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Investigation of efficiency of amberlite XAD-16 on adsorption of 2,4,6-trinitrophenol (TNP)

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ABSTRACT

2,4,6-trinitrophenol (picric acid or TNP) is toxic and hazardous material for the environment. It must be treated from any environmental contaminant. In this study, separation of TNP was aimed from aqueous solutions by Amberlite XAD-16 polymeric resin with adsorbtion method. Adsorption of TNP was investigated in terms of equilibrium, kinetics and thermodynamic conditions. For thermodynamic investigations the experiments carried out at three different temperatures (298 K, 308 K, 318 K). In the equilibrium studies, 1.5 g of Amberlite XAD-16 was determined as the optimal amount. The most used isotherms, Freundlich and Langmuir, were applied to the experimental data. The Langmuir isotherm gave good results with R square values about 0.99 at different temperatures. In the kinetic studies, pseudo-first, and pseudo-second order models were applied to the kinetic experiments. The pseudo second order was fitted to this adsorption system with an R square value of 0.987. In the thermodynamic studies, $\Delta H^0_{\text{ads}} = -24.6\ \text{kJ/mol} \cdot \Delta S^0_{\text{ads}} = -209\ \text{J/molK}$ and ΔG^0_{ads} for different temperatures were calculated.

Keywords: Adsorption; Picric acid; Amberlite XAD-16; 2,4,6-Trinitrophenol (TNP)

1. Introduction

Phenolic Compounds in wastewater have been found in many industries, such as chemical, plastic plants, refineries. Especially, nitrophenols enter the environment during manufacturing and processing. Those compounds are toxic even at low concentration. Most goes to water and soil; little goes to the air. It is very important to treat such contaminants from wastewater [1,2].

One of these pollutants is 2,4,6-trinitrophenol (picric acid or TNP). TNP is explosive, very toxic and highly oxidative yellow crystalline solid, also soluble in water, ethyl alcohol, chloroform, most organic solvents. It is derived from phenol through nitration reaction. TNP, itself as well as its metallic salts are highly explosive. Lyddite and melinite, high explosives, are composed mostly of compressed or fused TNP. It is also used as a booster for other explosives and rocket fuel. TNP is also used to make dyes (yellow color), antiseptic agents, external medicines, insecticides (chloropicrin, nitrotrichloromethane), batteries and etching copper as well as a colorimetric reagent to determine creatinine [3].

Several methods have been proposed in literatures on techniques for removal phenolic compounds from wastewater such as photocatalytic, microbial degradation, chemical-biological oxidation and catalytic oxidation process [4–7]. However, the adsorption process

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appears to be the most applicable method for removing trace amount of contaminant from wastewater effluent [8–11]. Adsorption process is broadly used for removal of odor, oil, colours and organic contaminants from liquid-phase system. The potential of granular and powdered activated carbon have been proven as an effective adsorbent used in adsorption technology over the century. It provides large surface area, high adsorption capacity and high degree of surface reactivity [12].

The aim of this study was to investigate adsorption efficiency of Amberlite XAD-16 polyimeric adsorbent for TNP from wastewater streams. The thermodynamic parameters, $\Delta H^0_{\mathrm{ads}}$ $\Delta S^0_{\mathrm{ads}}$ and $\Delta G^0_{\mathrm{ads}}$, were calculated. Regeneration was not within the scope of this study.

2. Material and methods

2.1. Material

2,4,6-trinitrophenol (TNP) (purity> 97%) and Amberlite XAD-16 were obtained from Merck Co. hydrophobic polyaromatic (dipole moment 0.3) used to remove hydrophobic compounds up to 40.000 MW; separation of large organic molecules, especially proteins. It was used without further treatment due to its high purity. Fig. 1 shows scheme of TNP.

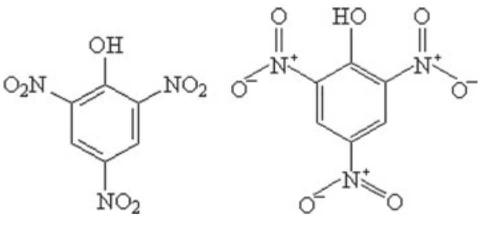

Fig. 1. Different Scheme of TNP.

2.2. Equilibrium methods

Five different concentrations of TNP were prepared for the equilibrium studies. These concentrations were 4.00 g/l, 6.00 g/l, 8.00 g/l, 10.00 g/l, and 14.00 g/l. The concentration 14.00 g/l is the maximum solubility of TNP in water at 298 K[10].

The prepared solutions 15 ml of TNP and different amount of adsorbent was putted to series of Erlenmeyer flaks 100 ml capacity and equilibration studies were carried out in a thermostatic shaker. The preliminary test shows that the period for equilibrium was determined as 90 min. The samples were shaken for 120 min, and the optimum amount of adsorbent was determined as 1.4 g for XAD-16. After equilibration, an aqueous phase sample was titrated to determine the amount of TNP by NaOH (0.1 N) with phenolphthalein as indicator [13,14]. In most cases the deviation between the amount of acid analyzed and the amount of acid known by preparing the solutions by weighing did not exceed 3%. All the adsorption experiments were carried out at room temperature (25 ± 2°C) except where the effect of temperature was being investigated.

2.3. Kinetic methods

The kinetic experiments were carried out in a 100 ml stirred cell. The cell was equipped with a fish agitator and was rotated at high speed (1100 to 1200) rpm to prevent bulk diffusion as a controlling step of adsorption kinetics as suggested by Azizian et al. [15]. The volume of solution was 15 ml and the amount of XAD-16 was 1.4 g as in the equilibrium experiments. The samples of 1 ml of solution were taken from the vessel and analyzed by a titration method with 0.1 N NaOH and phenolphthalein indicator at appropriate time intervals. The kinetic experiments were carried out at a 14.00 g/l concentration of TNP which was the maximum solubility of TNP in water at 298 K and each experiment was repeated twice but the average values were used [14].

3. Results and discussion

3.1. Equilibrium studies

4.00 g/l, 6.00 g/l, 8.00 g/l, 10.00 g/l, and 14.00 g/l of initial TNP concentrations were prepared to investigate effect of initial TNP concentration on the adsorption. It is presented from Table 1 and observed from Fig. 2 for increasing initial acid concentration from 4.00 g/l to 14.00 g/l the adsorbed TNP concentration decreased. The removal efficiency, which is defined as the amount of TNP adsorbed from aqueous solution on to the adsorbent, decreased from 93.00% to 59.42% with increasing initial concentration of TNP. The saturation of accessible exchangeable sites of the adsorbent explains this situation.

Adsorption dose experiments were also performed with 4.00 g/l to 14.00 g/l of TNP solution with the following adsorbent masses 0.5, 1.0, 1.5, 2, 2.5 g at different temperatures 298 K, 308 K, 318 K. The results in Table 1 show that the the adsorption capacity of TNP decreased rapidly with increase in the dose of XAD-16. However, conversion of adsorption capacity to TNP uptake gave an increase in % adsorbed. An increase in TNP uptake from 53.28 to 93% as the dose concentration was increased from 0.5–2.5 g was observed. However, the sorption efficiency showed a reverse trend to the removal percentage adsorptions. The maximum adsorption capacity was reached in 2.5 g of XAD-16, but after 1.5 g of Amberlite XAD-16, the capacity of adsorption increased very slowly. Hence, 1.5 g of adsorbent is the optimum amount for this adsorption study.

Table 1
Experimental results of the adsorption of TNP onto Amberlite XAD-16 at different temperatures (298 K, 318 K, 328 K)

Initial Conc. C_o (g/l)	Amount of XAD-16 (g)	Equilibrium Conc. 298 K C (g/l)	Equilibrium Conc. 318 K C (g/l)	Equilibrium Conc. 328 K C (g/l)	Removal of TNP 298 K (%)	Removal of TNP 318 K (%)	Removal of TNP 328 K (%)
4	0.5	0.93	1.12	1.28	76.75	72	68
4	1	0.71	0.83	1.04	82.25	79.25	74
4	1.5	0.41	0.56	0.78	89.75	86	80.5
4	2	0.35	0.48	0.68	91.25	88	83
4	2.5	0.28	0.42	0.56	93	89.5	86
6	0.5	1.74	1.91	2.04	71	68.16	66
6	1	1.49	1.62	1.66	75.16	73	72.33
6	1.5	1.13	1.3	1.39	81.16	78.33	76.83
6	2	0.98	1.19	1.29	83.66	80.16	78.5
6	2.5	0.89	1.08	1.22	85.16	82	79.66
8	0.5	2.6	2.74	2.94	67.5	65.75	63.25
8	1	2.15	2.37	2.53	73.125	70.37	68.37
8	1.5	1.7	1.97	2.16	78.75	75.37	73
8	2	1.61	1.92	2.06	79.875	76	74.25
8	2.5	1.54	1.83	1.99	80.75	77.12	75.12
10	0.5	3.62	3.87	4.15	63.8	61.3	58.5
10	1	3.14	3.5	3.72	68.6	65	62.8
10	1.5	2.73	3.12	3.31	72.7	68.8	66.9
10	2	2.66	3.05	3.26	73.4	69.5	67.4
10	2.5	2.6	2.98	3.19	74	70.2	68.1
14	0.5	5.68	6.16	6.54	59.42	56	53.28
14	1	5.11	5.47	6.1	63.5	60.92	56.42
14	1.5	4.5	4.85	5.49	67.85	65.35	60.78
14	2	4.42	4.76	5.4	68.42	66	61.42
14	2.5	4.35	4.62	5.33	68.92	67	61.92

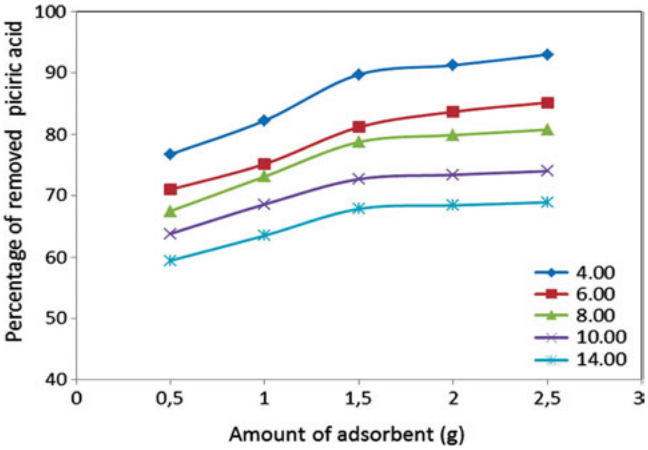

Fig. 2. A plot of effect of initial acid concentration on the adsorption of TNP at 298 K.

3.2. Adsorption isotherms

Langmuir and Freundlich isotherms were studied to find the equilibrium characteristics of adsorption.

The Langmuir equation [15–17],

$$q_A = \frac{K_A Q_0 C_e}{1 + K_A C_e} \tag{1}$$

where, q_A and Q_0 denote the adsorbent-phase concentrations of TNP and saturation capacity, respectively. C_e is the equilibrium concentration of TNP. K_A is the inverse of the Langmuir constant.

The values of K_A and Q_0 are determined by the following equation into which Eq. (1) was transformed.

$$C_e = -K_L + C_e \frac{Q_0}{q_A} \qquad K_L = \frac{1}{K_A} \tag{2}$$

Table 2
Freundlich and Langmuir isotherm parameters of TNP onto Amberlite XAD-16

	Freundlich parameters				Langmuir parameters			
Temperature (K)	Log K_f	K_f (mg·mg^{-1})	n	R^2	1/ Q_0	Q_0	K_L mg/l	R^2
298	1.78	60.25	1.56	0.981	0.0164	60.97	0.0572	0.989
308	1.48	30.20	1.45	0.964	0.0226	42.24	0.0410	0.993
318	1.29	19.50	1.31	0.979	0.0441	22.67	0.0306	0.995

The values of K_L and Q_0 are determined from the intercept and slope of the straight line by plotting a graph between C_e and $\frac{C_e}{q_A}$. The calculated parameters of the Langmuir equation are presented in Table 2.

The Freundlich isotherm was used in this study as a second isotherm [18–20].

$$q_A = K_f C_e^{1/n} \quad (3)$$

A logarithmic plot linearizes the equation by enabling the exponent n and the constant K_f to be determined,

$$\log q_A = \log K_f + (1/n) \log C_e \quad (4)$$

The values of K_f and $1/n$ at different concentrations were determined from the slope and intercept of the linear plots of log q_A and log C_e. The results of the Freundlich equation are presented in Table 2.

The obtained linear plot with a good correlation coefficient confirms that the Langmuir isotherm is a suitable isotherm for adsorption of TNP onto Amberlite XAD-16. The R square value is about 0.99 for each studied temperature in this study. However, the Freundlich isotherm does not obey the results of adsorption at each temperature. Especially, at 308 K some deviations were observed with the Freundlich isotherm.

3.3. Kinetic studies

3.3.1. Effect of contact time

The effect of contact time on the adsorption of TNP by Amberlite XAD-16 was studied for a period of 120 min for initial TNP concentrations of 14.00 g/l at 298 K. The equilibrium was established after 120 min. The Amberlite XAD-16 dosage was 1.5 g. Table 3 presents the data about effect of contact time on removal of TNP and Fig. 3 shows trend of removal of TNP. At the initial stages of the contact period, the uptake of adsorbate species is very fast, and thereafter, it becomes slower near the equilibrium. In between these two stages of uptake, the rate of adsorption was found to be nearly constant. This situation can be explained by many vacant sufrace sites at the initial stage. After initial stage, it is very hard to fill other vacant surface sites because of repulsive forces amoung the solutes and adsorbent bulk [21].

Table 3
Effect of contact time on the adsorption of TNP at 298 K

Initial conc. (g/l)	Amount of amberlite XAD-16 (g)	Equilibrium Conc. Ce (g/l)	Removal of TNP (%)	Time (min)
14.00	1.5	9.5	32.14	15
14.00	1.5	7.71	44.92	30
14.00	1.5	6.35	54.64	45
14.00	1.5	5.63	59.78	60
14.00	1.5	4.78	65.85	75
14.00	1.5	4.43	68.35	90
14.00	1.5	4.31	69.21	105
14.00	1.5	4.23	69.78	120

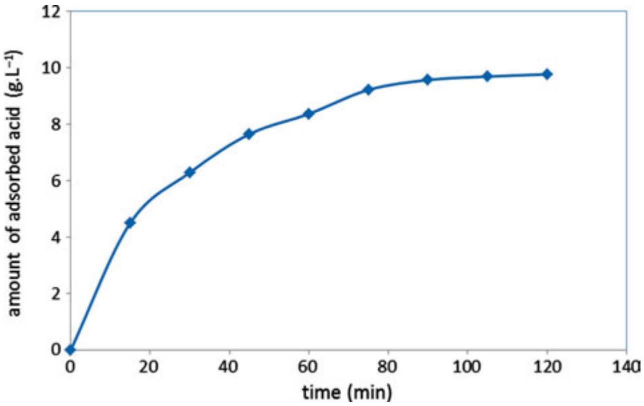

Fig. 3. A plot of effect of contact time on the adsorption of TNP at 298 K.

3.3.2. Adsorption rate

Lagergrensuggested the pseudo-first-order rate for the sorption of solutes from a liquid solution [22].

$$\frac{dq}{dt} = k_1(q_A - q) \quad (5)$$

Integrating Eq. (5) with the boundary conditions t=0 to t=t and q=0 to q=q gives the following equation:

$$\ln\frac{(q_A - q)}{q_A} = -k_1 t \quad (6)$$

where q and q_A are the grams of solute absorbed per gram of sorbent at any time and at equilibrium, respectively, and k_1 is the rate constant of first-order sorption. The pseudo-first-order equation was used extensively to describe the sorption kinetics [23,24].

If the adsorption kinetics obey a pseudo-first-order model, then a plot of ln (q_A–q) versus t should be linear. A plot for adsorption of TNP onto Amberlite XAD-16 was shown in Fig. 4. The constants of Eq. (7) were obtained from Fig. 4 at different concentrations and listed in Table 4 with the coefficients of determination, R^2. As is seen clearly from Fig. 4 and Table 4, the fitting of the experimental data to the pseudo first-order model was not so good. Although the coefficients of determination are good as can be seen in Fig. 4 there is a deviation from this model especially at elevated times when t is more than 75 min.

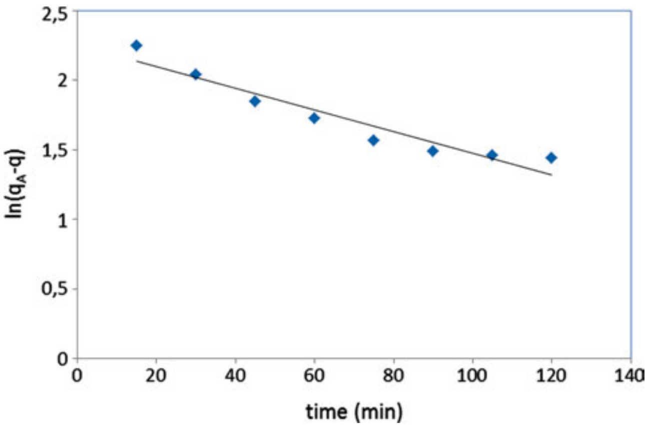

Fig. 4. The linear plot of pseudo first order kinetic model at 14 g/l initial TNP concentration and 1.5 g Amberlite XAD-16.

Another model for the analysis of sorption kinetics is the pseudo-second-order model. The rate law for this system is expressed as

$$\frac{dq}{dt} = k_2(q_A - q)^2 \quad (7)$$

Integrating Eq. (7), for the boundary conditions t=0 to t=t and q=0 to q=q, gives

$$\frac{1}{\left(q_A - q\right)} = \frac{1}{q_A} + k_2 t \quad (8)$$

where k_2 is the pseudo-second-order rate constant of sorption. Eq. (8), can be rearranged to obtain a linear form,

$$\frac{t}{q} = \frac{1}{k_2 q_A^2} + \frac{1}{q_A} t \quad (9)$$

A plot of $\frac{t}{q}$ versus t gives a straight line with slope of $\frac{1}{k_2 q_A^2}$ and intercept of $\frac{1}{q_A}$. So the gram of solute sorbed per gram of sorbent at equilibrium (q_A) and sorption rate constant (k_2) can be evaluated from the slope and intercept, respectively. The pseudo-second-order model was recently applied to the analysis of sorption kinetics from liquid solutions by Ho et al. [25,26].

The values of q_A and k_2 were obtained from the slopes and intercepts of plots in Fig. 5. These constants with the coefficients of determination are listed in Table 4. As can be seen in Fig. 5 this model fits these experimental results very well.

3.4. Effect of temperature

The adsorption of TNP from aqueous solution was investigated at different temperature to learn effect of

Table 4
Obtained constant for pseudo first order and pseudo second order kinetic models at 14 g/l initial TNP concentration

Pseudo-first-order kinetic model			Pseudo-second-order kinetic model		
q_A(g/g)	k_1(min^{-1})	R^2	q_A(g/g)	k_2 (g/g min)	R^2
1.42x10^{-1}	0.0132	0.918	2.54x10^{-1}	60.31	0.987

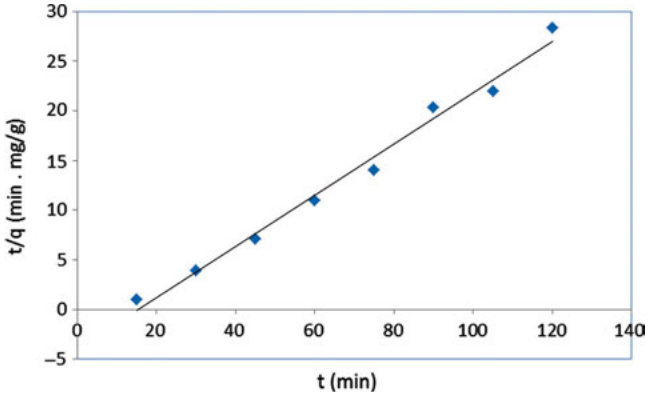

Fig. 5. The linear plot of pseudo second order kinetic model at 14 g/l initial initial TNP concentration and 1.5 g Amberlite XAD-16.

temperature in this adsorption system. The removal efficiencies was presented in Table 1 at three different temperatures (298 K, 308 K, 318 K). It can be seen from the experimental results the adsorption capacity of the XAD-16 is decreasing with increasing temperature.

As related to the temperature effect, the thermodynamic parameters have been calculated for this adsorption system. The free energy change of adsorption ΔG^0_{ads} was calculated by using the equation:

$$\Delta G^0_{\text{ads}} = -RT \ln K_L \qquad (10)$$

where R is the universal gas constant and T is the Kelvin temperature. K_L is the thermodynamic equilibrium constant for the adsorption process. It was determined by plotting ln (C_e/q_A) versus C_e and extrapolating to zero C_e as suggested by Khan and Singh [27].

The other thermodynamic parameters, like the enthalpy change ΔH^0_{ads} and the entropy change ΔS^0_{ads} were calculated from the slope and intercept of the plots of ln K_L against $1/T$ according to the following equation:

$$\ln K_L = \frac{\Delta S^0_{\text{ads}}}{R} - \frac{\Delta H^0_{\text{ads}}}{RT} \qquad (11)$$

ΔH^0_{ads} was obtained from the slope of the straight line and ΔS^0_{ads} was determined from the intercept of the graph [28,29]. In order to evaluate the thermodynamic equilibrium constant K_L, the C_e/q_A values were plotted versus C_e values at 298 K, 318 K and 328 K. Linear graphs were obtained for all temperatures. The obtained K_L parameters were used to calculate the ΔG^0_{ads} function [30]. The calculated thermodynamic parameters (ΔH^0_{ads}, ΔS^0_{ads} and ΔG^0_{ads}) at different temperatures are given in Table 5. ΔH^0_{ads} and ΔS^0_{ads} were obtained from plots of ln K_L versus $1/T$ as shown in Fig. 6.

Table 5
Thermodynamic parameters for adsorption of TNP onto Amberlite XAD-16 at different temperatures (298 K, 308 K, 318 K)

Temperature (K)	ΔG^0_{ads} (kJ/mol)	K_L (mol/l)	ΔS^0_{ads} (j/molK)	ΔH^0_{ads} (kj/molK)
298	−37.666	2.497x10^{-7}	−209	−24.6
308	−39.783	1.790x10^{-7}		
318	−41.848	1.336 x10^{-7}		

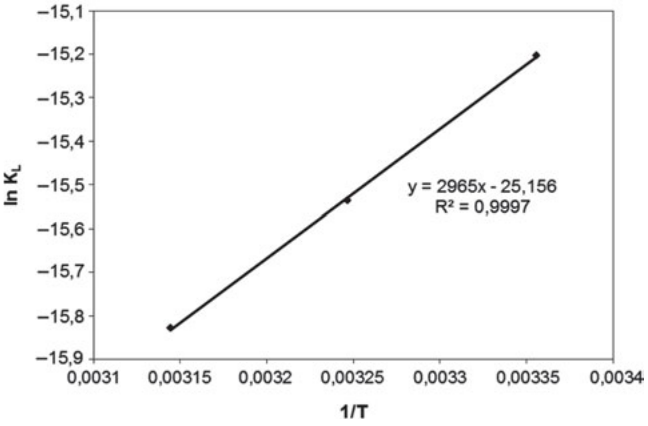

Fig. 6. Plot of ln K_L against $1/T$.

4. Conclusions

Batch studies showed the dependence of adsorption on characteristics of adsorbent and adsorbate (TNP). The removal of TNP from aqueous solutions strongly depended on amount of adsorbent, initial TNP concentration, and contact time. For TNP the maximum adsorption was obtained with 2.5 g adsorbent mass at 298 K temperature. In this adsorbent mass the maximum removal of TNP was 93%. The applied isotherms especially the Langmuir isotherm showed good results to predict data with an R square value of about 0.99 at different temperatures. The reaction rate was found as pseudo-second order. Thermodynamic studies show that this adsorption proces is exothermic (ΔH^0_{ads} = −24.6 kJ/mol).

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