

## Phenol removal from aqueous solution using silica and activated carbon derived from rice husk

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### Abstract

Phenol and its derivatives are organic pollutants with dangerous effects, such as poisoning, carcinogenicity, mutagenicity, and teratogenicity in humans and other organisms. In this study, the removal of phenol from aqueous solution by adsorption on silica and activated carbon of rice husk was investigated. In this regard, the effects of initial concentration of phenol, pH, dosage of the adsorbents, and contact time on the adsorption of phenol were investigated. The results showed that the maximum removal of phenol by rice husk silica (RHS) and rice husk activated carbon (RHAC) in the initial concentration of 1 mgL<sup>-1</sup> phenol, 2 gL<sup>-1</sup> adsorbent mass, 120 min contact time, and pH 5 (RHS) or pH 6 (RHAC) were obtained up to 91% and 97.88%, respectively. A significant correlation was also detected between increasing contact times and phenol removal for both adsorbents ( $p < 0.01$ ). The adsorption process for both of the adsorbents was also more compatible with the Langmuir isotherm. The results of this study showed that RHS and RHAC can be considered as natural and inexpensive adsorbents for water treatment.

**Key words:** adsorption, isotherm, phenol, rice husk activated carbon, rice husk silica

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### ABBREVIATIONS

RE removal efficiency  
SEM scanning electron microscope  
RHS rice husk silica  
RHAC rice husk activated carbon

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### INTRODUCTION

Phenol and its derivatives are among compounds that have been classified as toxic pollutants due to their toxic nature and potentially dangerous effects on human health and the environment, and hence have attracted the attention of many environmentalists (Chaichanawong *et al.* 2010; Rahmani *et al.* 2011; Asmaly *et al.* 2015). Phenol (chemical formula C<sub>6</sub>H<sub>5</sub>OH), which is an aromatic hydrocarbon and a benzene derivative with a very high water solubility, is one of the most commonly used organic compounds in the production process of many industries, such as pharmaceuticals, resin synthesis, plastics, paper, refineries, petrochemicals, leather etc. (Manojlovic *et al.* 2007; Busca *et al.* 2008; Khosravi *et al.* 2012; Moyo *et al.* 2012; Biglari & Bazrafshan 2013; Hashemi *et al.* 2014). Phenolic compounds are organic pollutants with dangerous effects, such as poisoning, carcinogenicity,

mutagenicity, and teratogenicity on humans and other organisms. The World Health Organization has set the maximum permissible concentration of phenol in drinking water as  $0.001 \text{ mgL}^{-1}$  (Mehralipour & Asgari 2015). The US Environmental Protection Agency has identified phenol in the group of top pollutants and set the limit for phenol in industrial wastewater that is discharged into rivers as less than  $0.1 \text{ mgL}^{-1}$ . This organization has also reported that the presence of more than  $1 \text{ mgL}^{-1}$  of phenol in aquatic environments will have a significant impact on aquatic life (Chaichanawong *et al.* 2010). Different methods, such as biological removal, chemical oxidation, photocatalytic oxidation, electrochemical oxidation, the ozonation process, ion exchange, and so on, can be used to remove phenol (Cherifi *et al.* 2009; Daffalla *et al.* 2010; El-Naas *et al.* 2010; Liu *et al.* 2010; Khosravi *et al.* 2013; Mohammad *et al.* 2014; Biglari *et al.* 2016). Most of these methods have some disadvantages. Due to their high removal ability, the biological processes face constraints, such as the slow rate of decomposition and the need for relatively long stagnant periods. Electrochemical processes involve the use of electrical energy and an electrode, which can be defined as a limiting factor (Kulkarni & Kaware 2014). At present, adsorption technology has been widely considered for the removal of organic and inorganic pollutants from aqueous solutions. The adsorption process is the easiest, fastest, most effective, and economical option to remove phenol (Zeng *et al.* 2009; Al-Sultani Kadhim & Al-Seroury 2012; Mehdinia *et al.* 2012; Song *et al.* 2013). Commercial activated carbon is one of the most popular adsorbent materials for the removal of various pollutants due to its high adsorption capacity. However, its high cost and problems with regeneration has limited its use to only developing countries, which has led many researchers to seek more economically viable options. Recently, attention has been paid to the removal of pollutants from water and wastewater by adsorbents obtained from agricultural wastes and some other wastes such as CD and DVD wastes (Pajoohehshfar & Saeedi 2009; Noorimotlagh *et al.* 2019).

The annual rice husk production in the world is about 100 million tons, and in Iran it is about 0.45 million tons. Large quantities of husk are burned, which causes air pollution. Due to its grain structure, rice husk is insoluble in water and has high chemical strength and mechanical persistence (Kazmi *et al.* 2013). Two of the components of rice husk are carbon and silica, which can be converted to silica and activated carbon (Tongpoothorn *et al.* 2011; Ghosh & Bhattacharjee 2013; Cheenmatchaya & Kungwankunakorn 2014). They were used as an adsorbent for removal of different pollutants regarding their suitable adsorption properties such as porous structure and high surface area (Ajmal *et al.* 2003; Yi *et al.* 2016). Also, silica is used as an adsorbent in other forms such as zeolite (Gao *et al.* 2017). However, no scientific results are found regarding the efficiency of the derived adsorbents from rice husk for phenol removal. Therefore, the main purpose of this study was to investigate the applicability of RHS and RHAC in the removal of phenol from aqueous solutions.

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## MATERIALS AND METHODS

This study is an experimental and laboratory-scale study, which was carried out in the Chemistry Laboratory of Water and Wastewater at Babol University of Medical Sciences in 2018.

### Preparation of studied adsorbents

The RHS was prepared according to the Jamwal and Mantri methods (Jamwal & Mantri 2007; Moeinian *et al.* 2019). In this method, raw husk was first washed clean with tap water, and then dried for 24 hours at  $110 \text{ }^\circ\text{C}$ . Then it entered the process of acid leaching. Acid leaching was performed for 50 g of husk with 1 liter mixture of chloridric acid (3%, volumetric) and sulfuric acid (10%, volumetric) for 2 hours. The husk was then washed with distilled water and dried in an oven at  $100 \text{ }^\circ\text{C}$  for 4 hours. Finally, rice husk silica was prepared in a furnace at a temperature of  $800 \text{ }^\circ\text{C}$  for 4 hours

To prepare RHAC, 200 g of rice husk was strained with 1 liter of 3 molar phosphoric acid at 80 °C for 3 hours. Then, the husk was washed with distilled water to reach pH 7. The sample was dried in an oven at 100 °C for 24 hours. After drying, it was carbonated in a furnace at 700 °C for 2 hours (Cheenmatchaya & Kungwankunakorn 2014).

### Batch experiments

In order to carry out the phenol adsorption tests using these adsorbents, a stock solution of phenol was prepared first with a concentration of 1,000 mgL<sup>-1</sup>, and then the samples were prepared at the concentrations of 0.1, 0.2, 0.3, 0.4, and 0.5 mgL<sup>-1</sup> for drawing the calibration curve (Rand *et al.* 1976). All experiments were carried out under the same condition (initial concentration of 5 mg/l phenol, contact time of 60 min, adsorbent dose of 2 g/l and pH for RHAC adsorbent of 6 and for RHS of 5). Each criterion was evaluated as an independent variable. To determine the efficiency of initial phenol concentrations on the RE by the adsorbents, different phenol concentrations of 1, 5, 10, and 15 mgL<sup>-1</sup> (Daraei *et al.* 2014) were studied. To assess the effect of acid and alkaline pH conditions on the phenol removal process at the initial pH, pH levels of 5, 6, 7, 8, and 9 were obtained by adding sulfuric acid and sodium hydroxide 0.1 and 1 molar. To determine the effect of adsorbent dosage, different doses (0.5, 1, 1.5, and 2 gL<sup>-1</sup>) were prepared (Sarker & Fakhruddin 2017). Also, to investigate the effect of time, different contact times (30, 45, 60, 75, 90, and 120 min) were studied (Biglari *et al.* 2016).

After preparing the samples based on the type of variable, the adsorbent concerned was added and placed on the shaker. The mixing and contact procedure was performed at 150 rpm. After the desired contact time, the samples were filtered using filter paper and the phenol concentration was measured in both standard and unknown samples using a UV-Vis spectrophotometer (T80+ model by PG Instruments Ltd, UK), and the calibration curve was plotted at 500 nm wavelength. All the experiments were carried out at a temperature of 25 ± 2 °C and all the tests were performed with three replications. All experiments were carried out three times and the averages were reported as results.

The adsorption capacity was calculated by Equation (1) and the RE from Equation (2).

$$q_e = \frac{(C_i - C_e) \cdot V}{m} \quad (1)$$

where  $q_e$  is the adsorption capacity (mgg<sup>-1</sup>),  $C_i$  is the initial concentration of phenol in the solution (mgL<sup>-1</sup>),  $C_e$  the concentration of phenol after equilibrium (mgL<sup>-1</sup>),  $V$  is the volume of the liquid inside the reactor (L), and  $m$  is the mass of the adsorbent (g).

$$RE = \frac{C_{Gi} - C_{Go}}{C_{Gi}} \times 100 \quad (2)$$

where ( $C_{Gi}$ ) is the initial concentration of phenol (mgL<sup>-1</sup>),  $C_{Go}$  is the secondary phenol concentration (mgL<sup>-1</sup>), and  $RE$  denotes the removal efficiency (%) (Mehdinia *et al.* 2013; Nassehinia *et al.* 2016).

After determining the appropriate conditions for the adsorption analysis of phenol by RHS and RHAC, the Langmuir and Freundlich adsorption isotherms were used to indicate the proper compliance of the isotherms and the adsorption process. For this, the linear models of Langmuir and Freundlich isotherms were used, respectively, in accordance with Equations (3) and (4).

$$\frac{C_e}{q_e} = \frac{1}{q_{\max} \cdot k_l} + \frac{C_e}{q_{\max}} \quad (3)$$

$$\ln q_e = \ln k_f + \frac{1}{nf} (\ln C_e) \quad (4)$$

where  $C_e$  is the equilibrium liquid phase concentration (mol/L),  $q_e$  is the equilibrium state adsorption capacity, which represents the amount of adsorbed lead per amount of adsorbent,  $q_{max}$  is maximum monolayer adsorption capacity (mol/g) and  $k_l$  is Langmuir constant related to energy of adsorption (L/mol),  $k_f$  is the constant of the Freundlich isotherm related to adsorption capacity ((mol/g) (L/mol)<sup>1/n</sup>), and  $n$  is the constant of the isotherm related to the adsorption intensity. The main characteristic of the Langmuir isotherm is the non-dimensional constant called Equation (5).

$$R_L = \frac{1}{1 + k_l C_0} \quad (5)$$

when  $R_L = 0$  the adsorption process is irreversible,  $0 < R_L < 1$  is desirable,  $R_L = 1$  is linear, and  $R_L > 1$  is undesirable.

In the Freundlich isotherm, if the coefficient  $n$  is in the interval 1–10, the isotherm is mathematically desirable. Also, if the value is within the range of 0.1–1, it indicates proper compliance of the Freundlich isotherm, and thus the adsorption process is suitable (Azar & Momeni 2009).

### Data analysis

For data analysis in this research, the Pearson correlation was used to analyze the correlation between contact time and phenol removal efficiency. Meanwhile, descriptive statistics were used to indicate the percentage of removal efficiency and removal capacity.

## RESULTS

### The result of CHNS and SEM

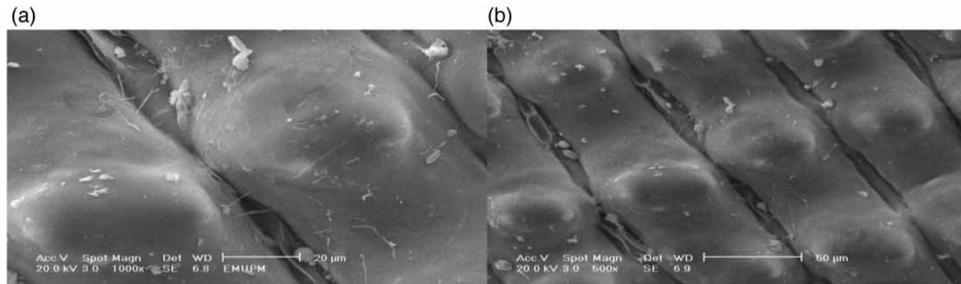
The results of some of the important elemental analysis of CHNS for RHS and RHAC have been presented in Table 1. As is clear in the table, the CHNS measure the carbon, hydrogen, nitrogen, and sulfur content based on complete and instantaneous oxidation of the sample in combustion (Fadeeva *et al.* 2008). Regarding the results, C, N, and H are notably decreased, while S is increased for RHS compared to RHAC. Considerable contents of carbon (22%) in RHAC, and sulfur (0.03%) in RHS show carbonization and silica structure formation in RHAC and RHS respectively. Also the morphologic images of the raw rice husk, RHS and RHAC have been presented in Figures 1–3, respectively. In the figures, deformation of the rice husk and formation of a more porous structure in the RHS and RHAC can be seen.

**Table 1** | The results of CHNS determination of RHS and RHAC

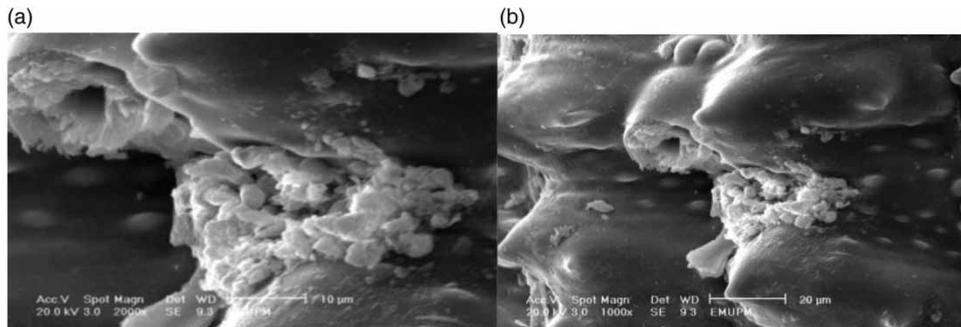
Adsorbents	Elements			
	S(%)	N(%)	H(%)	C(%)
RHS	0.03 ± 0.01	0.36 ± 0.05	0.27 ± 0.01	0.05 ± 0.01
RHAC	0.002	0.71	0.87	22

### The results of the pH effect

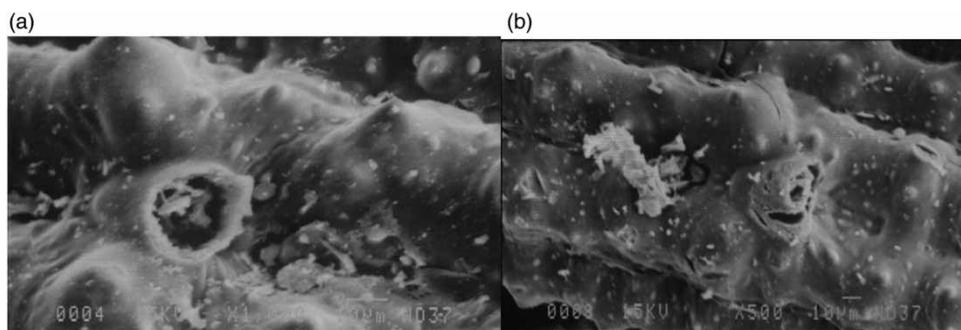
In this study, the effect of initial pH of the solution (range of 5–9) on the RE of phenol by RHAC and RHS (adsorbent dose of 2 gL<sup>-1</sup> in 250 mL of solution with 5 mgL<sup>-1</sup> phenol and a contact time of 60 min) was investigated. According to Figure 4, it has been determined that the best pH for RHAC adsorbent is 6, while for RHS it is 5. The RE for RHAC (pH 6) was obtained as 34.8% and for RHS (pH 5) as 25%.



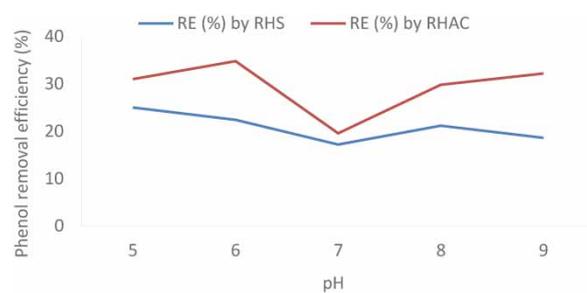
**Figure 1** | Scanning electron microscope (SEM) of raw rice husk with magnification (a) 1,000 and (b) 500.



**Figure 2** | Scanning electron microscope (SEM) of RHS with a magnification of (a) 2,000, and (b) 1,000.



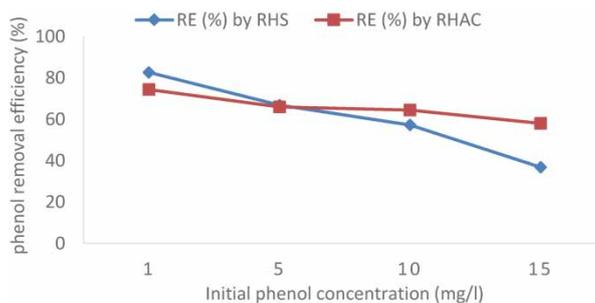
**Figure 3** | Scanning electron microscope (SEM) of RHAC with a magnification of (a) 1,000 and (b) 500.



**Figure 4** | The effect of initial solution pH on the RE of phenol by RHAC and RHS.

### Results of the effect of initial phenol concentrations

In order to investigate the effect of the initial concentrations of phenol on RE, the phenol variable was investigated at different concentrations, keeping the value of other effective factors constant. In [Figure 5](#), phenol RE is provided for RHAC and RHS at pH 6 and pH 5, respectively, with an adsorbent

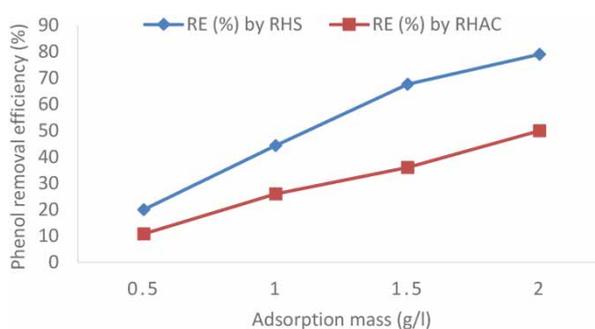


**Figure 5** | The effect of initial concentration of phenol on the phenol RE by RHAC and RHS.

dose of  $2 \text{ gL}^{-1}$ , and contact time of 60 min at different concentrations of phenol (1, 5, 10, and  $15 \text{ mgL}^{-1}$ ). As shown in [Figure 5](#), by increasing the initial concentration of phenol, the RE for both adsorbents decreased. The RE for RHAC at an initial concentration of  $1 \text{ mgL}^{-1}$  is obtained as 74.4%. After increasing the phenol concentration to  $15 \text{ mgL}^{-1}$ , the RE decreased to 58%. The same results were also obtained for RHS; RE at the concentration of  $1 \text{ mgL}^{-1}$  of phenol was 82.6%, while it decreased to 36.8% for  $15 \text{ mgL}^{-1}$  of phenol concentration.

### Effects of adsorbent mass

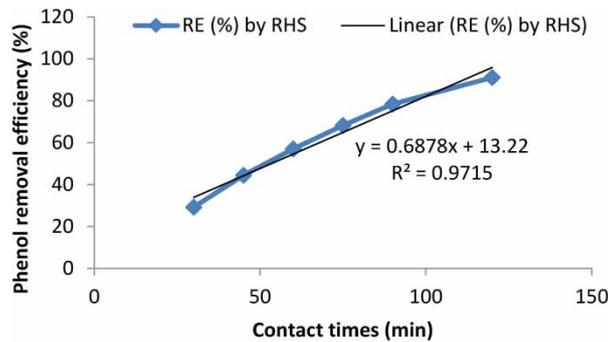
In order to investigate the effect of various doses of adsorbent mass on the RE, the variable of adsorbent mass in different doses was investigated, while keeping the values of other effective factors constant. The results of phenol removal experiments for different doses of RHAC and RHS adsorbents ( $0.5, 1, 1.5,$  and  $2 \text{ gL}^{-1}$ ) at a phenol concentration of  $1 \text{ mgL}^{-1}$ , pH 6 for RHAC and pH 5 for RHS, in a volume of 250 ml, and for a period of 60 min at laboratory temperature, have been shown in [Figure 6](#). According to this graph, with an increase in the amount of adsorbent, the RE increased significantly. For an adsorbent dose of  $0.5 \text{ gL}^{-1}$ , the RE for RHAC and RHS were 10.8% and 20%, respectively. After increasing the amount of adsorbent to  $2 \text{ gL}^{-1}$ , the RE increased to 50% and 79%, respectively.



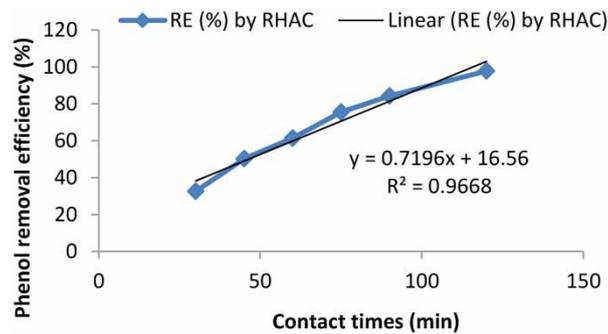
**Figure 6** | The effect of adsorbent mass on the phenol RE by RHAC and RHS.

### The results of the impact of contact time

In order to investigate the effect of different contact times of adsorbents with phenol in aqueous solution, the Pearson correlation test was carried out. The contact time variable was investigated from half an hour to two hours, while maintaining constant values of other factors that affect adsorption. The results from the effect of different contact times (30, 45, 60, 75, 90, and 120 min) at initial concentration of phenol  $1 \text{ mgL}^{-1}$ , adsorbent dose of  $2 \text{ gL}^{-1}$ , and optimal pH for RHAC and RHS (6 and 5, respectively) have been presented in [Figures 7](#) and [8](#). Based on the results presented in these graphs, increasing the contact time resulted in an increase in phenol RE by both adsorbents.



**Figure 7** | The correlation between increasing contact time and phenol RE by RHS at pH 5, adsorbent dosage of  $2 \text{ gL}^{-1}$ , and  $1 \text{ mgL}^{-1}$  of initial concentration of phenol.



**Figure 8** | The correlation between increasing contact time and phenol RE by RHAC at pH 5, adsorbent dosage of  $2 \text{ gL}^{-1}$ , and  $1 \text{ mgL}^{-1}$  of initial concentration of phenol.

The RE for RHAC and RHS at 30 minutes of contact time was 32.6 and 29.2%, respectively. By increasing the contact time to 120 minutes, the RE for these two adsorbents increased to 97.88% and 91%, respectively. The effect of contact time on phenol absorption capacity has also been shown in Figure 5. The results of the correlation test between contact time and phenol RE by RHS and RHAC are shown in Table 2. As shown in Figures 7 and 8 and Table 2, a strong and significant correlation between increasing contact times and RE was detected for both RHS and RHAC adsorbents ( $p < 0.01$ ).

**Table 2** | The results of the correlation test between contact time and phenol RE by RHS and RHAC

Correlation				
$Q_{H_0} > Z_{0.975}$	$Q_{H_0}$	$\omega$	$r$	Adsorbents
4.19 > 1.96	4.19	2.39	0.9856	RHS
4.31 > 1.96	4.31	2.46	0.9834	RHAC

Where  $r$  is the correlation coefficient,  $\omega$  is the confidence interval,  $Q_{H_0}$  is the statistical test index.

### Results of adsorption isotherms

In this study, Langmuir and Freundlich isotherms were used to analyze the adsorption of phenol by RHS and RHAC. The adsorption isotherms were studied using Langmuir and Freundlich models after determining the optimal parameters for phenol removal by RHS and RHAC. Correlation coefficients of the curves for these two adsorption models show a better matching of phenol adsorption on

both adsorbents with the Langmuir isotherm model. The coefficients of these isotherms have been presented in Table 3 regarding the correlation coefficient of the models; phenol adsorption follows the Langmuir model.

**Table 3** | Langmuir and Freundlich isotherm constants on the adsorption of phenol on RHA and RHAC

Isotherm							Adsorptions
Freundlich		Langmuir					
R <sup>2</sup>	n	K <sub>f</sub>	R <sup>2</sup>	K <sub>l</sub>	q <sub>max</sub>	R <sub>l</sub>	
0.8965	3.32	20.7	0.9986	1.27	45.24	0.136	RHS
0.9845	1.64	20.1	0.9981	4.36	103.09	0.043	RHAC

## DISCUSSION

The morphologic images of the raw rice husk, RHS and RHAC by scanning electron microscope (SEM) showed that when the rice husk goes under on acid leaching and incineration at high temperature, it reduces the crystallization of cells and increases its porosity. This phenomenon can thus increase the potential of adsorbents to remove environmental pollutants. Moreover, the results of this study have shown that increasing initial phenol concentration decreases the RE. This may be due to a decrease in the active level in the adsorbent surfaces. In high concentrations, active bands are less available to pollutants and the speed of mass transfer in these concentrations is reduced. This result is consistent with the result obtained from Kassahun *et al.* 2016, who studied the removal of phenol from water using activated carbon derived from the avocado grain. In addition, Pirzadeh & Ghoreyshi 2014 investigated the removal of phenol from aqueous solutions using activated carbon from the sludge of a paper mill. They reported that increasing the initial phenol concentration decreased RE. Another important and effective factor affecting the adsorption efficiency of pollutants is the pH of the environment (Biglari *et al.* 2016). This study has shown that initial pH changes do not apply a constant process in the removal of phenol by RHS and RHAC. However, the greatest removal occurs in an acidic environment. This conclusion is consistent with the results of studies conducted by other researchers as reported by Kavitha 2016, that the percentage of phenol adsorption on the thermally modified activated carbon would decrease with increasing pH. Similarly, in 2009, Pajooheshfar *et al.* obtained similar results in which phenol RE from contaminated water and sewage decreased by increasing pH after using Activated Carbon laboratory (ACL), almond shell carbon (ASC), and walnut shell carbon (WSC) (Pajooheshfar & Saeedi 2009). The study by Khanahmadzadeh *et al.* 2012 on the removal of phenol from aqueous solutions by a silica-aerogel-activated carbon nanocomposite also resulted in the best RE of phenol in acidic pH. By increasing the adsorbent dose, the phenol RE increased, which can be due to the increased availability of adsorbent sites for the phenol present in the solution. Thus, by increasing the amount of adsorbent, more sites are available for the phenol, and therefore more phenol is adsorbed. Sarker *et al.*, who in 2015 studied the removal of phenol from aqueous solution using rice straw as adsorbent, reported a similar result (Sarker & Fakhruddin 2017).

The results showed that phenol RE is increased by increasing the contact time. This shows that contact time and phenol RE in both RHS and RHAC adsorbents are positively and directly correlated ( $p < 0.01$ ). This result is consistent with the result of the study by Daraei *et al.* in 2012, which stated the optimal parameters for the removal of phenol from industrial wastewater using ostrich feather (Daraei *et al.* 2014).

The study of phenolic isotherm adsorption models by RHAC and RHS were found to be more consistent with Langmuir's isothermal adsorption model. Research conducted by Kavitha in 2016 on the removal of phenol by thermally modified activated carbon (Kavitha 2016), and Kassahun *et al.*, on the removal of phenol from water using activated carbon derived from the avocado grain (Kassahun *et al.* 2016), indicate that phenol adsorption is more consistent with the Langmuir isotherm model. Regarding the results, the higher phenol removal for RHAC could be due to the higher adsorption capacity ( $q_{\max}$ ) of RHAC compared to RHS, which is obtained in the Langmuir isotherm. In a study conducted by Pirzadeh & Ghoreyshi in 2014 on phenol removal from aqueous solutions using activated carbon produced from sludge in a paper mill (Pirzadeh & Ghoreyshi 2014), phenol adsorption was found to be more consistent with the Freundlich model. According to the results of this study and similar studies, it can be concluded that it is not possible to provide a single model for adsorbing pollutants by different adsorbents. However, adsorption models follow from pollutants and the characteristics of the adsorbents that are being used.

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## CONCLUSION

The general results of this study have shown that pH plays a key role in the adsorption of phenol by RHAC and RHS. Moreover, RE increases with increasing contact time and adsorbent mass, while it decreases with increasing initial phenol concentration. Maximum adsorption capacity for RHAC and RHS under optimal pH conditions for RHAC (6) and RHS (5), adsorbent mass  $2 \text{ gL}^{-1}$ , initial phenol concentration of  $15 \text{ mgL}^{-1}$ , and contact time 60 min were obtained as  $4.35 \text{ mgg}^{-1}$  and  $3 \text{ mgg}^{-1}$ , respectively, which shows better performance of RHAC compared to RHS. Therefore, RHS and RHAC have good potential in the removal of phenol and can be considered as cheap and effective adsorbents for the removal of phenol from the aqueous environment.

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## ETHICAL ISSUES

The research was approved by the Ethics Committee of Semnan and Babol University of medical sciences.

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## CONFLICT OF INTEREST

The authors declare that they have no conflict of interests.

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## AUTHORS CONTRIBUTION

Seyed Mahmoud Mehdinia and Hossinali Asgharnia contributed to the design of the study along with data analysis, interpretation and preparing the manuscript. Hamidreza Nasehinia, Roohollah Rostami and Marzieh Rahmani contributed to data analysis and interpretation.

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## FUNDING/SUPPORTS

The authors would like to acknowledge Semnan and Babol University of Medical Sciences for the financial support through the project No: 1149.

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