

Adsorption of Phenol And Resorcinol on Parthenium Based Activated Carbon (PAC) in Basal Salt Medium: Equilibrium and Kinetics

Khushi Gandhi¹, Shambhatee Srivastav¹, Huda Afreen^{*2}, Ravi Kant Singh^{*1}

¹Amity Institute of Biotechnology, Amity University, Noida, U.P., India.

²Department of Biotechnology, Dr. D. Y. Patil Biotechnology and Bioinformatics Institute Dr. D. Y. Patil Vidyapeeth, Pune, Maharashtra, India.

ABSTRACT

The adsorption studies were carried out for the removal of Phenol and Resorcinol on *Parthenium* Based Activated Carbon (PAC) at 301 K and neutral pH ~7. To determine the adsorption equilibrium isotherms, the experiments were performed at the initial concentration of adsorbates with adsorbent doses from 1 g/l to 10 g/l. The experimental data were fitted to six isothermal models, and a nonlinear least square method has been used to estimate model parameters. It has been observed that for adsorption on PAC, Freundlich model was best fitted for the study and data was more closely related for the removal of both the pollutants from industrial wastewater. In this study, the adsorption kinetics were also studied in three distinct phases-rapid, medium & slow for the time period till 12 hours. The parameter values found in this study shall be useful in designing and activated carbon adsorber for treatment of industrial wastewater at large scale. The present work shows that *Parthenium* based activated carbon (PAC) was an effective method for the adsorption of phenol and resorcinol and p-cresol from aqueous solution. The adsorption of phenol and its derivatives are in the following order: Phenol is adsorbed in maximum amount Resorcinol is comparatively adsorbed lesser than phenol.

KEY WORDS: PHENOL, RESORCINOL, ACTIVATED CARBON, ADSORPTION EQUILIBRIUM ISOTHERM, ADSORPTION KINETICS, ADSORPTION STUDIES.

INTRODUCTION

Adsorption is considered as one of the most preferred and cost-effective methods for treatment of waste water contaminant constituents such Phenol and resorcinol the pollution of industrial wastewater, which can reduce the concentration of pollutants discharged into the plant (Agency for Toxic Substances and Disease Registry (ATSDR), 1990; Daifullah and Girgis, 1998; Mourão, Carrott and Ribeiro Carrott, 2006). Naturally, this will inevitably lead to a decline in water quality, especially causing scarcity of drinking water. These organic compounds in water pollution are mainly caused by heavy industrialization (Tanada et al., 1990; Wu and Tseng, 2006) most scientific research is moving towards an innovative process of implementing environmentally friendly treatments. These organic pollutants are non-biodegradable or poorly biodegradable. Like most pollutants, phenol is harmful, and it has been found in the wastewater of many industrial units (D'Ari

and Barker, 1985; Rathi and Puranik, 2002, Mourão and Ribeiro Carrott, 2006).

Phenol has an unpleasant odor and taste and is toxic to aquatic organisms, plants, and humans at concentrations of 5 µg/L-1 Long-term ingestion of phenol at concentrations between 10 and 240 mg L-1 can cause oral irritation, blurred vision, and diarrhea (World Health Organization (WHO), 1971; Fawell and Hunt, 1988). Therefore, this pollutant often appears in rivers, seawater, industrial waste, urban wastewater, and even groundwater. Among all the methods of treatment of wastewater loaded with organic compounds, the adsorption of phenol on solid supports such as activated carbon, clay, and transition metal oxides allows their removal from water with a high removal rate without the addition of chemicals.

Parthenium hysterophorus (Linn), sometimes known as carrot grass, is an invasive species that was introduced to India through a planned import from the United States (PAC). PAC is made by chemical activation using concentrated H₂SO₄. The produced activated carbon was examined and determined to be a useful adsorbent (Singh et al., 2008). This

Article Information:*Corresponding Author: rksingh1@amity.edu, huda.

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paper highlights the use of parthenium activated carbon's adsorption characteristics for the elimination of pollutants like phenol and others. The work being presented aims to investigate the adsorption of phenol and resorcinol on activated carbon made of parthenium.

MATERIAL AND METHODS

Preparation of adsorbent: Activated carbon act as an effective adsorbent material because of its highly porous nature and provides a large surface area for adsorption (Kadirvelu, Sivasankari, et al., 2002; Singh et al., 2008). There are two preferred ways for the preparation of activated carbon first one is called as physical method and a second method called as chemical method. During the preparation of activated carbon there are several parameters which is required and needed to keep in mind such as temperature and heat duration and pressure. Dried activated carbon is pulverized and sieved to get the particles then the next step for the process of preparation of activated carbon is carbonization also called as pyrolysis which is carried out under heat treatment in the absence of air, Dried activated carbon is then pulverized and sieved to get the particles This leads to the initialization of porosity formation in activated carbons (Ajmal et al., 2006; Singh et al., 2008).

Characterisation of Activated carbon: A high porosity and this microporosity enable great adsorption and selectivity, making activated carbon the most effective adsorbate. Both PAC and GAC forms of activated carbon are offered. Basal zone, heterogeneous groups (mostly oxygen-containing surface groups), and inorganic ash are the typical three zones that make up the AC surface (Kumar et al 2003). Adsorption is only permitted in the mesoporous region when the adsorbent possesses liquid

adsorbate, leading to a greater parthenium activated carbon concentration in the ash. The basal zone contains the majority of the adsorption sites for aromatic chemicals (Li et al., 2002). The chemical properties of the carbon surface are defined by heterogeneous groups, who are more active (Kadirvelu, Sivasankari, et al., 2002). Physical, chemical, and electrochemical processes can change the nature of the surface groups (Pandey & Singh, 2014).

Adsorbate solution: For the preparation of synthetic adsorbate solution of phenol and resorcinol. Phenol and Resorcinol were used each with known concentration of 500 mg/l. And for the stock solution preparation and storage of (1%w/v) for both 10g of phenol and resorcinol were dissolved in 90 ml of water separately to make up the volume to 100 ml each. This solution is stored in cool and well- ventilated area and same way phenol is stored. For the experiment, the solution of desired concentrations was prepared by successive dilution of the stock.

Determination of phenol and resorcinol in a solution : The determination of phenol and resorcinol in a solution was done with the help of UV-vis spectrophotometer onto carbonaceous adsorbent at maximum wavelength of 217nm for phenol adsorption and 273nm wavelength for resorcinol adsorption (model Lambda 35; Perkin Elmer, Massachusetts 02451, U.S.A) (Kumar, Kumar and Kumar, 2003; Ajmal et al., 2006).

Experiment procedure: The experimental studies were carried out to observe the adsorption capacity. The process was carried out as follows: stems of parthenium plants were collected from fellow lands near Amity University Noida campus and then the batch experiment was carried out for the study of adsorption potential of *Parthenium* activated carbon.

Table 1. Major Characteristics of the PAC

S. No.	Properties	Parthenium based activated carbon PAC
1	BET surface area, m ² /g	350
2	pH (1% solution)	6.8
3	Moisture content (%)	2.6
4	Ash content (%)	7.2
5	Bulk density (g/ml)	0.62
6	Average particle size (mm)	0.512
7	Average pore diameter (Ao)	36.74
8	Water soluble matter (%)	3.2
9	Acid soluble matter (%)	3.8

In this adsorption experiment, 10g of resorcinol aqueous solution sample (whose initial concentration and pH were known) was poured to a known adsorbent dose of 100ml in a 500ml stoppered conical flask and shaken a bit then for a constant time period of 24 hr and at varied temperature adsorption was studied with the help of different isotherm models, where adsorption kinetics were studied by keeping

the temperature constant at 301K, and according to the residual concentration of resorcinol and phenol their percent removal were calculated (Kumar et al 2003; Singh et al., 2008).

The waste effluents concentration varies at high range from 1gm to 1000 gm/l and sometimes more, so the experimental

studies the concentration resorcinol taken was 500 gm/l. The adsorbent dose concentrations in each 500 ml flask used ranges from 1gm then 2gm then 3gm and so on to 10 gm at an initial temperature of 301K with initially maintained at neutral pH 7. The samples were taken at some specific time intervals and filtered this study were carried out on Parthenium activated carbon. The quantity of adsorped phenol and resorcinol by 1 gram of adsorbent, q_t (mg/g) at any given time (t) was calculated by (Pandey and Singh, 2014).

$$q_t = [(C_0 - C_t) V] / m \quad (1)$$

where C_0 is the concentration of phenol and resorcinol at the initial time while C_t is the concentration at time t, respectively, V is the volume of adsorbate solution, and m is the mass of adsorbent dose used in the run. At equilibrium condition, the amount of adsorption per gm of adsorbent, q_e (mg/g), was calculated by:

$$q_e = [(C_0 - C_e) V] / m \quad (2)$$

Adsorption equilibrium isotherms: For the study of adsorption different equilibrium isotherms are used. This equilibrium is generally expressed in terms of concentration as of adsorbate (C_e) which is industrial wastewater in liquid state and adsorbent (q_e) on which the process of adsorption occurs at equilibrium conditions which are keeping temperature and pressure constant. These isotherm models help us to study about interaction between adsorbate and adsorbent. In orders to corelate the data six different isotherm models are used which are: Freundlich isotherm model, Langmuir isotherm model, Radke isotherm model, Toth isotherm model, Redlich isotherm models and Fritz isotherm models (Abuzaid & Nakhla, 1996, Li et al., 2001; Nouri, Haghsresht and Lu, 2002).

The Langmuir model equation is expressed as:

$$q_e = (Q_0 b C_e) / (1 + b C_e) \quad (3)$$

here b is the equilibrium adsorption constant. It is a measurement of adsorption affinity and is associated with the adsorption free energy. The quantity of phenol or resorcinol required to completely create a monolayer on the surface of a gram of activated carbon is known as Q_0 , which represents the adsorption capacity (Abuzaid and Nakhla, 1996). This model assumes that all of the activated carbon's surface has the same level of activity. Moreover, it is constrained to a particular concentration range.

The Freundlich, an empirical model, can be expressed in the equational form as:

$$q_e = K_F C_e^{1/n} \quad (4)$$

K_F is the measure of the adsorption capacity and n is a measure of the adsorption intensity This model is applicable to the system involving heterogeneous surface and organic compound (e.g. activated carbon—phenolic compound). The Radke–Prausnitz, Toth, and Fritz–Schlunder models

are not common in use to describe adsorption of phenolic compounds on activated carbon.

Kinetic study of adsorption: Following the rate of adsorption and ultimate equilibrium conditions, the fluctuation in the concentration of unadsorbed or remaining phenol and resorcinol in the solution is almost minimal. Pseudo-first order and pseudo-second order kinetic models are the most used ones for phenolic chemical adsorption on activated carbon (Moreno-Castilla et al., 1995; Abuzaid and Nakhla, 1996). The ability of these two categories of kinetic models to predict the adsorption of phenol and resorcinol on PAC is investigated in this work.

As written, the pseudo-first order kinetic model is:

$$dq_t/dt = k_1(q_e - q_t) \quad (5)$$

where k_1 is pseudo-first order adsorption rate constant (min^{-1}). The model with pseudo-second order kinetics can be expressed as:

$$dq_t/dt = k_2(q_e - q_t)^2 \quad (6)$$

where k_2 is the pseudo-second order adsorption rate constant ($\text{g mg}^{-1} \text{min}^{-1}$). When it is integrated with boundary condition, $q_t(t=0) = 0$ becomes:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (7)$$

Here, q_e and q_t represents the amounts of phenolic compounds studied which are phenol and resorcinol adsorbed per unit mass of adsorbent at equilibrium and at time t, respectively, and can be calculated by using the above equations. Based on the above equations order of kinetics is calculated which shows which order of reaction is followed by the studied interactions of adsorption (Moreno-Castilla et al., 1995; Abuzaid and Nakhla, 1996).

RESULTS AND DISCUSSION

Effect of adsorbent's dosage: In order to know the effect of dose of adsorbent on the adsorption of resorcinol and phenol a number of adsorption experiments were carried out with varying amounts of PAC (1–10 g/l) were contacted with *phenol* and *resorcinol* Solutions. The analysis of this experimental series demonstrates that a greater adsorbent dosage increases the amount of available active sites for adsorption, leading to an increase in removal efficiency (Lata, Garg and Gupta, 2007). The % elimination changes very little incrementally as the dosage of PAC is increased further. Adsorbent dosage is not very effective after 99% removal, and the majority of active sites are left vacant. This causes a decrease in the amount of phenol and resorcinol adsorption decreases with increase in adsorbent dose. This research approximates the ideal adsorbent dosage needed to remove phenol and resorcinol under current operating circumstances when employing activated carbons (Kumar, Kumar and Kumar, 2003).

It takes 10 g/l of PAC dosage. The ideal dose of adsorbent for PAC is then required, which is 10 mg/l, to treat 500 mg/l

of phenol and resorcinol solution since the removal rate is so low. The final percentage of removal is represented in the below mentioned data the final removal percentage of phenol is estimated as 93.6% by 10g/l PAC dose, and the final percentage removal of resorcinol is estimated as 87.2% by 10g/l PAC dose (Pandey and Singh, 2014).

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Table 2. Deviation Percentage for Phenol and Resorcinol in different isotherm models

Isotherm Models	% Deviation (R^2)	
	Phenol	Resorcinol
Freundlich $q_e = KFC^{1/n}$	13.5	11.64
Langmuir $q_e = (q_0 b C_e) / (1 + b C_e)$	50.75	11.09
Redlich–Peterson $q_e = (K_1 C_e) / (1 + K_2 C_e^b)$	13.45	11.67
Toth $q_e = (q^{\infty} C_e) / [a + C_e]^{1/n}$	14.17	11.92
Radke–Prausnitz $1/q_e = 1/KC_e + 1/KC_e^{1/n}$	14.93	9.27
Fritz–Schlunder $q_e = \alpha_1 C_e^{\beta_1} / (1 + \alpha_2 C_e^{\beta_2})$	14.45	11.62

Isotherm studies: By creating a dynamic phase equilibrium between the solid surface of the adsorbent and the liquid adsorbate, equilibrium is studied. This equilibrium is often stated in terms of the adsorbate's concentration and the amount of adsorbate that has been put onto the adsorbent. Adsorbate loading on the adsorbent is shown as (q_e) in adsorption equilibrium isotherms, and the liquid phase concentration of adsorbate is shown as (C_e) at equilibrium while maintaining temperature constant. Adsorption isotherm, then, depicts how adsorbate and adsorbent interact. Many models used to characterise the experimental isotherm data are empirical equations based on the form of the isotherm curve to correlate the isotherm data of this sort, six isotherm models are selected (Li et al., 2002).

The data mentioned below shows a comparative result of all the isotherm models studies. The data mentioned below shows that Freundlich isotherm model is best suited for Phenol PAC system, p-cresol PAC (Moreno-Castilla et al., 1995) system and Resorcinol PAC system whereas Langmuir isotherm model is least for Phenol PAC system and Resorcinol PAC system. To represent the whole concentration range with a single isotherm model, six isotherm models were tested. The correlation coefficient and greatest variation from the anticipated value served as the selection criteria for the isotherm model (Kadirvelu, Sivasankari, et al., 2002).

On this basis, Langmuir model could not fit the data for phenol with the maximum deviation percentage of 50.75 %. but it suited for resorcinol with the maximum deviation percentage of 11.09 %. The results of the Toth and Radke-Prausnitz are nearly similar. The only four parameter isotherm model studied is by Fritz Schlunder. This model gave the similar result to three parameter models with maximum deviation percentage for phenol of 14.45% and for resorcinol of 11.62%.

Kinetic study: Kinetics studies were performed in simple glass flask experiments. The calculated amounts of adsorbate were poured into 500 ml stoppered conical glass flask and activated carbon at a range of 1mg/l to 10mg/l variably was added to each flask. These flasks were kept untouched at 301K. The pre-determined amount of stock solution was added to each flask to obtain desired initial phenol and resorcinol concentration with working solution. The flasks were taken out and filtered one at a time at certain time intervals (Kadirvelu, Sivasankari, et al., 2002). The filtrate was used for phenol analysis. These experiments were done for 24 hours. There was very gradual decrease in concentration after 24 hours keeping the adsorbent dosage at 10 g/l, the temperature at 301 K, and the pH of the solution constant at 7. Using Kinetic equation, the values of (q_t) corresponding to each expected concentration of phenol and resorcinol at various time intervals were determined.

According to the experiments carried on each set of trials, each activated carbon and phenolic interaction, which is a phenol-PAC system, followed a different pace of reaction. Pseudo first order kinetics was followed by pseudo second order kinetics and the resorcinol-PAC system. The estimation of time for the maximum removal of phenolic compounds were with the help of studying diffusion coefficient as the diffusion coefficient determines the time it takes a solute to diffuse a given distance in a medium (Pandey and Singh, 2014). The maximum time taken for phenol-PAC system was 4hrs with the diffusion coefficient $R^2 = 0.975\%$, whereas for resorcinol-PAC system it estimated to be 10hrs, with diffusion coefficient $R^2 = 0.9926\%$.

Effect of Temperature: When the temperature rises, phenol and its derivatives' ability to adsorb substances increases. It was well understood that rising temperature would speed up the rate of adsorbate molecule diffusion through the exterior boundary layer and into the pores of the adsorbent particles, resulting in a reduction in the solution's viscosity (Li et al., 2002). The equilibrium capacity of the adsorbate

for a certain adsorbate will also fluctuate with temperature. The following equations were used to determine the thermodynamic parameters such as free energy change (G), entropy (S) and enthalpy change (H):

$$\Delta G^0 = - R T \ln K_c$$

$$\Delta G = \Delta H - (T \Delta S)$$

$$\ln K_c = \Delta H/RT + \Delta S/R$$

$$\Delta G = + Ve$$

i.e. $\Delta G \propto 1/\text{Temperature}$

K_c = equilibrium constant.

$K_c = C_{as}/C_e$

C_{as} = equilibrium const. (mg/L) of adsorbate on the solution.

C_e = equilibrium const. (mg/L) of adsorbate on the solution.

This relation with free energy changes along with temperature pointed towards the direct proportionality of adsorption capacity with temperature. The positive value of (ΔS) shows the affinity of PAC for phenol and its derivatives (Phenol and Resorcinol). During the adsorption process, some structural changes occur that result in increased randomness at the interface of the solid-solution.

Effect of pH of the solution: The surface charge distribution, which results from the interaction of the surface with adsorbate ions, plays a significant role in the interaction between the adsorbent and the adsorbate. This demonstrates how pH has an impact on the adsorbent's surface charge and the amount of ionisation of the adsorbate, which has an impact on the adsorption phenomena. The initial pH of an aqueous solution, which is defined as the pH at which the surface of activated carbon has zero charge and is thus neutral (Moreno-Castilla et al., 1995; Lata, Garg and Gupta, 2007), is one of the parameters that is frequently examined. As the pH rises over its original pH, the surface of the activated carbon becomes negatively charged and the positive charges are diffused, allowing the adsorption to take place.

Table 3. Effect of Temperature on Phenol, p-Cresol and Resorcinol

S.No.	Adsorbate	Temperature (0K)	ΔG (KJ/mole)	ΔH (KJ/mole)	ΔS (J.K-1 min-1)	R2
1	Phenol	288	2.42	13.24	40.86	0.98
		298	2.04			
		308	1.68			
		318	0.82			
2	p-Cresol	288	3.18	14.02	30.62	0.97
		298	2.67			
		308	1.82			
		318	1.02			
3	Resorcinol	288	4.06	14.46	22.46	0.99
		298	3.46			
		308	2.72			
		318	1.88			

The surface of the activated carbon becomes negatively charged as the pH drops below the starting pH, and the positive ions in the solution are attracted to the surface via electrostatic attraction. Diffusive is now used to help in the adsorption process. The fluctuation in pH has little impact on the effects of pH on the adsorption of phenol and its derivatives. Adsorption isotherm were studies at different pH values. The observed results do not show significant change in the adsorption process at different pH values (AYRANCI and DUMAN, 2005). At low and high pH, their hydrophilic behaviour increases and makes them more surface-active (pH 4 – 9) studies. Preferred pH ~ 7 neutral due to significance difference in adsorption of pollutant.

Effect of Particle Size: The effect of particle size for a const. weight of PAC on the adsorption of Phenol and its

derivatives were carried out. The result reveals that the adsorption is higher on smaller particle size of the adsorbent (Kadirvelu, Senthilkumar, et al., 2002; Rajeshwarisivaraj and Subburam, 2002). The reason for the same is that adsorption is a surface phenomenal, so the greater surface area for adsorption lead to the greater accessibility to pores and hence increased the uptake of smaller particles.

CONCLUSION

The present work shows that Parthenium based activated carbon (PAC) was an effective method for the adsorption of phenol and resorcinol and p-cresol from aqueous solution. The adsorption of phenol and its derivatives are in the following order. Shows the best adsorption of phenol then resorcinol. The generalised Freundlich model

was demonstrated to provide the best correlation for the adsorption for the adsorption of Phenol, Resorcinol and P-cresol onto PAC. The Langmuir isotherm model was found the least corelative for the adsorption studies on the PAC-phenol and PAC- Resorcinol system. As a result, it was stated that the phenol-PAC system, P-cresol-PAC system, and resorcinol-PAC system may all be employed with the Freundlich isotherm. Since activated carbon is a costly substance, repension is crucial.

In contrast, PAC is chosen since it is less costly and made from waste or harmful materials (Rajeshwarisivaraj and Subburam, 2002). In India, parthenium is a widespread weed that is inexpensive or free to purchase in the countryside. According to the aforementioned findings, it can be said that the PAC has outstanding adsorptive properties and may be successfully used to remove phenol and its derivative from industrial effluent (Kadirvelu, Sivasankari, et al., 2002; Rajeshwarisivaraj and Subburam, 2002; Singh et al., 2008). The adsorption rate forms from these experiments may be helpful in developing techniques for phenol and its derivatives' elimination from wastewater, that are both affordable and long-lasting (Singh, Vats and Tyagi, 2011; Singh et al., 2013).

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