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A comparative study for adsorption of carbolic acid by synthetic resins

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Abstract. Carbolic Acid which is called phenol is one of the important starting and/or intermediate materials in various industrial processes. However, its excessive release into environment poses a threat to living organisms, as it is a highly carcinogens and hazardous pollutant even at the very low concentration. Thus removal of phenol from polluted environments is very crucial for sustainable remediation process. We developed a low cost adsorption method for separating phenol from a model aqueous solution. The phenol adsorption was studied using two adsorbents i.e., Amber lite XAD-16 and Amber lite XAD-7 HP with a constant amount of resin 0.1 g at varying aqueous phenol concentrations (50-200 mg L^{-1}) at room temperature. We compared the efficacy of two phenol adsorbents for removing higher phenol concentrations from the media. We investigated equilibrium and kinetics studies of phenol adsorption employing Freundlich, Temkin and Langmuir isotherms. Amberlite XAD-16 performed better than Amberlite XAD-7 HP in terms of phenol removal efficiency that amounted to 95.52%. Pseudo second order model was highly fitted for both of the adsorption systems. The coefficient of determination (R^2) with Langmuir isotherm was found to be 0.98 for Amberlite XAD-7 HP. However, Freundlich isotherm showed R^2 value of 0.95 for Amberlite XAD-16, indicating that both isotherms could be described for the isotherms on XAD-7 HP and Amberlite XAD-16, respectively.

Keywords: carbolic acid; phenol; Amberlite XAD-16; Amberlite XAD-7 HP; freundlich; langmuir; Temkin

1. Introduction

Waste water management is a major concern for many developing countries, where escalating rate of industrial outflows have substantially polluted the soils and waters with persistent hazardous chemicals. As the industrial waste water contains variety of harmful chemicals, appropriate treatment of waste water is crucial before its discharge, especially if it is discharge into the water used for consumption or agricultural purpose. Thus development of an efficient and economical waste water treatment technique is of utmost importance.

Phenols are one of the extremely hazardous pollutants that could be highly toxic to the environment (plants, animals and human) even in small concentration. Because of widespread use

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of phenols for various industrial processes, it is commonly discharged into ecosystems from the industrial and metropolitan waste water, pesticides industry and oil refineries (Cárdenas *et al.* 2005). Acute exposure of human beings to phenol is extremely toxic due to the rapid skin absorption and subsequent effects on metabolism. Toxic effects on living tissues range from modification in metabolic processes such as chromosomal changes, hemotoxicity, reproductive and developmental toxicity to death of tissues through carcinogenesis (Deshpande and Madras 2010). Considering the widespread use of phenol in industrial operations, and toxicity to living organisms, its separation from wastewater or treatment of wastewater containing phenolic residues become very critical for environmental protection.

Adsorption, solvent extraction, catalytic degradation, chemical oxidation are commonly used methods for separating phenols from wastewater. The processes of liquid phase adsorption have been expeditiously used for separation of colors, organic compound and odors from wastewater (Tseng *et al.* 2003, Wu *et al.* 2005). A diversity of adsorbents used for removal of phenols from aqueous solutions with activated carbon (ACs) (Qadeer and Rehan 2002, Fierro *et al.* 2008). The ACs has been produced from many sources including agricultural wastes (Panumati *et al.* 2008, Rengaraj *et al.* 2002), carbon fibers (Liu *et al.* 2010), water-insoluble cationic starch (Chan and Fu 1997), polymeric XAD-4 (Bilgili 2006) and XAD-16 (Abburi 2003) resins.

Due to their high sorption ability on molecular surfaces, ACs are very effective in adsorption processes (Altenor *et al.* 2009, Zhang *et al.* 2006). In addition, in terms of regeneration capacity, ACs are among the best adsorbents although, a substantial fraction may be lost in each regeneration (Cárdenas *et al.* 2005). Recently, synthetic resins have been used as an alternative adsorbent, due to their easy regeneration and environment friendly nature (Juang *et al.* 1999). Various other alternative adsorbents have also been developed; i.e. polymeric adsorbents with wide surface area and high porosity were used instead of Acs, for selective removal of specific organic compounds from waste water (Zhang *et al.* 2009). Thus there has been great attention on use of polymeric adsorbents for adsorption of phenol. Previous studies were focused on modifying the polymeric materials to improve adsorption capacity and selectivity for an organic compound (Huang *et al.* 2008, 2011). The present study, investigates adsorption isotherm and kinetics of phenol on the two different structured synthetic resin adsorbents namely Amberlite XAD-16 and Amberlite XAD-7 HP. Batch isotherms have been tested and modeled by Langmuir, Freundlich and Temkin isotherms.

Materials and methods

Phenol was obtained from Merck Co., Amberlite XAD-16, and Amberlite XAD-7 HP was obtained from Sigma-Aldrich. Table 1 shows physical properties of adsorbents. Since all chemicals and reagents had an analytical purity they were used without further treatment. The

Table 1 Physical properties of adsorbents

Adsorbent	Dipole moment	Dry density (g/mL)	Surf. area (sq. m/g)	Pore diam, Ave (Angstroms).	West mesh size (somimal)	Pore vol. (mL/g)	$q_{e,max}$
Amberlite XAD-16	0.3	1.08	900	100	20 to 60	1.82	16.10
Amberlite XAD-7 HP	1.8	1.24	450	90	20 to 60	1.14	10.23

adsorbent and 20 mL phenol solution were thoroughly mixed in Erlenmeyer flaks, and adsorption equilibrium was carried out in a thermostatic shaker.

The period of adsorption equilibrium for Amberlite XAD-16 was determined as approximately 40 minutes. The samples have been shaken for 2 hours and the finest adsorbent particles were obtained as 0.1 g for Amberlite XAD-16 from q_e (mg g^{-1}) values. The period of equilibrium state for Amberlite XAD-7 HP was determined as approximately 1 hour. The samples were re-shaken for another 2 hours, and the optimum amount of adsorbent was determined as 0.1 g for Amberlite XAD-7 HP from q_e (mg g^{-1}) values.

Adsorption isotherms have been obtained with the batch adsorption process. Keeping in view of importance of phenolic concentration in adsorption processes, we investigated the phenol adsorption on Amberlite XAD-16 and Amberlite XAD-7 HP at a constant amount of resin 0.1 g and, varying aqueous phenol concentrations (50-200 mg L^{-1}) at room temperature under continuous shaking until equilibrium state. Experimental data obtained from this study were evaluated with Freundlich, Langmuir and Temkin isotherms models.

Adsorption experiments were performed by batch samples were added to several 100 mL volume flasks, a 20 mL aqueous solution containing known concentration of each adsorbate was then added into each flask. Afterwards the flasks were shaken for 2 h at a rate of 120 rpm. At particular time intervals, shaking was halted for a moment, and the sample solution was taken with an injection syringe and analyzed for phenol.

A stock solution of phenol was prepared in deionized water and the phenol solutions with desired concentrations (50-200 ppm) were obtained by successive dilutions of the stock solution. The phenol concentrations in sample solution were analyzed using an UV-is spectrophotometer at 270 nm. Linear calibration curves were developed and used for determining phenol concentrations. These curves were based on standards in the concentration *r* mode in conical flasks. First 0.1 g resin ange from 0-200 ppm.

The amounts adsorbed by resin particles at time *t*, q_t (mg g^{-1}), was calculated by

$$q_t = \frac{V(C_0 - C_t)}{W} \tag{1}$$

Where C_0 and C_t are the liquid concentrations at the start and at time *t*, respectively, *V* the volume of aqueous solution and *W* the mass of resin.

Phenol removal (%) was calculated using the following equation

$$\text{Phenol removal (\%)} = \frac{C_0 - C_t}{C_0} x100 \tag{2}$$

Where C_0 and C_t are the liquid concentrations at the start and at time *t*, respectively.

2. Results and discussion

Effect of emount of adsorbent

Under finest terms of shaking time, the varying dose from 0.1 to 0.5 g of both Amberlite XAD-16 and XAD-7 HP with an initial phenol concentration 117.2 ppm and at 298 K have been studied. The results showed that increasing the adsorbent amount significantly raised the phenol adsorption from aqueous phase on both adsorbent (Table 2). The maximum removals of phenol

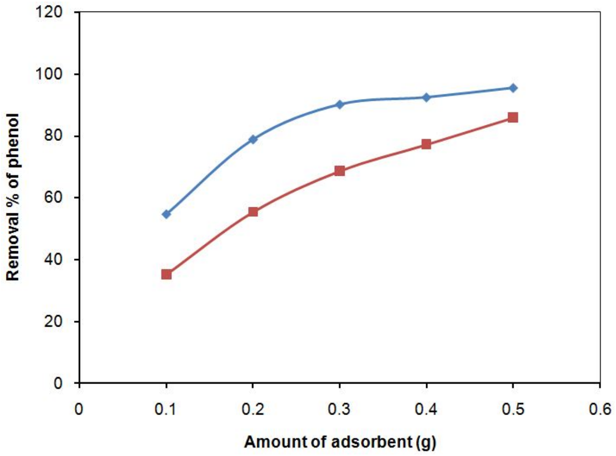

Fig. 1 Plot of the effect of the amount of adsorbent on the adsorption of phenol: ♦ XAD-16; ■ XAD-7 HP

Table 2 Effect of amount of XAD-16 and XAD-7 HP on the adsorption of phenol

Amount of XAD-16 (g)	**C_o (mg/L)**	**C_e (mg/L)**	**q_e**	**Removal (%)**
0.1	117.2	53.28	12.78	54.54
0.2	117.2	24.82	9.24	78.82
0.3	117.2	11.54	7.04	90.15
0.4	117.2	8.84	5.42	92.45
0.5	117.2	5.25	4.48	95.52
Amount of XAD-7 (g)	**C_o (mg/L)**	**C_e (mg/L)**	**q_e**	**Removal (%)**
0.1	117.2	75.99	8.24	35.16
0.2	117.2	52.29	6.49	55.38
0.3	117.2	36.76	5.36	68.63
0.4	117.2	26.71	4.52	77.21
0.5	117.2	16.56	4.02	85.87

(%) determined was 95.52 % and 85.87 % for 0.5 g of XAD-16 and XAD-7 HP of adsorbents, respectively.

Effect of initial phenol concentration

Effect of initial phenol concentrations on the adsorption efficiency was studied using different concentrations of phenol (53.46 ppm, 82.90 ppm, 118.81 ppm, 174.46 ppm) for 0.1 g of each of XAD-16 and XAD-7 HP used as adsorbents. Results have been presented in Table 3. It was obviously shown from Fig. 2 with the raising initial phenol concentration in solution from 53.46 ppm to 174.46 ppm linearly decreased the amount of adsorbed phenol for both adsorbents, resultantly, the adsorption efficiency of XAD-16 and of XAD-7 HP dropped from 55.92% to 46.15% with the higher phenol at initial solution. At the same conditions for phenol adsorption with XAD-7 HP, the efficiencies declined from 54.24% to 29.3%. It showed that the adsorbents were unable to remove higher phenol concentrations from the solutions due to the saturated sites in

Table 3 Effect of initial phenol concentration on the adsorption with XAD-16, and XAD-7 HP

Amount of XAD-16 (g)	C_o **(mg/L)**	C_e **(mg/L)**	q_e	**Removal (%)**
0.1	53.46	23.57	5.98	55.92
0.1	82.90	39.99	8.58	51.76
0.1	118.81	67.10	10.34	43.52
0.1	174.46	93.94	16.10	46.15
Amount of XAD-7HP (g)	C_o **(mg/L)**	C_e **(mg/L)**	q_e	**Removal (%)**
0.1	53.46	24.46	5.80	54.24
0.1	82.90	44.30	7.72	46.56
0.1	118.81	77.87	8.19	34.45
0.1	174.46	123.30	10.23	29.33

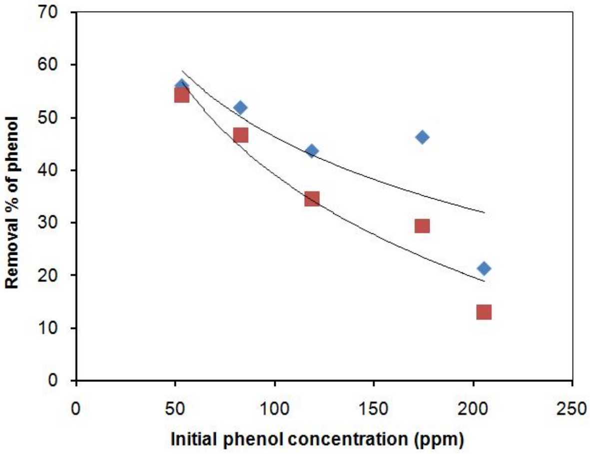

Fig. 2 Plot of the effect of the initial phenol concentration on the adsorption of phenol: ♦ AD-16; ■ XAD-7 HP

structure of the adsorbent. Therefore, selecting appropriate initial phenol concentrations are critical for attaining higher removal efficiency by these adsorbents.

Adsorption isotherms

There are many isotherm to explain characterization of adsorption mechanism in the literature. In this study, we applied Langmuir isotherm, Freundlich isotherm and Temkin isotherm to the experimental data.

The Langmuir equation (Langmuir 1916, Azizian *et al.* 2009)

$$q_e = \frac{K_A . q_m . C_e}{1 + K_A . C_e} \tag{3}$$

q_e indicates the adsorbent-phase concentrations of phenol, and q_m is a particle restricting adsorption capacity as the surface is wholly covered with phenol molecules that aids for comparison of adsorption performance. K_A is the Langmuir constant connected to the closeness of binding sites (L/mg). The K_A and q_m are developed by linearizing the Eq. (3)

$$\frac{C_e}{q_e}=\frac{1}{K_A q_m}+\frac{C_e}{q_m} \tag{4}$$

As seen from Eq. (4), K_A and q_m can be found by intercept and slope of the straight line. Linearized figure of Langmuir equation for XAD-16 and XAD-7 HP, and obtained parameters of Langmuir equation are presented in Table 4.

Freundlich isotherm (Freundlich 1906, Iftikhar *et al.* 2009, Coutrin *et al.* 2008) is the most widely used isotherm equation in the literature for adsorption mechanism. The general equation is

$$q_e = K_f.C_e^{1/n} \tag{5}$$

This equation can be linearized by a logarithmic formed Eq. (6).

$$\ln q_e = \ln K_f + (1/n) \ln C_e \tag{6}$$

Eq. (6) enables to determine constant n and K_f. Slope and intercept gives n and K_f values respectively. Freundlich adsorption isotherms of adsorption of phenol have been drawn for both adsorbents and all results related Freundlich isotherm have been presented in Table 4.

The value of n lies 1.507 and 3.079 for XAd-16 and XAD-7 adsorbents, which represents a favorable adsorption. The adsorption capacity K_f value of 2.107 for XAD-7 was higher than values of 0.723 for XAD-16. As seen from Table 4 Langmuir adsorption capacities for both adsorbent were higher the other equilibrium isotherms. The values present in the form of monolayer adsorption capacity.

Temkin isotherm agrees linearly decreasing of heat on the surface with increasing thickness of the cover of adsorbent layer. It includes a special factor adsorptive-adsorbent interactions (Azizian 2005).

$$q_e = \frac{RT}{b}\ln(K_T C_e) \tag{7}$$

Table 4 Results of adsorption isotherms for both of two adsorbents on adsorption of phenol

Langmuir isotherm					
XAD-16			XAD-7 HP		
q_m	K_A	R^2	q_m	K_A	R^2
32.68	$8.7\text{x}10^{-3}$	0.6823	12.29	0.034	0.9777

Freundlich isotherm					
XAD-16			XAD-7 HP		
K_f	n	R^2	K_f	n	R^2
0.723	1.507	0.9491	2.107	3.079	0.9466

Temkin isotherm							
XAD-16				XAD-7 HP			
B_1	$\ln K_T$	K_T	R^2	B_1	$\ln K_T$	K_T	R^2
6.6443	-2.3564	0.0947	0.8775	2.5235	-0.8753	0.4167	0.9423

Eq. (7) can be linearized as Eq. (8)

$$q_e = B \ln K_T + B \ln C_e \tag{8}$$

$$B = \frac{RT}{b} \tag{9}$$

Due to Eq. (8) is a linear A drawing between q_e and ln C_e, and gives a linear line. Slope and intercept presents the constants B and K_T, respectively. Isotherm parameters are shown in Table 3.

Kinetic studies

Studying adsorption kinetics in wastewater treatment is significantly important, as it provides valuable insights into the reaction pathways and the mechanisms of sorption reaction. Contact time between adsorbent and adsorbate is highly important in adsorption process, and it depends on the nature of the system used.

As a part of kinetic studies, the effect of contact time for the phenol adsorption by Amberlite XAD-16 and XAD-7 HP was studied for a period of 2 hours at initial phenol concentrations of 111.63 mg L^{-1} and 298 K (Table 5). Both adsorbents concentrations used in the solution were kept as 0.1 g. For both adsorbents, it has been observed that the adsorption was faster at beginning of

Table 5 Effect of contact time on the adsorption of phenol with XAD-16, and XAD-7 HP

Amount of XAD-16 (g)	**C_o (mg/L)**	**C_t (mg/L)**	**q_t**	**Removal (%)**	**t (min)**
0.1	111.63	89.185	4.49	20.10	3
0.1	111.63	82.363	5.85	26.22	6
0.1	111.63	72.309	7.86	35.22	10
0.1	111.63	68.000	8.73	39.08	20
0.1	111.63	63.242	9.68	43.34	30
0.1	111.63	60.819	10.16	45.52	40
0.1	111.63	61.716	9.98	44.71	50
0.1	111.63	60.729	10.18	45.60	60
0.1	111.63	60.549	10.22	45.76	90
0.1	111.63	60.998	10.13	45.36	120
Amount of XAD-7 HP (g)	**C_o (mg/L)**	**C_t (mg/L)**	**q_t**	**Removal (%)**	**t (min)**
0.1	111.63	89.185	2.71	12.14	3
0.1	111.63	82.363	3.57	16.00	6
0.1	111.63	72.309	4.44	19.86	10
0.1	111.63	68.000	5.49	24.61	20
0.1	111.63	63.242	6.12	27.42	30
0.1	111.63	60.819	6.39	28.63	40
0.1	111.63	61.716	6.50	29.11	50
0.1	111.63	60.729	6.72	30.08	60
0.1	111.63	60.549	6.68	29.92	90
0.1	111.63	60.998	6.66	29.83	120

the process. Hereafter, the adsorption rate slowed down until reached the equilibrium, and then it proceeded with a constant rate. This is a result of many vacant surface sites at beginning of adsorption. After vacant surface sites is filled by phenol molecules, the rate normally slows down. Then, it is very difficult to fill sites because of repulsive interactions among adsorbent and phenol molecules (Rajoriya *et al.* 2007).

The following equation describes the pseudo second order model (Azizian 2004, Urano and Tachikawa 1991)

$$\frac{dq_t}{dt} = k_s(q_e - q_t)^2 \tag{10}$$

In this equation, k_s is the rate constant (mg.g^{-1}.min^{-1}). When the Eq. (8) is integrated, a linear equation is obtained. Eq. (11) is a linear form of Eq. (10)

$$\frac{t}{q_t} = \frac{1}{k_s q_e^2} + \frac{1}{q_e} t \tag{11}$$

Γ (g.mg^{-1}min^{-1}) is the sorption rate at the beginning, when $t \rightarrow 0$, it is defined as

$$\Gamma = k_s q_e^2 \tag{12}$$

k_s and q_e are obtained from the slope of the plot t/q_t versus t. Γ and k_s are recorded from the intercept. Table 4 presents the pseudo second order model parameters.

Also the Elovich model equation is explained by following equation (Costa and Rodrigues 1985)

$$\frac{dq}{dt} = \alpha.\exp(-\beta.q) \tag{13}$$

In Eq. (13), α is the adsorption rate at the initial stage (mg.g^{-1}.min^{-1}) and β is the desorption constant (g.mg^{-1}). If $\alpha\beta t >> t$ is assumed and the conditions $q = 0$ at $t = 0$ and $q = q$ at $t = t$

Table 6 Results of rate models for both of two adsorbents on adsorption of phenol

Elovich model					
XAD-16			XAD-7 HP		
α	β	R^2	α	β	R^2
13.948	0.620	0.895	5.043	0.853	0.940

Pseudo second order model							
XAD-16				XAD-7 HP			
q_e (mg·g^{-1})	Γ (g.mg^{-1}.min^{-1})	k_s (mg.g^{-1}.min^{-1})	R^2	q_e	Γ (g.mg^{-1}.min^{-1})	k_s (mg.g^{-1}.min^{-1})	R^2
10.548	3.122	0.028	0.999	7.022	1.460	0.030	0.999

***W-M* Intra particle diffusion model**					
XAD-16			XAD-7 HP		
k_{id} (mg·g^{-1}min$^{-1/2}$)	I (mg·g^{-1})	R^2	k_{id} (mg·g^{-1}min$^{-1/2}$)	I (mg·g^{-1})	R^2
0.567	5.388	0.701	0.423	3.039	0.775

equation to simplify

$$q = \frac{1}{\beta}.\ln(\alpha.\beta) + \frac{1}{\beta}.\ln(t) \tag{14}$$

Plotting between q_t and $\ln(t)$ gives a linear relationship with a slope of $(1/\beta)$ and an intercept gives $(1/\beta)\ \ln(\alpha\beta)$. Calculated constants from figure are presented in Table 6.

Weber-Morris intraparticle diffusion model (Weber and Morris 1963) are using with following equation

$$q_t = k_{id}t^{1/2} + \mathrm{I} \tag{15}$$

3. Conclusions

This study shows that an efficient method of phenol adsorption from aqueous solution using Amberlite XAD-16 and XAD-7 HP. Amberlite XAD-16 is more effective phenol adsorbent compared with XAD-7 HP. In similar experimental conditions, XAD-16 removed 95.52 % of phenol compared with XAD-7 HP, which removed 85.87 % of phenol from the aqueous solution. Langmuir isotherm has good agreement with an R^2 = 0.98 for Amberlite XAD-7 HP and the Freundlich isotherm has good agreement with R^2 = 0.95 for Amberlite XAD-16. Elovich, pseudo second order and W-M diffusion models were tried to fit experimental kinetic results. Pseudo second order model showed very good agreement with an $R^2 > 0.99$.

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CC

Nomenclature

C_0	initial concentration of phenol in aqueous solution (mg/L)
$\mathrm{C_e}$	concentration of phenol at adsorption equilibrium (mg/L)
C_t	concentration of phenol at any time t (mg/L)
q_e	the amount of adsorbed phenol per gram of adsorbent at equilibrium (mg/g)
q_t	the amount of adsorbed phenol per gram of adsorbent at any time t (mg/g)
W	mass of the adsorbent (g)
V	volume of the aqueous solution used in adsorption (L)
k_s	pseudo-second-order rate coefficient (mg·g^{-1}·min^{-1})
k_{id}	intra-particle diffusion rate constant (mg·g^{-1}·min$^{-1/2}$)
$t^{1/2}$	the half adsorption time of phenol (min$^{1/2}$)
R^2	linear regression coefficient