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Optimization of Remazol Brilliant Blue R Adsorption onto Xanthium Italicum using the Response Surface Method

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Abstract

In this study, the adsorption capacity of toxic and recalcitrant organic pollutant(s) Remazol Brilliant Blue R (RBBR) onto the invader Xanthium İtalicum (XI) plant was assessed using the Response Surface Method (RSM). The adsorp-tion behavior of RBBR was determined by the spectrophotometric method, FTIR and SEM analysis were used to characterize XI, Brunauer, the Emmett and Teller (BET) analysis was accomplished to find the surface area of the ad-sorbent. The experiment procedures were planned by the Central Composite Design (CCD) whereby a model was built to examine the effects of the different parameters and the possible interaction between them. The correlation factor (R2) for the developed model was 0.985, suggesting that the model could achieve 98.5% of RBBR removal through an adsorption process. The desirability of 0.992 can be attained using the identified optimum conditions: XI = 3g, pH = 9, Time = 84min, RBBR = 50ppm. Under these conditions, the adsorption capacity reached 1.59 mg.g-1, and the removal percentage reached 95%. Freundlich isotherm had the best-fitted values with R2 of 0.999. Furthermore, the adsorption of RBBR onto XI was found to be pseudo-second-order kinetic with R2=0.999. Also, the recovery of RBBR from XI was studied through desorption experiments in presents of 0.5M NaOH. The recovery percentage after 120 min reached 47%. The maximum recovery percentage reached 73% at 24h.

Keywords

Xanthium İtalicum, Remazol Brilliant Blue R, Adsorption, Response Surface Method, bio adsorbent

1. Introduction

Dye sources can be natural or synthetic. From an environmental view, natural dyes are more favorable [1]. Chemical dyes can cause serious problems not only to human health, but to the whole ecosystem [2]. Synthetic dye production increased as the demand for textile industries increased. Indeed, the textile industry produces a large number of dyes through different processes [3].

In order to remove dyes from waste-water, conventional treatment methods such as biological treatment processes [4], coagulation and flocculation [3], as well as non-conventional treatments such as advanced oxidation techniques [5,6,7,8,9], membrane filtration [10], adsorption [11] and Nano-technique [12,13,14,15] are utilized.

Remazol Brilliant Blue R (RBBR) belongs to toxic and recalcitrant organic pollutants [16]. It is often used in the production of polymeric colorants as a starting material in the textile industry [17]. Techniques such as adsorption on agro-industrial waste materials (wheat bran) [18], Fenton oxidation [19], ozonization [20], hydrolysis [21], electrochemical [22], and bio-sorption on *Candida sp*. [23] are adopted in order to remove RBBR.

Dye removal by adsorption techniques is used extensively because of its simplicity and flexibility [24]. Moreover, it does not generate sludge or any harmful by-product [25]. In t recent years, the adsorption process has also been used for dealing with toxic and radioactive ion metals [26]. Activated carbon is the most known adsorbent material, and is used world-wide. Problems of high cost accompanied by mass demand led to use of low-cost adsorbent materials. Bio-adsorbents [27] and nanoparticle adsorbent [28] were investigated.

According to related literature, there are numerous parameters involved in the adsorption process [29]. Thus, optimization and modeling processes have been examined to attain maximum efficiency. Unfortunately, traditional methods deal with one parameter at a time. As a result of this constraint, the optimization process is inefficient both related to cost and time [30]. The collection of statistical and mathematical techniques are called Response surface methodology (RSM). These are used to fit the experimental model with an empirical model [31,32] to solve a problem.

Xanthium Italicum is one of the invader plants which is found in the farms near Mersin. The farmers don't favor this type of plants and work to get rid of it. They try to treat it by physical and chemical methods.

This study aims to provide an environment-friendly sustainable solution at minimum costs using invader plant (Xanthium İtalicum) as an adsorbent material to remove RBBR dye. A mathematical model has been developed by RSM Central Composite Design (CCD) to determine the effect of four parameters: initial pH, dye concentration, time and adsorbent mass on the efficiency of adsorption of RBBR onto *Xanthium İtalicum* (*XI*).

2. Experimental design

2.1. Adsorbent

In this study, the *Xanthium İtalicum* (*XI*) plant was used as an adsorbent. The plant was collected from farmlands near Mersin city, Turkey. It was cleaned by distilled water, then dried for 24 h by placing it in an oven at 105 °C. The plant was finely grounded using a blender and sieved at 35 mesh. The passing materials were used as adsorbents without further treatment. Fig. 1 shows the preparation process for using *XI* plants as adsorbents.

2.2. Characterization of adsorbents

Measurement of the weight of adsorbents and dyes was carried out with the precision scale (Uhaus Corp Adventure). The magnetic stirrer (Edmünd Bühler GmbH) was used to mix the solutions; the pH adjusted by a pH meter (Probe Cond HQ40D); the UV−Vis spectrophotometer T 90 was used to read the dye concentrations; the Fourier transform infrared spectrophotometer was used to study the Fourier transform infrared spectra of *XI*; the scanning electron microscope (SEM) (Gemini Zeiss Supra 55) was used to explore the surface morphology of the *XI*; and the content of the metal was obtained from literature [33]. The surface area of the adsorbent was determined by the method of BET analysis.

2.3. Dye preparation

Remazol Brilliant Blue R (RBBR) is an Anthraquinonoid synthetic dye with the chemical formula

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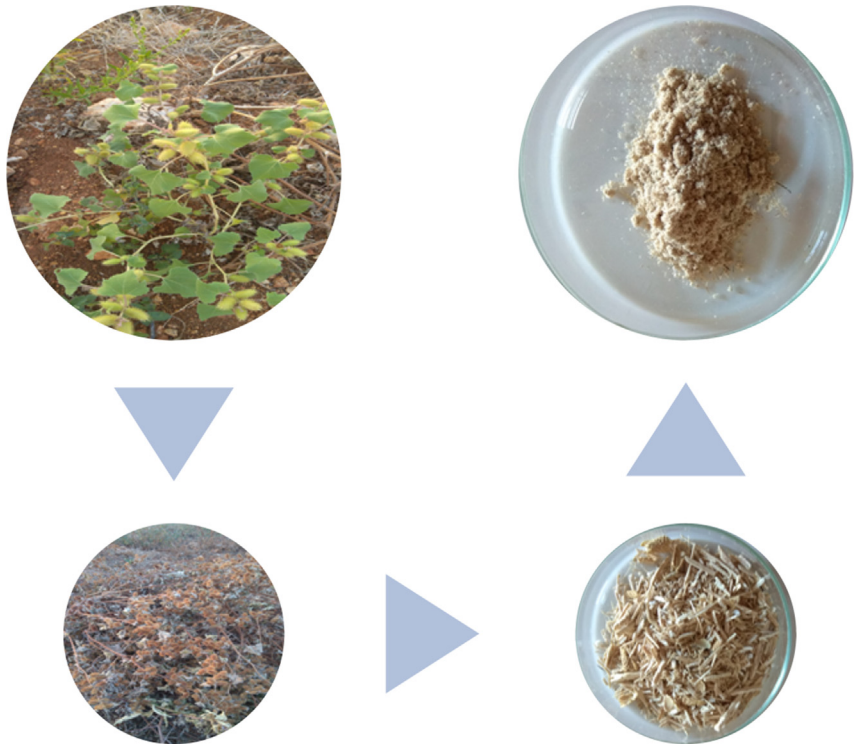

Fig. 1. *XI* preparation.

$C_{22}H_{16}N_2Na_2O_{11}S_3$, and a molecular weight of 626.54 g mol^{-1}. A stock solution with 2000 ppm was prepared of which target concentrations were prepared. pH values were controlled by HCl and NaOH. The results were obtained at a wavelength of 582 nm. Fig. 2 shows the chemical structure of the RBBR dye.

2.4. Batch analysis

The adsorption process is affected by the following factors: contact time, pH, adsorbent mass, and initial dye concentration [34]. Exploratory experiments were undertaken using batch studies to investigate these effects. Different dye concentrations, pH values and adsorbent masses were explored at different contact times. All experiments were carried out at 150 rpm. At the end of this experiment, the ranges of the factors were identified. The adsorption percentage (%A) and the amount of adsorbed dye (q_e) were calculated using Eqs. (1) and (2) respectively [35]:

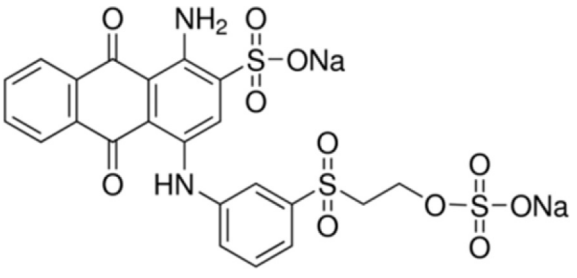

Fig. 2. Chemical structure of RBBR dye.

$$\%A = (C_i - C_e)/C_i * 100\% \quad (1)$$

$$q_e = (C_i - C_e) \times V/m \quad (2)$$

where C_i is RBBR initial concentration (mg L^{-1}); C_e is the final RBBR concentration (mg L^{-1}); q_e is the solid phase concentration (mg g^{-1}); V is solution volume in (L); and m is the *XI* mass (g).

The adsorption isotherm was identified using Freundlich and Langmuir isotherm equations as shown in Eqs. (3) and (4) respectively [36,37].

$$Log(q_e) = Log(k_L) + (1/n) \times Log(C_e) \quad (3)$$

$$C_e/q_e = a_L C_e/K_L + 1/K_L \quad (4)$$

where K_L is the energy of the adsorption (L mg^{-1}); n is the factor of heterogeneity; and a_l is the Langmuir constants ($L.mg^{-1}$). To select the optimum isotherm, the correlation coefficients (R^2) for both were compared

and the isotherm with the maximum R^2 value was selected.

Adsorption process thermodynamics is a critical issue. It impacts the process feasibility, spontaneity and randomness [1]. In this regard, different kinetic models were tested to ascertain the adsorbent–adsorbate process. In this study, the adsorption rate was tested based on the adsorbent capacity using Lagergren's Pseudo-first order [38], and Pseudo-second order kinetic models [39] as shown in Eqs. (5) and (6) respectively.

$$Log(q_e - q_t) = \log(q_e) - (k_1/2.303) \times t \quad (5)$$

$$t/q_t = 1/(k_2.q_e{}^2) + (1/q_e) \times t \quad (6)$$

where q_e and q_t are solid phase concentration (mg g^{-1}) at equilibrium and at time t; k_1 (min^{-1}) is the Lagergren's first order rate constant; and k_2 (gm g^{-1} min^{-1}) is the second order adsorption rate constant.

Desorption experiments were conducted at optimum adsorption condition. The experiments were performed by shaking the exhausted adsorbent in 0.5 M of NaOH and 0.5 M HCl at the same manner of adsorption. The recovery percentage was calculated through Eq. (7):

$$\%\ \text{Recovery} = \text{Amount of dye desorbed}/\ \text{Amount of dye adsorbed} * 100\% \quad (7)$$

2.5. Response surface method

The performance of RBBR removal by XI was modeled using the Response Surface Method (RSM). Four independent factors were represented using a Central Composite Design (CCD), which is quite adequate for up to five determinants [40]. These are: pH, *XI*, time and RBBR concentrations. The number of experiments necessary to complete the modeling process were calculated using Eq. (8) [41].

$$N = 2^n + 2n + Cp \quad (8)$$

where N is the experiment runs; n is the independent factors number; and *Cp* is the replicates of center point's numbers.

Independent factor's ranges were entered into the established model. The ranges of the independent variables are shown in Table 1. Accordingly, a sequence of experiments were undertaken (a total of 30 experiments were prepared), and the results of these experiments were entered again into the model. The experimental runs and responses are shown in Appendix A. Finally, a second order quadratic empirical model was developed to correlate RRBR removal as given in Eq. (9).

Table 1
Variables and level considered in CCD.

Variables	Unit	Factors	Low	High
pH	–	A	5	9
XI mass	G	B	3	6
Time	Min	C	45	125
RBBR concentration	mg L^{-1}	D	20	50

$$\boldsymbol{Y} = \boldsymbol{\beta}_\circ + \sum_{i=1}^{k} \boldsymbol{\beta}_i \boldsymbol{X}_i \sum_{i=1}^{k} \boldsymbol{B}_{ii} \boldsymbol{X}_i^2 + \sum_{i=1}^{k} \sum_{j=1}^{k} \boldsymbol{B}_{ij} \boldsymbol{X}_i \boldsymbol{X}_j + \boldsymbol{\epsilon} \quad (9)$$

where β_o is the offset term; β_i and β_{ii} are the linear and quadratic effects of input factor X_i; β_{ij} is the linear effect between the independent factor X_i and X_j; and ϵ is the error [42]. The experimental data was analyzed and fitted by RSM using Design Expert 11 software.

3. Results and discussion

3.1. Adsorbent characteristics

The chemical compositions of the XI plant were obtained from literature. The following elements were present on the XI structure: Na, K, Ca, Mg in addition to Cr, Ni, Pb, Cu, Fe, Mn, Zn, Cd, Co, Se, Al and As [33,43]. In this study, the analyses of FTIR and SEM were undertaken before and after the adsorption process. The surface morphology of XI was relatively rough and porous as shown in Fig. 3a. The SEM showed that the pores were filled with the dye after the adsorption process (Fig. 3b).

In this study, the analysis of FTIR was undertaken before and after the adsorption process. The peak, which could be corresponded to the functional group N–H bonding, shifted from 1600 cm^{-1} before adsorption to 1614 cm^{-1} after adsorption.

The functional group S=O stretching was detected at the peaks of 1418 and 1030 cm^{-1}. As discussed in previous studies, the change and shift in peak frequency confirmed the role of the functional group in the adsorbent–adsorbate relationship [44,45]. Fig. 4 shows FTIR spectra.

The adsorbent (*XI*) surface area was measured by the BET method. The measured surface area was 3.55 m^2/g for 500 mm adsorbent particle. This value is larger than those obtained for previous bio sorbents (Sargassum Tenerrimum powder) with a value of

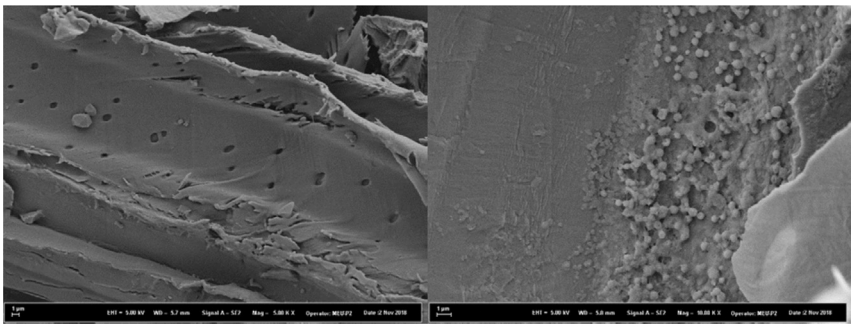

Fig. 3. SEM of *XI* a. without dye and b. with dye.

4.06 m^2/g for 0.045 mm [46]. But still less than molecularly imprinted biopolymers (MT-MIBP) with a value of 77 m^2/g which has an average pore diameter of 6.46 nm [47].

3.2. Response surface method

As shown previously, the effect of pH (A), adsorbent (B), time (C), and the concentration of RBBR (D) on the adsorbate capacity were studied by the CCD method. The quadratic model suggests the relationship between the adsorbate capacity and these factors as shown in Eq. (10).

$$\begin{aligned}(\text{Adsorbate}+0.25)^{0.5} &= 1.00434+0.022069\text{A}\\ &-0.205452\text{B}-0.003721\text{C}+0.024344\text{D}\\ &-0.002691\text{AB}+0.000114\text{AC}-0.000596\text{AD}\\ &+0.000433\text{BC}-0.001943\text{BD}+0.000053\text{CD}\\ &+0.000617\text{A}^2+0.017727\text{B}^2-7.35484\times10^{-6}\\ &\times\text{C}^2-0.000030\text{D}^2\end{aligned} \tag{10}$$

Results show the impact of the independent variables. These may be double (quadratic) or individual. Furthermore, a negative sign signifies a negative effect on the adsorbate while the positive signifies a positive effect. The analysis of variance (ANOVA) test examined the model adequacy. 2FI, linear, quadratic and cubic models were fitted to obtain the optimal representative model. According to the value of the regression coefficient (R^2), the quadratic model was selected to represent the model, and the transforms method was the square root method used, with a constant of 0.25. The model adequacy is shown in Appendixes B1 and B2.

The model was significant since the Model F-value was 359.54, with the chance of noise only 0.01%. Furthermore, the lack-of-fit test was performed. The F-value of this test was 2.27 which implied the lack-of-fit was not significant relative to pure error. The high F-value for the lack-of-fit test source may be due to noise. Appendix B3 shows the ANOVA analysis.

The adequate precision test measures the signal to noise ratio. The model indicated an adequate signal since the ratio was 69.233. This meant the optimum conditions could be ascertained by the developed model. The predicted and adjusted R^2 are in reasonable agreement since the difference between them was 0.009052 which is less than 0.2. According to the adjusted regression coefficient, the model can describe 99.4% of the RBBR removal by *XI* plants. This demonstrates an excellent correlation between the predicted results and the actual results [48]. Fig. 5 shows

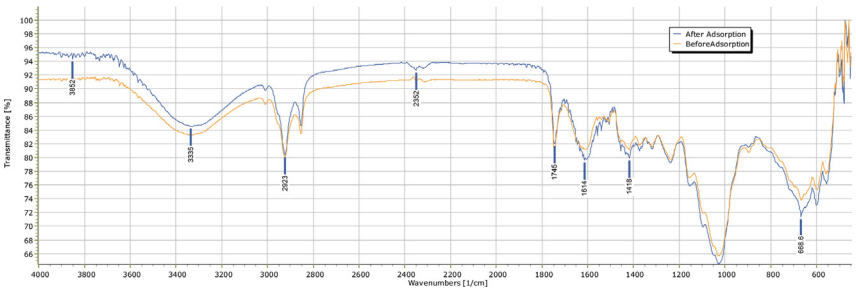

Fig. 4. FTIR spectra of *XI* before and after RBBR adsorption.

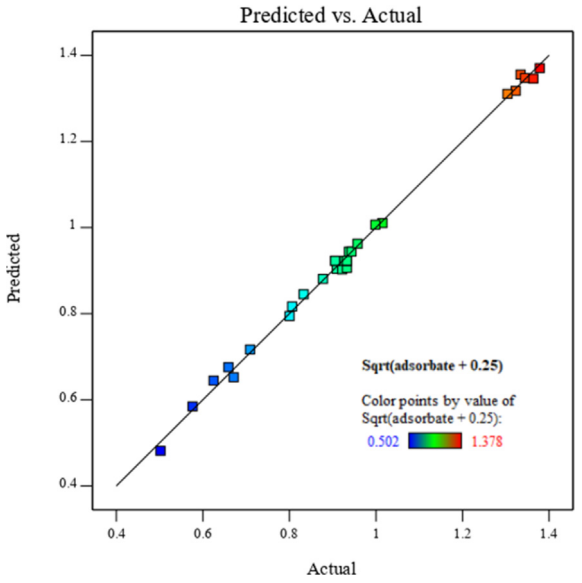

Fig. 5. Predicted and actual values for RBBR removal by *XI*.

the relationship between the predicted and actual values for the removal of RBBR by *XI*.

3.2.1. Model optimization

The developed model was optimized to identify the optimum conditions. For each variable, a set of recognized weighted goals were examined to meet the desirability, which can range from zero at outline points to one at the optimum position [49]. The choices of "in range" were selected for pH, and contact time with values of (8–9) and (45–125) min respectively. The "minimum" amount of adsorbent dose and "maximum" dye concentration were proposed to attain maximum adsorbate value. The optimum variable values are shown in Table 2.

3.2.2. Effect of independent factors on the removal of RBBR

The effects of the four independent variables (i.e. A: pH, B: adsorbent, C: contact time, and D: dye concentration) were analyzed in the ANOVA test; they proved to be significant. The model terms are significant if the P-value works out to be less than 0.05. In this case, A, B, C, D, AD, BC, BD, CD, B^2, C^2 were significant model terms. The model terms with P-value greater than 0.10 are not significant. Regarding the F-value, this affects the variable significances reversely: as F-values appear small, the significance becomes large. The pH and contact time had the lowest F-values. pH had 16.19 and contact time had 17.05, which means that the pH and contact time have maximum effects on this process. Similar results were obtained in previous studies [49,25]. Fig. 6 shows the relationship between the pH and RBBR removal by *XI*.

Table 2
Desirability table of RBBR adsorption onto XI.

pH	Adsorbent	Time	Dye Concentration	Capacity	Removal (%)
9.00	3.00	83.38	50.00	1.59	95%

The adsorption process was carried out at pH ranges from 3 to 11. In this study, the adsorption capacity increased with increasing pH. The removal of RBBR by the *XI* plant had an optimal pH range with values of 8–9. This result varied from the result obtained in previous studies [39] which suggested that the removal of RBBR by Pistachio Hull should be on pH 2. This result could be explained by the relationship between the dye functional group and the surface of the adsorbent. The dose variations of *XI* were studied in this model. The adsorption capacity increased as the *XI* decreased. Fig. 7 shows that the maximum capacity could be reached when applying 3 gm of *XI* as an adsorbent. Similar results were obtained in previous studies [50,51]. Fig. 8 reflects the effect of *XI* and RBBR on the adsorbate. It is clear that a maximum adsorbate can be obtained at maximum RBBR concentration and at minimum *XI* dose. Gadekar and Ahammed (2019) suggested that a maximum color removal occurs at a maximum concentration of dyes [30].

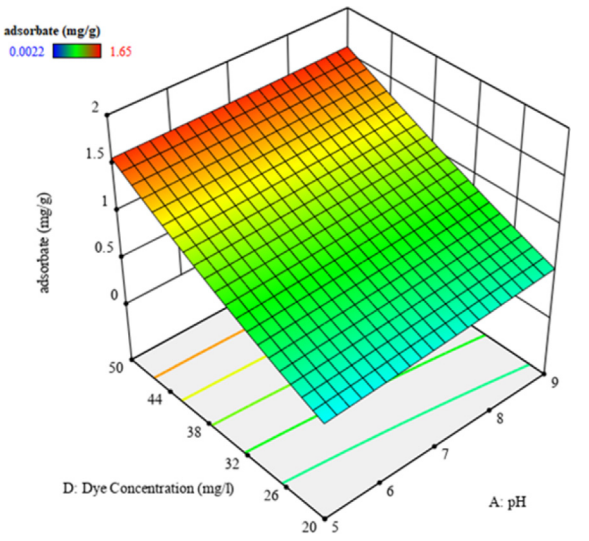

Fig. 6. Interaction effect of RBBR and pH on the adsorption capacity (at 3 gm *XI* and time of 84 min).

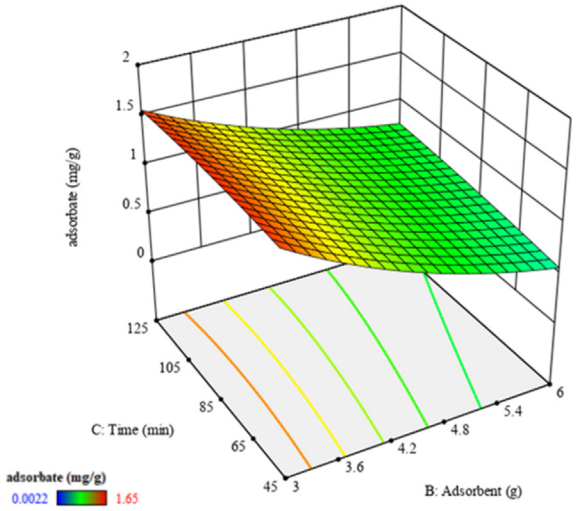

Fig. 7. Interaction effect of *XI* and time on the adsorption capacity (at RBBR of 50 mg/l and pH 9).

Fig. 9 represents the relation between the adsorbate, time and RBBR concentration. It shows a curvature in the graph of time, where the maximum adsorbate was obtained (t = 84 min).

3.3. Isotherms

Langmuir and Freundlich's isotherms were utilized herein to describe the adsorption mechanism. The constants of isotherms were obtained by plotting (Ce vs Ce/qe) and (Log qe vs Log Ce) in Langmuir and Freundlich's isotherms respectively. The slope and intercept were determined and summarized in Table 3.

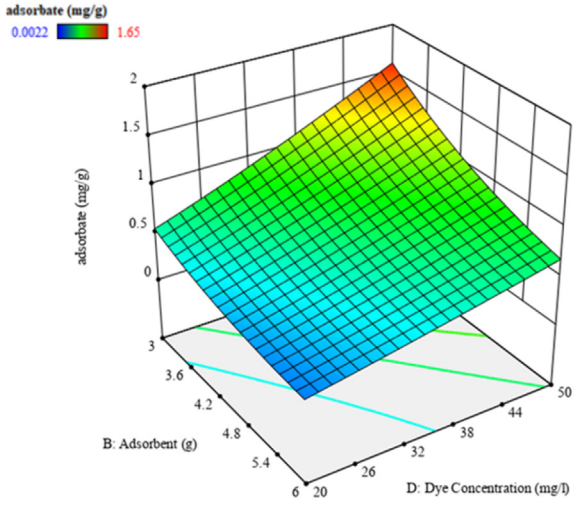

Fig. 8. Interaction effect of RBBR and *XI* on the adsorption capacity (at pH 9 and time of 84 min).

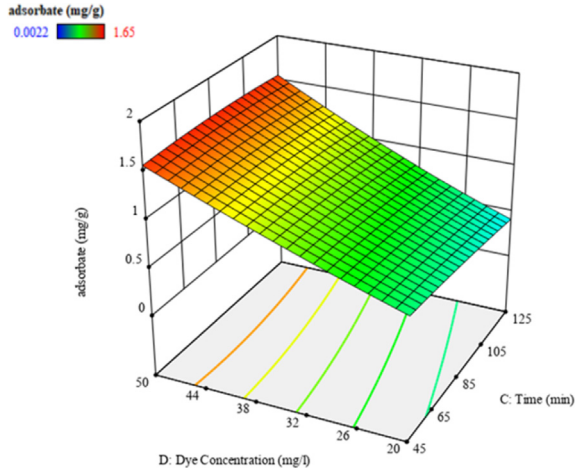

Fig. 9. Interaction effect of RBBR and time on the adsorption capacity (at 3 gm *XI* and pH 9).

The plots for Langmuir and Freundlich's isotherms are shown in Appendix C1 and C2 respectively.

Regression coefficients R for both Langmuir and Freundlich were high. However, because the Freundlich's isotherm had a higher correlation value, it was selected. In this type of isotherm, the adsorbent surface is heterogonous, the adsorption can be reversible and can occur in a multilayer. Freundlich's isotherm was used in the removal of RBBR by durian seed activated carbon [52] and in Rhodamine B dye removal by *Xanthium strumarium L.* seed hull [25].

3.4. Adsorption kinetics

In this study, the adsorption kinetic was assumed to follow one of these: Lagergren's Pseudo-first order or Pseudo-second order which could be calculated by Eqs. (5) and (6). The linearity graphs of the previous equations reflected the adsorption kinetics based on the correlation values in Fig. 10 and Fig. 11.

Based on the correlation factor ($R^2 = 0.9999$), the adsorption process of the RBBR onto *XI* followed the

Table 3
Isotherms parameters.

Isotherm	Parameter	Unit	Value
Langmuir	K_L	$L.mg^{-1}$	0.787
	a_L	$L.mg^{-1}$	0.195
	R^2	–	0.946
Freundlich	n	0	0.837
	K_F	$L.mg^{-1}$	0.938
	R^2		0.999
	Q max	$mg.g^{-1}$	1.595

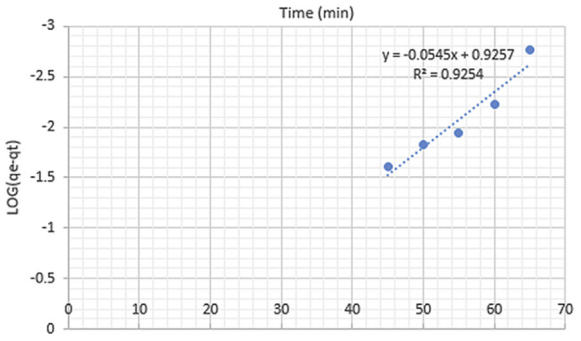

Fig. 10. Lagergren's Pseudo-first order.

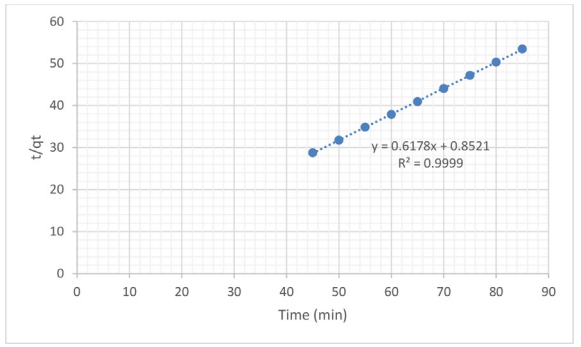

Fig. 11. Pseudo-second order.

Pseudo-second order kinetic model. This result fits with the result obtained when Rhodamine B removed by *X. strumarium L.* seed hull [25]. The equilibrium constant value from the Pseudo-second order reaction was 0.46 mg g^{-1}. min^{-1}.

3.5. Desorption

Desorption experiments were conducted at the same conditions as for the adsorption. Firstly, the optimum conditions of adsorption of RBBR onto XI were applied, then desorption experiments were conducted by using 0.5 M of NaOH and HCl in parallel. At the end of the experiments, the flask containing HCl had no effect on the desorption process. By contrast, the recovery of RBBR reached 47% and 73% after 120 min and 24 h respectively in NaOH flasks.

3.6. Comparison with other sorbent

In this study, the using of low-cost adsorbent material was investigated to remove RBBR from the solution. In other studies, several adsorbents were used to eliminate dyes. Table 4 shows a comparison between them.

4. Conclusion

In this study, the adsorption capacity of toxic and recalcitrant organic pollutants RBBR onto the invader *Xanthium İtalicum* plant was assessed. The experiment procedures were planned by CCD. The developed model was used to examine the effects of different parameters, as well as to study the possible interaction between them. The model proved that it can be used to achieve 98.5% removal of RBBR onto *XI*. A desirability value of 0.992 can be achieved using the identified optimum conditions: *XI* = 3 g, pH = 9, Time = 84 min, RBBR = 50 ppm. Freundlich isotherm had the best-fitted values with R^2 of 0.999. Furthermore, the adsorption of RBBR onto *XI* was determined to be pseudo-second-order kinetic with R^2 = 0.999. This study demonstrated the potential of using XI as an environmentally friend, cheap, and available adsorbent material for removing RBBR dye.

Table 4
Comparison between XI and other adsorbent.

Adsorbent	Dyes	Adsorption Capacity (mg/g)	References
Pistachio hull	Remazol Brilliant Blue R	16.74	[50]
Peanut hull-based activated carbon by microwave	Remazol Brilliant Blue R	149.25	[53]
Activated Carbon Prepared from Pinang Frond	Remazol Brilliant Blue R	232.59	[54]
Activated carbon from industrial laundry sewage sludge	Remazol Brilliant Blue R	33.47	[55]
rice husk	Rhodamine B	$5.87*10^{-5}$	[56]
Xanthium strumarium L. seed hull	Rhodamine B	2.17	[25]
Argemone Mexicana	Rhodamine B	1.3	[57]
agro-industrial waste Jatropha curcas pods as an activated carbon	Remazol Brilliant Blue R	0.31	[58]
cigarette ash	Remazol Brilliant Blue R	178.57	[59]
Using Pineapple Leaf Powder and Lime Peel Powder	Remazol Brilliant Blue R	9.58	[60]

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Appendix A. Supplementary data

Supplementary data to this article can be found online at 10.1016/j.kijoms.2019.01.004

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