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Response surface methodology approach for adsorptive removal of Reactive Blue 19 onto green pea pod

Elçin Demirhan

ABSTRACT

In the present study, removal of Reactive Blue 19 dye by using green pea pod as a low-cost adsorbent was investigated. Box–Behnken design was used to determine the independent and interaction influences of process variables of pH, temperature and adsorbent amount. The variance analysis (ANOVA) results showed that the second order model with high coefficient of determination value ($R^2 = 0.9997$) was statistically significant. The experimental results stated that the removal efficiency increased when the pH value decreased and the adsorbent amount increased. The maximum removal (99.42%) was obtained at pH 2, temperature of 35 °C and adsorbent amount of 1.5 g/100 mL. The equilibrium data investigation showed that the Freundlich isotherm model fitted better for removal of dye than did the Langmuir isotherm model. Furthermore, the adsorption kinetic was also evaluated and it was found that the adsorption followed the pseudo second order model for the Reactive Blue 19 removal onto green pea pod.

Key words | adsorption, anionic dye, Box–Behnken design, isotherm, kinetic, low-cost adsorbent

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HIGHLIGHTS

- The agricultural wastes may be good potential adsorbents according to their physicochemical characteristics and low cost.
- The feasibility of waste residue of green pea pod as low cost adsorbent for the removal of RB19 was investigated.
- Box–Behnken design was used for used for the prediction of the optimum process parameters for the wastewater treatment.
- RB19 removal using green pea pods fitted well to the Freundlich isotherm model and the pseudo second order model.
- Green pea pod can be employed as a novel, low cost, effective and alternative adsorbent for removal of dye and other environmental pollutants.

INTRODUCTION

The major source of water pollution is the wastewater discharge of industries especially textile, leather, paint, paper and pulp (Hong & Yang 2017; Oyekanmi *et al.* 2019). The total yearly consumption of dyes is predicted to be over 700,000 tons and 75% of this is accounted for mainly by textile plants textile plants (Andrade *et al.* 2007; Ahmadi *et al.* 2018). Because of the mutagenic and carcinogenic effects of dyes, discharging of dye without any treatment can have severe damage for the aquatic environment and human beings (Basu 2016; Dehvari *et al.* 2016). Reactive dyes, the most extensively used coloring agents in the textile industry, are based on azo and anthraquinone functional groups and they are water soluble dyes (Du *et al.* 2017; Ahmadi *et al.* 2018; Holkar *et al.* 2018). Reactive Blue 19 (RB19), an anionic dye, is resistant to chemical oxidation due to its aromatic anthraquinone structure which is highly stabilized by resonance. (Isah *et al.* 2015; Dehvari *et al.* 2016; Du *et al.* 2017; Ahmadi *et al.* 2018; Dalvand

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et al. 2018; Holkar *et al.* 2018). RB19 exists for long periods in the natural environment because of its resistance to degradation. In aquatic environment, the existence of RB19 decreases the light penetration and increases oxygen consumption (Holkar *et al.* 2018; Sadeghi *et al.* 2018). Therefore, it is of great importance to eliminate the negative effect of these dyes before discharge to the natural environment (Radaei *et al.* 2014; Holkar *et al.* 2018).

There are several treatment methods available for the removal of dyes in wastewater, such as membrane filtration (Xu *et al.* 1999), adsorption (Dod *et al.* 2012; Dalvand *et al.* 2018; Çelebi 2019), coagulation (Sadri Moghaddam *et al.* 2010; Mohtar *et al.* 2017), biological (El Atouani *et al.* 2019) and advanced oxidation (Ahmadi *et al.* 2018). Taking into consideration the operational costs and adaptability of the methods, among all methods, adsorption is an efficient, easy and economic method for the treatment of the dye-containing wastewater (Basu 2016; Dandge *et al.* 2016; Tepe 2018).

In recent years, for the adsorption of the pollutants, research on the usage of economical, easily available and highly effective adsorbents such as agricultural wastes as an alternative to commercial activated carbon has increased (Sallah *et al.* 2011; Dehvari *et al.* 2016). The agricultural wastes may be good potential adsorbents according to their physicochemical characteristics and low cost. Agricultural wastes are lignocellulosic materials that consist of the main structural components of lignin, cellulose and hemicelluloses. Agricultural wastes are available in large quantities, renewable and less expensive as compared to other adsorbents (Sallah *et al.* 2011).

Taking into account the economy and sustainability principles, a wide variety of the agricultural wastes as adsorbents have been tested for the dye removal, such as peanut hull (Gong *et al.* 2005); rice husk (Patabandige *et al.* 2019); orange peel (Sivaraj *et al.* 2001); banana peel (Munagapati *et al.* 2020); wood waste (Azizi *et al.* 2012); hazelnut shell (Doğan *et al.* 2009); pomegranate seed (Ghaneian *et al.* 2015); tea leaves (Sarkar *et al.* 2019); watermelon seed (Benkaddour *et al.* 2018); walnut shell (Almasi *et al.* 2017); lemon peel (Kumar 2007); apple pomace and wheat straw (Robinson *et al.* 2002); palm kernel fibre (Ofomaja & Ho 2007); coconut husk (Jain & Shrivastava 2008); sunflower seed shell (Osma *et al.* 2007); garlic peel (Hameed & Ahmad 2009); bamboo shoot skin (Zhu *et al.* 2019) and pumpkin seed hulls (Çelebi 2019).

Pea (*Pisum sativum*) is the second most important food legume worldwide after the common bean. About 11.7 million tons are produced annually and 30% (fresh weight basis) of the total pea weight is owing to pea pods. Thus, based on the annual production, approximately 4 million tons of pea pod are generated, which is discarded as waste (Verma *et al.* 2011; Upasana & Vinay 2018).

Therefore, in the present study, it was aimed to study the feasibility of waste residue of green pea pod as low cost adsorbent for the removal of RB19 from aqueous solution. In addition, going beyond classical adsorption applications, response surface methodology (RSM) based on Box–Behnken design (BBD) was used for the prediction of the optimum process parameters (pH, adsorbent amount and temperature) under different adsorption conditions for the wastewater treatment. Moreover, adsorption isotherms and kinetics were used to evaluate the experimental results to comprehend the dye removal and the feasibility of green pea pods as adsorbent.

MATERIALS AND METHODS

Materials

RB19 used in this study was obtained from Gülerçin Kimya A.Ş., Istanbul, Turkey. All other chemicals of analytical grade were purchased from Sigma Aldrich. The molecular structure is shown in Figure 1 and the physical and chemical properties of RB19 are given in Table 1.

Adsorbent

Green pea pods (GPP) were purchased from a local greenhouse supplier in Antalya, Turkey. The shells were washed with distilled water to remove the surface-adhered particles, and then dried in an oven at 80 °C until constant weight was reached. Finally, GPP were crushed and sieved to obtain 100 mesh particles. The GPP powder was stored in airtight bottles for experimental use.

The physicochemical characteristics of the adsorbents are important in order to evaluate their adsorption

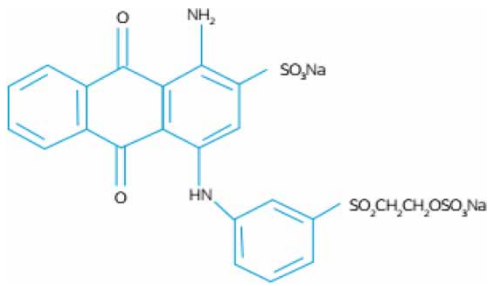

Figure 1 | Molecular structure of Reactive Blue 19.

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Table 1 | General characteristics of Reactive Blue 19 and green pea pod

Parameter	Characteristic
Reactive Blue 19	
Generic name	Reactive Blue 19
Commercial name	Remazol brilliant blue R
Type	Anionic
Functional group	Anthraquinone
Chemical formula	$C_{22}H_{16}N_2Na_2O_{11}S_3$
Molecular weight (g/mol)	626.54
Wavelength of maximum absorption (nm)	595
Molar extinction coefficient ε595 (M^{-1} cm^{-1})	10,000
Solubility	30 g/L in water (80 °C)
pH of stock solution	6.20 (28 °C)
Green pea pod	
Bulk density (g/cm^3)	0.65
Porosity (%)	61.52
Proximate analysis (%)	
Moisture	6.48
Ash	3.87
Volatile matter	79.41
Fixed carbon	10.24
Ultimate analysis (%)	
Carbon	38.51
Hydrogen	5.74
Nitrogen	2.15
Sulfur	0.19
Oxygen	53.41

capacities. The bulk density, porosity, surface charge, and point of zero charge (pH_{pzc}) were determined and also proximate and ultimate analyses of GPP were done. The physicochemical properties of GPP are presented in Table 1.

Experimental design by response surface methodology

RSM is essentially a collection of mathematical and statistical methods used to design experiments, to develop models by considering the interactions of parameters, and optimize the process. The principal aim of RSM is finding the optimum operational conditions for the process or specification of a region that satisfies the performance responses set by the researcher (Karimifard & Alavi Moghaddam 2018; Tepe 2018). Among several design strategies of RSM, BBD was applied to determine the individual and complex effects of process variables for the RB19 removal. The pH, temperature and adsorbent amount were selected as independent variables and the response was dye removal efficiency (%). Independent variables, their experimental range and three levels of these variables are presented in Table 2 for RB19 removal. A BBD with 17 experiments (5 of centered points) carried out using Stat-Ease software (Design-Expert 11.0 trial version, Stat-Ease, Inc.).

Adsorption experiments

The adsorption experiments by varying the process variables obtained from BBD design were conducted in 250 mL Erlenmeyer flasks containing 100 mL of 100 mg/L dye concentration in an orbital shaker. At desired process variable values the experiment was started by the addition of adsorbent to dye solution. The pH of the solution was adjusted with 0.1 N HCl or NaOH solutions. The flasks were shaken at 175 rpm agitation speed. The samples were taken at desired time intervals during the experiments. In order to remove the adsorbent particles, the samples were centrifuged. The dye concentration was measured by using a UV/vis spectrophotometer (Schimadzu UV-1800 UV-VIS spectrophotometer) at a wavelength of 595 nm after centrifuging (Isah *et al.* 2015; Ahmadi *et al.* 2018). The adsorption equilibrium was reached after 1 hour. The dye removal efficiency (R (%)) and amount of dye adsorbed at equilibrium q_e (mg/g) were determined according to Equations (1) and (2), respectively:

$$R\ (\%) = \frac{C_0 - C_e}{C_0} \times 100 \quad (1)$$

$$q_e = \frac{(C_0 - C_e) \cdot V}{m} \quad (2)$$

where C_0 is the initial dye concentration (mg/L), C_e is the equilibrium dye concentrations (mg/L), V is the volume of the solution and m is the weight of adsorbent (g).

Table 2 | Independent variables, their experimental range and three levels of these variables

	Coded levels		
Independent variable	**−1**	**0**	**+1**
pH, x_1	2	4	6
Temperature (°C), x_2	25	35	45
Adsorbent amount (g/100 mL), x_3	0.5	1	1.5

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Adsorption isotherm and kinetic studies

The adsorption isotherm represents the relationship between the amount of the dye adsorbed and its concentration in the equilibrium (Amin 2009; Kyzas *et al.* 2012; Dehvari *et al.* 2016; Tanyol 2017). Langmuir and Freundlich models were chosen to evaluate the data obtained from the RB19 adsorption experiments.

Langmuir model

The Langmuir model (Equation (3)) is used to define the adsorption that occurs on one single active site on the surface of the adsorbent (Dada *et al.* 2012; Haldorai *et al.* 2015).

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{b \cdot q_m} \tag{3}$$

where q_m is the maximum adsorption capacity (mg/g) and b is the equilibrium constant related to affinity of binding sites (L/mg).

Freundlich model

The Freundlich model (Equation (4)) describes the multilayer adsorption on a heterogeneous surface with nonuniform distribution of energy and active sites (Piccin *et al.* 2011; Dehvari *et al.* 2016; Hong & Yang 2017).

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

where K_F and n are coefficients indicating the adsorption capacity and adsorption intensity, respectively (Oyekanmi *et al.* 2019).

Adsorption kinetics are used to explain the mechanism of the adsorption processes and adsorption behavior (Dehvari *et al.* 2016; Hong & Yang 2017). Among the several methods available for investigation of adsorption mechanism, in this study, the data obtained from the RB19 adsorption process were analyzed with two kinetic models (pseudo first order model (Equation 5) and pseudo second order model (Equation 6)) as follows:

Pseudo first order model

$$\log \frac{q_e - q_t}{q_e} = \frac{k_1 t}{2.303} \tag{5}$$

where q_t (mg/g) is the adsorbed amount at time t; k_1 (1/min) is the rate constant of pseudo first order model (El Atouani *et al.* 2019).

Pseudo second order model

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{6}$$

where k_2 (g/mg·min) is the rate constant of pseudo second order model (El Atouani *et al.* 2019).

RESULTS AND DISCUSSION

Characterization of adsorbent

The pH_{pzc} is an important factor which provides information about the degree of ionization on the adsorbent surface and its interactions with the adsorbate. The graph of the ($pH_i - pH_f$) versus pH_i is given in Figure 2. As can be seen from this figure, the pH_{pzc} value of the GPP adsorbent was determined as 6.02. The pH_{pzc} value of GPP adsorbent indicates that the surface of the GPP is positively charged at pH values below pH_{pzc} and negatively charged above the pH_{pzc} values.

Optimization of process variables

Box–Behnken design and regression model

The values of the pH, temperature and adsorbent amount for the experiments, and observed and predicted results for removal efficiency are presented in Table 3.

According to a statistic evaluation done by the Stat-Ease program with the experimental data from Table 3, the second order model was used to illustrate the mathematical relationship between the independent process variables and

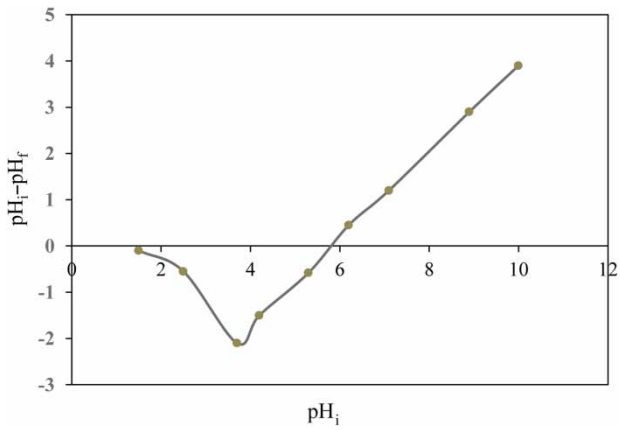

Figure 2 | Point of zero charge of the GPP adsorbent.

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Table 3 | Box–Behnken design matrix

Run	x_1	x_2	x_3	% Removal (observed)	% Removal (predicted)	q_e (mg/g)
1	4	35	1.0	87.11	87.06	8.71
2	4	35	1.0	86.85	87.06	8.69
3	2	35	0.5	94.42	94.18	18.88
4	6	25	1.0	74.21	74.05	7.42
5	4	45	1.5	91.04	90.96	6.07
6	4	45	0.5	82.53	82.62	16.51
7	4	35	1.0	87.26	87.06	8.73
8	4	25	0.5	82.41	82.49	16.48
9	4	25	1.5	90.12	90.04	6.01
10	2	25	1.0	96.58	96.74	9.66
11	2	45	1.0	95.28	95.44	9.53
12	4	35	1.0	87.09	87.06	8.71
13	6	35	1.5	81.02	81.26	5.40
14	6	35	0.5	70.47	70.54	14.09
15	2	35	1.5	99.42	99.35	6.63
16	4	35	1.0	86.98	87.06	8.70
17	6	45	1.0	76.56	76.40	7.66

the response values. The model equation that showed the removal efficiency as a function of pH (x_1), temperature (x_2) and adsorbent amount (x_3) could be presented as Equation (7):

$$\begin{aligned} Removal\ (\%) = {} & 102.10 - 6.61x_1 + 0.23x_2 + 0.42x_3 \\ & + 0.05x_1x_2 + 1.39x_1x_3 + 0.04\,x_2x_3 \\ & - 0.19x_1^2 - 0.006x_2^2 + 0.28x_3^2 \end{aligned} \quad (7)$$

It is essential to carry out an analysis of variance (ANOVA) in order to determine whether or not the second order model is significant (Zhang *et al.* 2016; Tepe 2018). The results of ANOVA for removal of RB19 is listed in Table 4. As seen from Table 4, the second order model was statistically significant ($P < 0.0001$). The *P*-value of 'lack of fit' is 0.1232, which indicates that lack of fit is not significant relative to the pure error. A non-significant lack of fit was considered good and desirable for the model to fit. Moreover, according to the significance of the model coefficients, pH, temperature, adsorbent amount, interaction terms of x_1x_2 and x_1x_3 and quadratic terms of pH and temperature all had significant effect on the RB19 removal. In addition, the value of the coefficient of determination ($R^2 = 0.9997$) indicated that 99.97% of total variation of the response was explained by the model. The high R^2

Table 4 | Analysis of variance of the fitted second order model for RB19 removal

Source	Sum of squares	df	Mean square	F-value	*P*-value*
Model	1,012.62	9	112.51	2,260.24	<0.0001
x_1, pH	870.28	1	870.28	17,482.76	<0.0001
x_2, Temp.	0.5460	1	0.5460	10.97	0.0129
x_3, Ads. amount	126.17	1	126.17	2,534.52	<0.0001
x_1x_2	3.33	1	3.33	66.91	<0.0001
x_1x_3	7.70	1	7.70	154.70	<0.0001
x_2x_3	0.1600	1	0.1600	3.21	0.1161
x_1^2	2.67	1	2.67	53.66	0.0002
x_2^2	1.54	1	1.54	30.86	0.0009
x_3^2	0.0212	1	0.0212	0.4264	0.5346
Residual	0.3485	7	0.0498		
Lack of fit	0.2546	3	0.0849	3.62	0.1232
Pure error	0.0939	4	0.0235		
Corrected total	1012.96	16			

*$P < 0.05$ indicates statistical significance.

and adjusted R^2 ($R^2_{adj} = 0.9992$) values also showed a high correlation between the experimental results and the results predicted by the model. Also, from Figure 3, the correlation between the predicted response values versus actual response values could be clarified, and the cluster points around the diagonal line indicated the good fit of the model.

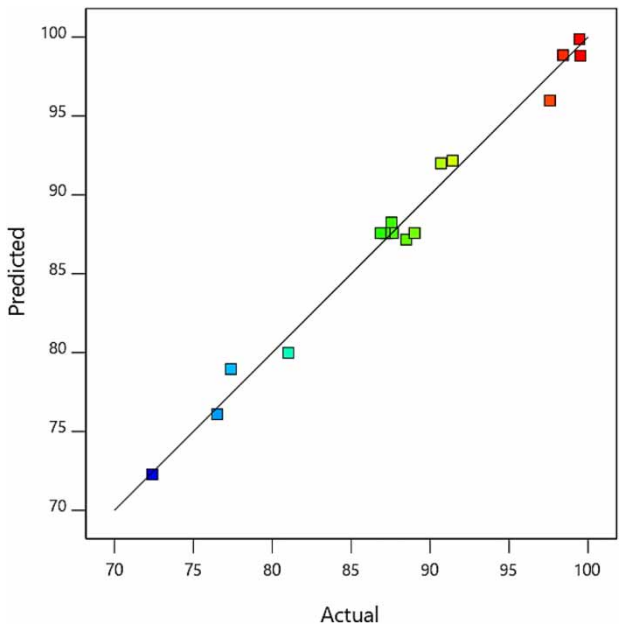

Figure 3 | Plot of the predicted response values versus actual response values of RB19 removal.

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Effects of process variables

The overall influences of pH, temperature and adsorbent amount (Ads. Amount) are pointed out in Figure 4. As seen from this figure, the influence of pH indicated that decreasing of pH resulted in a linear increase in the removal efficiency. The pH_{pzc} of GPP adsorbent was obtained as 6.02. At the pH values below the point of zero charge, the surface of adsorbent is positively charged. The adsorption of RB19 is favorable in the acidic environment due to the electrostatic attractions between the negatively charged functional groups of the anionic dye and positively charged

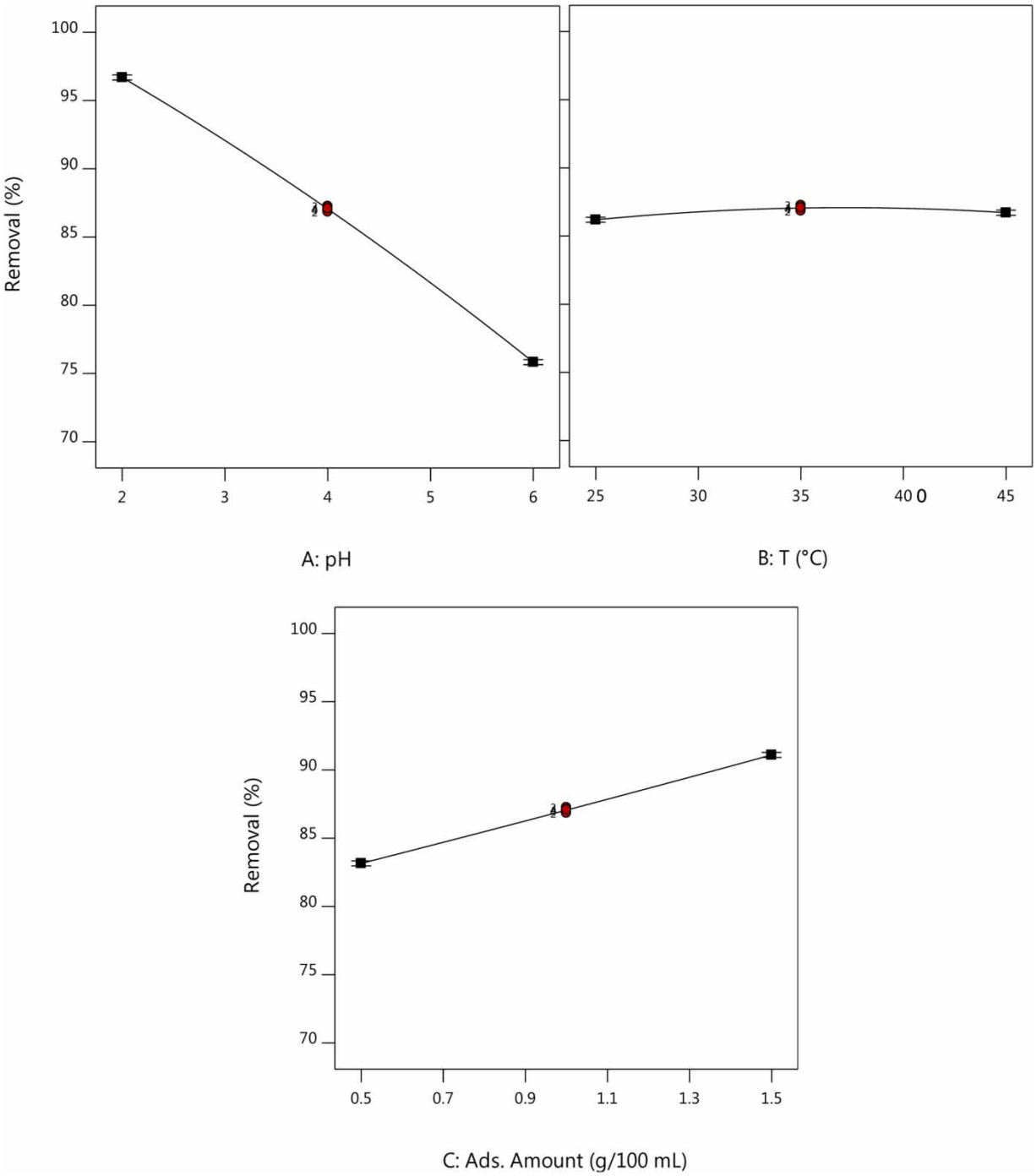

Figure 4 | Overall effects of process variables on RB19 removal by using GPP.

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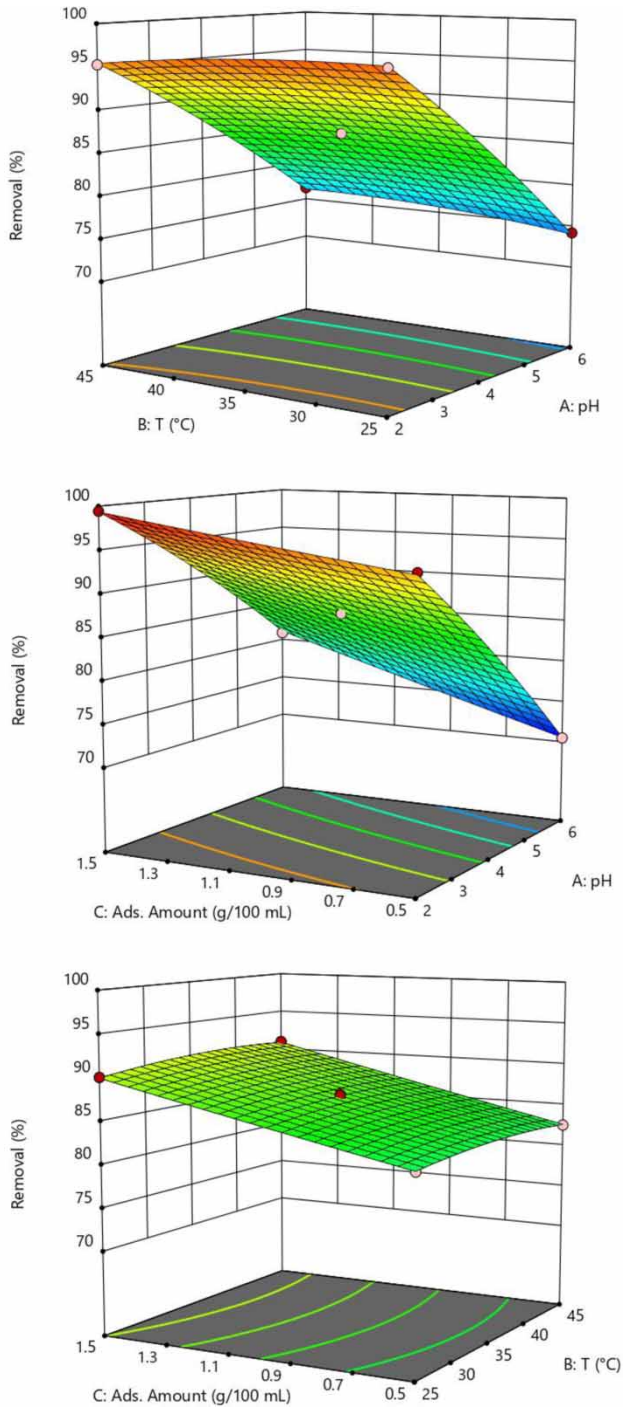

Figure 5 | Three-dimensional response surface plots for removal of RB19.

adsorbent surface. As the pH increases, the number of positively charged sites decreases on the adsorbent surface. Thus, it is concluded that the dye adsorption is less favorable.

The increase in temperature caused a slight increase in removal efficiency in the studied range. Moreover, increasing the adsorbent amount induced a linear increase of the removal efficiency. Eventually, it can be concluded that the pH and adsorbent amount have the most profound effect on RB19 removal.

Response surface plots for the RB19 removal were drawn in order to determine the effects of variables' interactions and are given in Figure 5(a)–5(c). The maximum removal (99.42%) was achieved at pH 2, temperature of 35 °C and adsorbent amount of 1.5 g/100 mL. The effects of pH and temperature are shown in Figure 5(a). As seen from this figure, the highest removal was obtained at lowest pH value. The decrease in the pH resulted in enhanced adsorption rate of the dye. Figure 5(b) shows the effects of pH and adsorbent amount. The highest removal was achieved at lowest pH value and highest absorbent amount. Likewise, the effects of adsorbent amount and temperature are shown in Figure 5(c). The maximum removal was obtained at highest adsorbent amount in the studied range. However, from Figure 5(a) and 5(c), it can be concluded that the temperature had little effect on the RB19 removal. Thus, as a conclusion, the pH and adsorbent amount are considered to be most effective parameters in influencing the dye removal process.

Adsorption isotherms

The prediction of dye adsorption on GPP was assessed according to the well-known Langmuir and Freundlich isotherm models. The estimated parameters and statistical values of the models are presented in Table 5. The Freundlich isotherm model was determined as the best as indicated by the high values of correlation coefficient (R^2) and low values of standard error in comparison with the Langmuir isotherm in the studied range. In the Freundlich isotherm, the $1/n$ constant indicates the sorption intensity of the adsorbent and mechanism of adsorption. If $0 < 1/n < 1$, it indicates that the isotherm is favorable; if

Table 5 | Isotherm model parameters and statistical values for RB19 removal onto GPP

Langmuir model				Freundlich model			
q_m (mg/g)	b (L/mg)	R^2	σ	K_F ((mg/g)(mg/L)^n)	$1/n$	R^2	σ
32.79	0.146	0.9768	0.0962	5.17	0.49	0.9981	0.0602

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$1/n = 0$, the isotherm is irreversible; and if $1/n > 1$, it is unfavorable. (Tang *et al.* 2012). And also, if $1/n$ is below 1, it points out the adsorption is a chemisorption process. In the present study, the $1/n$ was found as 0.49 and it showed that the isotherm is favorable for RB19 adsorption. Moreover, this result indicates that the dye is mainly adsorbed onto the heterogeneous surface of adsorbent (Hong & Yang 2017) and the adsorption mechanism may be chemisorption (Dehvari *et al.* 2016).

Adsorption kinetics

The estimated constants and statistical data of the pseudo first order and pseudo second order kinetic models are given in Table 6. As seen from this table, the pseudo second order kinetic model was observed as the most appropriate one for all the experimental data as indicated by the high value for the coefficient of determination and low value for the standard error between these models. The pseudo second order kinetic model is also based on the sorption capacity of the solid phase and on the assumption that the sorption process involves a chemisorption mechanism involving valence forces through sharing or exchange of electrons between sorbent and sorbate (Ho & McKay 1999; El-Bindary *et al.* 2019).

Table 6 | Kinetic model parameters and statistical values for RB19 removal onto GPP

Pseudo first order kinetic model			Pseudo second order kinetic model		
k_1 (1/min)	R^2	σ	k_2 (g/mg·min)	R^2	σ
0.166	0.8668	3.0220	0.841	0.9998	0.0382

Similar results were obtained by the studies for RB19 removal using coconut shell based activated carbon (Isah *et al.* 2015), pomegranate seed powder (Dehvari *et al.* 2016), pineapple leaf powder and lime peel powder (Rahmat *et al.* 2016) and corn silk (Değermenci *et al.* 2019).

Comparison of anionic dye removal with different adsorbents reported in literature

Several studies have been carried out for the removal of anionic dye from aqueous solution by using adsorbents from various waste materials. The comparison between the adsorption capacity of GPP and other adsorbents is given in Table 7. As can be seen from this table, GPP was used without any modification as a native adsorbent with good capacity for RB19 dye.

CONCLUSION

In the present study, the removal of RB19 from aqueous solution using GPP as low-cost adsorbent was investigated. The effect of pH, temperature and adsorbent amount on the removal efficiency was studied using BBD. The pH and adsorbent amount were considered as the most effective variables for the dye removal process. The isotherm data for RB19 removal using GPP fitted well to the Freundlich isotherm model indicating that the RB19 was adsorbed onto the heterogeneous surface in multilayers. Furthermore, it was determined that kinetics of RB19 adsorption follows the pseudo second order model. Finally, according to the results, it can be concluded that GPP can be employed as

Table 7 | Comparison of the adsorption capacities of anionic dye on GPP and other adsorbents

Adsorbent	Adsorbate	Adsorption capacity (mg/g)	References
Orange peel	Acid violet 17	19.88	Sivaraj *et al.* (2001)
Banana peel	Congo red	18.12	Annadurai *et al.* (2002)
Peanut hull	Sunset Yellow	13.99	Gong *et al.* (2005)
Canola hull	Reactive Blue 19	2.00	Mahmoodi *et al.* (2011)
Sunflower seed hull	Reactive Orange 16	22.10	Suteu *et al.* (2011)
Pumice	Reactive Black 5	12.85	Heibati *et al.* (2014)
Walnut wood (activated carbon)	Reactive Black 5	19.34	Heibati *et al.* (2014)
Pomegranate seed powder	Reactive Red 198	10.95	Ghaneian *et al.* (2015)
Rice husk ash	Reactive Orange 16	18.69	Akbar *et al.* (2020)
Tea leaves	Reactive Green 19	26.32	Sarkar *et al.* (2019)
Green pea pod	Reactive Blue 19	18.88	This study

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a novel, low cost, effective and alternative adsorbent for dye removal and other environmental pollutants.

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