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Research Article

Removing Acidic Yellow Dye from Wastewater Using Moringa Peregrina

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Keywords

Moringa Peregrina, Industrial wastewater treatment, RSM experiments, Kinetics models, Isotherm models.

Abstract

The efficacy of Moringa Peregrina seeds, a natural, non-toxic and environmental friendly adsorbent, in the treatment of dyed wastewater is investigated in this research. First, an isothermal model and absorption kinetics are studied. Freundlich isothermal, langmuir isothermal, and pseudo-second-order model are used in explaining the adsorption process Influencing variables like major and minor factors are established, and the amount of dye removal is calculated. Optimization of three important variables; including color concentration, pH and adsorbent dose were performed using central composite method in Design-Expert software. Acidic yellow dye at the concentration of 250 mg/L with pH=8, and adsorbent at a concentration of 0.875 g were purified to 80 % of the aqueous solution. The findings were obtained at the mixing speed of 200 rpm for a period of 60 minutes with 0.93 reliability. The response surface methodology (RSM) findings showed that Moringa Peregrina is effective in removing pigments such as acidic yellow (code19) from aqueous solutions given the specific conditions.

1. Introduction

There are more than 100,000 different forms of dye exceeding one million tons of annual production limit [1]. Millions of liters of colored effluent are produced daily by the dyeing industry and associated factories. Each liter of these colored effluents has indestructible materials that causes environmental problems if not treated before discharge [2].

Removing dyes from sewage in the last decade is one of the most challenging topics for water and wastewater treatment. Colored wastewater produced by various industries, including textile, paper, rubber, and plastics, may cause significant environmental problems in the event of discharge to the environment [3]. Such dyes are human health threats because they are carcinogenic and mutagenic in nature. Besides its apparent reduction in water transparency, turbidity also constitutes a disorder in the ecosystem of the region [4]. Demand of the pure water is increasing and many residents in non-urban areas are preparing a healthy, turbid-free water.

Physical, chemical, biological or compliant methods are typically used to extract color from aqueous solutions. It is vital to minimize the side effects of chemicals before discharging the wastewater in the environment [5]. In general, there are two ways to reduce the effects of pollutants: experimental method and through machine learning [6]. In the biological method, an experimental process, the contamination caused by textile wastewater is removed by the activity of microorganisms [7]. Bacillus and Aeromonas hydrophilia microorganisms in aqueous solutions decreased the concentration of dispersing blue and acidic yellow dyes to less than 1.5 mg / L for 48 hours [8]. The lack of versatility of this approach is a time for the activity of microorganisms [9]. Nevertheless, due to the high cost of disposal of effluent, biological methods have not been

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wholly accepted [10, 11]. Chemical methods include coagulation, flocculation, oxidation, and ozonation, or in the form of a compilation. The drawbacks of these approaches are usually hazardous substance formation, sludge production, and high investment costs [12]. In general, membrane methods (such as nanofiltration and reverse osmosis) and adsorption are common physical techniques. Membrane methods such as filtration systems are not appropriate for removing dye since the chemical composition remains within the microfilter product effluent [13]. Adsorption, unlike membrane methods, is effective in removing sewage colors and is relatively easy to implement and requires a lower design cost [14].

Adsorption is a good alternative approach for the removal of dyes, which is the process of transferring molecules that occurs in the accumulation of two substances in the liquid and solid phases [15]. The adsorbent may then be regenerated or stored in a dry area without any interaction with the environment. Essentially, the process of adsorption is not prone to poisonous compounds; however, the high price of adsorbents limits its use.

A variety of different adsorbents are used to adsorb kinetic dyes. Surface adsorbents are classified into two types: industrial and natural adsorbents. Industrial materials are used to a great degree owing to excessive chemical stability; however, the origin of their production is from non-renewable sources that are destructive to the environment [16]. The most powerful industrial accessible adsorbent, activated carbon can remove wastewater pollution; however, it is not effective at reducing dispersing and vat dyes [15]. Herbal products and agricultural waste are considered inexpensive and environmentally friendly adsorbents [17]. Items such as rice bran and pomegranate peel have a color removal potential of 99 per cent and 55 per cent of the aqueous solution respectively [18]. Therefore, the usage of wastewater treatment plants tends to be rational and more affordable than industrial adsorbents.

There are a wide variety of methods for turbidity to be assessed and minimized. Water turbidity in laboratory processes is now removed by coagulation and adsorption [4]. For centuries villages discover that some plants like Moringa have the power to purify the opaque water. Moringa Oleifera, another branch of the Moringa plant, is a suitable coagulant for removing water turbidity [19]. Besides coagulation, the Moringa plant is also effective in the adsorption of heavy metals [20, 21]. Owing to the structural similarity of Moringa Oleifera and moringa peregrina, it seems that the two functions in the field of wastewater removal are the same but in a different method [22].

Moringa Peregrina has also been observed in hot and humid areas such as Saudi Arabia, India, South Africa, and the southern provinces of Iran (Sistan and Baluchestan and Hormozgan). This plant can be found either at the height of 0 to 300 meters above sea level or higher elevations (1600 to 2200 meters above sea level) [23]. Due to the initial presence of a xerophytic substance's initial presence that helps propagate, the plant is drought resistant [24]. Moringa plant is quite well known globally for its benefits in the medicinal, pharmaceutical, food, and agricultural industries. This plant has a higher nutritional value than Moringa Oliefra, and its low-fat content has created a significant role in healthy diets [25]. Moreover, high doses of antioxidants in plant oil activate the battle against the free radicals in the human body and prevent cancer cells from emerging [26].

Acid yellow 19 (AC) is widely used in textile factories to dye cotton, silk, modified acrylic, and wool fibers. This pigment is called acidic due to the massive presence of sulfonic acid or carboxylic acid [27]. Acid yellow belongs to azo clusters of pigments, which are of potential concern to the environment. In most studies, acid yellow indicates satisfactory performance in research as an absorbent [28]. The Acid yellow chemical structure is shown in Figure 1.

Acid Yellow 19

Figure 1. Chemical structure of an acid yellow dye

Effect of various variables, including initial pH, adsorbent dose, initial dye concentration, and contact time, were studied and the experimental results obtained were correlated with three adsorption isotherm models, namely Langmuir, Freundlich, and BET. The optimization of initial pH, adsorbent dose, and dye concentration, as influential parameters, were carried out via Central Composite Face-Centered RSM experimental design.

2. Method

2.1. Dyestuff

Analytical grade (Merck, Germany) of AC was described with molecular weight 601.35 g/mol and wavelength λmax=234 nm. The AC($C_{20}H_{12}C1_2N_4Na_2O_7S_2$) C.I no is 18967. Dye solutions with concentrations ranging from 100 to 300 mg / L were prepared for the treatment procedure. Assessments with specific pH values were performed by changing this solution to the individual pH using HCl 0.5 M and NaOH 0.5 M.

2.2. Preparation of Adsorbent

Moringa Peregrina (MP) purchased from Fanouj, Sistan and Baluchestan, Iran, was washed, and then the kernels of the seeds were removed. The seeds were sieved in powder and used as an adsorbent. Then, the seed oil was extracted with a Soxhlet extractor machine to minimize the existing oil. After oil extraction, the adsorbent was dried in the oven at 60°C for 24 hours to achieve the lowest moisture content and placed in a closed container for re-use. Scanning electron microscopy (SEM) was used to establish the adsorbent's structural properties. Figure 2 shows the fourier transform infrared (FTIR) spectrum of adsorbents (after oil extraction). The peaks showed a composition of different groups related to adsorption characteristics.

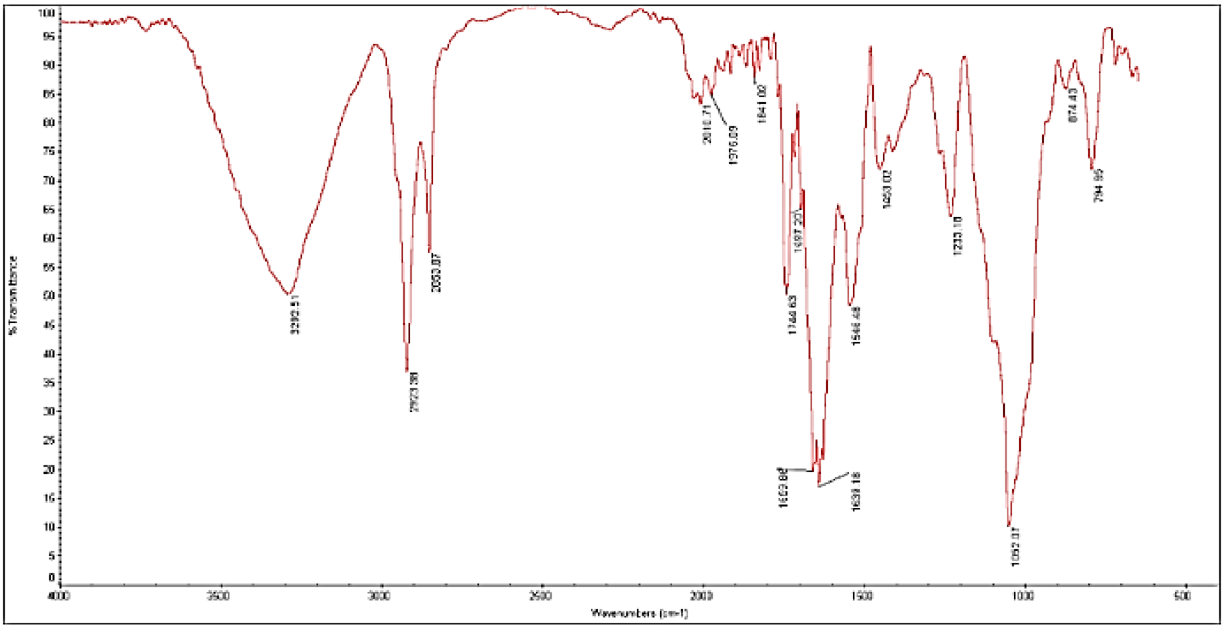

Figure 2. FTIR test of an adsorbent made from Moringa Peregrina

2.3 Batch Studies for RSM Experiment

Primarily, the sorption mixture consisted of pH=8 with a unique adsorbent size (mesh 30-60) was conflated at 200 rpm for 1 hrs. The pH influences study on adsorption was undertaken by adjusting the pH to values in the range 8-12. The dye concentration effect was accomplished with an initial concentration range of 100-300 mg / L and the effect of the adsorbent was varied from 0.5-2 g.

2.4 Equilibrium Studies for Isotherm Model

For determining isotherm and kinetic models, studies were conducted at 25±1°c. Five adsorbent dosages of 0.5, 1, 1.5, 2, and 2.5 g and initial dye concentrations of 100 mg/l were considered. The pH was set to the amount of 8. After mixing, a shaker operated at 200 rpm was used to rotate solutions for 1 hour. The design process was equipped with UV-VIS spectrophotometer (URMIN-1240) setting at a wavelength of 234 nm to calculate the final dye's overall absorption wavelength. After the final concentration was defined using a spectrophotometer, the volume of dye absorbed was computed using the Eq. (1)

$$q_e = \frac{(C_i - C_f).V}{m} \tag{1}$$

where, q_e is the dye absorbed (mg dye/ g adsorbent), V is the total solution volume (mL), c_i, c_f are the initial and final dye concentration(mg/L), and m is the sum of an adsorbent applied on a dry basis(g).

Kinetic tests were carried out by stirring 1 g adsorbent with 100 mg / L of dye solution on a magnetic stirrer at 200 rpm. Samples were investigated for 10, 20, 30, 50, 60, 80, 90, 100, 120, and 180 minutes to ascertain the best contact time and the best kinetic model. The concentration of the dye was ultimately determined. This procedure was performed at constant temperature and pH of 25±1 °C, and 8, respectively.

2.5 Experimental Design for Optimization Process

According to the central composite design (CCD) specification, the optimization of the adsorption capacity was performed by three selected independent control parameters such as initial pH, adsorbent dose, initial dye concentration. The CCD option was designed by factorial experiment with two-star points and five replicates at center points. The quadratic equation model for estimating the optimum point was described according to Eq. (2)

$$Y = \beta_0 + \sum_{i=1}^{K} \beta_i x_i + \sum_{i=1}^{K} \beta_{ii} x_i^2 + \sum_{i=1} \sum_{j=i+1} \beta_{ij} x_i x_j + \varepsilon \tag{2}$$

where, Y is the response, β_0 is the constatnt coeficient , β_i, β_{ii}, β_{iii} are coefficients for linear, quadratic, and interaction effect, x_i, x_j are factors and ε is the error.

The levels and rates of the variables involved in the analysis are presented in Table 1. Thirty-three assays were conducted in parallel, according to the model. Regression and graphic interpretation of the data were made by the Design Expert software (version 10.0.7, Stat Ease, Inc., USA). The variation of the independent variables was demonstrated by the multiple coefficients of determination, R^2. The chosen variable's optimum conditions were achieved by integrating the regression equation and evaluating the surface contour response plots [28].

Table 1. Experimental range and level of independent variables

Factors		Range and levels				
		-2	-1	0	+1	+2
Dye Concentration(mg/L)	A	100	150	200	250	300
pH	B	7	8	9	10	11
Adsorbent dose(g)	C	0.5	0.875	1.25	1.625	2

3. Results and Discussion

3.1. Characteristics of the Adsorbent

The FTIR analysis in Figure 2 depicts: the peak of 1052 cm^{-1} can be referred to as the presence of alkyl halide, which is linked to halogens and typically has a justification for plant photosynthesis. The recorded peak at 1639 cm^{-1} could confirm the c = c bond, and the peak 3292 cm^{-1} can be attributed to free fatty acids in the sample and the presence of this functional group may indicate the ability to exchange ions in the adsorption process. Furthermore, SEM test was carriedout to reveal the surface texture of MP before and after oil extraction as shown in Figure 3. To evaluate the extracted oil of moringa seed, SA Muyibi and the authors stated that the reduction of the water turbidity in the extracted oil sample was higher than the other samples and that it was reported to be 97.9 percent [29]. According to Warhurst, F[30], the more porosity, the more absorption is produced to minimize turbidity. Therefore, more porosity allows permeability and absorption to increase. In Figure 3b, a porous structure with excessive porosity made up of minute aggregated components can be implicated. SEM analysis also shows that the adsorbent has heterogeneous pores on the surface.

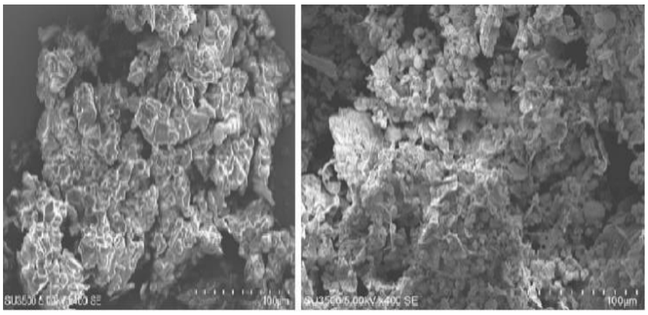

Figure 3 SEM test results a) before (left) and b) after (right) oil extraction in Moringa Peregrina adsorbent

3.2. Adsorption Isotherm Studies

Within the Langmuir model, it is assumed that there is single layer adsorption [31], and Langmuir is not the case for natural adsorbents due to their heterogeneous chemical groups. Whereas Freundlich expresses adsorption in circumstances where the adsorption is multilayer and heterogeneous [32]. Moreover, in the Langmuir model, enthalpy and the energy absorption of molecules is constant. The Langmuir model can be described as Eq. (3)

$$\frac{1}{q_e} = \left(\frac{1}{Q_0}\right) + \left(\frac{1}{KQ_0}\right)\left(\frac{1}{C_e}\right) \tag{3}$$

where C_e is the concentration of adsorbate (mg/l) at equilibrium, q_e is the amount of solute adsorbed at equilibrium (mg/g), constant Q_0 signifies the adsorption capacity (mg/g) and K (l/mg) is related to the energy of adsorption.

The Freundlich isotherm is more commonly used but does not include details about the monolayer adsorption capacity as opposed to the Langmuir model and can be defined as Eq. (4)

$$\log q_e = \log K_F + \frac{1}{n}\log C_e \tag{4}$$

where q_e is the amount of adsorbate at equilibrium (mg/g), C_e the adsorbate concentration in the solution (mg/L) at equilibrium, and K_F and n are constants incorporating all factors affecting the adsorption process such as adsorption capacity. If n is close to 1, the heterogeneity of the surface could be considered less important and, as n reaches 10, the heterogeneity of the surface becomes more substantial. As the K_F value rises, the adsorption capacity of the adsorbent increases for the provided adsorbate. In the brunauer-emmett-teller (BET) model, the possibility of multilayer absorption is investigated. This model is classified as a subcategory based on Langmuir model assumptions. The BET model can be expressed in its linear form as Eq. (5)

$$\frac{C_e}{q(C_s - C_e)} = \left(\frac{1}{BQ_0}\right) + \left(\frac{B-1}{BQ_0}\right)\left(\frac{C_e}{C_s}\right) \tag{5}$$

where B is constantly proportional to the surface interaction's energy, and C_s is the absorbed saturated concentration.

Parameters related to each isotherm have been evaluated using a linear regression model; a correlation coefficient (R^2) values are tabulated in Table 2. The highest correlation of 0.975 was found in case of Freundlich model, while the lowest of 0.91 was found in the case of Langmuir model. The best fit Freudlich isotherm model is given by Eq (6).

$$\ln(q) = 9.2631 - 1.9395\ln(c) \tag{6}$$

Table 2. Regression parameters for three isotherm model

Isotherm model	R^2	Equation
Langmuir	0.91	y=-38.31x+0.92
Freundlich	0.975	y=-1.9395x+9.26
BET	0.96	y=1.37x-0.20

The n-parameter of the Freundlich equation indicates that the adsorption sites have a restricted distribution of energy as their amount was close to 1 (n = 1.939). On this assumption, the information obtained shows that the Freundlich equation can be fitted with a favorable R^2=0.975 with a multilayer capacity of 22.85 mg / g. Aminna A. Attia achieved a value of 75 mg/g for the removal of yellow acidic dye by activated carbon at 25 °C [33]. In contrast, According to a 2017 study based on acid yellow uptake using natural zeolite, an adsorption capacity of 1.17 mg/g has been reported [34]. It implies the superiority of MP in AC removal in comparison to other adsorbents.

3.3. Kinetic Studies

Batch trials were performed to investigate the rate of AC adsorption by MP of 1 g at pH 8.0 and initial dye concentrations of 100 mg/l. Generally, after the contact time there is no significant improvement in the amount of adsorbent and dye concentration. Hence, the equilibrium time for acid yellow, 60 minutes has been estimated. Compared with kinetic models such as pseudo-first order and Elovich, dye abstraction from the aqueous phase by a certain adsorbent is represented by pseudo-first-order kinetics. The coefficient determination of pseudo-second order is 0.99 which indicates the highest R^2 between models; therefore, the model is based on reaction. The Pseudo-Second - Order Kinetic Model is represented as Eq. (7)

$$\frac{t}{q_t} = \frac{1}{k_{2p} q_e^2} + \frac{1}{q_e} t \quad (7)$$

where k_{2P} (g/min mg) is the rate constant of pseudo-second-order model adsorption. If pseudo-second order kinetics is valid, the t / q_t versus t plot will have a linear relationship. Figure 4 displays the exceptional kinetic model and distinguishes the final relationship (Eq. (8)).

$$\frac{t}{q_t} = -59.754 + 2071t \quad (8)$$

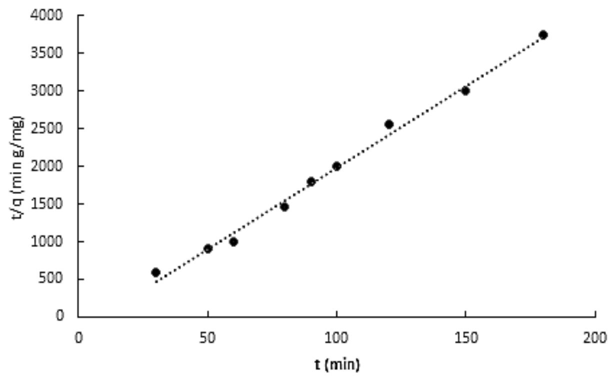

Figure 4. Pseudo-second-order kinetic model for the adsorption of AC by Moringa Peregrina

3.4. Statistical Analysis

Results based on the experimental design at each points are reported in Table 3. Considering that the full quadratic model could not meet the requirements, the model was modified by minimizing additional terms. The following model demonstrates an analytical relationship between the removal parameter and the independent variables:

$$remove^{1.49} = 0.31 + 0.014A - 0.084B + 0.037C - 0.076AC + 0.063BC + 0.1A^2 - 3.822E + 0.11C^2 \quad (9)$$

Table 3. Experimental design and results for dye removal

Trial number	Coded values of the variables			Dye removal (%)
	Dye concentration (A)	pH (B)	Adsorbent dose (C)	(R)
1	+1	- 1	- 1	78
2	- 1	+1	- 1	75
3	0	0	+2	59
4	0	0	- 2	81
5	- 1	- 1	+1	79
6	0	0	0	44
7	- 2	0	0	85
8	- 1	+1	+1	77
9	+1	+1	+1	64
10	0	0	0	50
11	+1	+1	- 1	56
12	- 1	- 1	- 1	70
13	0	0	0	71
14	- 2	0	0	71
15	- 1	+1	+1	66
16	+1	- 1	+1	71
17	+1	- 1	- 1	10
18	- 1	+1	- 1	40
19	0	- 2	0	66
20	+2	0	0	46
21	0	0	0	2
22	0	0	- 2	77
23	0	+2	0	79
24	- 1	- 1	- 1	59
25	+1	+1	+1	77
26	+2	0	0	81
27	+1	+1	- 1	60
28	0	0	0	62
29	0	- 2	0	60
30	- 1	- 1	+1	67
31	0	0	+2	50
32	0	+2	0	19
33	+1	- 1	+1	55

The analysis of the response variance is summarized in Table 4. To verify the model's perfection, the coefficient of variation (the ratio of the standard error of estimation to the mean value expressed as a percentage) and the F-value tests were also carried out. The coefficient of determination (R^2= 0.8746) was moderately strong, as proven by 87.46% of the response's total variance. In an overall estimate, the proposed model is valid and significant if “Prob>F” is less than 0.05. Also, based on being the non-significant value of lack of fit, the conclusion is that the model is sufficiently descriptive of the data to eliminate dye. The validity of the parameter's coefficients and the associated standard error of any Equation term is set out in Table 5. According to p values (< 0.05 is meaningful), it can be inferred that all the main second-order effects (A^2 and C^2) are incredibly significant. Furthermore, the first-order parameters' negative coefficient indicates the maximum response value within the specified parameters ranges. Other factors, such as AB and B^2, have a negligible effect on the removal of AC owing to p values of more than 0.05.

Table 4. Analysis of variance for the quadratic model for dye removal

	Sum of square	DF[a]	Mean square	F-value	Prob.>*F*
Model	0.58	1	0.082	14.94	<0.0001
Residual	0.083	15	5.506E-003		
Lack of fit	0.010	6	1.745E-003	0.22	0.9614
Pure error	0.072	9	8.013E-003		
Total	0.66	22			

R^2=0.8746, CV[b]=14.06%.
[a]DF= degree of freedom
[b]CV= coefficient of variation

Table 5. The significance of components in the modified model

Factor (coded)	Coefficient estimate	Standard error (SE)	*p* value
A	0.014	0.016	0.0376
B	-0.084	0.015	<0.0001
C	0.037	0.019	0.0774
AC	-0.076	0.021	0.0025
BC	0.063	0.021	0.0094
A^2	0.10	0.014	<0.0001
C^2	0.12	0.018	<0.0001

3.5. Optimization of Adsorption Process

To obtain a better perception of the AC adsorption process, a contour plots were studied. Curved contour lines represent that there is an interaction between initial concentration, dose adsorbent, and pH. In each graph, the influence of two factors on the adsorption potential was examined while the other variable remained at the optimum value. The response surface of adsorption is shown in Figure 5 and 6. Figure 5 demonstrates the combined influence of the initial dye concentration and pH on the AC adsorption efficiency. In solutions with a dye concentration of about 200 to 250 mg/L and a pH of about 8-9, the best dye removal mode occurs. Besides, the AC adsorption capacity decreases in constant initial concentration with pH augmentation. The zero charge point (pzc) is commonly defined as the pH at which the total particle adsorbent surface's net charge is equal to zero [35]. It seems that pH_{pzc} is equal to 8, and the percentage of dye removal is higher at high pH since the adsorbent surface has a positive electrical charge and is appropriate for acidic yellow, which is considered ionic dye. In the acidic zone, reaction between H+ ion and acidic dyes leads to a decrease in the active sites' sorbet surface area due to dyes. Other AC adsorption research also reported that an increase in pH could increase in adsorption capacity [36]. The reason for this phenomenon is the separation of the OH^- bond and the uptake of oxygen by functional groups of acidic dye. In the case of specific and constant pH of 8, an increase in initial concentration causes to increase the dye removal efficiency up to 75%.

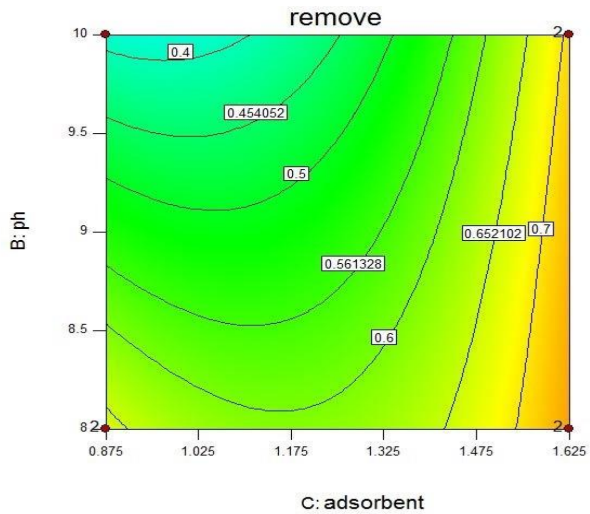

Figure 5. Response surface contour plot indicating the effect of interaction between dose adsorbent and pH on dye removal while holding initial concentration, 250 mg/L

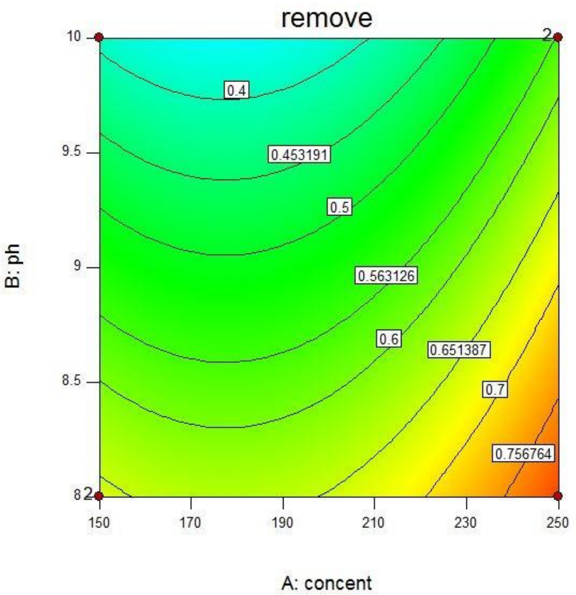

Figure 6. Response surface contour plot indicating the effect of interaction between dye concentration and pH on dye removal while holding initial concentration, 0.875 g

It can be noticed that the impact of the interaction between pH and the adsorbent dose is also significant. Figure 6 shows that relatively higher dye concentration and a very low pH contributes to higher dye removal at constant adsorbent dosage. The highest removal efficiency is about 70%. This process is initially performed very quickly, and the majority of the dye is eliminated by the adsorbent in the first few minutes. Observation can be explained in terms of the amount of MP molecules existing in the solution. The explanation for this phenomenon is to fill active adsorption sites by increasing dye molecules [37].

In addition to Figure 5 and Figure 6, Figure 7 can be considered to define the final relationship at constant pH (=8). In Figure 7, simultaneous factors such as dye concentration and adsorbent dose are displayed in a 3D graph to estimate the dye removal efficiency. Dye removal efficiency increases with increasing adsorbent content and decreasing dye concentration, and these two variables are inversely related to reducing dye removal. This is likely due to the increased activity of the surface sites, mobility of dye molecules; and the change in adsorbent pores size [38]. However, between 0.875g to 1.175 g of adsorbent, dye removal efficiency has a reverse result. The reason behind this is the accumulation of absorbent molecules and the formation of the overwhelming adsorption surface [39].

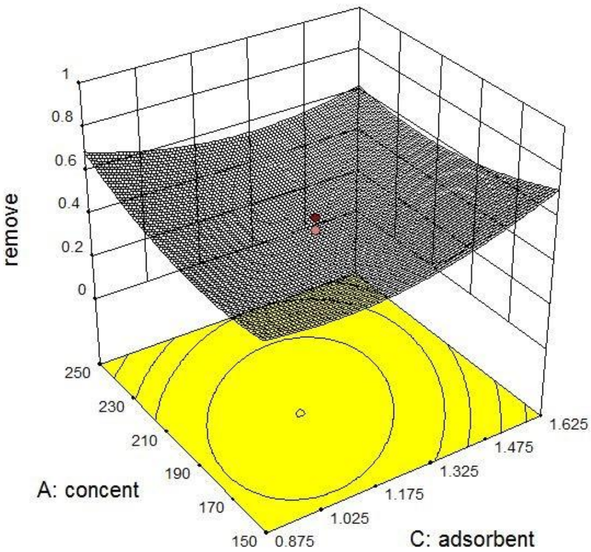

Figure 7. Effect of the initial dye concentration and adsorbent dosage on dye removal of AC (pH=8).

The design was given a set of solutions (Table 6) to achieve the operation's optimum conditions. Based on the solution provided by the design, four experiments were carried out under fixed conditions. It was observed that the maximum removal dye or adsorption of AC of 80% was obtained when 250 mg/L of initial dye concentration and 0.875 g of MP were used, and the optimum pH value was found to be 8.

Table 6. Optimum conditions defined by the Design Expert for the adsorption process

Run	A	B	C	Removal efficiency (%)	Desirability
1	250.00	8.00	0.875	80.5	0.93
2	250.00	8.00	0.878	80.3	0.92
3	250.00	8.01	0.878	80.1	0.92
4	248.98	8.00	0.875	80.0	0.92

4. Conclusion

The research focused on the acidic yellow dye adsorption by Moringa Peregrina (MP) from the aqueous solution. MP adsorption was studied in batch mode and was highly reliant on the solution's pH value, the adsorbent dosage, and the initial dye concentration. The most significant results of this study are summarized as follows:

- Freundlich isothermal model showed better equilibrium data than the Langmuir isotherm.
- The pseudo-second-order model adequately represented the adsorption kinetics.
- Optimum conditions were identified as an initial pH of 8, an initial dye concentration of 250 mg/L, an adsorbent dose of 0.875 g, and the maximum removal efficiency was found to be 80 %.
- The RSM technique's findings are based on evaluation results that showed: RSM (R^2= 0.875) is a valid and effective method for predicting adsorption.
- Moringa Peregrina is considered a natural and inexpensive adsorbent and is efficient in eliminating dye materials from aqueous solutions.

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