

Competitive Adsorption of Xylene and Toluene on Modified and Unmodified Sodium Bentonite Clay Mineral

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

Cetyltrimethylammonium bromide (CTAB) – modified and unmodified sodium bentonite were both used for the competitive adsorption of m-xylene and toluene from their aqueous solution. Infrared spectroscopy (IR) and expansion tests (adsorption capacity and Foster swelling) measurement were performed in order to evaluate the performance of the adsorbents. The FT-IR results indicated that the quaternary ammonium salts (CTAB) were intercalated between the layers of the sodium bentonite clay. The Foster swelling test results indicated that the affinity of CTAB was more in the treated clay for all other organic compounds tested. The result showed that modified sodium bentonite swelled more than the unmodified and the order of the swelling factor is; m- xylene > toluene > gasoline > diesel > kerosene > water. However, when the assay was performed under agitation, it yielded higher swelling capacity. The modified clay exhibited very high capacity of adsorption compare to unmodified samples. The m-xylene and toluene uptake follows a pseudo second order kinetics mechanism, suggesting that the rate determining step of adsorption involves the two adsorbates competitively adsorbed on the adsorbent sites. Therefore this organo-modified bentonite can be used for the removal of organics from polluted aqueous effluents.

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1. INTRODUCTION

The advancement and development of petrochemical industries has contributed to pollution of the environment such as water and land pollution owing to the hydrocarbons and other discharge present in the waste or used water engendered in the industries during production [1-2]. Large amount of water is being utilized by these petrochemical industries causing them to spawn a big amount of waste water that contains some pollutants such as heavy metals, oils, organic compounds, benzene, toluene, ethylbenene, xylene, cyanide, grease and suspended solids in different concentration [2,3]. Some of these petrochemical industries discard their effluent and waste water without proper treatment and in so doing, they constitute some environmental menace and health nuisance to aquatic organism and human [3]. To avert the effect of this menace and nuisance, the environmental protection agency (EPA) is effectively at work to make sure that this petrochemical waste are treated prior to discharge [1]. Xylene and toluene which are insoluble in water are one of the major hydrocarbons present in the waste water of these petrochemical industries [4]. These hydrocarbon can be removed through several methods but a better and an effective way which is more economical is by adsorption method and adsorption has always been carried out with the aid of a seemly adsorbent such as bentonite [4]. Bentonite is readily available in nature, cheap, recyclable, have a high adsorption capacity and can be seen in most countries [5]. Bentonite exist in two natural form which are sodium bentonite and calcium bentonite but other forms of bentonite such as magnesium bentonite, potassium bentonite, iron bentonite and aluminium bentonite could be synthesized from the natural forms of bentonite through ion exchange method or mechanism [6-7]. The use of organo -modified bentonite has been demonstrated to be more effective in the removal of hydrocarbon from waste water and aqueous solution [4].

The objective of this study is to determine the efficiency and the effectiveness of CTAB modified sodium bentonite in the removal of m-xylene and toluene from aqueous solution. The effect of contact time, adsorbent and initial concentrations were also determined.

2. MATERIALS AND METHODS

2.1 Materials

The sodium bentonite used in this work was obtained from Mansid Nigeria Limited, Portharcourt Rivers State, Nigeria. Cetyltrimethylammonium bromide, m- xylene and toluene were purchased from Sigma-Aldrich Company, Switzerland at >98% purity, and were used as received without further purification. The petroleum products were purchased from Nigeria National Petroleum Company, Portharcourt, Rivers State, Nigeria.

2.2 Modification of Adsorbent

The modification of the adsorbent was carried out following the method outlined by [8]. Specifically 10 g of sodium bentonite was weighed and steeped in 200 ml of distilled water for a period of 24 hours. Thereafter it was stirred for 30 minutes and exactly 200 ml of 0.02 M of CTAB (cetyltrimethylammonium bromide) was added to the mixture. The mixture was stirred with reflux at a temperature of 75°C for two hours. Finally the mixture was filtered and oven dried at 105°C for a duration of three hours. The dried CTAB modified bentonite was properly stored and preserved in a sample tube for characterization and batch adsorption. The whole process was repeated for different concentration of CTAB.

2.3 Adsorbent Characterization

2.3.1 FT- IR analysis

The FT- IR analysis was carried out using Fourier transformed infrared spectrometer (model number IR AFFINITY-1) at a range of 400 – 4000 cm^{-1} . The samples were analyzed using KBr technique. Exactly 1 mg of the sodium bentonite samples were successively homogenized with 100 mg of pure KBr and the mixture was carefully screwed and compressed to form a disc which was afterward placed in the machine and then scanned.

2.4 Foster Swelling Test

This test was carried out following previous reported method [9-10]. Exactly 0.5 g of the adsorbent was added to a 50 ml glass tube with internal diameter of 1.0 cm and the volume

occupied by the adsorbent was recorded. Exactly 50ml of distilled water was added to the glass tube containing the sample, after which the mixture was uninterruptedly left to stand for a period of 24 hours. The new volume, V_B of the adsorbent was recorded after 24 hours and agitation was subsequently carried out. After shaking, the mixture was left to stand for 24 hours and the final volume, V_A was recorded. The Foster swelling test was repeated using other solvent (toluene, xylene, gasoline, diesel and DPK). The swelling factor (F_S) is calculated using equation 1 and 2.

$$F_{SB} = \frac{V_B - V_I}{V_I} \quad (1)$$

$$F_{SA} = \frac{V_B - V_I}{V_I} \quad (2)$$

F_{SB} and F_{SA} are the swelling factor before and after agitation respectively, V_B and V_A are the volume of the adsorbent before and after agitation respectively, While V_I is the initial volume of the adsorbent in the glass tube before solvent addition.

2.5 Adsorption Capacity Measurement in A Static Condition

This analysis was carried out in accordance with previous reported method [11]. In this method, a glass mesh bucket containing the adsorbent sample was weighed and the exact value recorded. The test cell was filled with the test solvent of interest and the adsorbent sample and bucket lowered into the test cell. The sample was allowed to float freely within the test cell for a duration of 30 minutes. The bucket was removed manually in a vertical direction and allowed to drain for 15 seconds \pm 3 seconds. The adsorption capacity (AC) in a static condition is calculated using equation 3

$$Ac = \frac{W_F - W_I}{W_I} \quad (3)$$

W_F and W_I are the weight of the adsorbent after adsorption and before adsorption respectively.

2.6 Batch Adsorption Experiment

The batch adsorption technique used in this study is in line with the method used by Aamir *et al.* [12]. Exactly 0.5 g of the adsorbents were successively added to 120 ml capacity plastic

container and 20 ml of m-xylene - toluene mixture (100 mg/l) was added and the container was tightly covered to avoid evaporation. The mixture was subjected to 60 minutes shaking using a mechanical shaker (model number, J.P. SELECTA, 7000384) at 400 rpm and the supernatant solution was carefully decanted and was analyzed properly using Uv- visible spectrometer (model number, GENESYS 10 uv) at a wavelength of 315 nm and 325 nm for toluene and meta - xylene respectively. The adsorption process was repeated by varying adsorbent weight, adsorbate concentration and contact time. A blank analysis was similarly carried out in order to determine the possible loss of the adsorbate solution by evaporation. The percentage removal (% R) and the adsorption capacity (q_e) are calculated using equation 4 and 5

$$\text{Percentage removal, \%R} = \frac{C_0 - C_e}{C_0} \times 100 \quad (4)$$

$$\text{Adsorption capacity, } q_e = \frac{C_0 - C_e}{m} \times V \quad (5)$$

Where m is the mass of the adsorbent, V is the volume of the adsorbate, C_0 and C_e are the initial and final concentration of the adsorbate respectively.

3. RESULTS AND DISCUSSION

3.1 Adsorbent Characterization

3.1.1 FT – IR analysis

The FT- IR spectral result of CTAB modified bentonite and natural bentonite is shown in Fig. 1. At 3400 – 3642 cm^{-1} , a broad band was observed, which shows the presence of O-H vibration of the water adsorbed silica surface [13]. Another adsorption band appeared at 1640 cm^{-1} which shows the presence of H-O-H bending vibration whereas the broad band around 1011.1 cm^{-1} and 1038.1 cm^{-1} is related to the stretch vibration of Si-O-Si groups. With respect to the modified bentonite, the two peaks that appeared at 2985.64 – 2860.02 cm^{-1} shows the presence of $-\text{CH}_3$ and $-\text{CH}_2$ stretching vibration respectively. A H-O-H deformation peak was also seen at 1566.92 – 1601 cm^{-1} [14]. The presence of Cetyltrimethylammonium bromide in the modified sodium bentonite is proved by the existence of methyl group and methylene group, strictly in the modified sodium bentonite.

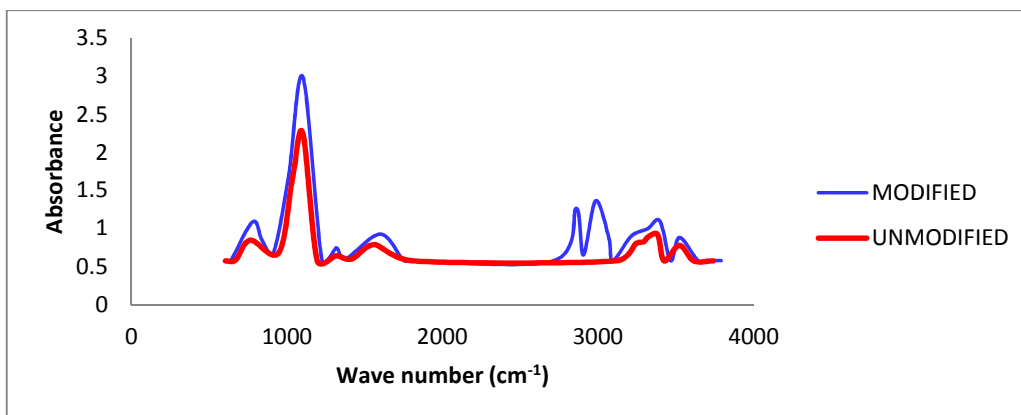


Fig. 1. FT- IR spectral of unmodified (red) and CTAB modified (blue) sodium bentonite

3.2 Foster Swelling Test

The swelling factor of both CTAB modified and unmodified sodium bentonite in petrochemical solvent are shown in Figs. 2 and 3, while that of petroleum product are shown in Fig. 4 and 5. From the results in Figs. 2 to 5, modified sodium bentonite swelled higher in petrochemical/hydrocarbon solvent (m- xylene and toluene) than in petroleum product (diesel, DPK and gasoline) but completely failed to swell in water. The swelling factor increased as the CTAB concentration increased. The Foster swelling results show that the organo-modified bentonite has acquired an organophilic character. Unmodified sodium bentonite swelled in water but failed to swell in other organic solvents. The swelling of clays in water occurs because of the capacity of the external and internal surfaces of the clay mineral layers to be hydrated. Sodium as an exchangeable cation promotes layers of "oriented water". As the clay is immersed in

water, it adsorbs several water layers between the clay mineral galleries, due to the hydrogen bonds and, consequently, the swelling occurs. The swelling occurs exclusively in the (001) planes, causing delamination of the layers, but the crystallographic integrity is kept during the expansion process [15-16].

During the test, unmodified sodium bentonite showed no swelling in any of the organic solvents investigated. As expected, the absence of swelling is due to the organophobic character of Na- bentonite that does not permit interaction with the organic solvent. For the organophilic bentonite in contact with the organic solvents, the swelling occurred due to the diffusion of the molecules of the solvent between the clay mineral layers. This process is favored by the balance between the nature of the hydrocarbon solvents - compounds of low polarity, and the alkyl chains of the CTAB modifier that decrease the polarity of the clays.

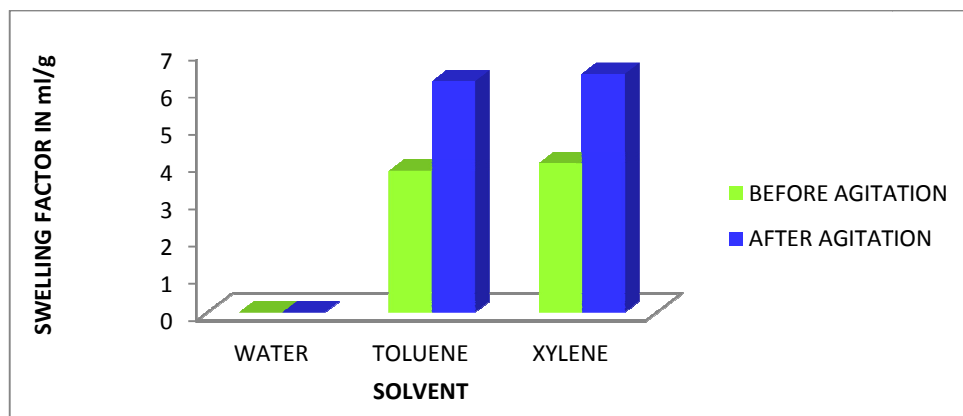


Fig. 2. The swelling factor of modified sodium bentonite in hydrocarbon and water

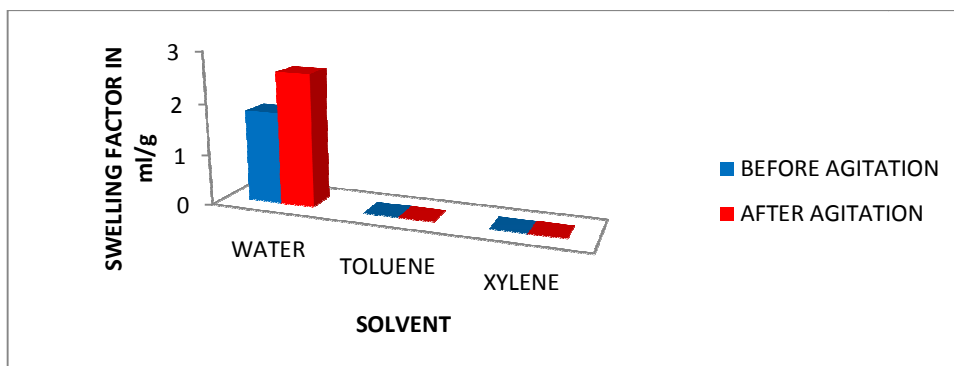


Fig. 3. The swelling factor of unmodified sodium bentonite in hydrocarbon and water

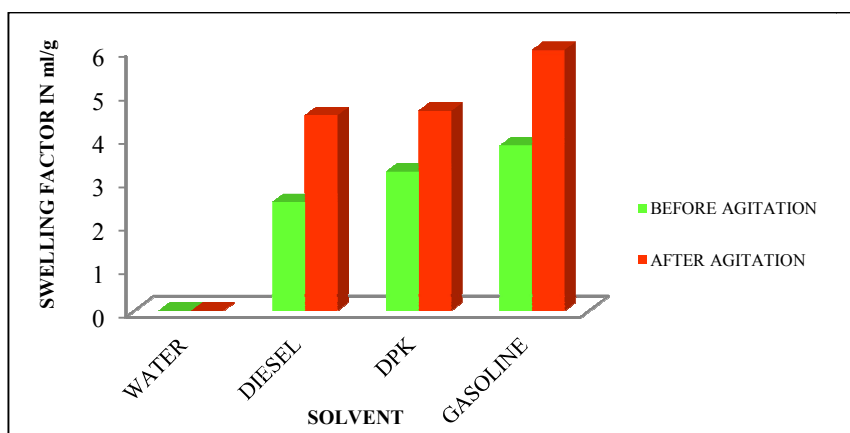


Fig. 4. The swelling factor of modified sodium bentonite in petroleum products and water

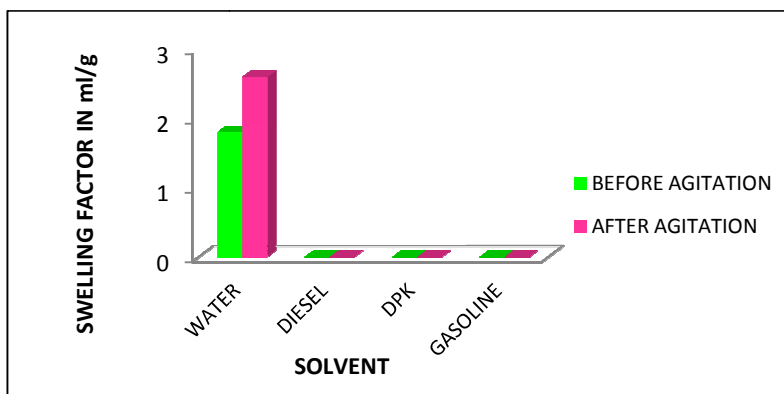


Fig. 5. The swelling factor of unmodified sodium bentonite in petroleum products and water

3.3 Adsorption Capacity in a Static Condition

Fig. 6 and 7 showed the adsorption capacity measurement of sodium bentonite using water, petroleum products and hydrocarbons. The Modified bentonite adsorbed more of organic

solvent than water whereas unmodified sodium bentonite adsorbed more water than the organic solvent. The result of the adsorption capacity measured under a static condition is given as; gasoline > m- xylene > toluene > diesel > DPK > water. Unmodified Sodium bentonite adsorbed more water due to the isomorphous substitution of

the cation present in the octahedral lattice which results to excessive electrons on the bentonite surface. These electrons enabled the formation of the hydrogen bonds of partially covalent character between the water molecule and the bentonite surface. The excess electron helps to strengthen the bentonite – water bond and also facilitate the bonding of additional layers of water. The negative charge and the hexagonal arraignment of oxygen atoms on the bentonite surface facilitate the formation of the first few oriented water layers. Water adsorption also occurred as a result of the net negative charge and the presence of sodium ion which produces an osmotic pressure that draws water molecules into the layer [17].

This Foster swelling test confirmed the organophilicity of the CTAB modified sodium bentonite.

3.4 Batch Adsorption

Fig. 8 shows the effect of adsorbent weight on the adsorption process. The percentage removal

of meta- xylene and toluene increased from 50.2% to 83.33% and from 41.21% to 74.78% respectively as the adsorbent weight increased from 0.1 g to 0.5 g. The observed increase may be due to increase in adsorption site which is in good agreement with the work recently reported by Aamir et al. [12].

Fig. 9 shows the effect of initial concentration. As the initial concentration of the adsorbate increases, the adsorption capacity and the amount of m- xylene and toluene removed from the aqueous solution also increased. The result showed that the affinity of CTAB modified bentonite towards meta – xylene and toluene increased with increasing time. M-xylene is heavier than toluene and its removal is therefore more favoured compared to toluene. Toluene is lighter and more hydrophilic than m-xylene, and therefore has the tendency to exhibit a minimal inclination to be attracted to the organo - bentonite during the adsorption process. This result is in good agreement with the work recently reported [12,18].

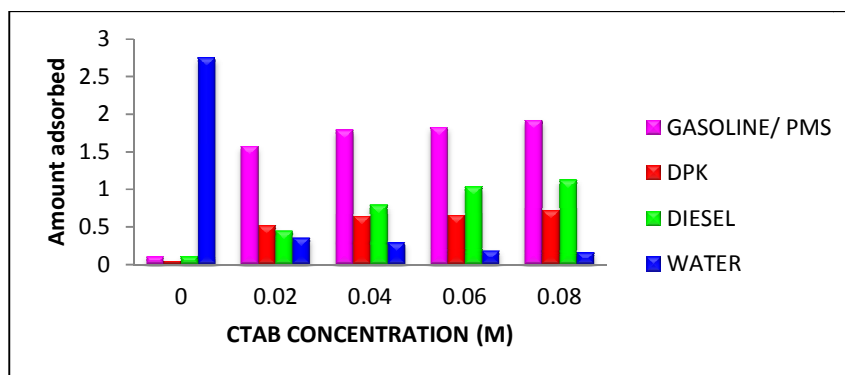


Fig. 6. Effect of CTAB concentration on the adsorption capacity of sodium bentonite using petroleum products and water

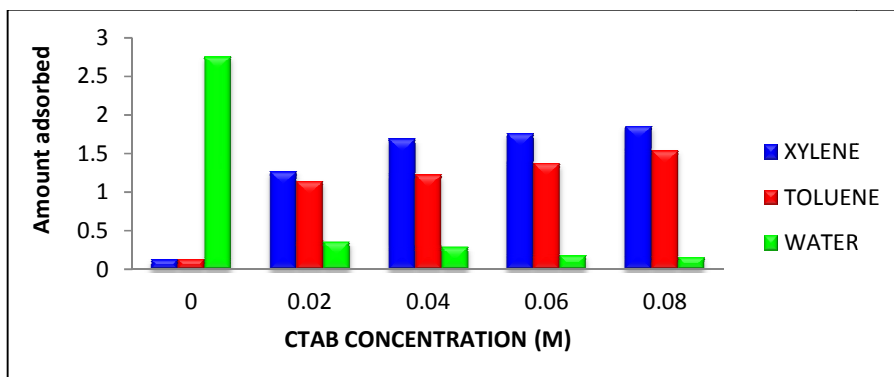


Fig. 7. Effect of CTAB concentration on the adsorption capacity of sodium bentonite using petrochemical solvents and water

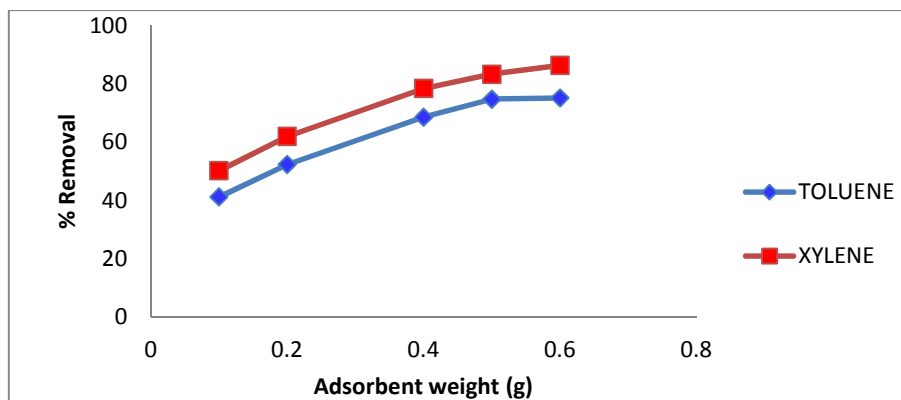


Fig. 8. Effect of adsorbent weight on the percentage removal of meta- xylene and toluen using modified sodium bentonite

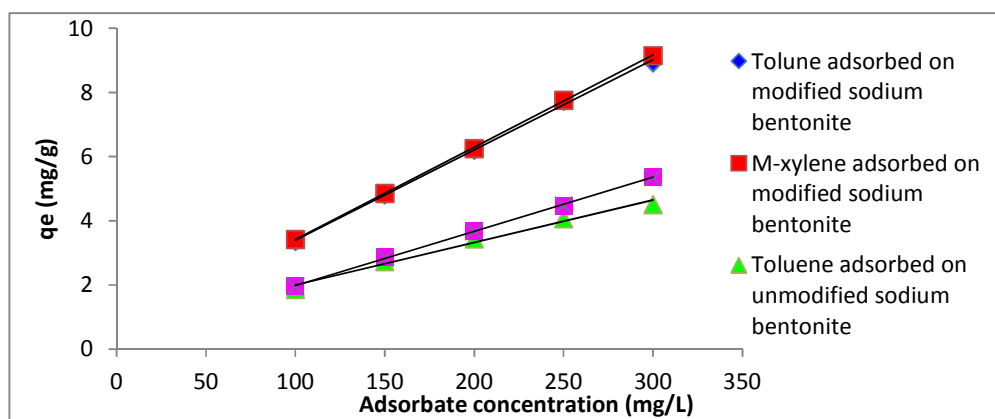


Fig. 9. Effect of initial concentration on the adsorption capacity

3.5 Langmuir Adsorption Isotherm Model

Mathematically, the Langmuir adsorption isotherm model [19] (Langmuir, 1918) is expressed as

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{q_m K_L} \quad (6)$$

C_e is the concentration (mg/L) of the adsorbate at equilibrium while q_e , which is the adsorption capacity refers to the quantity or amount of adsorbate removed or adsorbed from the solution per unit weight of the adsorbent. K_L is the langmuir constant while q_m is the maximum capacity of the adsorbent [19]. The values of K_L and q_m are obtained from a linear plot of C_e/q_e against C_e . K_L is the slope of the graph while q_m is the y-axis intercept. Langmuir separation factor, R_L is an important equilibrium parameter which helps to analyze the properties of the Langmuir isotherm model. The mathematical

expression of the Langmuir separation factor, R_L [20], is given as;

$$R_L = \frac{1}{1 + (K_L C_0)} \quad (7)$$

K_L and C_0 are Langmuir constant (L/mg) and the adsorbate's initial concentration (mg/L) respectively. If the separation factor, $R_L = 0$, adsorption is considered to be irreversible, if $R_L = 1$, it is considered to be linear but if $R_L > 1$, it is considered to be unfavorable. Adsorption is only considered to be favorable if $1 > R_L > 0$. [3,2].

The correlation factors, R^2 obtained from Langmuir model for m- xylene and toluene removal using organo-philic bentonite are 0.9135 and 0.9511, while that of unmodified bentonite are 0.9460 and 0.9773 as shown in Table 1. The Langmuir separation factors, R_L for m- xylene and toluene removal using modified sodium bentonite are given as 0.204 and 0.217

respectively. While that of unmodified bentonite are given as 0.149 and 0.323 for m-xylene and toluene respectively as shown in Table 1. This shows that the adsorption bonds between the adsorbent and the adsorbate are very strong and adsorption is highly favoured since the R_L values are within 0 and 1.

3.6 Freundlich Isotherm Model

In contrast to the Langmuir isotherm model, Freundlich isotherm model is an empirical adsorption isotherm model which describes the equilibrium relationships that exist between the adsorbate and the adsorbent molecules and assume multiple layer adsorptions on the heterogeneous site of the adsorbent. The Freundlich isotherm model [3,5] is given as;

$$\ln q_e = \ln k_f + \frac{1}{n} \ln C_e \quad (8)$$

q_e and C_e are the adsorption capacity (mg/g) and the concentration (mg/L) of the adsorbate at equilibrium. The values of $1/n$ and k_f are gotten from the linear plot of $\ln q_e$ against $\ln C_e$. $1/n$ is the slope of the graph while k_f is the intercept on y-axis [21,2].

The correlation factors, R^2 obtained from Freundlich isotherm model for m-xylene and toluene removal using modified bentonite are 0.9911 and 0.9948, while that of unmodified bentonite are 0.9992 and 0.9858 as shown in Table 1. The Freundlich constant with respect to the heterogeneous surface, $1/n$ for the removal of m-xylene and toluene using modified sodium bentonite are given as 0.6182 and 0.636

respectively while the values for the unmodified bentonites are given as 0.8424 and 0.710. These results clearly demonstrate that adsorption is said to be favourable since the values of $1/n$ obtained are less than unity.

The removal of m-xylene and toluene from the aqueous solution shows a great affinity of the adsorbate towards the adsorbent and also a stronger interaction of the adsorbate-adsorbent molecules. The correlation factor, R^2 obtained from the Freundlich isotherm model are closer to unity and are higher than those of Langmuir isotherm as shown in Table 1 which indicate and strongly show that multi layer adsorption mechanism was favoured on sodium bentonite surface for the competitive adsorption of m-xylene and toluene from the aqueous solution which is in good agreement with the recent reports by several authors [21,22,23,24].

3.7 Kinetics Studies

The effect of contact time on the adsorption of hydrocarbon (m-xylene and toluene) onto CTAB-modified and unmodified sodium bentonite are represented in Fig. 10. The result revealed that the optimum time of the sorption process was 120 mins. The adsorption rate was rapid in the first 30 mins then became a little subdued until equilibrium in the sorbents. Therefore a 120 mins shaking time was found to be appropriate and used in all subsequent measurements. Similar observation was reported by Obi and Apemiye, [4]; on the adsorption of m-xylene on a modified sodium bentonite.

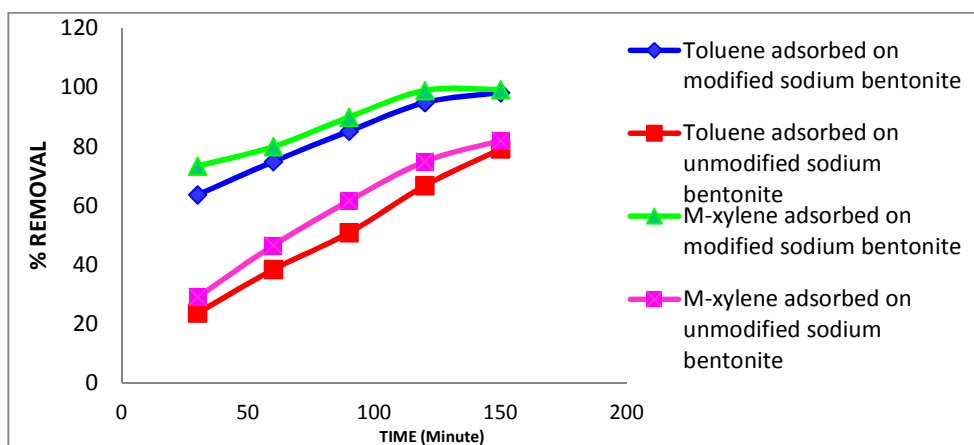


Fig. 10. Effect of contact time on the % removal of meta – xylene and toluene using modified and unmodified sodium bentonite

Table 1. Parameters obtained from Langmuir and Freundlich adsorption isotherm models

Adsorption Isotherm Model	Co (mg/L)	Meta- Xylene		Toluene	
		MSB	USB	MSB	USB
		300	300	300	300
Langmuir	qm	18.55	6.91	18.46	8.28
	K _L	0.013	0.019	0.012	0.007
	R _L	0.204	0.149	0.217	0.323
	R ²	0.9135	0.946	0.9511	0.9773
Freundlich	K _F	0.634	0.072	0.568	0.115
	1/n	0.6182	0.8424	0.636	0.710
	R ²	0.9911	0.9992	0.9948	0.9858

MSB = Modified Sodium Bentonite, USB = Unmodified Sodium Bentonite

In order to investigate the adsorption kinetics of the hydrocarbons onto the adsorbents, Lagergren's pseudo first order, pseudo second order and intraparticle diffusion kinetics were applied to the experimental data. The linearized form of the pseudo first order rate equation by Lagergren [25], is given as;

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (9)$$

Where q_e and q_t are the amount of the hydrocarbon adsorbed at equilibrium (mg/g) and t (min), respectively, and k_1 is the rate constant of the equation (min^{-1}). The adsorption rate constant (k_1) can be determined experimentally by plotting of $\ln(q_e - q_t)$ versus t . The plot of $\ln(q_e - q_t)$ versus t (not shown here) reveals that the data obtained from the hydrocarbon adsorption using CTAB – modified and unmodified sodium bentonite as shown in Table 2 are not well fitted with pseudo first order kinetics.

The linearized form of pseudo second order equation [26] is expressed as;

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e} \quad (10)$$

Where k_2 ($\text{mg.g}^{-1} \text{min}^{-1}$) is the rate constant for the second order equation, q_t (mg/g) is the amount of hydrocarbons adsorbed at a particular time t (min) and q_e (mg/g) is the amount of hydrocarbons adsorbed at equilibrium.

A plot of t/q_t versus t of equation 10 should give a linear relationship, from which q_e and k_2 can be obtained from the slope and intercept of the plot respectively (Fig 11 and 12). The values of the rate constant (K_2) and correlation factor (R^2) are given in Table 2.

The mathematical expression of the intraparticle diffusion kinetics [27] is written as;

$$q_t = k_{id} t^{1/2} + c \quad (11)$$

k_{id} is the intraparticle diffusion constant and its value and that of c are obtained from a linear plot of q_t against $t^{1/2}$. [28]

The correlation factor obtained from intraparticle diffusion kinetics using modified bentonite for the removal of m-xylene and toluene are given as 0.9632 and 0.9913, while that of unmodified are given 0.9956 and 0.8870 for m-xylene and toluene removal respectively as shown in Table 2.

Linear plots were obtained with high correlation coefficients (R^2), which are quite closer to unity compared to pseudo first order and intraparticle diffusion kinetics suggesting that the interaction between the adsorbent and the two adsorbates follow second order mechanism. This observation clearly demonstrates that both m-xylene and toluene competitively adsorbed on the surface of the bentonite, and the rate determining step is bimolecular involving the two adsorbates. The result equally showed that above 120 mins, the plots begin to deviate a little from linearity [29]

The results presented in Table 2 shows that the value of the adsorption capacity, q_e increased for the CTAB - modified sodium bentonite. The intercalation of cetyltrimethylammonium bromide (CTAB) between the layers of the bentonite increased the adsorption power, the basal spacing and the organophilicity of the bentonite, thereby increasing its affinity and interaction with organic compounds (m-xylene and toluene). Similar results were recently reported for the adsorption of m-xylene and toluene on different adsorbent which have similar chemical structures with that of natural bentonite [30-31] as shown in Table 3.

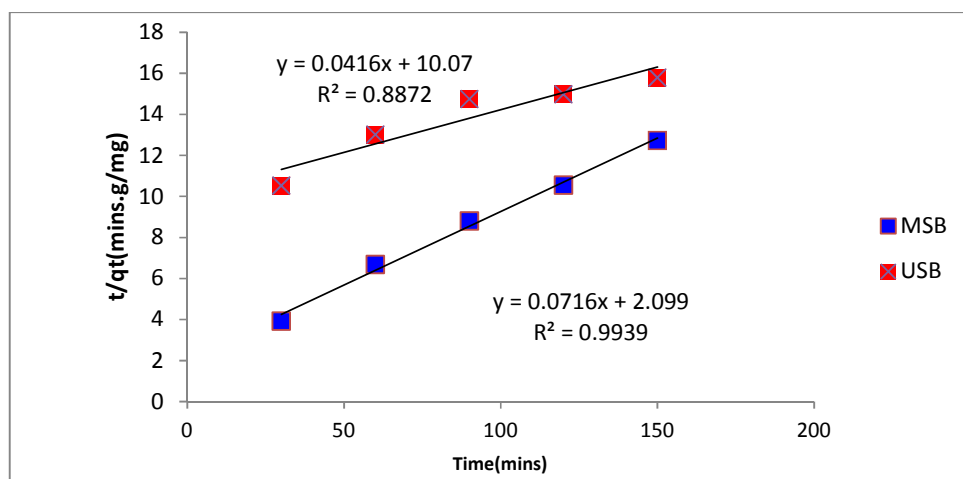


Fig. 11. Pseudo-second order kinetic model for toluene removal from aqueous solution using modified and unmodified sodium bentonite

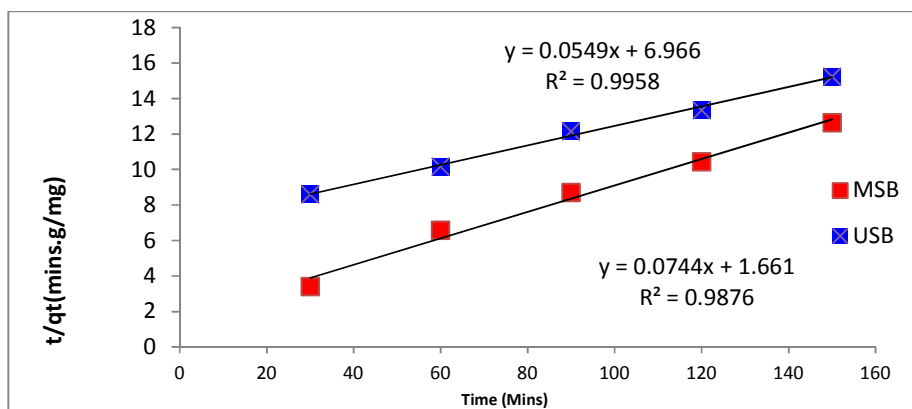


Fig. 12. Pseudo-second order kinetic model for m-xylene removal from aqueous solution using modified and unmodified sodium bentonite

Table 2. Parameters obtained from pseudo first order, pseudo second order and intraparticle adsorption kinetics models

Kinetics Model	Co (mg/L)	M- Xylene		Toluene	
		MSB 300	USB 300	MSB 300	USB 300
Lgergren Pseudo- First Order	qe(mg/g)	11.86	9.84	11.78	9.50
	K_1	0.0265	0.0195	0.0844	0.0178
	R^2	0.9623	0.9543	0.9613	0.9517
Pseudo- Second- Order	qe(mg/g)	11.86		11.78	9.50
	K_2	0.0039	0.00043	0.0024	0.00017
	R^2	0.9959	0.9958	0.9939	0.8872
Intraparticle Diffusion	qe(mg/g)	11.86		11.78	9.50
	K_{id}	0.5078	0.964	0.6393	0.9853
	R^2	0.9632	0.9956	0.9913	0.8870

MSB = Modified Sodium Bentonite, USB = Unmodified Sodium Bentonite

Table 3. Comparison of the adsorption capacity of m- xylene and toluene using different adsorbent

S/N		q_e		Reference
		Toluene	Meta- xylene	
1	HDTAB CLAY	—	9.328	[4]
2	ACTIVATED CARBON	5	6.5	[32]
3	TTAB CLAY	5.15	6.98	[12]
4	DIATOMITE	0.037	0.042	[31]
5	HDTMA CLAY	6.8	7.21	[33]
6	CTAB CLAY	11.78	11.91	This study

HDTAB = Hexadecyl trimethylammonium bromide, TTAB = Tetradecyl trimethylammonium bromide, CTAB = Cetyl trimethylammonium bromide

4. CONCLUSION

The competitive adsorption of m- xylene and toluene was successfully carried out using natural or unmodified and CTAB modified sodium bentonite. The modification improved the efficiency and effectiveness of the sodium bentonite during the adsorption process.

The FT-IR analysis confirmed the presence of cetyltrimethylammonium bromide in the organo modified bentonite.

Organo-modified sodium bentonite has a higher percentage removal and higher adsorption capacity than natural bentonite. m- xylene was removed from the solution more than toluene due to difference in their molecular weight. CTAB modified sodium bentonite has proven to be an effective adsorbent for removal of both m- xylene and toluene from their aqueous solution. Hence water treatment industries and petrochemical industries and refineries should utilize this adsorbent for removal of hydrocarbon (m- xylene and toluene) from petrochemical waste water and effluents.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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