# ORIGINAL PAPER

# The structure of montmorillonites modified with zwitterionic surfactants and their sorption ability

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Abstract In this work, a novel organo-clays, zwitterionic surfactant modified montmorillonites (ZSMMs) were synthesized by using sulphobetaine and montmorillonites. The structures of ZSMMs were characterized by X ray diffraction (XRD) methods; the surfactant loading levels were measured by Total organic carbon (TOC) analysis, and their sorptive characteristics toward p-nitrophenol and nitrobenzene were investigated. XRD and TOC measurements indicated that the amount of adsorbed surfactants and the basal spacing of the ZSMMs increase with alkyl chain length and surfactant concentration. Sorption experiments showed that the capacity of p-nitrophenol to sorb onto the ZSMMs is higher than that of nitrobenzene. Both capacities increase with surfactant loading level; However, sorption capacity decreases when the surfactant concentration is higher than 2.0 CEC. Under the same surfactant loading level, the sorption capacities of pnitrophenol and nitrobenzene increase with alkyl chain length. Under this experimental condition, the longer alkyl chain leads to a higher sorption capacity for hydrophobic organic compounds. On the basis of the ability of p-nitrophenol and nitrobenzene to sorb onto the montmorillonites, we conclude that the contaminant sorption coefficients, normalized with organic carbon content, highly depend on surfactant loading levels.

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# Introduction

Montmorillonite (Mt) has attracted much interest as sorbents because of their high cation exchange capacity, excellent swelling, and sorption, as well as their special nanometer scale interlayer space, large specific surface area, and consequential sorption. However, raw Mt weakly adsorb hydrophobic organic compounds (HOCs) from water because of the strong hydrophilicity of their surfaces and interlayers. Montmorillonite modification using surfactants is a strategic measure in improving the affinity of the mineral toward organic matter. When the exchangeable metal cations in the interlayer of Mt are replaced by organic cations, the surface properties of Mt can be transformed from hydrophilic to hydrophobic. As a result, the capacity of Mt to sorb onto organic contaminants can be dramatically enhanced (Boyd et al. 1988; Khaodhiar and Changchaivong 2009; Liao et al. 2006). As effective sorbents, organo-montmorillonites (OMts) may therefore have potential applications in the removal of organic contaminants from wastewater (He et al. 2008; Li et al. 2010); their sorption mechanisms have been widely investigated (He et al. 2006, 2008; Wang et al. 2010; Zampori et al. 2009; Zhu et al. 2008b).

In the past two decades, numerous kinds of surfactants have been used to synthesize OMts. The structural characteristics and sorption mechanisms of the resultant OMts have been extensively reported (Zhu and Ma 2007; Zhu et al. 2008c). To date, the most commonly used surfactants in the synthesis of OMts are cationic surfactants, such as quaternary ammonium, which can be easily intercalated into the clay interlayer space via cation exchange (Boyd et al. 1988; Frost et al. 2007; He et al. 2006, 2008; Sheng et al. 1998). Aside from cationic surfactants, anionic (Yang et al. 2007; Zhu and Chen 2000), nonionic (Guegan 2010; Rodriguez-Cruz et al. 2005), and amphoteric surfactants (McLauchlin and Thomas 2008, 2009) have also been used to modify montmorillonites. Research on the sorption of charged or uncharged polar

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organic compounds onto OMts and the mechanisms by which this occurs indicate that the electrochemical properties of the surfactants have a tremendous effect on sorption capacity (Gennari et al. 2009; Sanchez-Martin et al. 2008). However, including our recent works about the preparation and characterization of zwitterionic surfactant modified montmorillonites (Zhu et al. 2011), only a few studies on zwitterionic surfactant-modified montmorillonites (ZSMMs) have been published (Grandjean 2001; Meneghetti and Qutubuddin 2004; Yamaguchi and Hoffmann 1997), and the physical and chemical properties of ZSMMs, such as the micro-structure, intercalation mechanism and sorption ability towards environmental contaminant still remain unclear.

As a kind of zwitterionic surfactant with a positively charged trimethylammonium group and a negatively charged sulfonate group, sulfobetaine (SB), consistently showed zwitterionic characteristics and a tendency toward cationic and anionic interaction across all pH ranges. Given their cationic properties, zwitterionic surfactants can enter the interlayer space of montmorillonites by inorganic cation exchange, similar to the process of cationic surfactants penetrate into the interlayer. On the other hand, the negatively charged sulfonate group may be the potential adsorption sites for some heavy metal cations. In addition, zwitterionic surfactants have unique properties, such as excellent water solubility, ideal biodegradability, and biological safety (Qi et al. 2008). Hence, ZSMMs are expected to address the issue of secondary pollution caused by surfactants released from traditional organo-montmorillonites. They can be used for wastewater treatment and effectively remove organic contaminants with both negative and positive charges.

In the present study, three series of ZSMMs were prepared at different surfactant concentrations (0.2–3.0 CEC). Montmorillonites and zwitterionic surfactants with different alkyl chain lengths were used. The ZSMMs were characterized by X-ray diffraction (XRD) to obtain the states of basal space swelling in water, and total organic carbon (TOC) methods to derive the amount of adsorbed surfactant. These ZSMMs were used as sorbents to remove the organic contaminants p-nitrophenol (PNP) and nitrobenzene (NB) from aqueous solutions by the batch sorption technique. Surfactant loading level, alkyl chain length, and the nature of organic pollutant effect on sorption properties were investigated. The results will contribute to the development of environmentfriendly and highly efficient sorbent materials.

## Materials and methods

# Materials

compositions of the montmorillonite are SiO<sub>2</sub> 58.16 %, Al<sub>2</sub>O<sub>3</sub> 16.95 %, Fe<sub>2</sub>O<sub>3</sub> 5.26 %, CaO 2.29 %, MgO 3.57 %, K<sub>2</sub>O 0.15 %, Na<sub>2</sub>O 0.19 %, MnO 0.027 %, TiO<sub>2</sub> 0.2 %, P<sub>2</sub>O<sub>5</sub> 0.08 %, and the ignition loss is 13.23 %. The cation exchange capacity (CEC) was 110.5 meq/100 g.

Chemically pure 3-(N,N-dimethyldodecylammonio) propane sulfonate (SB12), 3-(N, N-dimethylmyristylammonio) propane sulfonate (SB14), and 3-(N, N-dimethypalmitylammonio) propane sulfonate (SB16) were purchased from Nanjing Robiot Co., Ltd. (China). The PNP and NB (Shanghai Chemical Co., China) were of analytical grade. All of the reagents were used as received.

#### Preparation of ZSMMs

The synthesis procedure of ZSMMs has been depicted in the literature (Grandjean 2001). A specific amount of surfactant was dispersed in distilled water, and then completely dissolved by ultrasound at 60 °C for 0.5 h. Afterward, 30 g of montmorillonite was slowly added to the mixture. The initial amounts of surfactants were equivalent to 0.2, 0.4, 0.6, 0.8, 1.0, 1.5, 2.0, 2.5, and 3.0 times of the montmorillonite CEC. The water: montmorillonite mass ratio was 20: 1. The reaction was carried out at 60 °C for 15 min by ultrasonic method with 100 Hz. All products were washed three times with distilled water, dried at 80 °C, grinded in an agate mortar, and passed through a 100-mesh sieve. The surfactant concentration and wet state was appended to the label (i.e., SB12-0.2 W) to indicate a 0.2 CEC wet sample. All other products were labeled in the same manner.

## Characterization methods

The organic carbon contents (foc) of the raw Mt and the ZSMM solid samples were obtained using a LECO-C230 organic carbon analyzer. The organic carbon content at the solution were determined using a Shimadzu TOC-VCPH analyzer. The XRD patterns of the wet samples were recorded using a Bruker D8 Advance diffractometer with Cu K $\alpha$  radiation (40 kV and 40 mA) at a relative humidity of 60-70 % and 27 °C. The samples were scanned between 1° and 20° (20) at a speed of 2° min<sup>-1</sup> and the testing was performed twice in a row, the difference between the two results is tenuous, and so the structure changes caused by water evaporation in about 15 min could be ignored.

#### Sorption of PNP and NB onto the ZSMMs

The sorption of PNP and NB in the ZSMMs were studied in batch experiments. 0.2 g ZSMM powder was combined with 20 mL solution PNP or NB solution with designed concentration (pH,  $\sim$ 7.0) in 25 mL glass centrifuge tubes. The tubes were sealed with Teflon-lined caps, and then were shaken at 150 rpm for 6 h under 25 °C (Preliminary experiments showed that the sorption equilibrium could be reached within 4 h). After being centrifuged at 4,000 rpm for 20 min, The 1~10 mL supernatant, according to the solution concentration, was analyzed using a UV-260 spectrophotometer at wavelengths of 399 nm for PNP and 268 nm for NB to obtain the residual concentration of PNP and NB. The adsorbed amounts of PNP and NB were calculated based on the difference between their initial and equilibrium concentrations in the solution. The UV absorbance of three zwitterionic surfactants is lower than 0.04 at 268 and 399 nm, so their effects on the adsorbed amounts of PNP and NB are negligible. The blank samples showed that the sorption of PNP or NB onto the tube is negligible and the loss of PNP and NB by photochemical decomposition and volatilization are negligible during sorption.

# Results

# Structures of the ZSMMs

In previous work, the structure of ZSMMs has been studied by XRD method (Zhu et al. 2011). However, the previous structure information about the ZSMMs was obtained from dried powder, which may be deviated with real situation in aqueous

state. As we know, the ZSMMs work in water when used as sorbent in waste water treatment, so the XRD data obtained from the hydrated samples will be more accurately reflect the structure of ZSMMs in water, which is important for understanding the sorption mechanisms by ZSMMs.

The effect of alkyl chain length and surfactant loading on the basal spacing of organoclays and arrangement of intercalated surfactants has been the subject of many investigations (Chen et al. 2005; He et al. 2006; Lagaly 1981; Zhou et al. 2007). Fig. 1 shows the XRD patterns of wet ZSMMs prepared with SB12 (Fig. 1 (a)), SB14 (Fig. 1(b)), and SB16 (Fig. 1 (c)) at a series of surfactant concentrations (0.2–3.0 CEC). The basal spacing of the wet ZSMMs increased with surfactant loading while the maximum basal spacing increased as the alkyl chain length increased. The maximum basal spacings of SB12, SB14, and SB16 modified Mts were 4.17, 4.73, and 5.37 nm respectively. The basal spacing ( $d_{001}$ ) depended on surfactant loadings and length of alkyl chains.

Compared to the  $d_{001}$  value of dry ZSMMs, the hydrated ZSMMs have a larger basal spacing (Zhu et al. 2011), meanwhile the surfactant concentration under which the maximum  $d_{001}$  reached is lower than the dry OMts. Fox example, the SB16–0.8 W sample had a 4.73 nm  $d_{001}$  value at 0.8 CEC concentration. Obviously, due to the water molecular present, the wet OMts obtained a higher interlayer spacing than the dry OMts. The exact structure of wet OMts might be helpful to understand the adsorption mechanism to HOCs.



Fig. 1 XRD patterns of ZSMMs modified by SB12(a), SB14(b) and SB16(c) under wet state

#### Sorption of surfactant onto Mt

The relationship and amount of surfactant added is shown in Fig. 2. The sorbed amount increases with the amount of surfactant added and alkyl chain length. At low concentration (added amount <1.5 CEC), the  $f_{oc}$  increases quickly. As the concentration increased further, the  $f_{oc}$  reaches a plateau. Based on the organic-carbon contents in the supernatants during preparation of ZSMMs, the amounts of surfactant adsorbed were calculated, and the exact loading level of zwitterionic surfactant (Csorbed/CEC) was derived (foc: organic carbon content of the sorbates; Csorbed/CEC: saturated CEC by the intercalated surfactants;  $K_{d}$ : sorption coefficient;  $K_{sf}$ : organic carbon content normalized with the sorption coefficient ( $K_{sf} = K_d / f_{oc}$ ).). In the case of sorbed levels ( $C_{sorbed}$ /CEC) of <1.0, most of surfactant was taken up by montmorillonite. At higher surfactant loading levels, C<sub>sorbed</sub>/CEC increased gradually.. The zwitterionic surfactant uptakes by Mts via a two-step process too. Both cation-exchange and nonexchange processes contribute to the overall uptake of long alkyl-chain organic surfactant by clays (Chen et al. 2005).

# Sorption of PNP and NB by ZSMMs

Figures 3 and 4 present the sorption isotherms of PNP and NB in the ZSMMs. The initial concentrations of PNP and NB are 20, 50, 80, 110, 140, 170, and 200 mg/L respectively. As illustrated in the figures, the sorption isotherms of PNP (Fig. 3 (a), (b), and (c)) and NB (Fig. 4 (a), (b), and (c)) in all the ZSMMs are linear and can be defined as C type isotherm, indicating that the sorption is dominated by the partition process. Linear regression was used to fit these isotherms, and sorption coefficient  $K_d$  was determined from the slope of the obtained linear equation.

When the added amount of surfactant is 0.2 CEC, the amounts of PNP and NB adsorbed onto SB12-Mt and SB14-Mt are both less than 1 mg/g, and less than 2 mg/g in



Fig. 2 The relation of total organic carbon and amount of added surfactant of organo montmorillonites

SB16-Mt. With the increase in surfactant loading amounts, the  $K_d$  levels of PNP and NB rapidly increase. For example, as surfactant concentration is increased to 0.6, 1.0, 2.0, and 3.0 CEC, the  $K_d$  of SB12-Mt increases by 4.4, 11.9, 18.1, and 39.1 times for PNP, and 2.6, 6.1, 8.0, and 11.6 times for NB, respectively. By contrast, the  $K_d$  of SB14-Mt dramatically decreases when the amount of added surfactant increases to 3.0 CEC.

Figure 5 shows that the  $K_d$  value of PNP is higher than that of NB in the ZSMMs, indicating that the capacity of PNP to sorb onto the ZSMMs is higher than that of NB. The S and  $K_{ow}$  of PNP are 1.6 µg/mL and 81, respectively. Meanwhile, those of NB are 0.2 µg/mL and 71, respectively. The order of the sorption capacity on the ZSMMs is consistent with that of the S and  $K_{ow}$  values of PNP and NB. So the water-soluble organic (S) and octanol-water partition coefficient ( $K_{ow}$ ) of HOCs are important factors that affect the sorption capacity of HOCs onto OMTs.

The  $K_d$  values in Fig. 5 also indicate that the sorption capacity of these ZSMMs follows the order SB16-Mt>SB14-Mt>SB12-Mt when the surfactant loading level is lower than 1.8 CEC. However, the order changes when the surfactant loading is higher than 1.8 CEC. The  $K_d$  values of PNP and NB in SB12-Mt continually rise, whereas those in SB14-Mt and SB16-Mt show the reverse. As a result, the sorption capacity of SB12–3.0 is higher than that of SB14–3.0.

Given that surfactant modifiers contribute most to the costs involved in the preparation of organo-montmorillonites, the  $f_{oc}$  normalized sorption coefficient  $(K_{sf}=K_d/$  $f_{\rm oc}$ ) was suggested as a more suitable indicator of the sorption capacity of OMts (Chen et al. 2005; Wang et al. 2010). The calculated  $K_{sf}$  of PNP and NB against sorbed surfactant levels (Csorbed/CEC) are presented in Fig. 6 (a) and (b). The  $K_{sf}$  of PNP and NB are strongly dependent on surfactant loading. In the case of SB12 ZSMMs, The  $K_{sf}$  increase with surfactant levels. Conversely, the  $K_{\rm sf}$  values of SB14 and SB16 ZSMMs initially increase with surfactant loading levels until the maximum is reached, at which point they begin to decrease. The maximum  $K_{sf}$  of PNP appears at 1.8 CEC (Fig. 6 (a)), and the maximum  $K_{sf}$  of NB appears at 1.0 CEC for SB16 and 1.8CEC for SB14(Fig. 6 (b)).

Previous studies showed that the carbon-normalized sorption coefficients of nitrobenzene (Ksf of NB) on the cetyltrimethylammonium bromide (CTMAB) modified montmorillonite were approximately in the range of 50–500 mL/g [8], which is bigger than those on the SB16 (123–249 mL/g) (Fig. 6(b)). However, in consideration of the negatively charged sulfonate group may be the potential adsorption sites for some heavy metal cations inorganic contaminants, ZSMMs can perhaps sorption organic and inorganic contaminants simultaneously. Of course further sorption



Fig. 3 Sorption isotherms of p-nitrophenol on the ZSMMs of SB12(a), SB14(b), and SB16(c)

experiments of ZSMMs toward to organic and inorganic contaminants are needed.

## Discussion

Previous research on montmorillonite modified by cation surfactant show that surfactant loading levels and alkyl chain length can influence sorption properties toward HOCs (Frost et al. 2008; Zhou et al. 2007). The comparative study on the capacity of PNP and SB to sorb onto three series of ZSMMs reflect similar results. The longer the alkyl chain and the higher the amount of loaded zwitterionic surfactants, the greater the sorption capacity for HOCs in the ZSMMs. This result can be ascribed to the strong hydrophobic interaction.

In more recent studies, researchers attributed the synergistic effect (caused by hexadecyltrimethyl- ammonium organo-montmorillonites) on the uptake of HOCs in binary solute systems from water to the occurrence of multiple sorption mechanisms, including solvation with ammonium cations and mineral surfaces, and partition with the hexadecyltrimethylammonium organic phase (Boyd et al. 1988; Sheng et al. 1996). Sorption on montmorillonites modified with small quaternary ammonium cations exhibit mainly adsorption; the adsorption isotherms of OMts are non-linear. The sorption on montmorillonites modified with quaternary ammonium cations containing large alkyl groups is more complex, but controlled mainly by the partition process (Khaodhiar and Changchaivong 2009; Zhu et al. 2008a).

According to the results on structural characteristics, the long-chain alkyls intercalate into the interlayer space of the ZSMMs, making the interlayer surface more hydrophobic. These long-chain alkyls can adopt different arrangements and conformations under different surfactant loading levels. Thus, the alkyls



Fig. 4 Sorption isotherms of nitrobenzene on the ZSMMs of SB12(a), SB14(b), and SB16(c)



Fig. 5 Relationship between the  $K_d$  of PNP and NB with the sorbed zwitterionic surfactant levels ( $C_{\text{sorbed}}$ /CEC)

aggregate in the interlayer space, and compose partition phases similar to what they exhibit in the interlayer of montmorillonites were modified with hexadecyltrimethylammonium. The sorption isotherms of PNP and NB in SB12, SB14, and SB16 ZSMMs are linear, indicating that the sorption is a partition process, and the sorption force is mainly due to the hydrophobic interaction distribution between the surfactant alkyl chains.

When the amount of adsorbed surfactant is higher than 1.8 CEC, the amounts of PNP and NB sorbed onto SB14 organo-montmorillonites dramatically decrease. The sorption capacity of NB by SB14–3.0 and SB16–3.0 is lower than that of SB12–3.0, even though they have the higher organic carbon content. Because of the confined interlayer spacing, the stack density of



alkyl chains in the interlayer space will increase with the surfactant loading levels. As a result, large parts of the interlayer space on SB14–3.0 and SB16–3.0 are occupied by the surfactants, leading to the "steric effect" during the sorption of PNP or NB and causing the decrease in sorption capacity (Chen et al. 2005; Zhu et al. 2008b). In the case of SB12–3.0 sample, the exact loading amount is only 2.08 CEC, the highest stack density of alkyl chains is not reached yet. Therefore, the capacity of HOCs to sorb onto the ZSMMs is dependent not only on the strength of hydrophobic interaction, but also on the amounts of available sorption sites.

# Conclusions

ZSMMs can be effective sorbents for HOCs in water. The sorption capacity of ZSMMs toward PNP is higher than that of NB. The sorption processes of PNP and NB in the ZSMMs are dominated by the partition mechanism. The alkyl chain length and the amount of surfactant loading considerably influence the sorption capacity of the ZSMMs. Under these conditions, better sorption properties are achieved with a longer alkyl chain length and a higher surfactant loading level. The capacities of organic compounds to sorb onto the three ZSMMs rapidly increase when the amount of adsorbed surfactant is lower than 1.8 CEC, but decrease when the surfactant loading levels continually increase. To improve the sorption capacity of ZSMMs, both surfactant loading levels and alkyl chain length should be properly controlled. These results are expected to aid in the understanding, prediction, and maximization of ZSMM sorption capabilities. Moreover, this study can be a reference in the design of new families of organo-montmorillonites for wastewater treatment.

Fig. 6 The relationship between carbon-normalized solute sorption coefficients by sorbed zwitterionic surfactant ( $K_{sf}$ ) and sorbed surfactant levels ( $C_{sorbed}$ / CEC); (a)PNP; (b)NB



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