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Adsorption of phenol onto activated carbon from *Rhazya stricta*: determination of the optimal experimental parameters using factorial design

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Abstract A novel activated carbon was prepared from *Rhazya stricta* leaves and was successfully used as an adsorbent for phenol removal from aqueous solution. The prepared activated carbon was characterized by FTIR and SEM analysis. Three factors (namely, temperature, pH and adsorbent dose) were screened to study their effect on the adsorption of phenol by *R. stricta* activated carbon. A 2^3 full factorial design was employed for optimizing the adsorption process. The removal of phenol by adsorption onto *R. stricta* carbon reached 85 % at a solution pH of 3, an adsorbent dose of 0.5 g/l and a temperature of 45 °C. The temperature and adsorbent weight had a positive effect on phenol removal percentage, when both factors were changed from low to high and the opposite is true for the initial solution pH. The results of the main effects showed that the three studied factors significantly affected phenol removal by *R. stricta* carbon with 95 % confidence level. The interaction effects revealed that the interaction between the temperature and pH had the most significant effect on the removal percentage of phenol by *R. stricta* activated carbon. The present work showed that the carbon prepared from a low-cost and natural material which is *R. stricta* leaves is a good adsorbent for the removal of phenol from aqueous solution.

Keywords Adsorption · Activated carbon · Phenol · Factorial design · *Rhazya stricta*

Introduction

Phenols are among the most common water pollutants (Nayak and Singh 2007), and considered as major toxic pollutant, even at a concentration of as low as 0.1 ppm (Subramanyam and Das 2009). The US Environmental Protection Agency (EPA) regulations call for lowering phenol content in the wastewater to <1 mg/l (Hameed and Rahman 2008).

Phenols are introduced into surface water from industrial effluents such as those from the coal tar, gasoline, plastic, rubber proofing, disinfectant, pharmaceutical and steel industries and domestic wastewaters, agricultural run-off and chemical spills (Lin and Juang 2009).

The ingestion of such contaminated water in the human body causes protein degeneration, tissue erosion and paralysis of the central nervous system and also damages the kidney, liver and pancreas (Hameed and Rahman 2008). Ingestion of a small amount of phenol by human beings may cause nausea, vomiting, paralysis, coma, greenish or smoky colored urine and even death from respiratory failure or cardiac arrests. Fatal poisoning may also occur by adsorption of phenol by skin, if a large area of the body is exposed (Mohanty et al. 2005).

The most common methods used for removal of phenol from industrial effluents include stripping, solvent extraction, oxidation, ion exchange, biodegradation and

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adsorption methods (Subramanyam and Das 2009). Out of all these treatment methods, adsorption process is recognized as the most efficient, promising and widely used fundamental approach in wastewater treatment processes, mainly hinging on its simplicity, economically viable, technically feasible and socially acceptable (Foo and Hameed 2010).

In our previous researches, the potential use of different low-cost and eco-friendly adsorbents have been achieved. Recently, *Typha domingensis* biomass was successfully used as a biosorbent for aluminum (Abdel-Ghani et al. 2009), iron and zinc ions removal from aqueous solution (Hegazy et al. 2011).

Activated carbons are the most versatile and commonly used adsorbents because of their extremely high surface areas, micropore volumes, large adsorption capacities, fast adsorption kinetics and relative ease of regeneration (Tongpoothorn et al. 2011). Carboxylic, carbonylic, lactonic, phenolic, aldehydic and other organic functional groups are located at the edges of hexagonal carbon layer planes and are responsible for surface reactivity of activated carbon (Momcilovic et al. 2011).

Materials that can produce activated carbons from plant origin include wood, peat, lignin, coal, vegetable waste from food processing, coconut shells, nut shells (Poinern et al. 2011). Many previous studies reported successful preparation of activated carbons from various plant biomasses including *Euphorbia rigida* (Gerçel et al. 2007), *Typha orientalis* (Zhang et al. 2008), *Enteromorpha prolifera* (Li et al. 2010), *Jatropha curcas* fruit shell (Tongpoothorn et al. 2011), *Zizania latifolia* (Huang et al. 2012), etc.

In the present study, *Rhazya stricta* leaves were used as a precursor material for the preparation of activated carbon. *Rhazya stricta* Decne. (Apocynaceae) is a small glabrous, erect herb that grows in deserts of South Asia and the Middle East Countries (Gilani et al. 2007). The aim of the present work was to achieve the optimal conditions for phenol removal by a novel activated carbon prepared from *R. stricta* leaves using factorial experimental design.

Materials and methods

Adsorbent preparation and characterization

Rhazya stricta leaves were collected from wadi Hermela, central Saudi Arabia (GPS location: 25°02′46.7″E). The leaves were washed several times with distilled water to remove any adherent dust. After that, they were air dried until constant weight. The dried leaves were then grinded to a fine powder. An aqueous solution of H_2SO_4 at the concentration of 25 % was prepared. The powdered leaves

of *R. stricta* (250 g) were soaked in the aqueous solution of H_2SO_4 (1,000 ml) for 5 days. The mixture was separated by filtration and the resultant product was washed several times until clearness and neutral pH. The resulting product was then oven dried at 100 °C for 24 h, then transferred to a muffle furnace and burnt at 600 °C for 2 h. The weight loss during carbonization was about 85 %. The prepared chemically activated carbon was collected and stored in a desiccator until its use in the adsorption experiments.

The solution pH was adjusted at the beginning of each experiment using a solution of 0.5 M HCl and/or NaOH and the pH value was recorded using a THERMO Scientific ORION pH meter.

FTIR spectrum of the activated carbon prepared from *R. stricta* leaves was recorded using Thermo *Nicolet Avatar 370 FTIR* Spectrometer and the SEM imaging of the activated carbon was obtained using a Quanta 250-FEI scanning electron microscope.

Adsorption experiment

To optimize the adsorption of phenol by *R. stricta* activated carbon, a 2^3 factorial design was employed. Three factors at two levels were chosen to study their effects on the removal of phenol by *R. stricta* activated carbon. The chosen factors were temperature (T), solution pH (pH) and adsorbent weight (X). The other experimental factors such as the initial phenol concentration (100 mg/l), speed of agitation (100 rpm) and agitation time (4 h) were kept constant. The temperature, pH and adsorbent weight employed are shown in Table 1. The lower and upper levels of pH were chosen based on previously reported studies (Badr 2007; Rathinam et al. 2011).

At the beginning of each experimental run, 50 ml of phenol solution was mixed with the adsorbent in a conical flask and the flasks were agitated on a shaking water bath at the required temperature. After the predetermined equilibrium time (4 h), the adsorbent was separated from the solutions by filtration and the remaining phenol concentration was determined by measuring its absorbance at 270 nm (Ioannou and Simitzis 2009) using a UV–Vis Spectrophotometer Double Beam Model UVD-3500. The phenol removal percentage ($R\%$) was calculated as

Table 1 High and low level factors employed in the 2^3 factorial design

Factor	Factor level	
	Low level	High level
Temperature (°C)	25	45
Adsorbent weight (g/l)	0.5	5
pH	3	9

Fig. 1 FTIR spectrum of *R. stricta* leaves activated carbon
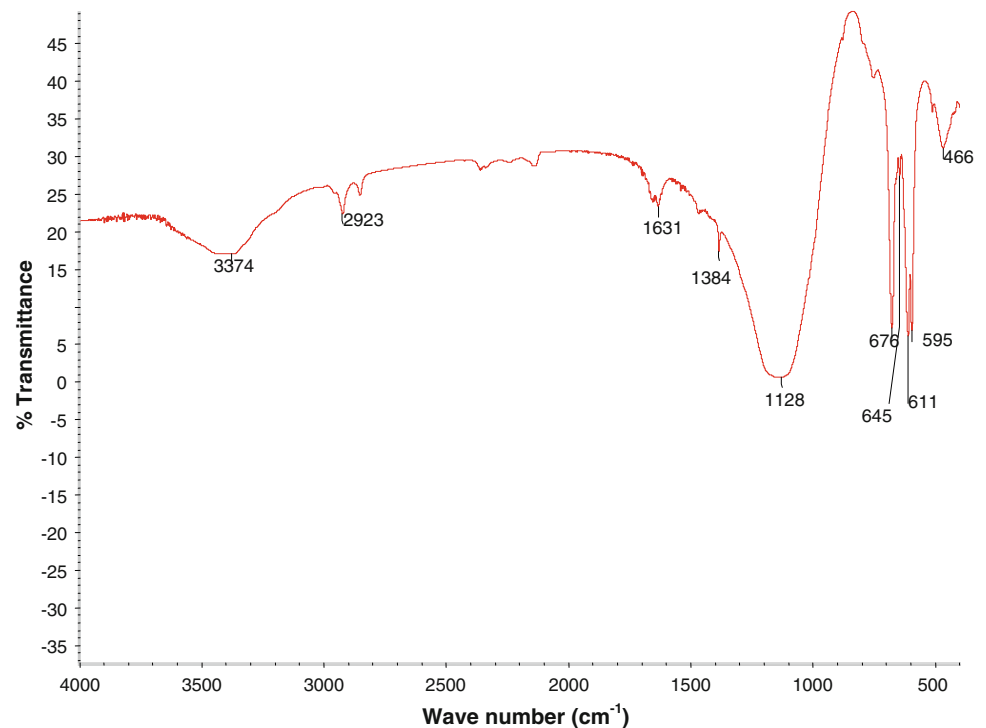
$R\% = (C_i - C_f/C_i) \times 100$; where C_i and C_f are initial and final concentrations of phenol, respectively. The experimental results were analyzed using the Minitab 15 software, and the main effects and interactions between factors were determined.

Results and discussion

Adsorbent characterization

To determine the surface functional groups of *R. stricta* activated carbon, its FTIR spectrum was obtained as shown in Fig. 1. The spectrum revealed the presence of a number of characteristic bands in the range of 400–4,000 cm^{-1}. A broad band at 3,374 cm^{-1} was assigned to OH stretching vibration, and may be also the characteristic peak of carbonyl and phenolic hydroxyl groups or adsorbed water (Huang et al. 2012). A weak peak at 2,923 cm^{-1} was ascribed to aliphatic group stretching vibration, and according to Huang et al. (2012) the low intensity of this peak is due to the decrease in aliphaticity in the activated carbon by increasing temperature. The band observed at 1,631 cm^{-1} was assigned to carbonyl C–O present in carbonyls, ketones, aldehydes or ester groups and to C–C present in olefinic vibrations in aromatic region (Virote et al. 2005; Apaydın-Varol and Pütün 2012). The band at 1,128 cm^{-1} is indicative of the C=O and –CO stretching vibrations which indicate the existence of carbonyl containing groups (Huang et al. 2012). The bands around
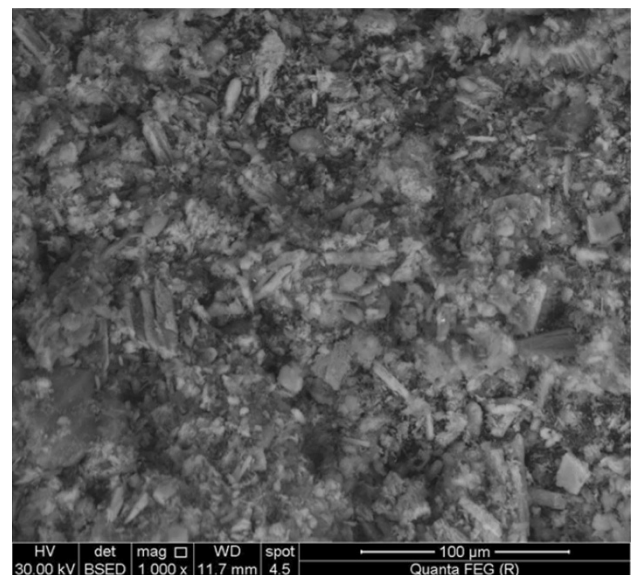

Fig. 2 SEM micrograph of *R. stricta* leaves activated carbon ($\times 1,000$)

600 cm^{-1} was attributed to normal vibration modes of SO$_2$ tetrahedral configuration (Olivares-Marín et al. 2012).

The surface morphology of *R. stricta* activated carbon as obtained by scanning electron microscopy is given in Fig. 2. The adsorbent surface contains particles of different sizes and different shapes indicating that the external surface of the activated carbon is heterogeneous.

Table 2 Removal percentages (%) of phenol by *R. stricta* activated carbon

Factors			Removal of phenol (%)		
T	X	pH	Trial (1)	Trial (2)	Average (%)
−1	−1	−1	69.8	69.8	69.9
−1	−1	1	73.2	73.3	73.2
−1	1	−1	80.2	80.1	80.2
−1	1	1	79.1	78.7	78.9
1	1	1	75.5	75.5	75.5
1	1	−1	84.4	82.0	83.2
1	−1	−1	87.9	82.6	85.2
1	−1	1	75.0	74.4	74.7

Table 3 Estimated effects and coefficients for % removal (coded units)

Term	Effect	Coefficient	Standard error coefficient	T	P
		77.596	0.3689	210.34	0.000
T	4.156	2.078	0.3689	5.63	0.000
X	3.674	1.837	0.3689	4.98	0.001
pH	−4.024	−2.012	0.3689	−5.45	0.001
$T \cdot X$	−4.302	−2.151	0.3689	−5.83	0.000
$T \cdot$pH	−5.076	−2.538	0.3689	−6.88	0.000
$X \cdot$pH	−0.446	−0.223	0.3689	−0.60	0.563
$T \cdot X \cdot$pH	1.863	0.931	0.3689	2.52	0.036

$S = 1.47564$, $R^2 = 95.61$ %, R^2 (adjusted) $= 91.77$ %

Adsorption study

All possible combinations of variables were used, and a matrix was established according to their high and low levels, represented by +1 and −1, respectively (Hegazy et al. 2011). Table 2 gives the design matrix of coded values for the studied factors and the response in terms of phenol removal percentage.

The results of phenol removal using *R. stricta* activated carbon were analyzed using the software MINITAB 15 for windows. The factors affecting the removal of phenol by adsorption onto *R. stricta* activated carbon were evaluated using factorial plots including main effect, interaction effect, the Pareto chart and normal probability plots. Analysis of variance (ANOVA) and *P* value significant levels were used to check the significance of the studied effect on phenol removal percentage.

Analysis of variance (ANOVA)

The main effects of factors (T, X and pH) and the interaction between factors ($T \cdot X$, $T \cdot$pH, $X \cdot$pH and $T \cdot X \cdot$pH) were determined. The effect of a factor is defined as the change in response produced by the change in level of that factor. This is frequently called a main effect as it refers to the primary factor of interest in the experiment (Rathinam et al. 2011).

Table 3 displays the main factors' effect, factors' interaction effect, coefficients of the model, standard deviation of each coefficient and the probability as well as Student's *t* test values for each effect for the full 2^3 factorial design. The significance of the regression coefficients was determined by applying a Student's *t* test (Bingol et al. 2010). The results showed that all factors' effects were significant with 95 % confidence level, with the exception of the interaction effect $X \cdot$pH ($P = 0.563$). The R^2 statistic indicated that the first-order model explained 95.61 % of the variability of phenol removal percentage. The model

also presented an adjusted square correlation coefficient R^2 (adjusted) of 91.77 %, fitting the statistical model quite well.

The codified mathematical model employed for the 2^3 factorial design is given by (Abdel-Ghani et al. 2009):

$$R = A_o + A_1 T + A_2 X + A_3 pH + A_4 TX + A_5 TpH + A_6 XpH + A_7 TXpH \tag{1}$$

where A_o represents the global mean and A_i the other regression coefficients.

Substituting the coefficients A_i in Eq. (1) by their corresponding values from Table 3, phenol removal by *R. stricta* activated carbon could be expressed as:

$$\begin{aligned} R\,(\text{phenol}) = &\, 77.596 + 2.078\,T + 1.837\,X - 2.012\,pH \\ &- 2.151\,T \cdot X - 2.538\,T \cdot pH - 0.223\,X \\ &\cdot pH + 0.931\,T \cdot X \cdot pH \end{aligned} \tag{2}$$

According to Rathinam et al. (2011), when the effect of a factor is positive, removal efficiency increases as the factor is changed from low to high levels. In contrast, if the effects are negative, a reduction in removal efficiency occurs for the high level of the same factor. Thus, it can be concluded from Eq. (2) that a change in the temperature from low to high level results in 2.078 % increase in the phenol removal efficiency. Also, when the adsorbent dosage was increased from low to high value, a 1.837 % increase in the phenol removal was observed. On the other hand, an increase in pH from low to high level resulted in 2.012 % decrease in phenol removal.

The increased phenol removal by adsorption onto *R. stricta* activated carbon observed as the temperature was raised from 25 to 45 °C can be explained on the basis of hydrogen bonding. According to Nayak and Singh (2007), there exists extensive hydrogen bonding between the phenol molecule and water in aqueous solution of phenol resulting in appreciable solubility. This hydrogen bonding

gets broken at higher temperatures and, this would cause phenol to be less soluble and, therefore, exhibit a higher tendency to go to the adsorbent surface and adsorbed rather than remaining in solution.

Regarding the effect of pH on phenol removal by adsorption onto *R. stricta* activated carbon, it has been generally known that the solution pH influences the degree of the ionization of phenolic compounds and surface charge of adsorbents and thereby controls the adsorption processes (Park et al. 2010). Especially, this phenomenon is noted in the case of phenol adsorption on AC (Thinakaran et al. 2008); (Hameed and Rahman 2008). In the present study, there was a slight reduction in the phenol removal percentage with increasing initial pH from 3 to 9. According to Hameed and Rahman (2008), phenol adsorption decreases at high pH values due to ionization of adsorbate molecules. The reason could be also the electrostatic repulsions between the negative surface charge and the phenolate–phenolate anions in solution (Moreno-Castilla 2004). While at acidic pH, the percentage removal was higher because phenol was undissociated and the dispersion interaction predominated (Hameed and Rahman 2008).

The increase in adsorbent weight from 0.5 to 5 g/l resulted in an increase in phenol removal percentage due to the greater availability of the exchangeable sites or surface area of the adsorbent (Kilic et al. 2011).

After estimating the factors' main effects, the determination of the significant factors affecting the removal efficiency was performed by ANOVA. Table 4 shows the sum of squares being used to estimate the factors' effect and the F ratios, which are defined as the ratio of the respective mean-square-effect to the mean-square-error (Bingol et al. 2010). From the P value, defined as the lowest level of significance leading to the rejection of the null hypothesis (Abdel-Ghani et al. 2009), it can be seen that the main factors, two-way interactions and three-way interactions were all significant at 5 % of probability level ($P < 0.05$).

Table 4 Analysis of variance for % removal (coded units)

Source	df	Seq SS	Adj SS	Adj MS	F	P
Main effects	3	187.84	187.84	62.614	28.75	0.000
2-way interactions	3	177.88	177.88	59.293	27.23	0.000
3-way interactions	1	13.88	13.88	13.877	6.37	0.036
Residual error	8	17.42	17.42	2.178		
Pure error	8	17.42	17.42	2.178		
Total	15	397.02				

df degree of freedom, *Seq SS* sequential sum of squares, *Adj SS* adjusted sum of squares, *Adj MS* adjusted mean sum of squares, *F* factor F, *P* probability

The main effects

The mean changes that occur in the response (% of phenol removal) when the levels of each of the main factors were changed from low to high level are presented in Fig. 3. The statistical significance of a factor is directly related to the length of the vertical line (Palanikumar and Davim 2009). It has to be also noted that the larger the vertical line, the larger the change in the response when changing the main factor from level −1 to level +1 (Bingol et al. 2010). Thus, from Fig. 3, it can be concluded that the temperature and adsorbent weight have positive effect on phenol removal by *R. stricta* activated carbon, when both factors are changed from low to high and the opposite is true for the initial solution pH. From the length of the vertical lines, it can be noticed that the adsorbent weight had the lowest effect on phenol removal.

The interaction effects

An interaction is effective when the change in the response from low to high levels of a factor is dependent on the level of a second factor, i.e., when the lines do not run parallel (Mathialagan and Viraraghavan 2005). Figure 4 shows the interaction plots for phenol removal using *R. stricta* leaves activated carbon. It can be seen that there is a strong interactional effect between adsorbent dosage and temperature as well as between temperature and pH. Alternatively, the interaction between the adsorbent dosage and pH was not effective.

As can be seen from Fig. 4, when the temperature is increased from 25 to 45 °C, the phenol removal efficiency increased from 69.85 to 85.24 % at an adsorbent dosage 0.5 g/l and from 80.13 to 83.20 % at an adsorbent dosage of 10 g/L. This result suggests that the effect of temperature is high when the dosage is low but at higher dosage, effect of temperature is small. Similarly, when the temperature is increased from 25 to 45 °C, the phenol removal efficiency increased from 69.85 to 85.24 % at pH 3.0 and from 73.21 to 74.72 % at pH 9.0. Thus, it can be concluded that the effect of temperature is more pronounced at low pH than at high pH.

The Pareto chart

The relative importance of the main effects and their interactions is graphically presented by the Pareto chart given in Fig. 5. The vertical line in Fig. 5 indicates minimum statistically significant effect magnitude for a 95 % confidence level. It was observed that for a 95 % confidence level and eight freedom degrees, the t value was equal to 2.306. This value is in agreement with the values
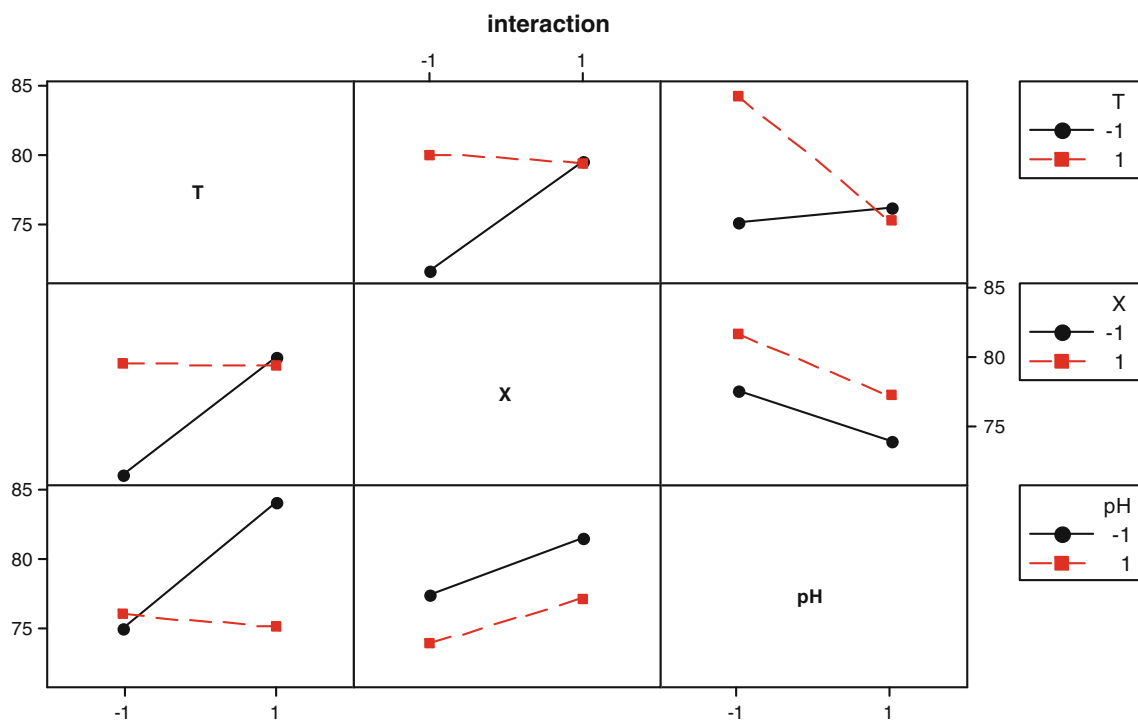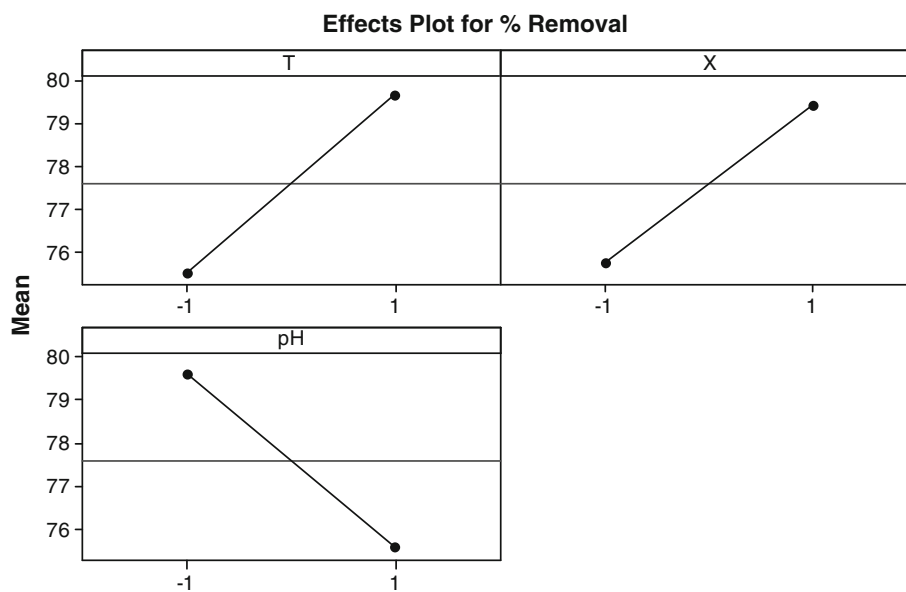
Fig. 4 Interaction effect plot for % phenol removal using *R. stricta* leaves activated carbon

previously reported in the literature (Carmona et al. 2005; Abdel-Ghani et al. 2009; Hegazy et al. 2011). Values shown in horizontal columns are Student's *t* test values for each effect. All the values presenting an absolute value higher than 2.306, which are located right of the vertical line, are significant. Analyzing the Pareto chart, it can be seen that the interaction between the temperature and pH

(*T*·pH) had the greatest effect on the removal percentage of phenol by *R. stricta* activated carbon. The second interaction affecting the phenol removal percentage was the interaction between the temperature and adsorbent dose (*T*·*X*) followed by the temperature (*T*), pH of solution (pH), adsorbent dose (*X*) and finally the interaction among the three main factors (*T*·*X*·pH).

Pareto Chart of the Standardized Effects
(response is % removal, Alpha = 0.05)

Normal Plot of the Standardized Effects
(response is % removal, Alpha = 0.05)

The normal probability

To better evaluate each factor and its interaction, the normal probability plot of standardized effects is presented in Fig. 6. According to Bingol et al. (2010), a normal probability plot is used to identify the "real" effects from those obtained by chance. One point on the plot is assigned to each effect. According to the normal probability plots, the points which are close to a line fitted to the middle group of points represent those estimated factors that do not demonstrate any significant effect on the response variables. Points far away from the line likely represent the "real" factor effects (Brasil et al. 2006; Palanikumar and Davim 2009). The plot (Fig. 6) is divided into two regions: the

region with percentage below 50, where the factors and their interactions presented negative coefficients (pH, $T \cdot X$ and $X \cdot$pH), and the region with percentage above 50, where the factors presented positive coefficients (T, X and $T \cdot X \cdot$pH). It can also be observed that the main factors (T, X and pH) as well as their interactions ($T \cdot X$, $X \cdot$pH and $T \cdot X \cdot$pH) are far away from the straight line and are therefore considered to be "real". The order in which the main factors and their interaction affect the phenol removal percentage by *R. stricta* carbon is as follows: $T \cdot$pH $> T \cdot X > T >$ pH $> X > T \cdot X \cdot$pH. This order is computed based on the distance of each "square" in Fig. 6 from the straight line and considering that when the factor's point lies farthest from the line, this means the factor's effect is higher (Bingol
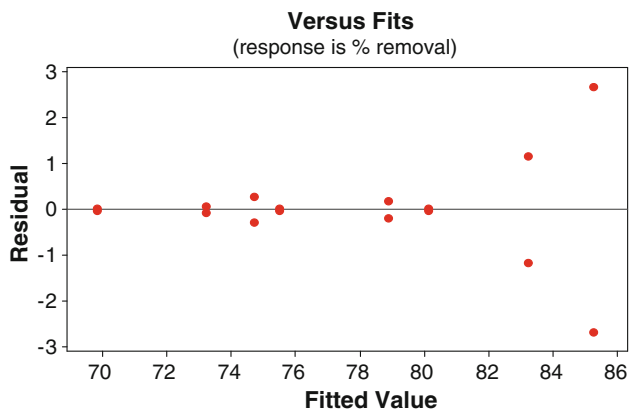
Fig. 7 Percentage phenol removal (predicted) vs. residual

et al. 2010). These results confirm the analysis of Pareto chart (Fig. 5) and the values of Table 3.

Residual analysis

The adequacy of the model was also evaluated by the residuals (difference between the experimental and the predicted values). Residuals are thought as elements of variation unexplained by the fitted model and then it is expected that they occur according to a normal distribution (Meski et al. 2011). The plot of percentage phenol removal predicted versus residual (Fig. 7) did not show any outliers, all the points were found to fall in the range of +3 to −3, which indicated that the model presents a minimal deviation of the fitted value from the observed (Carmona et al. 2005; Rathinam et al. 2011).

Comparison of maximum phenol removal

The maximum removal of phenol by adsorption onto *R. stricta* activated carbon at the optimum conditions was 85 %. In the previous studies reported in the literature, the removal percentage of phenol was found to be 87.6 % on activated carbon prepared from tobacco residues (Kilic et al. 2011); 95.31 % on activated carbon prepared from seaweed (Rathinam et al. 2011) and ranged from 79 to 98 % onto activated carbons prepared from African beech wood sawdust (Abdel-Ghani et al. 2013).

Conclusion

The preparation of a novel activated carbon from *R. stricta* leaves as a cheap and locally available material was successfully achieved. To determine the effects of various operating conditions (pH, adsorbent dose and temperature) and their interactions on the adsorption of phenol by *R. stricta* activated carbon, a full 2^3 factorial design was

performed. ANOVA, *t* test and *F* test showed that the three tested variables (*T*, *X* and pH) as well as their interactions were statistically significant except the interaction (*X*·pH). The prepared activated carbon showed high adsorption efficiency for the removal of phenol from aqueous solution. The present research highlights the use of a cheap activated carbon prepared from *R. stricta*, a locally grown plant for wastewater decontamination.

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