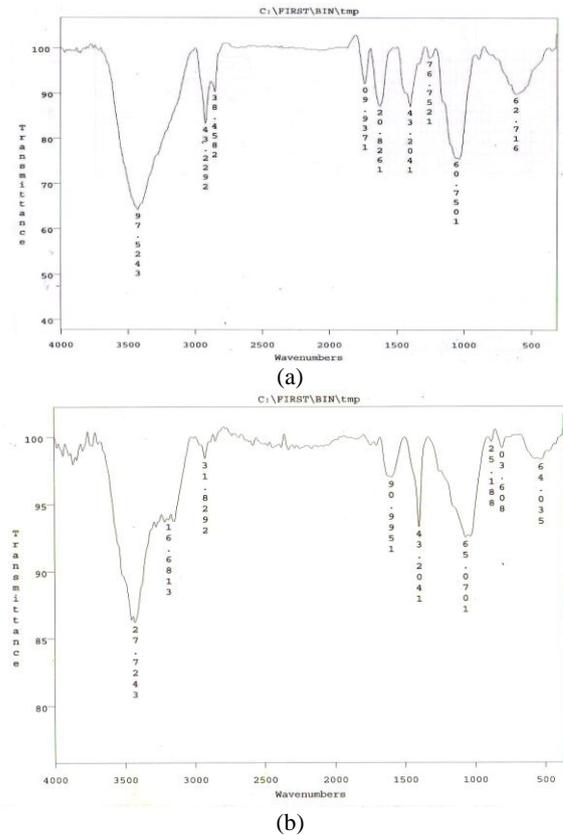


Figure 2. FT-IR spectra of (a) saffron petals powder and (b) blends of saffron petals powder with activated carbon after adsorption

The SEM images of saffron petals powder before adsorption in two forms, without activated carbon and with activated carbon, are shown in Figures 3(a) and 3(b), respectively.

According to these figures, the adsorbent surface is heterogeneous. In fact, adsorption process is a separation one in which adsorbent is concentrated from a bulk vapor or liquid phase on to the surface of a porous solid. A heterogeneous surface provides more available active sites which are more suitable for the adsorption process [26].

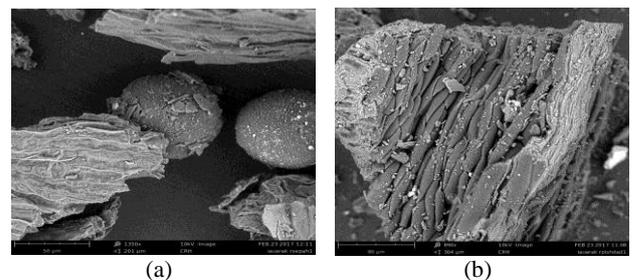


Figure 3. SEM of (a) saffron petals powder and (b) blends of saffron petals powder with activated carbon before adsorption

SEM imaging was prepared to show the morphological structure (in μm) of pure saffron petals powder and its blends with activated carbon [before adsorption process as shown in Figures 3(a) and 3(b)] while Figures 4(a) and 4(b) tried to show the internal structure changes for the both adsorbents after adsorption process.

3. 3. Statistical Analysis In order to optimize five independent variables of the current research, twenty five experiments were designed by the software. The matrix related to the parameters and the residual concentrations of MB as a response are illustrated in Table 2.

$$Y = 17.15 + 10.40X_1 + 0.31X_2 + 3.77X_3 + 0.12X_4 - 13.64X_5 - 0.03X_1X_2 - 0.29X_1X_3 - 0.02X_1X_4 + 0.63X_1X_5 - 0.01X_2X_3 - 6.85X_2X_4 + 0.02X_2X_5 - 0.01X_3X_4 + 0.87X_3X_5 + 0.03X_4X_5 - 0.13X_1^2 - 7.02X_2^2 - 0.14X_3^2 + 1.63X_4^2 - 0.27X_5^2 \quad (1)$$

where, Y is the residual concentration of MB in the solution after adsorption process. X_1 , X_2 , X_3 , X_4 and X_5 are pH, contact time (min), saffron petals powder dosage (g/l), initial concentration of methylene blue in solution (ppm) and activated carbon dosage (g/l), respectively.

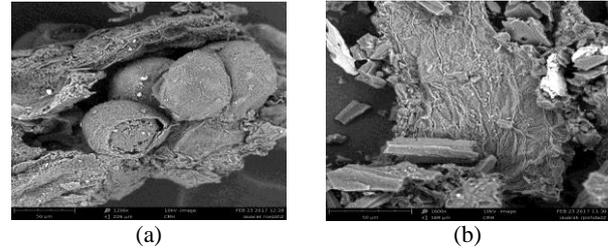


Figure 4 (a) SEM of saffron petals powder after adsorption (b) SEM of blends of saffron petals powder with activated carbon after adsorption

TABLE 2. Central composite design with five variables and the removal percentage of MB by the blends of saffron petals powder with activated carbon

| Trialno. | pH | Initial concentration (ppm) | Saffron petals powder dosage (g/l) | Contact time (min) | Activated carbon dosage (g/l) | Removal (%) |
|----------|-------|-----------------------------|------------------------------------|--------------------|-------------------------------|-------------|
| 1 | 10.52 | 236.87 | 2.33 | 210.26 | 2.33 | 55.06 |
| 2 | 4.48 | 83.13 | 2.33 | 64.74 | 2.33 | 57.36 |
| 3 | 4.48 | 83.13 | 7.77 | 210.26 | 7.77 | 75.63 |
| 4 | 7.50 | 300.00 | 5.05 | 137.50 | 5.05 | 58.23 |
| 5 | 7.50 | 160.00 | 0.10 | 137.50 | 5.05 | 65.31 |
| 6 | 4.48 | 236.87 | 7.77 | 210.26 | 2.33 | 46.87 |
| 7 | 7.50 | 160.00 | 5.05 | 270.00 | 5.05 | 76.52 |
| 8 | 2.00 | 160.00 | 5.05 | 137.50 | 5.05 | 50.12 |
| 9 | 7.50 | 160.00 | 5.05 | 137.50 | 10.00 | 66.32 |
| 10 | 7.50 | 160.00 | 10.00 | 137.50 | 5.05 | 75.89 |
| 11 | 7.50 | 160.00 | 5.05 | 137.50 | 0.10 | 68.43 |
| 12 | 10.52 | 236.87 | 7.77 | 64.74 | 2.33 | 62.34 |
| 13 | 7.50 | 160.00 | 5.05 | 137.50 | 5.05 | 65.19 |
| 14 | 10.52 | 236.87 | 2.33 | 64.74 | 7.77 | 73.62 |
| 15 | 4.48 | 236.87 | 7.77 | 64.74 | 7.77 | 54.67 |
| 16 | 7.50 | 160.00 | 5.05 | 137.50 | 5.05 | 73.05 |
| 17 | 10.52 | 83.13 | 7.77 | 210.26 | 2.33 | 79.29 |
| 18 | 13.00 | 160.00 | 5.05 | 137.50 | 5.05 | 90.28 |
| 19 | 7.50 | 160.00 | 5.05 | 137.50 | 5.05 | 73.73 |
| 20 | 7.50 | 160.00 | 5.05 | 137.50 | 5.05 | 73.19 |
| 21 | 4.48 | 236.87 | 2.33 | 210.26 | 7.77 | 53.76 |
| 22 | 10.52 | 83.13 | 7.77 | 64.74 | 7.77 | 95.31 |
| 23 | 7.50 | 160.00 | 5.05 | 137.50 | 5.05 | 73.81 |
| 24 | 10.52 | 83.13 | 2.33 | 210.26 | 7.77 | 84.09 |
| 25 | 7.50 | 20.00 | 5.05 | 137.50 | 5.05 | 83.48 |
| 26 | 7.50 | 160.00 | 5.05 | 5.00 | 5.05 | 69.12 |

3. 4. Effect of Process Parameters on Adsorption

The effect of solution pH on the MB adsorption was investigated in the pH range of 2-13. As shown in Figure 5, the adsorption sharply increased with increasing the initial concentration of MB and then decreased with pH from 4 to 10.

The low adsorption under low pHs can be explained by the competitive adsorption between the MB molecules and hydrogen ions on the available active sites. Another reason may be due to the carboxyl group presence on the saffron petals powder which can be transformed into the carboxylic acid group at the low pHs. In fact, hydrogen ions increase with pH reduction. It causes particles coagulation which decreases the adsorption process [27].

Figure 6 shows interaction effect of the saffron petals powder dosage and pH on the level of MB adsorption. As shown in this figure, the saffron petals powder dosage increment (from 0.1 to 10 g) gradually decreased the MB residual concentration. In the initial stage, it can be seen that the adsorption rose with increasing the saffron petals powder dosage. This is due to existing large number of active sites. Furthermore, there was slight change in the adsorption percentage with the further increment of saffron petals powder dosage.

Figure 7 shows the effect of pH and contact time on the MB adsorption. As shown in this figure, the adsorption level decreased with increasing the initial concentration.

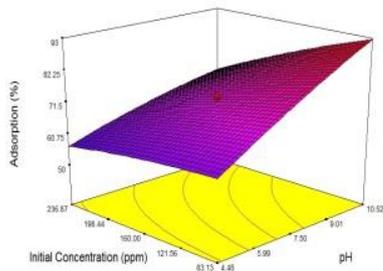


Figure 5. Effect of interaction between pH and initial concentration of MB (ppm) on MB adsorption

This is due to the restriction in the saffron petals powder spots. The rate of adsorption gradually decreased with increasing time. It is due to time consuming the internal diffusion of dye molecules into the saffron petals powder [28]

Figure 8 shows the interaction between pH and activated carbon dosage on the MB adsorption. As shown in this figure, activated carbon addition (up to 5 g/l) to the saffron petals powder improves the properties of saffron petals powder adsorbent while its increment (more than 5 g/l) will block the saffron petals powder pores. It seems that too much addition of activated carbon decreases the adsorption amount due to filling the saffron petals pores which can limit interaction between functional groups of saffron petals powder with MB active heads.

3. 5. Process Optimization

Adsorbent amount optimization is a key point in the process cost reduction and decrement of contamination and sludge production. The optimum conditions for 97% removal of MB by the blends of saffron petals powder with activated carbon were statistically obtained at pH of 10.51, MB initial concentration of 85.90 ppm, saffron petals powder dosage of 7.07 g/l, activated carbon dosage of 7.35 g/l and contact time of 182 min while it experimentally was around 96.5%. This shows good agreement between both data.

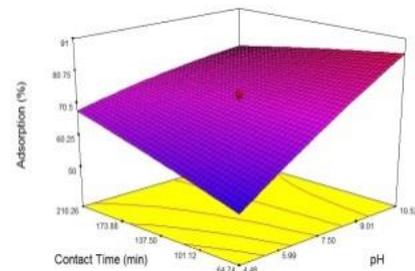


Figure 7. Effect of interaction between pH and contact time (min) on MB adsorption

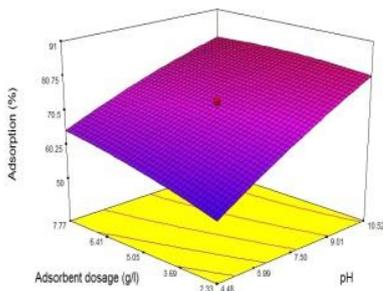


Figure 6. Effect of interaction between pH and saffron petals powder dosage (g/l) on MB adsorption

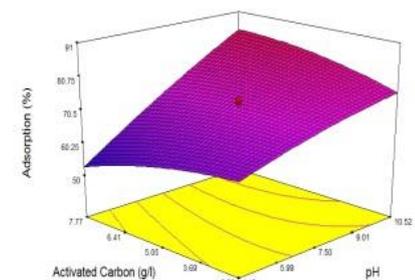


Figure 8. Effect of interaction between pH and activated carbon dosage (g/l) on MB adsorption

According to the literature, MB removal percentages with the other bio-adsorbents such as *Abelmoschus esculentus* seed, *Elaeagnus angustifolia* and *Birnessite*, respectively were at 86, 89 and 92% [29-31] while 96.5% removal of MB was obtained in this research when the blends of saffron petals powder with activated carbon was applied.

4. EQUILIBRIUM MODELING

The isotherm study is an interesting way to explore the structural properties of a material which is chosen as an adsorbent. An isotherm model selection depends on the system type. In this section, the adsorbent capacity in

the optimized conditions was evaluated by the equilibrium tests. The Langmuir model and Freundlich model are the most commonly used models to study the adsorption equilibrium. As shown in Table 3, q_{max} and K_1 (from Langmuir model) are the surface concentration in the monolayer cover and coefficient related to the adsorption energy while K_f and $1/n$ (from Freundlich model) are adsorbent capacity and reaction degree, respectively [32].

Table 3 shows the data of isotherm models parameters in three temperatures of 15, 25, 35 °C. However, two models properly legitimize the adsorption process but Langmuir model is excellently able to model the MB adsorption process on the blends of saffron petals powder with activated carbon.

TABLE 3. Isotherm model constants for MB adsorption on the blends of saffron petals powder with activated carbon

| Temperature (K) | Langmuir | | | Freundlich | | |
|-----------------|------------------|--------------|--------|--------------|--------------|--------|
| | Q_{max} (mg/l) | K_r (l/mg) | R^2 | K_f (mg/l) | $1/n$ (l/mg) | R^2 |
| 288 | 3506.4 | 0.01161 | 0.9929 | 84.86 | 0.6863 | 0.9848 |
| 298 | 3107.5 | 0.00700 | 0.9860 | 56.82 | 0.6714 | 0.9755 |
| 308 | 2991.5 | 0.00566 | 0.9972 | 47.92 | 0.6684 | 0.9914 |

5. CONCLUSIONS

In this study, methylene blue from an aqueous solution was adsorbed by the blends of saffron petals powder with activated carbon. The FT-IR tests showed that the saffron petals powder (as base adsorbent) was containing many active functional groups which are able to adsorb MB. The data analysis showed that the adsorbent capacity was dramatically increased in the blends of saffron petals with activated carbon. The CCD (under RSM) was applied to study the effects of five parameters including pH, contact time, initial concentration of MB, saffron petals powder dosage and activated carbon dosage for MB adsorption from an aqueous solution. Furthermore, Langmuir was excellently able to model the MB adsorption process on the blends of saffron petals powder with activated carbon. It was concluded that saffron petals powder as a biomass can properly modify activated carbon properties in the MB adsorption.

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Methylene Blue

Wastewater

متیلن بلو یکی از رنگهای مشهور که نمونه ای از رنگهای مضر است میباشد. در این تحقیق متیلن بلو با استفاده از مخلوطهای پودر گلبرگهای گیاه زعفران به همراه کربن فعال جذب سطحی شد. پنج متغیر مستقل شامل پی اچ (۱۳-۲)، زمان تماس (۲۷۰-۵ دقیقه)، دوز پودر گلبرگهای گیاه زعفران (۱۰-۰/۱ گرم بر لیتر)، غلظت اولیه محلول متیلن بلو (۳۰۰-۲۰ پی پی ام) و دوز کربن فعال (۱۰-۰/۱ گرم بر لیتر) بمنظور جذب سطحی متیلن بلو مطالعه شد. برای این منظور پودر گلبرگهای گیاه زعفران در حالت خالص و به همراه کربن فعال جهت جذب متیلن بلو با غلظتهای مختلف از آب در بازه های فوق بکار برده شدند. طراحی مرکب مرکزی تحت متد سطح پاسخ جهت پیشبینی اثرات متغیرهای مستقل بر روی جذب متیلن بلو بکار گرفته شد. شرایط بهینه برای حذف ۹۶/۵٪ بطور آزمایشگاهی در پی اچ ۱۰/۵۱، غلظت اولیه متیلن بلو ۸۵/۹ پی پی ام، دوز پودر گلبرگهای گیاه زعفران ۷/۰۷ گرم بر لیتر و زمان تماس ۱۸۲/۰۵ دقیقه و دوز کربن فعال ۷/۳۵ گرم بر لیتر بدست آمد درحالیکه شرایط عملیاتی برای حداکثر حذف ۹۵/۳۱٪ بطور آزمایشگاهی در پی اچ ۱۰/۵۲، غلظت اولیه متیلن بلو ۸۳/۱۳ پی پی ام، دوز پودر گلبرگهای گیاه زعفران ۷/۷۷ گرم بر لیتر و زمان تماس ۶۴/۷۴ دقیقه و دوز کربن فعال ۷/۷۷ گرم بر لیتر بدست آورده شد. نتیجه گیری شد که پودر گلبرگهای گیاه زعفران (بعنوان یک ماده دورریز) به همراه کربن فعال قادر است بنحو شایسته ای متیلن بلو را جذب نماید.

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