

Adsorption-Desorption of BTX (Benzene, Toluene and O-Xylene) on Fe, Fe-Al Pillared Clay

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Abstract

The studies are conducted in laboratory to determine the adsorption-desorption behavior of BTX (benzene, toluene and o-xylene) in gas phase on Fe, Fe-Al pillared clays adsorbents. In experimental conditions of constant atmospheric pressure, initial concentrations with an increasing volume (0.5 - 2)ml) injected benzene (2.25), toluene (1.89) and o-xylene (1.66) µmol/L at T (40°C, 60°C and 80°C), and the adsorption increases with increase of temperature, indicating that the adsorption process would be a chemical adsorption rather than physical one. The results are shown that the BTX adsorption data fitted very well ($R^2 > 0.999$) to the both equations Langmuire and Elovitch for the three samples: bentonite (B), Fe-bentonite (Fe/B) and Fe-Al/bentonite (Fe - Al/B). At 80°C, the BTX adsorption capacity increased in the following order: B < Fe/B < Fe - Al/B. The maximum adsorption capacity (q_m) at 80°C is 175.13, 171.84 and 171.81 µg/g respectively for benzene, toluene and o-xylene for Fe - Al/B; the last is a good adsorbent of BTX removal. The benzene diffuses faster than toluene and o-xylene. Thermodynamic parameters, such as ΔG° , ΔH° and ΔS° are also discussed and the results suggested that the BTX adsorption on all samples used is a spontaneous and endothermic process. Desorption studies show that BTX is very easily desorbed with Fe - Al/B.

Keywords

Fe, Al-Pillared, Benzene, Toluene, O-Xylene, Adsorption

1. Introduction

The natural gas and oil industry activities are known for some time to create harmful air emissions that emit volatile organic compounds (VOCs) and oxides of nitrogen (NO_x), which are precursors to tropospheric ozone. The major

VOCs, the benzene, toluene and xylene (BTX) volatile compounds are significant environmental concern and are listed as priority pollutants by the United States Environmental Protection Agency (US EPA) [1] because of their toxic and carcinogenic effects on humans. Besides, the benzene is already known as the leukemia agent in humans [2]. Another, VOCs characterized by their photochemical activity could undergo a series of photochemical reaction to from the secondary organic aerosol, which is one of the major components of airborne fine particles. The VOCs cause environmental concerns about their toxicity and malodor, even at very low concentrations, due to the obvious impacts on atmosphere and human health; it is necessary to limit and control this air emission. The difficulty, for decreasing the VOCs in gas phase at very low concentrations, requires a highly optimized process. Various processes can be used for abatement of VOCs which are broadly classified into two types: destruction (biofiltration, thermal oxidation, catalytic oxidation, reverse flow reactor) and recovery (adsorption, condensation, membrane separation) [3]. The adsorption by solid adsorbents is one of the best solutions for this treatment; the choice of adsorbent depends on these adsorptive properties and availability. Granular or powdered actived carbon is the most widely used adsorbent [4], but their use is usually limited due to their high cost. Over several decades, many researchers show their interests in searching for low-cost adsorbents with excellent adsorption characteristics, such as zeolites [5], organokaolinite [6], smectite [7], hectorite [8], organosilica [9], and montmorillonite [10] [11]. The pillared interlayer clay (PILC) attracts attention of many researchers, and constitutes one of the most widely studied series among the microporous materials with a wide range of potential applications in adsorption processes. PILCs are formed by insertion of polynuclear inorganic cation into their interlayer space, followed by calcinations to give stable metal oxide pillars (e.g. Al₂O₃, Fe₂O₃ etc.) having larger micropores. The purpose of this paper is to present the effectiveness of (Fe, Fe-Al)-pillared bentonite clay adsorbents to reduce the concentration of BTX and determine behavior of BTX with evaluating the influence of the temperature on BTX adsorption. Adsorption isotherm is measured at three different temperatures: 40°C, 60°C and 80°C.

2. Materials and Methods

2.1. Materials

The natural clay used in this work is a bentonite type from Maghnia (west Algerian). It is supplied by the Algeria Bentonite Company (ENOF). The natural bentonite is purified in laboratory [12] [13], using a sedimentation method to obtain the < 2 μ m montmorillonite rich faction. The carbonates are removed by sodium acetate/chloridric acid, iron oxide by sodium thiosulfate/sodium chloride and organic materials by hydrogen peroxide (30% vol.). To ensure complete transformation into the sodium from all samples, they are washed several times with 0.5 M NaCl. The exchange capacity or CEC 91 meq/100g (by methylene bleu exchange).

2.2. Preparation of Hydroxyl-Al

The pillaring solution of Al and Fe polycation are prepared separately [13]. 0.207 M NaOH solution is added slowly while stirring to a 0.207 M AlCl₃ solution until it reached an OH/Al^{3+} molar ratio of 2.5 in the mixture. The mixture is aged at room temperature during 6 days at room temperature.

2.3. Preparation of Hydroxyl-Fe

Fe polycations solution is prepared by slowly adding a 0.1 M NaOH solution to 0.1 M FeCl₂ solution under vigorous stirring, until the OH/Fe^{3+} ratio reaches the value 2.5. The mixture is aged for two weeks at room temperature [13].

2.4. Preparation of Fe-Al Pillared Bentonite

The pillaring solution containing hydroxyl-Al oligocations and hydroxyl-Fe oligocations are slowly added under vigorous stirring into the suspension purified bentonite while, until the mass ratio of M^{3+} ($M^{3+} = Fe^{3+}$, Al^{3+})/caly reached 6.25% [13]. The solids are filtered and washed with deionized water until it are free of Cl⁻ ions. The solids B, Fe/B and Fe-Al/B are calcined for 2 h at 300°C ground and sieved.

High-purity BTX: benzene (99 wt.%, Aldrich), toluene (99.5 wt.%, Aldrich) and o-xylene (99 wt.%, Aldrich) are used absorbate.

2.5. Characterization

The Analysis of the chemical composition of the purified bentonite is obtained fluorescence X. The surface area is measured with a Micromeritics ASAP 2010 instrument by adsorption of nitrogen at 77 K. Before measurement, the samples are degassed under vacuum of 20.8 Pa at 120°C for 2.

2.6. BTX Adsorption Kinetics

1 g of the samples in the nacelle is placed in glass enclosure (10 L), closed and thermostated degassed for 2 h using the means of a water-jet pump. Then, 2 ml of BTX (benzene, toluene and o-xylene) containing respectively, 2.25, 1.89 and 1.66 µmol/L is sprayed into the enclosure by injection (Figure 1). After, the nacelles are removed from the enclosure and the samples are weighed. The experiments are carried out at 40°C, 60°C and 80°C in a temperature controlled bath (Figure 1).

2.7. Equilibrium Isotherm

1 g of the samples in the nacelle is placed in glass enclosure (10 L), closed and thermostated degassed for 2 h using the means of a water-jet pump. Then, different volume ranging from (0.5 - 2 ml) is sprayed into enclosure by injection of liquid BTX: benzene, toluene and o-xylene with initial concentration respectively 2.25, 1.89 and 1.66 µmo/L. The experiments are carried out at 40°C, 60°C and 80°C in a temperature controlled bath (Figure 1). After reaching the adsorption equilibrium, the nacelles are removed from the enclosure and the samples



Figure 1. Experimental set-up for the adsorption of the BTX.

weighed by Sartorius 1219 MP balance type (accuracy $\pm 10^{-3}$ g). The amount of adsorbed BTX on adsorbents ($q_e \mu g/g$) is calculated as follows:

$$q_e = \frac{\left(C_0 - C_e\right) \cdot V}{m} \tag{1}$$

where C_0 and C_e are the initial and equilibrium BTX concentrations (µg/L), respectively; V is initial liquid volume (L) equal to glass enclosure volume; and m is the adsorbent weight (g).

2.8. Desorption Experiments

For desorption experiments, the nacelle in glass enclosure is subjected before to degassing for 2 h at constant pressure. 1 g of the samples are saturated in benzene, toluene and *o*-xylene of concentration 2.25, 1.89 and 1.66 μ mol/L respectively are desorbed for 2 h and at temperature $T(40^{\circ}C, 60^{\circ}C \text{ and } 80^{\circ}C)$ (Figure 1). The amounts of BTX retained are obtained from the difference between the initial concentration (C_0) and the final concentration (C_t), desorption rate is calculated as from Equation (2):

$$\tau = \frac{\left(C_0 - C_t\right)}{C_0} \cdot 100 \tag{2}$$

3. Results and Discussion

3.1. Characterization of the Adsorbent

The chemical composition of purified bentonite by X-ray fluorescence is reported in **Figure 2**, the results collected in **Table 1** showed the silica to alumina ration (SiO_2/Al_2O_3) confirms the montmorillonite variety as an almost exclusive component of our purified clay.

The N_2 adsorption/desorption isotherm of purified bentonite is shown in **Figure 3**. The adsorption isotherm is of type IV according to (B.D.D.T) classification, which is generally associated with capillary condensation in mesopore structures, with a well-defined H4 hysteresis loop. This behavior is the indication of a mono-multilayer adsorption on slit-shaped pores among plate-like particles [14]. The opening behaviors of the hysteresis loop indicated the formation



Figure 2. Spectrum of purified bentonite by X-ray fluoresence.

Table 1. Chemical analyses of purified bentonite by X-rays fluoresce	ence
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Corresponding Ovide	Purified ber	ntonite
Corresponding Oxide	Ζ	Concentrations
Na ₂ O	11	3.98
MgO	12	3
Al_2O_3	13	22.74
SiO ₂	14	61.68
P_2O_5	15	0.072
SO ₃	16	0.181
Cl	17	0.282
K ₂ O	19	1.3
CaO	20	0.192
TiO ₂	22	0.349
Cr_2O_3	24	1.1
Fe ₂ O ₃	26	5.175
NiO	28	0.00892

of irregular shape pores. This opening demonstrated the presence of mesopores in the purified bentonite. The inset of Figure 3 is the pore size distribution of the bentonite purified, in which different volume is plotted against pore size for the desopriton branches of the N₂ adsorption/desorption isotherms according to the BJH model [15]. The results are given in Table 2, for purified bentonite the total pore volume and micropore volume are 0.103 and 0.027 cm³/g, respectively. The increase of the specific surface area (Table 2) after pillaring with Fe, Fe-Al polycation solutions, suggest also the increasing of the micropores [16].

3.2. Adsorption Kinetics of BTX on Purified and Pillared Bentonite

Figure 4 presents the kinetic curves of all samples used at different temperatures.





Figure 3. N_2 adsorption/desorption isotherms and pore size distribution of the purified bentonite.

Table 2. Textural properties of the samples used calcined at 300°C.

Sample	Specific surface area (m ² /g)	Specific surface area (BJH) (cm ² /g)	Total pore volume (cm ³ /g)	Micropore volume (cm ³ /g)
В	99.78	100.21	0.103	0.027
Fe/B	177.00	-	-	-
Fe – Al/B	355.00	-	-	-









Figure 4. Adsorption kinetics of BTX onto samples used at various temperature.

It can be seen that the adsorption capacity increased with contact time, quickly in the first 15 min and then increased gradually with increasing contact time until the adsorption reached adsorption equilibrium at 3 h. It can also be observed that the lower the temperature is, the lower the saturated adsorption capacity is. When the adsorption reached the equilibrium at 80°C, the adsorption capacity (q_e) is 178.26 µg/g (benzene), 175.50 µg/g (toluene) and 170.56 µg/g (o-xylene) on Fe – Al/B; 172.95 µg/g (benzene), 160.15 µg/g (toluene) and 159.76 µg/g (o-xylene) on Fe/B; 139.70 µg/g (benzene), 121.33 µg/g(toluene) and 119.01 µg/g (o-xylene) on B. Generally, adsorption is exothermic process; the adsorption capacity would be expected to be decreasing with temperature.

The results showed that the adsorption capacity increased with an increase of temperature, indicating the adsorption process is endothermic and the adsorption process would be a chemical adsorption rather than a physical one.

3.3. Equilibrium Isotherms

For to assess efficacies for the three adsorbents: B, Fe/B and Fe – Al/B; at constant temperature, the quantity of BTX adsorbed onto pillared clay will be in equilibrium with BTX in the gas phase and the adsorption equilibrium data for these adsorbents are fitted by the saturated monolayer isotherm can be represented by Langmuir isotherms:

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

where C_e (µg/L) is the equilibrium concentration; q_e (µg/g) is the equilibrium amount of BTX adsorbed; q_m (µg/g) is a maximum adsorption capacity, K_L (L/µg/) is the adsorption equilibrium constant. Equation (4) can be linearized into the form as follows:

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

The results obtained by the applying the Freundlich model is not presented because the low values correlation coefficients ($R^2 < 0.99$) show poor agreement of Freundlich isotherm with the experimental data.

The adsorption isotherms are presented in **Figure 5**, from the results shown in **Table 3** where, the Langmuir constants q_m and K_L are determined from the slope and intercept of the plot Equation (4) (**Figure 6**); the $R^2 > 0.999$ values for all adsorbents suggest that the Langmuir isotherms provides a good model of the BTX adsorption, it is observed good fit of the Langmuir equation to the experimental data (**Figure 6**). The capacities of the aromatics compounds for BTX adsorption are significantly dependent on the temperature and the nature of polycation pillaring (Fe/B and Fe – Al/B), although the values of K_L and q_m increase when the temperature is increased about 60°C to 80°C (**Table 3**). These results also suggest that the BTX interaction must be an endothermic process. The positives ΔH^0 (**Table 4**) value confirms that the adsorption process is endothermic for BTX, which is an indication of the existence of a













Parameters	Langmuir					Elovitch			
	$T(^{\circ}C)$	Sample	$q_{\scriptscriptstyle m}$ (µg/g)	K_{L} (L/µg)	R^2	$q_{\scriptscriptstyle m}$ (µg/g)	$K_{_E}$ (L/µg)	R^2	
	40	В	70.180	0.325	0.99	66.40	0.0149	0.99	
		Fe/B	93.540	0.017	0.99	91.32	0.0010	0.99	
		Fe-Al/B	123.609	0.384	0.99	115.74	0.0086	0.99	
	60	В	117.500	0.425	0.99	107.17	0.0093	0.99	
Benzene		Fe/B	131.750	0.421	0.99	128.35	0.0078	0.99	
		Fe-Al/B	157.480	0.340	0.99	149.70	0.0066	0.99	
		В	136.990	0.474	0.99	129.61	0.0015	0.99	
	80	Fe/B	168.067	0.559	0.99	160.51	0.0062	0.99	
		Fe-Al/B	175.130	1.089	0.99	170.35	0.0058	0.99	
		В	68.400	0.273	0.99	64.107	0.0155	0.99	
	40	Fe/B	83.750	0.325	0.99	80.19	0.012	0.99	
		Fe-Al/B	104.710	0.323	0.99	99.18	0.0099	0.99	
	60	В	111.111	0.154	0.99	105.59	0.0094	0.99	
Toluene		Fe/B	127.880	0.194	0.99	121.21	0.0082	0.99	
		Fe-Al/B	142.850	0.275	0.99	139.66	0.0071	0.99	
	80	В	121.950	0.314	0.99	119.62	0.0083	0.99	
		Fe/B	155.040	0.149	0.99	149.03	0.0067	0.99	
		Fe-Al/B	171.840	0.481	0.99	164.74	0.0060	0.99	
		В	62.420	0.153	0.99	58.82	0.0016	0.99	
	40	Fe/B	78.430	0.102	0.99	71.42	0.014	0.99	
		Fe-Al/B	99.110	0.110	0.99	92.59	0.011	0.99	
	60	В	90.900	0.147	0.99	87.71	0.011	0.99	
O-xylene		Fe/B	110.010	0.082	0.99	99.31	0.0010	0.99	
		Fe-Al/B	141.640	0.150	0.99	140.84	0.0071	0.99	
	80	В	119.050	0.013	0.99	102.04	0.0079	0.99	
		Fe/B	140.650	0.145	0.99	136.79	0.0073	0.99	
		Fe-Al/B	171.82	0.416	0.99	169.49	0.0059	0.99	

Table 3. Langmuir and Elovitch isotherms constants at different temperatures for the adsorption of BTX onto samples used.











	Thermodynamic parameters						
Parameters	<i>T</i> (°C)	Sample	ΔG^0 (KJ/mo)	ΔH^0 (KJ/mo)	ΔS ⁰ (KJ/mol)	R^2	
Benzene		В	-6.89				
	40	Fe/B	-14.13				
		Fe-Al/B	-20.61				
		В	-16.49				
	60	Fe/B	-23.15				
		Fe-Al/B	-30.25				
		В	-26.09				
	80	Fe/B	-30.79				
		Fe-Al/B	-38.89	119.96	0.450	0.99	
		В	-2.04				
	40	Fe/B	-11.2				
		Fe-Al/B	-21.14				
		В	-11.46				
Toluene	60	Fe/B	-18.89				
		Fe-Al/B	-28.15				
		В	-20.25				
	80	Fe/B	-27.1				
		Fe-Al/B	-35.84	94.77	0.370	0.99	
		В	-0.7				
	40	Fe/B	-11.89				
		Fe-Al/B	-19.13				
		В	-9.47				
O-xylene	60	Fe/B	-20.22				
		Fe-Al/B	-23.44				
		В	-17.43				
	80	Fe/B	-28.02				
		Fe-Al/B	-28.16	53.03	0.230	0.99	

Table 4. Thermodynamic parameters for the adsorption of BTX onto samples used.

strong interaction between all samples used and the molecular structure of these aromatic hydrocarbons compounds. Also, at 80°C, the maximum capacity q_m for the benzene (175.130 µg/g) are higher than those for toluene (171.84 µg/g) and *o*-xylene (171.82 µg/g) by Fe-Al/B. Besides, BTX adsorption by B is very low compared to the Fe/B and Fe-Al/B, (**Table 3**); the adsorption of benzene is larger than those of toluene and o-xylene, due to the larger surface area in particular Fe-Al/B (355.00 m²/g) (**Table 2**). Again, o-xylene and toluene adsorption capacity on Fe/B and Fe-Al/B is almost the same at 60°C and 80°C. In this case, the toluene and o-xylene molecules are larger molecules than

the benzene. Therefore, the adsorption of toluene and o-xylene is more difficult and complicated by microporous adsorbents. The increase of temperature favors this adsorption to occur more easily given that the activation energy can be surmounted more readily. In the case of benzene (C_6H_6), the smaller size of the molecule leads to easy adsorption without need of large activation energies, this reflects the fact that the kinetic diameter of C₆H₆ molecule is smaller than those of toluene and o-xylene [17].

In multilayer adsorption, it is supposed that molecules are adsorbed in several layers on the adsorption surface. One of the equations that predicted multilayer adsorption with unlimited layers is Elovich equation [13]: Equation (5):

$$(x/m)/q_m = K_E \cdot C_e \cdot \exp\left(-(x/m)/q_m\right)$$
(5)

where x and m are masses of adsorbed and adsorbent respectively, C_e is the equilibrium concentration of the adsorbent (μ g/L). K_E and q_m are Elovitch equilibrium constant (L/ μ g) and Elovitch maximum adsorption capacity (μ g/g) respectively. Equation (5) can be linearized into the form as follows:

$$\operatorname{Ln}\left(\left(x/m\right)/C_{e}\right) = \operatorname{Ln}K_{E}q_{m} + \left(x/m\right)/q_{m}$$
(6)

The values of Elovitch maximum adsorption capacity (q_m) and K_E are computed the slopes and y-intercepts of the plot $Ln((x/m)/C_e)$ versus (x/m). The calculated results are shown in Table 3. The adsorption data of the all samples used is good fitted to the Elovitch equation (Figure 7) with the correlation coefficient $R^2 > 0.99$. The values of maximum adsorption capacity determined using the linear transformation of the Elovich equation (Table 3) are higher on Fe-Al/B at 80°C for: of benzene (170.3 μ g/g), toluene (164.74 μ g/g) and o-xylene (169.49 μ g/g). This means that the assumption of the exponential covering of adsorption sites that implies multilayer adsorption is in agreement with the experiment in the studied concentration range. Therefore, the Elovich model is able to describe the adsorption isotherms of aromatic BTX hydrocarbons.

3.4. Desorption of BTX

Figure 8 presented the desorption rate. The results shown that desorption are increased with increase the temperature, at 80°C desorption of Fe - Al/B is very quick: and desorption rate of BTX is high > 75% compared to Fe/B > 70%and B > 65%. The desorption of the BTX gives results matching those of the adsorption, indeed more than 90% of the quantities of the BTX are adsorbed on Fe - Al/B at 80°C.

3.5. Adsorption Thermodynamics

In any adsorption process, namely free energy (ΔG^0), enthalpy (ΔH^0) and entropy (ΔS^0) have an important role. The Gibbs free energy change, ΔG^0 , is an indication of spontaneity of a chemical reaction and therefore an important criterion for spontaneity. Both energy and entropy factors must be considered in order to determine the Gibbs free energy of the process. Reactions occur spontaneously at a given temperature if ΔG^0 is a negative quantity. The free energy















Figure 8. Desorption of BTX onto samples used.



of an adsorption, considering the adsorption equilibrium constant K_d is given by the following equation:

$$\Delta \mathbf{G}^0 = -RT \mathbf{L} \mathbf{n} K_d \tag{7}$$

$$K_d = \frac{C_{ads}}{C_e} \tag{8}$$

where ΔG^0 is the standard free energy change (J/mol), K_d is the equilibrium constant. C_{ads} and C_e are the equilibrium concentrations (µg/L) of the BTX on the adsorbent used and in the gas phase respectively. *R* the universal gas constant (8.314 J/mol·K), and T is the absolute temperature (K). Considering the relationship between ΔG^0 and K_d , change in equilibrium constant with temperature can be obtained in the differential from as follows [18] [19]:

$$\frac{\mathrm{d}\mathrm{Ln}K_{d}}{\mathrm{d}T} = \frac{\Delta H^{0}}{RT^{2}} \tag{9}$$

After integration, the integrated form of Equation (9) becomes:

$$LnK_d = -\frac{\Delta H^0}{RT} + y \tag{10}$$

where *Y* is a constant Equation (10) can be rearranged to obtain;

$$-RT \operatorname{Ln} K_d = \Delta H^0 - TRY \tag{11}$$

Let:

$$\Delta S^0 = RY \tag{12}$$

Substituting Equation (11) and Equation (12), ΔG^0 , can be represented as follows:

$$\Delta G^0 = \Delta H^0 - T \Delta S^0 \tag{13}$$

The equilibrium constant, K_d is obtained from using Equation (6) and Equation (13). A plot of ΔG^0 versus T (K) will be linear and the values of ΔH^0 and ΔS^0 are determined from the slope and intercept of the plot (Figure 9). The results are presented in **Table 4**, as it can be seen, at the temperatures of 40, 60°C and 80°C the negative values of ΔG^0 and positive values of ΔH^0 indicate that the adsorption of BTX on all samples used is spontaneous and endothermic process. The positive value of ΔS^0 reflects the affinity of the all samples used for BTX and suggests that entropy is responsible for making the ΔG^0 negative for the adsorption process to be spontaneous.

4. Conclusions

The study of adsorption of aromatic BTX hydrocarbons on B, Fe/B and Fe – Al/B solids has shown that the adsorption of BTX is a function of temperature. The adsorption capacity increased with increasing temperature. The adsorption capacity of purified bentonite and pillared bentonite increases with polycations of Fe or Fe-Al where Fe – Al/B is a good adsorbent with maximum capacity q_m for the benzene being 175.30 µg/g at 80°C; the adsorption isotherms of all BTX aromatic hydrocarbons are favorable at Langmuir model and Elovich





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model provides the best fit to the experimental data with high correlation coefficient ($R^2 = 0.99$).

The adsorption is easy for benzene compared with toluene and o-xylene. The Gibbs free energy (ΔG^0) demonstrated that the adsorption process is favorable for adsorption of all BTX aromatic hydrocarbons by purified bentonite or pillared bentonite and this adsorption is reflected in the positive values of entropy (ΔS^0).

It may be concluded that pillared bentonite may be used as a low-cost, natural and abundant source for the elimination of aromatic BTX hydrocarbons.

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