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RESEARCH ARTICLE

Phenol removal from synthetic wastewater with powdered activated carbon: Isotherms, kinetics and thermodynamics

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ABSTRACT

Phenol is the 11th most toxic 126 chemical substance and causes cancer by accumulating in the food chain. Adsorption of phenol is an effective and also environmentally friendly method for its removal. In this study, phenol removal by using powdered activated carbon (PAC) was optimized and modeled for various isotherms at constant mixing rate (150 rpm) and sample volume (100 mL); adsorbent dose (0.01-2 g), contact time (1-180 min), and initial phenol concentration (50-1000 mg L^{-1}). Moreover, adsorption studies were carried out at different temperatures for kinetic and thermodynamic calculations. In this study, optimum adsorbent dose and contact time of PAC were determined as 0.3 g 100 mL^{-1} (3 g L^{-1}) and 10 minute, respectively. It can be concluded that it provides discharge standards for a wastewater containing 100 mg L^{-1} phenol. It was observed that the adsorption capacity decreased with increasing temperature and the adsorption process fits well with Langmuir isotherm. It has been concluded that the adsorption of phenol with PAC is an exothermic reaction. As a result of the kinetic studies, it was found to be suitable for the Pseudo Second Order (R^2 0.9999-1.0000). ΔS, ΔH and ΔG were calculated as -0.02 J mol^{-1} K^{-1}, -14.15 kJ mol^{-1} and between -8.16 and -7.76 kJ mol^{-1}, respectively.

Keywords: Adsorption, isotherm, kinetic, phenol, powdered activated carbon (PAC)

1. INTRODUCTION

Phenol (C_6H_5OH), an aromatic compound, is one of the 126 most toxic chemicals. In addition to their toxicity, they cause carcinogenic effect by accumulating in the food chain. The permissible values in water are less than 0.002 mg L^{-1}. Phenol is obtained from coal tar or benzene. It is a common pollutant in wastewater as a result of its widespread use in different industrial fields (pharmaceutical, steel, textile industry, plastics, dyestuff, paper, epoxy, phenolic resin, pesticide, insecticide, petroleum refinery, coal gasification, olive water, etc.). Biological treatment methods (lagoons, aerated stabilization ponds, trickling filters and activated sludge) may be preferred due to their resistance to moderate phenol pollution, while adsorption is preferred for high pollution removal and toxic pollutants [1].

Adsorption is an environmentally friendly advanced treatment method for pollutant removal. Various adsorption studies on phenol removal have been conducted [2-10], especially high removal yields were obtained with commercial adsorbents. Adsorption method provides organic/inorganic pollutant removal as well as taste, colour and odour removal [11]. Phenol is also a compound that causes bad taste and odour in waters [12]. Therefore, the adsorption method provides solutions for many purposes.

In this study, the removal of phenol, which is an important pollutant in industrial wastewater, with powdered activated carbon (PAC) was investigated. PAC is a very active and wide surface area adsorbent to eliminate high concentrations in industrial production plants. For this purpose, at constant mixing speed and sample volume; adsorbent dosage (0.01-2 g), contact time (1-180 minutes), initial phenol concentration (50-1000 mg L^{-1}) were optimized. Different isotherms (Freundlich, Langmuir, Temkin, Elovich, Dubinin-Radushkevich and Redlich-Peterson), kinetics (Pseudo First Order, Pseudo Second Order and Interparticle Diffusion) and

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thermodynamic coefficients were determined by analysing at different temperatures under optimum conditions. Moreover, the adsorption process was evaluated for benefit, cost and risk.

2. MATERIALS AND METHODS

The powdered activated carbon used as adsorbent in this study was obtained from Sigma Aldrich (Activated Charcoal DARCO-100 mesh=<0.142 mm). The powdered activated carbon is commercial and has not been undergone any activation process (pre-treatment). Phenol (> 99% purity) from Sigma Aldrich was used as synthetic wastewater (SWW) (1000 mg L^{-1}). Phenol analyses were carried out according to APHA Standard Methods [13].

100 mL of the phenol solution diluted in the appropriate ratio was put into a 250 mL flask, an appropriate amount of PAC was added and operated at a mixing speed of 150 rpm. As a result of the study, it was filtered through a 0.45 µm filter and phenol analysis was performed. The system used in the study is given in Fig 1.

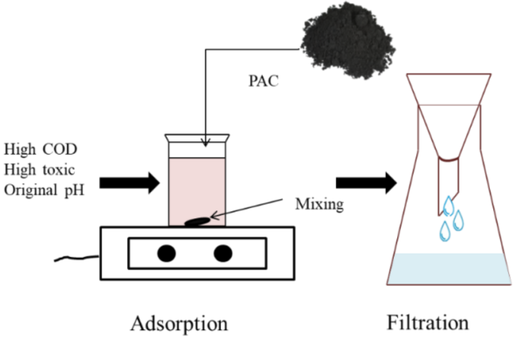

Fig 1. Schematic view of the system used in the study

At constant mixing speed (150 rpm) and sample volume (100 mL), adsorbent dosage (0.01-2 g), contact time (1-180 minutes), initial phenol concentrations (50-1000 mg L^{-1}) were optimized at 298 ºK. Analyses were carried out at 3 different temperatures (298-308-318 °K) under the optimum conditions. Different isotherm (Freundlich, Langmuir, Temkin, Elovich, Dubinin-Radushkevich and Redlich-Peterson), kinetic (Pseudo First Order, Pseudo Second Order and Interparticle Diffusion) and thermodynamic coefficients were determined and the mechanism of adsorption of phenol on PAC was explained.

3. RESULTS

3.1. Optimization

In the first step, the optimization of PAC amount was determined by shaking for 4 hours at 298°K, 150 rpm mixing speed in 100 mL SWW, 100 mg L^{-1} phenol concentration. The results obtained are given in Fig 2. As can be seen in Fig 2, the concentration decreased from 100 mg L^{-1} to 0.12 mg L^{-1} when 0.5 grams of PAC were added, and a large amount of phenol was removed. The removal efficiency is quite high. Even when 0.1 g of PAC was added, the concentration decreased to 15.6 mg L^{-1} and a removal efficiency of 84.4% was achieved. Accordingly, the addition of PAC resulted in high removal efficiencies, even in very small amounts. This high removal may be related to the high activity of PAC and the large surface area. In other words, increasing amount of adsorbent increases active regions for the retention of phenol pollutants and accordingly increases the removal efficiency. The initial activated carbon dosage at which 99% removal is achieved is 0.3 grams. After this value, the increase in removal efficiency was negligible. While the dosage amount can be selected higher, the dosage of 0.3 g 100 mL^{-1} PAC was chosen as the optimum value considering the supply and cost of PAC.

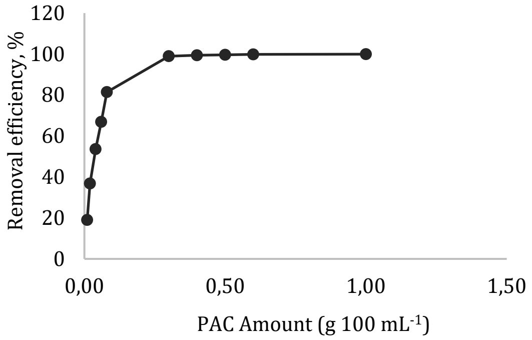

Fig 2. Optimizing the amount of adsorbent (150 rpm, 100 mL SWW, 100 mg L^{-1} phenol, 4 hours, 298°K)

After determining the optimum value of PAC amount (0.3 g 100 mL^{-1}), the optimum the contact time was studied at different reaction times (150 rpm, 298 °K, 100 mL SWW, 100 mg L^{-1}). After 5 minutes, the concentration of the solution decreased to 1.76 mg L^{-1} and 98.2% of the phenol was removed. As time was increased, the removal efficiency increased continuously and most of the phenol was removed within the first 5 minutes (Fig. 3). Subsequently, no significant increase in removal efficiency was observed with the increase in time. In other words, rapid phenol adsorption occurred at first and then slowed down. As a result of the observations, the optimum reaction time was selected as 10 minutes. It can be concluded that the increase of the adherence of phenol on the surface with the increase in the adsorbent amount shows the width of the surface area and the absence of decrease in the efficiency over time shows the suitability of mixing speed. It can also be explained by decreasing the active regions over time and decreasing the adsorption rate [14].

Different concentrations were studied to optimize the pollutant concentration, and in other words, to examine the effect of pollutant concentration on removal efficiency. The concentration range is kept wide (50-1000 mg L^{-1}), since PAC activity can occur at different concentrations in wastewater in industries.

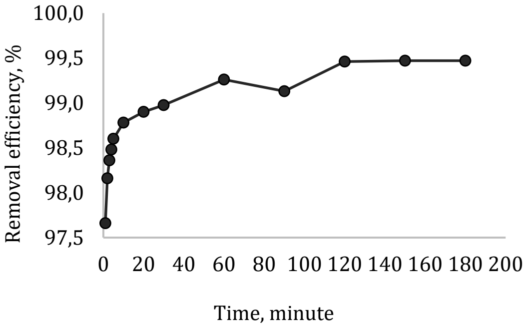

Fig 3. Optimizing contact time (150 rpm, 100 mL SWW, 100 mg L^{-1} phenol, 0.3 g PAC, 298 °K)

When the removal efficiency amounts were compared (Fig 4), the removal rates calculated as 99.0% at 50 mg L^{-1}; 97.7% at 100 mg L^{-1}; 94.7%, 90.0% and 84.5%, respectively, at 200 mg L^{-1}, 300 mg $L^{-1}$and 400 mg L^{-1}; and the efficiency decreased while the concentration increased. When there was a higher phenol content in water, it was more difficult to remove all of the phenol with the same amount of PAC. This was observed at all contact times. The decrease in efficiency as concentration increases indicates that the active area/surface area of the optimally determined 0.3 g is insufficient. In this case, a higher contact time may be provided, or more adsorbents may be added, so that the removal efficiency does not decrease as the initial concentration increases. It is stated that the removal efficiency increased as the concentration of pollutant increases in the adsorption of cationic dyes on the soil since there is not mass transfer limitation/it is the driving force for mass transfer [15]. However, there was no such effect on the removal of phenol pollutant by PAC. The reason for this difference may be related to the type of pollutant and/or type of adsorbent. Moreover, diffusion path may be prolonged for the phenols to reach to open active areas due to the decrease of active areas, because adsorbent is coated with pollutants [16]. This can also be explained by the increase in the adsorption efficiency by increasing the time as a result of prolongation of the diffusion path.

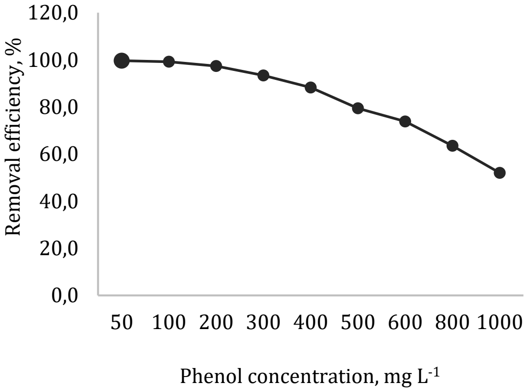

Fig 4. Optimization of phenol concentration (150 rpm, 100 mL SWW, 0.3 g PAC, 10 minute, 298°K)

3.2. Isotherms

Isotherms are equations that give an idea about the adsorption mechanism. Isotherm coefficients were determined for three different temperatures and values are given in Table 1. While temperature generally served as a catalyst for reactions, it had no positive effect on the removal of phenol by adsorption. As the temperature increased, the removal efficiency and adsorption capacity decreased and the best phenol removal was obtained at 298 °K. In the adsorption process, as the temperature increases, it may go away from the adsorbent as a result of the mobility of the pollutant molecules in the water; and a decrease in the amount of phenol retained by the adsorbent was observed as a result. Adsorption capacity decreased with temperature. This shows that the adsorption of phenol with PAC is an exothermic reaction. In a study carried out using wheat bran, the removal of pollutants increased with increasing temperature. It was stated that the temperature increased the active regions in the adsorbent [14]. Furthermore, the solubility of phenol may be increased with increasing temperature and the interaction of the pollutant with the adsorbent (physical bond) may be reduced. In other words, Brownian movement of phenol molecules may be increased [17].

Since R^2 values are very close to 1, it was shown that experimental data can be explained quite well with Langmuir and Elovich isotherms at all temperatures. Other isotherms were observed to vary depending on temperature. The suitability to Langmuir isotherm shows that the surface of the adsorbent is homogeneous, and the adsorbent surface is energetically identical (monolayer). The order of suitability of the isotherms is Langmuir=Elovich>Temkin=Redlich-Peterson>Freundlich>Dubinin-Radushkevich, respectively. The high K_F (Freundlich isotherm coefficient, L g^{-1}) values show the close proximity of the PAC with phenol and the strong relationship between them. The decrease of K_F values with increasing temperature gives an idea that the adsorption capacity decreased. This means that the temperature increase decreases the adsorption rate in the study [18]. In Dubinin-Radushkevich isotherm, the energy value calculated by formula 1 gives information that adsorption occurred as a result of physical or chemical interaction. Since energy (E) values calculated with K_{DR} (Dubinin-Radushkevich isotherm coefficient, (mol^2 J^{-2}) values obtained in the study are between 1-8 kJ mol^{-1}, it was concluded that the interaction between the adsorbent and the pollutant was physical [19].

$$E = \frac{1}{\sqrt{2 * K_{DR}}} \quad (1)$$

The balance factor (R_L) is calculated by the following formula.

$$R_L = \frac{1}{(1+K_L * C_0)} \quad (2)$$

where, R_L is balance factor (unitless) and Co is the initial phenol concentration (mg L^{-1}). The calculated R_L values are given in Table 2. Whereas $R_L>1$, it can be said that the adsorbent is suitable for the adsorption of pollutants. If $R_L=0$, it is irreversible and whereas $0<R_L<1$, it is suitable for adsorption [20]. Since $R_L<1$, it can be said that the interaction between the pollutant-PAC is physical, the adsorption process is favorable and the adsorption occurs spontaneously.

Table 1. Coefficients of different isotherms

Isotherm Models	Isotherm Equations	Isotherm Parameters	298°K	308°K	318°K
Freundlich	$Qe = K_F * Ce^{1/n}$	K_F	29.1455	26.2456	23.4179
		n	0.3714	0.3732	0.4022
		R^2	0.9847	0.9780	0.9626
Langmuir	$Qe = \frac{(b * K_L * Ce)}{(1 + b * Ce)}$	b	0.2531	0.2104	0.1797
		K_L	121.9512	117.6471	117.6471
		R^2	0.9926	0.9931	0.9949
Temkin	$Qe = b_T * Ln(K_T\text{*Ce})$	b_T	19.0600	18.8820	20.4750
		K_T	6.2146	4.7370	3.1215
		R^2	0.9837	0.9913	0.9975
Redlich-Peterson	Ln(Ce/Qe)=β*LnCe-LnA	β	0.6286	0.6268	0.5978
		A	29.1455	26.2456	23.4179
		R^2	0.9946	0.9921	0.9827
Elovich	Ln(Qe/Ce)=LnK_E*Qm-Qe/Qm	Qm	30.5810	30.1205	34.1297
		K_E	0.8630	0.8687	0.8936
		R^2	0.9955	0.9999	0.9924
Dubinin Radushkevich	LnQe=LnQs-K_{DR}*ε^2	Qs	75.5660	75.7170	77.4010
		ε	2.0990	1.7560	1.3650
		K_{DR}	0.1140	0.1620	0.2684
		R^2	0.7680	0.7990	0.8297

Table 2. R_L (equilibrium factor) values calculated for Langmuir isotherm

Inital Concentration (mg L^{-1})	Temperature (°K)		
	298	308	318
50	0.000164	0.000170	0.000170
100	0.000082	0.000085	0.000085
200	0.000041	0.000042	0.000042
300	0.000027	0.000028	0.000028
400	0.000020	0.000021	0.000021

3.3. Kinetics

Kinetic models are used to determine the speed of the adsorption process. Pseudo First Order (Lagergen) and Pseudo Second Order (Ho-McKay) considering the chemical interaction with the results obtained at 298-308-318 °K; and interparticle diffusion coefficients were determined (Table 3). It was concluded that it conforms to Pseudo Second Order kinetics. Pseudo Second Order kinetic model is based on the assumption that it may be a chemical sorption including valence forces through the share or exchange of electrons between the adsorbent and the solution [14].

3.4. Thermodynamics

During the adsorption, heat is generated due to the interaction between molecules attached to the solid surface. Since this heat occurs spontaneously at constant temperature and pressure, the free enthalpy change, in other words, the adsorption free enthalpy ΔG is always minus-signed. As the more irregular molecules in the gas or liquid environment become more stable by holding on the solid surface, the entropy change during adsorption, in other words, the adsorption entropy (ΔS) is always minus-signed. The fact that adsorption free enthalpy and adsorption entropy are always minus-signed requires the adsorption enthalpy in Equation 3 to be always minus-signed.

$$\Delta H = \Delta G + T*\Delta S \quad (3)$$

As the adsorption process is exothermic, the adsorption capacity decreases in parallel with the increase in temperature. As a result of thermodynamic calculations, enthalpy, Gibbs free energy and entropy changes were determined to be minus (-) (Fig 5 and Table 4). The fact that these thermodynamic coefficients are minus (-), shows that adsorption is applicable, it is physisorption, sorption reaction is exothermic/spontaneous (ΔH) and it is less structural change, less random adsorption (ΔS) [19, 21]. Entropy, in other words, irregularity decreased. The reason for this is that it becomes more regular with adsorbed agent accumulation/adhesion in the adsorption process. The fact that ΔH<84 kJ mol^{-1} and ΔG are obtained between -20 and 0 kJ mol^{-1}, means that adsorption occurs by physical interaction [19].

Table 3. Kinetic coefficients

Temperature (°K)	Pseudo First Order		Pseudo Second Order		Interparticle Diffusion	
	k_1 (dk^{-1})	R^2	k_2 (g mg^{-1} dk^{-1})	R^2	Kip (mg gr^{-1} dk$^{-0.5}$	R^2
298	0.2467	0.9595	0.1063	0.9999	0.1028	0.7604
308	0.1849	0.9441	0.0171	1.0000	0.1262	0.8505
318	0.1765	0.9398	0.0175	1.0000	0.0733	0.3232

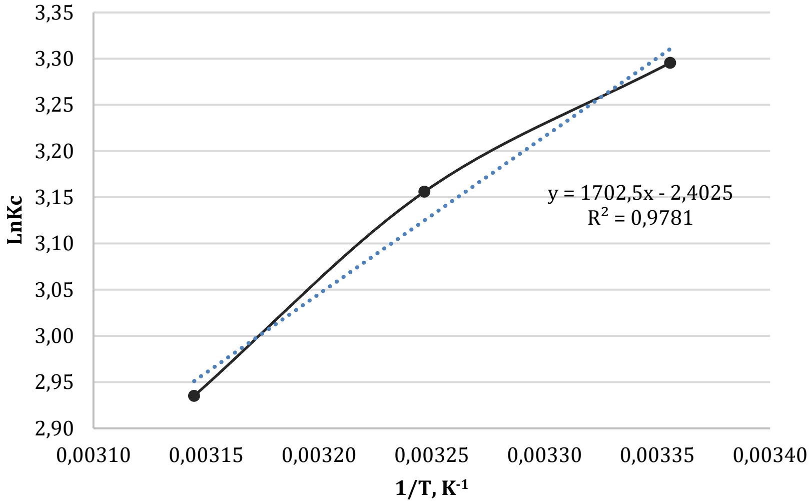

Fig 5. Thermodynamics coefficients

Table 4. Gibbs free, enthalpy and entropy energies

°K	ΔG (kJ mol^{-1})	ΔH(kJ mol^{-1})	ΔS (J mol^{-1} K^{-1})
298	-8.16	-14.15	-0.02
308	-8.08		
318	-7.76		

4. EVALUATION IN TERMS OF BENEFIT, COST AND RISK

Adsorption is one of the advanced treatment methods. It is a physical/physicochemical method based on the adsorption of pollutants on solids. The benefits of adsorption are that it can be used as a pre-treatment and can be used as a post-treatment; it provides recovery of water because it provides high efficiency removal, it is easy to operate and the design of the system is simple. Moreover, biological treatment of a toxic pollutant is difficult to perform while it can be successfully treated by adsorption [22, 23]. PAC has high specific surface area, wide usability, and structural stability at acidic/basic environment and high temperatures and it is an effective adsorbent used for removal of a wide range of pollutants (inorganic, organic, taste, odour, colour, etc.) [24]. Simplicity of the adsorption method is that, it does not require much manpower. Disadvantages are that it is disposable and sometimes causes turbidity [25].

When the cost of adsorption with PAC is taken into consideration, the investment cost is economic because it is not a complicated system. Considering 10 minutes reaction time, 10 years treatment unit life, treatment unit cost 10000 $ m^{-3} [26] and annual interest rate of 4%, The investment cost for treatment with adsorption was calculated as 0.31 $ m^{-3} by operating annual 30000 m^{3} wastewater in 8 hours a day, 300 days a year. The operating cost depends on the price of the adsorbent and the regeneration of the adsorbent. Disposal of the polluted adsorbent is also an element that increases the cost. However, it is not correct to say that there is a big problem because there is a waste that needs to be disposed as solid or liquid in all treatment methods. In the calculation of operating cost, adsorbent, energy consumption and maintenance cost (with 2% acceptance of investment cost) were considered. When the cost of commercial activated carbon is taken as 0.8-1.1 $ m^{-3} (according to optimum value 3 g L^{-1}), maintenance cost was calculated as 0.006 $ m^{-3} (2% of investment cost). The cost of electricity was accepted as 0.133 $ m^{-3} [26]. Thus, total operating cost was determined as 2.54 $ m^{-3} and total cost (investment + operation) was determined as 2.85 $ m^{-3}. PAC cost constitutes approximately 89% of the total cost and investment cost constitutes 11% of the total cost. Considering these data, it is very important to reduce the cost of adsorbent. Magnetic properties can be imparted to the PAC to eliminate the disadvantages such as difficult separation of the adsorbent from the water environment and a secondary pollution. If the adsorbent is magnetized (no necessary regeneration) or if new adsorbents can be produced more economically in commercial activated carbon properties (surface area, pore diameter, high stability, etc.), adsorption is a very economical method.

The adsorption process does not pose a great risk due to the simplicity of operation and no extreme operating conditions. Accidents that may occur during operation and spills, leakage during disposal of polluted adsorbents (especially if they contain hazardous contents) may pose a risk to human and environmental health. The burden of these methods (incineration, landfill) can also be increased with disposal by incineration or landfill of polluted adsorbents [27]. However, there is a waste generated/concentrated in each treatment process and management is required for this waste. The important thing is that the amount of waste is low. For this, much activated adsorbent or magnetic adsorbent should be preferred.

5. CONCLUSIONS AND RECOMMENDATIONS

In this study, the removal of phenol, which is a toxic substance and can cause negative effects on living organisms, by the adsorption method with PAC from water and the parameters affecting the adsorption process were investigated. It was determined that phenol was removed at high efficiency with PAC. Optimum operating conditions were determined as 0.3 g 100 mL^{-1} SWW (3 g L^{-1}), 10 min, and 100 mg L^{-1} phenol. It is found that it is suitable to Langmuir and Elovich isotherms for all temperatures, but it changes according to temperature for other isotherms. It is concluded from isotherm coefficients that adsorbent surface has homogeneous/same energy; adsorption occurs in one layer, interaction between pollutant-PAC is physical and adsorption occurs spontaneously. As a result of kinetic studies, it was found to be suitable for Pseudo Second Order kinetics (R^2>0, 99). As a result of thermodynamic calculations, enthalpy, Gibbs free energy and entropy change were determined to be negative (-). The negative (-) values of these thermodynamic coefficients show that the adsorption was applicable and physisorption; sorption reaction was exothermic/spontaneous; there was less structural change and less random adsorption.

Finally, adsorption is an effective method for phenol removal despite of some disadvantages (particularly adsorbent cost). Total cost (investment + operation) is determined as 2.85 $ m^{-3}. PAC cost constitutes approximately 89% of the total cost and investment cost constitutes 11% of the total cost. The adsorption process is an important alternative that can be preferred for wastewater treatment, especially with the use of low cost adsorbents. Studies were conducted with inexpensive adsorbents or magnetic adsorbents which can be easily regenerated under different operating conditions (pH, effect of different ions, real wastewater, etc.).

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