

## RESEARCH ARTICLE

# Competitive adsorption ability of aniline and pentachlorophenol with organoclay catalysts

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## Abstract

In this study, incorporated hexadecyltrimethylammonium (HDTMA) cations to smectite via ion exchange and formed the modified smectite-HDTMA, a kind of organoclay catalyst. Besides analyzing its physical properties, we also adopted it to conduct adsorption experiments on organic pollutants such as aniline and pentachlorophenol (PCP). In terms of the adsorption of organic pollutants, adsorption reaction reached the saturation point within 2 h and remained unchanged after reaching 8 h when smectite-HDTMA with a pH value of 7 was used to bind aniline. Moreover, adsorption reaction reached the saturation point within 2 h and remained unchanged after reaching 24 h when smectite-HDTMA with a pH value of 7 was used to bind PCP. This study also found that high adsorption efficiency of smectite-HDTMA towards aniline could be achieved; there was a slow but steady increase in the adsorption capacity of smectite-HDTMA when its equilibrium concentration reached 100 mg/L. Besides, batch adsorption experiments using two solvents showed that smectite-HDTMA mainly bound aniline and PCP via distribution, meaning that the adsorption capacity of the smectite-HDTMA remains the same no matter how many kinds of organic compounds are presented and that competitive adsorption does not occur.

## KEYWORDS

aniline, HDTMA, organoclay, pentachlorophenol, smectite-HDTMA

## 1 | INTRODUCTION

Adsorption is a process in water treatment that uses porous solids to adsorb pollutants in water and separate them from water. The porous solids used to adsorb pollutants in water and separate them from water are called adsorbents. Adsorbents that are applied in environmental engineering<sup>1,2</sup> include activated carbon, activated coal, coke, cinder, resin, sawdust, and others. Moreover, adsorption is a surface phenomenon related to surface energy. It is often categorized into physical adsorption (that relies on molecular interactions between adsorbents and adsorbates), chemisorption (that relies on chemical bonding),

and ion exchange adsorption (that relies on electrostatic attraction). During the wastewater treatment process, a filter bed is often used to adsorb pollutants in wastewater, whereby heavy metal ions (such as Hg, Cr, Ag, Ni, and Pb) can be removed from wastewater;<sup>3,4</sup> it can even be applied in advanced wastewater treatment. Furthermore, adsorption can be used to purify low-concentration inorganic gases in water. For instance, a fixed-bed adsorption device is generally used to purify gases, such as F and H<sub>2</sub>S, in order to adsorb inorganic materials that are difficult to be processed.<sup>5</sup>

The actual adsorption process is extremely complex and is not fully understood.<sup>6–8</sup> This is particularly the case

for adsorbents being applied to polluted water samples, due to their complexity. One traditional method to characterize adsorption is to use model compounds instead of actual polluted water samples.<sup>9,10</sup> Similarly, Newcombe et al.<sup>11</sup> evaluated the competitive effect of organic pollutant fractions and found that their smallest fraction, with molecular weight (MW<sub>w</sub>) of 949 g/mol, respectively, was the most detrimental to 2-methylisoborneol (168 g/mol) adsorption capacity and removal rates. In addition, chemisorption is a type of adsorption that involves a chemical reaction between the chemical bond of the surface of a solid and the chemical bond of an adsorbate. Some studies have pointed out that substances undergo physical adsorption at low temperatures, and that increasing such temperatures to certain levels induces chemisorption.<sup>12–14</sup> Sometimes physical adsorption and chemisorption occur simultaneously.

Organoclay materials are defined as hybrid materials resulting from the association of clay minerals (swelling ones such as montmorillonite (Mt), vermiculite, and saponite) with surfactants and/or other various organic compounds.<sup>15,16</sup> Organoclays have been studied for soil remediation, groundwater purification, industrial waste water treatment, and oil spill remediation using batch systems.<sup>17,18</sup> Studies have shown good adsorption capacity of organoclays for chlorinated compounds such as trichloroethylene and polychlorophenols.<sup>19–20</sup> With the incorporation of the surfactants in their internal structure through different chemical and physical processes, the obtained materials combine a large specific surface and a hydrophobic behavior, enhancing the applications of clay minerals in various fields. A large volume of the research work focused on and pointed out the use of organoclays as reinforcers in polymer nanocomposites, starting materials for photophysical films, rheological agents, and as adsorbents for organic pollutants in soil and aquatic environments.<sup>21,22</sup> Organoclay minerals may be affected by factors of influence, such as the degrees of protonation in the environment, prompting the clay minerals to adsorb different amounts of cations. When the pH value of the environment changes, deprotonation or protonation occurs, changing the cation exchange capacity. For instance, when the pH value of the environment drops, clay minerals adsorb fewer cations; when the pH value of the environment rises, clay minerals adsorb more cations. Thus, organoclay minerals carry variable charges.

This research aims to study the adsorption capacity of PCP and aniline and their adsorption mechanism via conducting adsorption experiments using smectite-HDTMA, which was formed by transferring HDTMA cations to smectite via ion exchange.

## 2 | MATERIALS AND METHODS

### 2.1 | Reagents

The main reagents used in this study include Hexadecyltrimethylammonium bromide (HDTMA) (C<sub>16</sub>H<sub>33</sub>(CH<sub>3</sub>)<sub>3</sub>NBr) (Merck, used in this study was purchased from Lancaster Synthesis), aniline (C<sub>6</sub>H<sub>5</sub>NH<sub>2</sub>) (Arcos Chimica), pentachlorophenol (PCP) (C<sub>6</sub>HCl<sub>5</sub>O) (Lancaster), sodium chloride (Merck), and ethanol (Merck, GR grade).

### 2.2 | Purification and preparation of smectite

To prepare for the sodium-saturated smectite, the smectite clay used in this study was purified and Cl<sup>-</sup> removed from the smectite clay, whereby ion exchange could be subsequently performed.<sup>16</sup> The catalyst carrier this study adopted was the Wyoming smectite bought from American corporation Colloid, a type of 2:1 clay mineral. The smectite clay sold in the market was taken and weighed, then soaked for days until it was completely inflated. The smectite clay suspension (250 ml) was taken and sand filtered out of it. The deionized water (1 L) was added, shaken, stirred, and placed still.<sup>23</sup> The top 10 cm of suspension in the cylinder was pumped using a high-speed centrifuge. A proper amount of NaCl was added to the smectite clay obtained by centrifugation and stirred continuously for 8 h. After freeze drying, the saturated smectite clay was ground into powder and became the smectite sodium saturated clay.

### 2.3 | Preparation of smectite-HDTMA

Organically modified clay has enhancing properties which can be applied for adsorption capacity. Total 0.5 g of smectite sodium saturated clay was taken and 30 ml of HDTMA solution with a value equivalent to 0.75 times the CEC value added.<sup>24</sup> It was continuously heated and stirred in a water bath at 80°C for 24 h. A solid was separated from the liquid using a high-speed centrifuge at 20,000 rpm. AgNO<sub>3</sub> was used to inspect whether the supernatant has white sediment after centrifugation, so as to ensure that the chloride ions have been completely removed. After the modified smectite-HDTMA was freeze-dried, it was ground using an agate mortar and pestle. The characteristic analysis and absorption test were subsequently conducted.

## 2.4 | Analysis of the various physical properties of the organoclay

### 2.4.1 | Measurement of the specific surface area

The Ethylene Glycol was used to measure the total specific surface area of the smectite and smectite-HDTMA.<sup>13</sup>

$$A = \frac{W_a}{(W_s \times K)} \quad (1)$$

A: Specific Surface Area (m<sup>2</sup>/g);

W<sub>a</sub>: Difference in weight before and after the absorption of the Ethylene Glycol;

W<sub>s</sub>: Weight (g) before the absorption of the Ethylene Glycol; K: 0.00031

### 2.4.2 | Fourier-transform infrared spectroscopy (FT-IR) of smectite and smectite-HDTMA catalyst

The smectite and smectite-HDTMA, as well as KBr powders, were put in an oven. After they were heated and dried for 48 h, they were mixed evenly in a 1:10 ratio. The mixture was then ground with an agate mortar. A hydraulic press was used to press the mixture at 10 tons/h, whereby it was made into a transparent foil. The foil was put in a spectrometer while an analysis was performed with a scan range of 4000–400 cm<sup>-1</sup>.

### 2.4.3 | XRD analysis

The XRD analysis was conducted using an x-ray diffractometer (Rigaku RINT-2000). With CuK<sub>α</sub> as the light source, an analysis on the crystal patterns of the smectite and smectite-HDTMA was performed to learn the spacing of layers of materials. The x-ray wavelength produced is 1.5418 Å; the operating current during testing is 10 mA; the voltage is 20 kV; the scanning speed is 5°/min; and the scanning angle is 2θ = 2~40°.

### 2.4.4 | Measuring the concentration of smectite-HDTMA

We concocted multiple HDTMA solutions with concentrations between 0–0.0125 M and a bromophenol blue (BPB) solution (5 × 10<sup>-4</sup> M) containing a NaH<sub>2</sub>PO<sub>4</sub> buffer solution with a pH value of 8. We then combined excessive BPB<sup>2-</sup> and HDTMA in the solutions to

form (HDTMA<sup>+</sup>)<sub>2</sub>•BPB<sup>2-</sup>, a blue compound. After purifying the compound via chloroform extraction, we used a spectrometer to produce its calibration curve.

We also collected the residual HDTMA solution after retrieving smectite-HDTMA, and mixed 0.3 ml of it with 5 ml of 5 × 10<sup>-4</sup> M BPB and 1 ml of NaH<sub>2</sub>PO<sub>4</sub>. We then added 15 ml of chloroform into the solution for chloroform extraction and processed it with ultrasonication. After 30 min, we extracted the chloroform solution at the bottom and diluted it. The diluted solution was then analyzed using a spectrometer for its optical density. The change in the concentration of HDTMA solution before and after modifying the smectite was considered the adsorption capacity of the smectite in this study.

## 2.5 | Adsorption Efficiency

### 2.5.1 | Adsorption kinetics

First, we mixed 0.02 g of smectite-HDTMA with 30 ml of aniline solution (100 mg/L) using a vortex mixer. Another 0.02 g of smectite-HDTMA was mixed with 30 ml of PCP solution (5.25 × 10<sup>-5</sup> M = 140 mg/L) using a vortex mixer. Both solutions were shaken for 5 min, 10 min, 20 min, 30 min, 60 min, 120 min, 240 min, 480 min, 720 min, and 1440 min, respectively. After reaching the designated time, the shaken solutions run through the Whatman 47 mm filter paper before being measured with an atomic absorption spectrometer to determine their concentrations. The time needed for the concentration of a solution to become stable was considered the equilibration time.<sup>7</sup>

### 2.5.2 | Adsorption isotherm

Aniline solutions with concentrations between 15 and 120 mg/L were concocted; 30 ml of each was mixed with 0.02 g of smectite-HDTMA by mixers for 12 h. The solution was processed with a high-speed centrifuge; its supernatant was removed and diluted afterwards. An ultraviolet spectrometer was then adopted to test the optical density of the liquid at 230.0 nm to measure the concentration of the remaining aniline. PCP solutions of 18.75 and 250 mg/L with a pH value of 7 were also produced; 30 ml of each was mixed with 0.02 g of smectite-HDTMA by mixers for 12 h. The solution was processed with a high-speed centrifuge; its supernatant was removed and diluted afterwards. An ultraviolet spectrometer was then adopted to test the optical density of the liquid at 216.4 nm to measure the concentration of the remaining PCP.

### 2.5.3 | Adsorption experiment with two solvents

We took 0.02 g of smectite-HDTMA and mixed it with 15 ml of aniline solution of 125 mg/L and 15 ml of PCP solutions of concentrations varying between 18.75 and 250 mg/L in centrifuge tubes. We then conducted adsorption experiments at pH 7. Next, we carried out another set of experiments in which 0.02 g of smectite-HDTMA was mixed with 15 ml of PCP solution of 125 mg/L and 15 ml of aniline solutions of different concentrations (18.75–250 mg/L), respectively. A UV spectrometer was adopted to measure the concentrations of aniline and PCP in order to observe the adsorption capacity and mechanism of smectite-HDTMA when PCP and aniline are both present.

## 3 | RESULTS AND DISCUSSION

### 3.1 | FTIR analysis

Figure 1A–C indicate the results of the FT-IR analyses on HDTMA, as well as smectite and smectite-HDTMA, respectively. Figure 1B shows many distinctive and complex peaks inside the fingerprint region between 750 and 1170  $\text{cm}^{-1}$ , which reflects the existence of impure silicate (Si–O) and silicate (O–Si–O) bonds in silicate minerals. The –OH on the metal ion layer (octahedral layer) bends and vibrates, resulting in different absorption properties because  $\text{Al}^{+3}$  partially replaced  $\text{Si}^{+4}$  in the silicate layer (tetrahedral layer), and many metal ions could enter the octahedral layer. Besides, from Figure 1B, we can see that absorption peaks of octahedral –OH on the smectite only occur at wavenumbers between 3400 and 3700  $\text{cm}^{-1}$ , but after being modified with HDTMA, the absorption peaks of – $\text{CH}_2$ – and – $\text{CH}_3$  appear at 2910, 2846, and 1461  $\text{cm}^{-1}$ , respectively; this suggests that methyl molecules or carbon and hydrogen atoms in the alkyl carbon chain were successfully inserted into the structure of the smectite.<sup>25</sup> The results prove that smectite-HDTMA should have enough capacity to degrade pollutants during catalytic reactions.

### 3.2 | XRD analysis

#### 3.2.1 | XRD analysis of smectite

The XRD analysis of the smectite clay presents noticeable diffraction peaks after the purification and saturation processes using potassium and magnesium. The X-crystal lattices of the smectite are susceptible to the humidity of the atmosphere, allowing water molecules easy entry

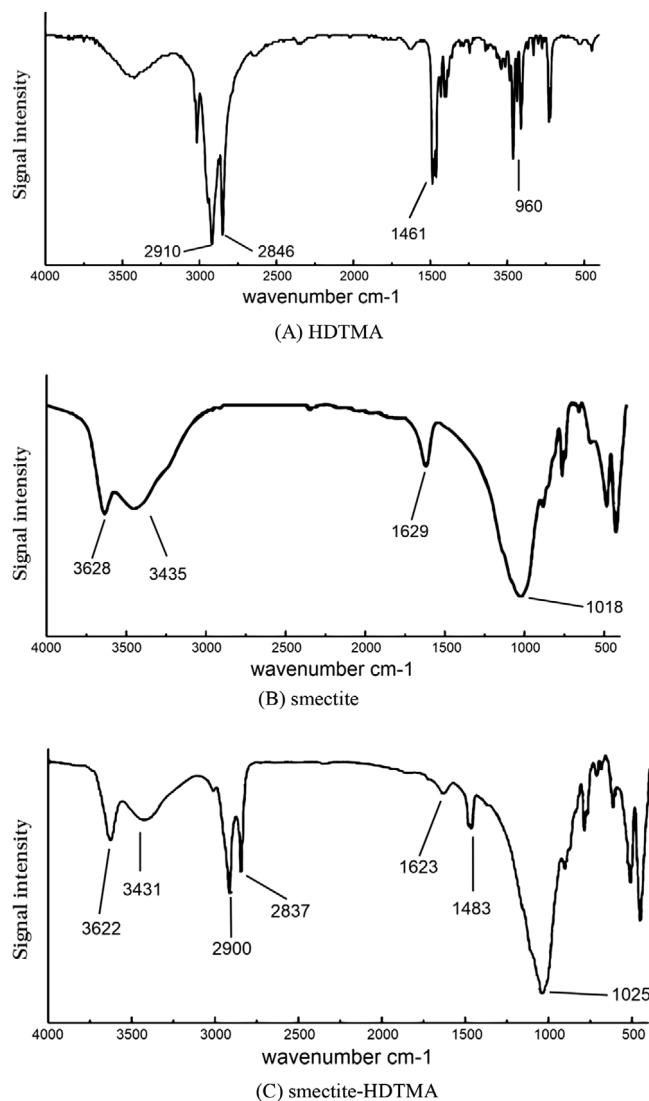


FIGURE 1 FT-IR spectrum of HDTMA (a), smectite (b) and smectite-HDTMA (c)

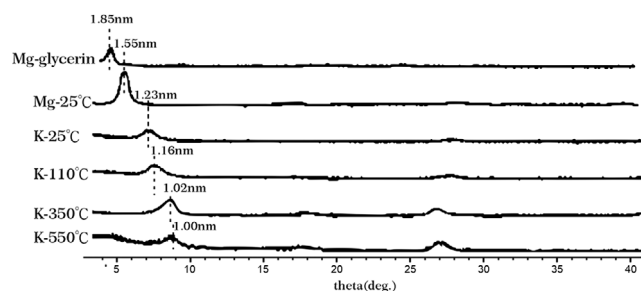


FIGURE 2 XRD analysis of smectite

into its interlayers. After the smectite clay was saturated with magnesium and steamed with glycerol, its interlayers expanded.<sup>26</sup> On the other hand, after it was saturated with potassium and heated, its interlayers contracted due to moisture loss. From Figure 2, we know that the diffraction

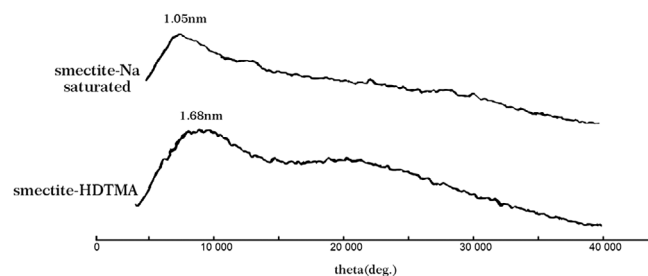


FIGURE 3 XRD analysis of Na-saturated smectite and smectite-HDTMA

peak of the potassium-saturated smectite at room temperature is 1.229 nm. When heated to 110°C, 350°C, and 550°C, the spacing of layers of the potassium-saturated smectite declined to 1.155 nm, 1.017 nm, and 1.004 nm, respectively. In contrast, that of the magnesium-saturated smectite at room temperature increased from 1.551 to 1.846 nm after being steamed with glycerol. From the XDR graph below, we can see that the contraction and expansion of the smectite are relatively remarkable.

### 3.2.2 | XRD analysis of smectite-HDTMA

Figure 3 shows the observation of the changes in the spacing of layers before and after modifying the smectite clay with HDTMA. The results indicate that the spacing of the layers was 1.05 nm before the smectite was modified with HDTMA; the spacing of the layers increased by 0.63 nm after the clay was modified with HDTMA. The results suggest that alkyl ammonium was inserted into the interlayers of the smectite clay and formed a double-layered alkyl ammonium structure, indicating that the application of the smectite-HDTMA as a catalyst was feasible and that the modified smectite can facilitate the electronic transition in the clay.<sup>27</sup>

### 3.3 | Efficiency of smectite-HDTMA

Bromophenol blue is a kind of diprotic acid indicator which forms (HDTMA)<sub>2</sub>BPB chains with HDTMA when its pH value is 8. In this study, we extracted (HDTMA)<sub>2</sub>BPB chains with high-extractability chloroform and analyzed them with a spectrometer. The spectrometer was used to scan the visible spectrum between wavelengths 700–300 nm, in order to identify characteristic adsorption peaks that were used to draw standard curves. As shown in Figure 4, the results obtained by scanning the wavelengths of solutions of different concentrations suggest a stable

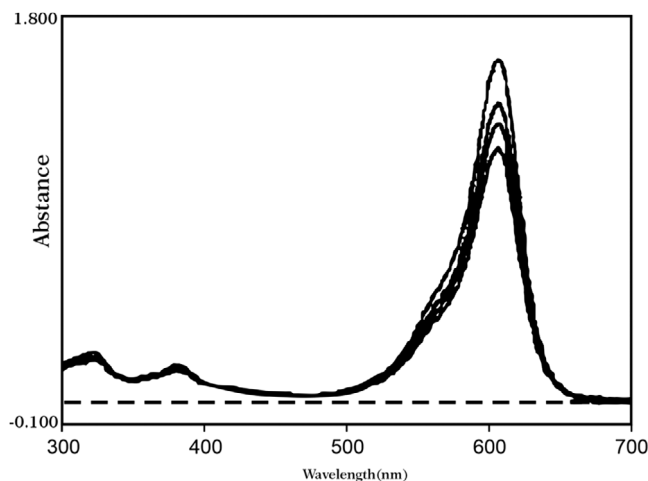


FIGURE 4 Results of scanning wavelengths of HDTMA solutions of different concentrations

TABLE 1 Specific surface area of smectite and smectite-HDTMA

Types	Specific surface area (m <sup>2</sup> /g)
Smectite	545
smectite-HDTMA	496

characteristic adsorption peak at 605 nm, which we used to measure the concentration of HDTMA. Moreover, in terms of 0.0047 M HDTMA solution's efficiency concerning modifying smectite, we measured the concentration of the remaining solution and found that the average was  $1.41 \times 10^{-4}$  M, reflecting a 97% decline.

### 3.4 | The determination of specific surface area of smectite and smectite-HDTMA

This study utilizes ethylene glycol to determine the specific surface area of smectite and smectite-HDTMA. The result is shown in Table 1. The specific surface area of the smectite carrier is bigger than that of the modified smectite-HDTMA. The specific surface area of smectite-HDTMA is smaller than that of smectite because hydroxyl groups on the surface of smectite-HDTMA bond with each other by condensation, then reactions such as nucleation and crystal growth occur, during the synthesis of smectite-HDTMA. This result suggests that although the specific surface area of synthesized smectite-HDTMA is smaller than that of smectite, it helps to improve the ability of smectite-HDTMA to adsorb organic pollutants while enhancing its chemical stability.

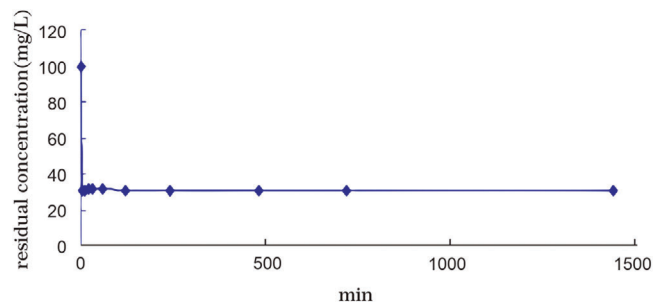


FIGURE 5 Results of the kinetic adsorption experiment of aniline with smectite-HDTMA

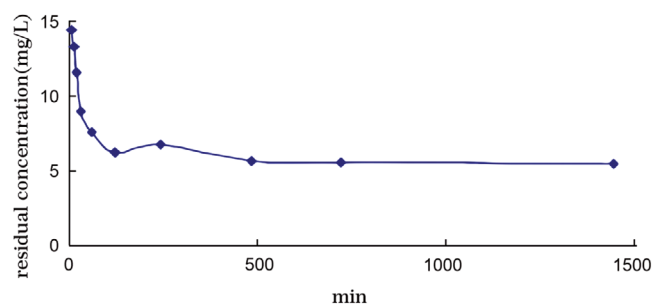


FIGURE 6 Results of the kinetic adsorption experiment on PCP with smectite-HDTMA

### 3.5 | Experiments on the adsorption ability of aniline and PCP

#### 3.5.1 | Adsorption kinetic experiment

The purpose of an adsorption kinetic experiment is to measure the time needed for an adsorption reaction to reach chemical equilibrium. Moreover, it aims to measure the time needed for reaching adsorption equilibrium by examining how adsorption capacity changes with adsorption time. The result can be used to determine the reaction time of the subsequent adsorption isotherm test and obtain the maximum adsorption capacity.<sup>28</sup> Figure 5 illustrates the relationship between time and the concentration of remaining aniline in the adsorption experiment conducted at pH 7 with smectite-HDTMA as the adsorbent. Figure 5 shows that adsorption kinetics is flat and that adsorption reaction reached the saturation point within 2 h and remained unchanged after reaching 8 h because smectite-HDTMA was bound to aniline mainly via distribution.<sup>21,29</sup> Therefore, future aniline adsorption experiments should be conducted for more than 2 h to ensure that adsorption reactions reach chemical equilibrium. Figure 6 shows how the concentration of PCP attracted to the smectite-HDTMA at a pH value of 7 changed by time. We found that the smectite-HDTMA almost reached saturation within 2 h

TABLE 2 Comparison of Freundlich model isotherm parameters of sorption for aniline and PCP

Components	Freundlich Equation	
	K(L/g)	R <sup>2</sup>
Aniline	2.304	0.7681
PCP	2.980	0.9997

into the experiment, and the concentration of PCP mostly remained the same after 24 h. The above results suggest that future PCP adsorption experiments should be carried out for more than 2 h to ensure that adsorption reactions reach chemical equilibrium.

#### 3.5.2 | Isothermal adsorption experiments

The curve for the isothermal adsorption experiment shows how adsorption capacity changes with equilibrium concentration. Under the condition that the temperature remains constant, adsorption capacity increases as the equilibrium concentration of adsorbate increases. In addition, said curve helps us understand the surface area of the adsorbent, pore volume, and pore-size distribution while determining the adsorption performance of the adsorbent toward adsorbed solvents.<sup>28</sup> To obtain the constant and maximum adsorption capacity of the adsorption isotherm model, we applied the data acquired from the isothermal adsorption experiment, in which smectite-HDTMA was used to bind aniline and PCP, to the Freundlich Equation. The results are presented in Table 2. Constant K in the Freundlich Equation, commonly treated as a distribution coefficient, indicates the ratio of 1-Octanol to water, which suggests the adsorption capacity of the organic phase. Besides, we also obtained the constant and maximum adsorption capacity of the Freundlich Equation by conducting isothermal adsorption experiments on aniline and PCP with smectite-HDTMA as the catalyst. The results are shown in Figures 7 and 8. The following is the Freundlich Equation:

$$\frac{X}{m} = KC^n \quad (1)$$

where  $C$  represents the equilibrium concentration of the adsorption capacity, and  $\frac{X}{m}$  indicates the adsorption capacity of the adsorption agent per unit. The  $K$  value is usually treated as the distribution coefficient and  $n$  as both empirical constants. Since the adsorption curve of the Freundlich Equation is linear, we can simplify the equation below by designating constant  $n$  as 1:<sup>30</sup>

$$\frac{X}{m} = KC \quad (2)$$

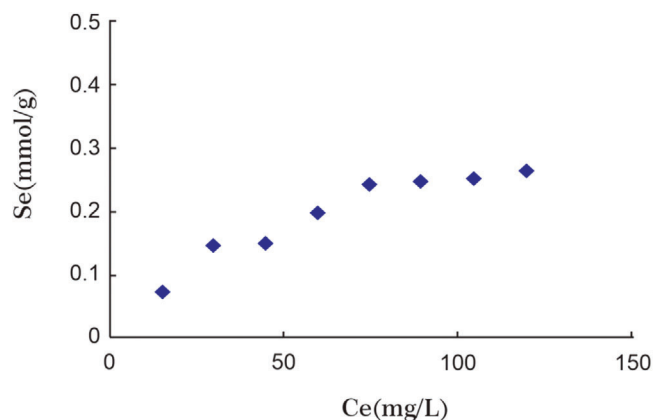


FIGURE 7 Results of the isothermal adsorption of aniline by smectite-HDTMA

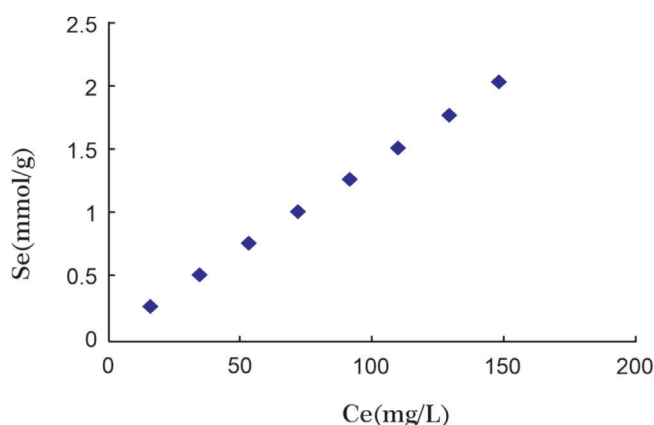


FIGURE 8 Results of the isothermal adsorption of PCP by smectite-HDTMA

Table 2 shows that smectite-HDTMA attracted more aniline than PCP, suggesting that the component with larger spacing of layers has higher adsorption priority.

In terms of the aniline and PCP isothermal adsorption experiments with smectite-HDTMA, the results presented in Figure 7 indicate that smectite-HDTMA could efficiently bind aniline; the adsorption capacity per unit rose with the equilibrium concentration.<sup>22,31</sup> However, when the equilibrium concentration reached 100 mg/L, the adsorption capacity did not increase as fast as it had before 100 mg/L. Figure 8 shows that the PCP adsorption capacity of smectite-HDTMA experienced linear growth as the concentration of smectite-HDTMA increased. The PCP adsorption capacity per unit was also found to be higher than that of aniline, probably because aniline (34,900 mg/L) had higher solubility and polarity than PCP (14 mg/L).

### 3.5.3 | Adsorption experiments on two solvents

This study conducted adsorption experiments on two solvents to observe how smectite-HDTMA binds aniline and PCP when both compounds are present, and whether competitive adsorption occurs between them. Figure 9 presents PCP and aniline adsorption capacity per unit obtained by conducting adsorption experiments with smectite-HDTMA on PCP of different concentrations and aniline of fixed concentration, as well as measuring the concentration of remaining aniline and PCP in the solution. From the graph, we found that the concentration of aniline barely changed as the concentration of PCP increased, meaning that the presence of PCP did not decrease aniline adsorption capacity, which was almost the same when aniline was the only component present. Figure 10 shows PCP and aniline adsorption capacity per unit obtained by conducting adsorption experiments with smectite-HDTMA on aniline of different concentrations and PCP of fixed concentration, as well as measuring the concentration of the remaining aniline and PCP in the solution. In terms of HDTMA-modified organoclay adsorption of PCP when both PCP and aniline are present in the solution, Figure 10 indicates that the PCP adsorption capacity of smectite-HDTMA could remain at a certain level even when the concentration of aniline increased. Furthermore, Figures 9 and 10 show that smectite-HDTMA with long alky chains does not have competitive adsorption ability, which proves that organoclay synthesized with HDTMA mainly attracts aniline and PCP via distribution processes and that competitive adsorption does not occur during distribution by organic compounds.<sup>10,15</sup> In other words, the adsorption capacity will remain the same no matter whether one or multiple organic compounds are present because the adsorption mechanism is a solution process, which has nothing to do with the adsorption position on the solvent surface.<sup>31–33</sup>

## 4 | CONCLUSION

In this study, we purified commercially available smectite and modified it with HDTMA. From changes in the spacing of layers, we assumed that the modified smectite featured a double-layered structure. In terms of kinetic adsorption experiments, smectite-HDTMA attracts aniline and PCP via distribution reaction, and that future adsorption experiments on aniline and PCP should be carried out for more than 2 h to ensure that the reaction reaches chemical equilibrium. In terms of isothermal adsorption

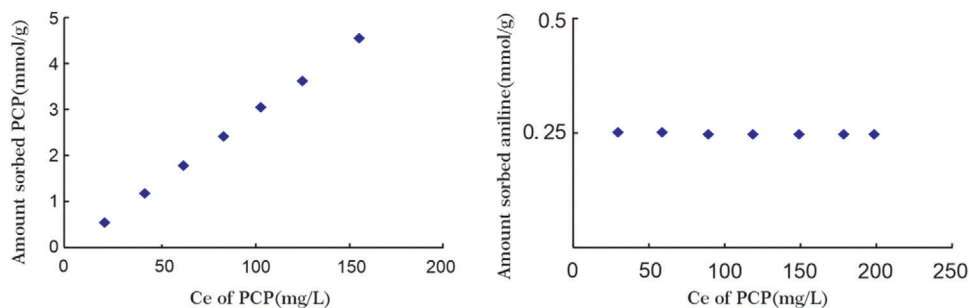


FIGURE 9 Adsorption of smectite-HDTMA when PCP of different concentrations and aniline of a fixed concentration were presented

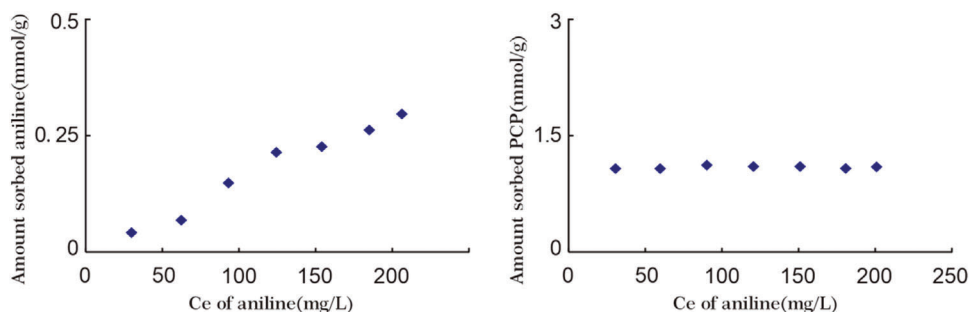


FIGURE 10 Adsorption of smectite-HDTMA when aniline of different concentrations and PCP of a fixed concentration were presented

experiments, PCP and aniline adsorption capacity of smectite-HDTMA increased with the equilibrium concentration. In double solvent experiments, the results show that the adsorption of HDTMA-modified smectite with long alky chains was not competitive, proving that organoclays formed with HDTMA mainly attract aniline and PCP via distribution.

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## CONFLICT OF INTEREST

The authors declare no conflicts of interest.

## DATA AVAILABILITY STATEMENT

Data are available upon request.

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