

# Effects of Surfactants on the Sorption of Sulfamethoxazole by Sediment

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## Abstract

Adsorption and desorption of sulfamethoxazole (SMX) at water-sediment interfaces from Taihu Lake, China, were investigated in this study. The effects of a cationic surfactant, cetyltrimethylammonium bromide (CTAB), and an anionic surfactant, sodium dodecylbenzene sulfonate (SDBS), on the adsorption/desorption processes were also studied. Results show that adsorption and desorption could be well described by the Freundlich isotherm model. CTAB significantly increased the SMX adsorption on sediments, while SDBS decreased the adsorption of SMX, probably because SDBS at high concentrations competed in the limited adsorption sites with SMX, and the addition of SDBS increased SMX solubility, thereby reducing the SMX adsorption on sediment. The degree of adsorption irreversibility was quantified by the Thermodynamic Index of Irreversibility (*III*) values. It shows that the *III* values increased with the increase of CTAB concentration, and decreased with the increase of SDBS concentration. These results imply that cationic and anionic surfactants may have contrasting impacts on the distribution and transport of SMX in the environment.

**Keywords:** sulfamethoxazole, adsorption, desorption, surfactant, adsorption irreversibility

## Introduction

Sulfonamides are one class of the most important antimicrobial agents that have been largely used in human therapy, livestock production, and aquaculture [1]. The consumption of sulfonamides is increasing in both industrialized and developing countries, resulting in their often being detected in sewage effluents [2], surface waters [3, 4], and sediments [5] around the world. In the aquatic environment sulfonamides mainly originate from sewage effluents from wastewater treatment plants, where these chemicals are only partly eliminated in treatment processes and finally reach ambient surface waters [6]. Their occurrence has

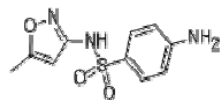
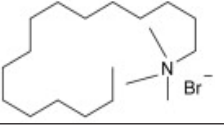
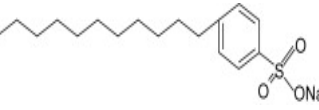
been reported in many publications [3, 4, 7], but knowledge on the transport and fate of sulfonamides in the aquatic environment is still not well documented. Understanding the distribution of these compounds in different environmental compartments (e.g. surface water, sediments) is essential for evaluating the transport and ultimate fate of these drugs in the aquatic environment.

Adsorption and desorption have been considered the most important processes controlling interaction between contaminants and sediments. After entering into the aquatic environment, chemicals will tend to be adsorbed onto sediment surfaces and, for organic compounds, partitioned into sediment organic matter phases. For sediment-associated contaminants, the irreversibly adsorbed fraction is the greatest concern and uncertainty, because it significantly

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Table 1. Characteristics of SMX, CTAB, and SDBS.

Chemicals	Formula	Molecular weight	CMC <sup>a</sup> (mmol·L <sup>-1</sup> )	HLB <sup>b</sup>	Structure
SMX	C <sub>10</sub> H <sub>11</sub> N <sub>3</sub> O <sub>3</sub> S	253	pKa1=1.7 pKa2=5.6	----	
CTAB	C <sub>19</sub> H <sub>42</sub> BrN	364	0.9 <sup>a</sup>	15.8	
SDBS	C <sub>18</sub> H <sub>29</sub> NaO <sub>3</sub> S	348	1.2 <sup>b</sup>	11	

<sup>a</sup> Quoted from [15].

<sup>c</sup> Quoted from [16].

<sup>b</sup> HLB (Hydrophile-Lipophile Balance Number) quoted from Internet.

affects chemical fate, toxicity, risk to human and aquatic life, and efficiency of most remediation technologies [8]. The sorption reversibility is crucial in controlling the physical/biological availability of sorbed contaminants and guiding sediment quality management [8, 9].

The adsorption and desorption processes may be affected by factors such as sediment properties, concentration and characteristics of contaminants, the presence of surfactant-like solutes, and environmental conditions, etc. [9]. For instance, Boxall et al. [10] found that the adsorption coefficient ( $K_d$ ) of sulfonamides increased from less than 1 up to 30 when pH values decreased from 8 to 4. Surfactants may significantly affect the adsorption and desorption of contaminants in the environment due to their amphiphilic structures, and their influence is concentration-dependent [11]. Adsorption of aniline and p-nitrophenol on sediment was enhanced with the presence of SDBS with concentrations increasing from 200 to 1,200 mg·L<sup>-1</sup> [12]. Low SDBS concentrations (lower than the critical micelle concentration (CMC)) promoted the desorption of aldicarb (a more hydrophilic pesticides) from spiked soil, while high SDBS concentrations (higher than CMC) would inhibit the desorption [13]. Surfactant micelles played an important role in the solubility enhancement of aldicarb, so that the solubility enhancement effect of surfactants could be used to improve the elution efficiency of aldicarb from soils [14]. The characteristics of transfer and transport of contaminants are strongly affected by the interaction between surfactants and contaminants.

In this study, the adsorption and desorption behavior of a selected sulfonamide, sulfamethoxazole (SMX), on sediment from Taihu Lake was investigated. Taihu is the third largest freshwater lake in China, and the contamination of antibiotics in the lake due to livestock farming and aquaculture has drawn much concern. Our previous study has shown that sulfonamides occurred in water, sediment and porewater samples from Taihu Lake (unpublished data). The objective of this study is to experimentally investigate adsorption and desorption of SMX on Taihu sediment.

The effect of cationic/anionic surfactants on the adsorption and desorption processes was also studied. The results will be helpful to better understand the environmental fate of sulfonamide antibiotics in the sediment-water system.

## Materials and Methods

### Chemicals and Materials

Sulfamethoxazole (99.9%) was purchased from Tokyo Chemical Industry (Tokyo, Japan). Cetyltrimethylammonium bromide (CTAB) and sodium dodecylbenzene sulfonate (SDBS) were purchased from Sigma-Aldrich (Seelze, Germany). Methanol (HPLC) was purchased from Fisher chemical (Fair Lawn, New Jersey, USA). Other used reagents such as NaOH, HCl, and CaCl<sub>2</sub> were all analytical grade. Structures and some relevant properties of SMX, CTAB and SDBS are given in Table 1.

The sediment was taken from Taihu Lake. After collection, the sediment was sealed in a stainless steel bottle on ice and transported to the lab. The sediment was freeze-dried and passed through a 0.5 mm sieve to remove coarse fragments and stored in the dark at 4°C. The XRD pattern of the Taihu sediment in Fig. 1 demonstrated that the sediment sample mainly contained SiO<sub>2</sub>, CaAl<sub>2</sub>Si<sub>2</sub>O<sub>8</sub>, and other components. The specific surface area of the sediment was determined by the Brunauer-Emmett-Teller method of N<sub>2</sub> adsorption. The sediment used for the experiment was analyzed and shown to be SMX-free. Salient physicochemical characteristics such as OM, BET surface area, and porous structure of the sediment are summarized in Table 2.

### Adsorption and Desorption Experiments

All the adsorption/desorption experiments were carried out in 100 mL polyethylene (PE) centrifuge tubes. Parallel control tests in the absence of sediment showed that the total SMX loss (e.g. sorption on the tube wall, photo-degradation)

Table 2. Characteristics of sediment used in this study.

Sediment	OM <sup>a</sup> (g·kg <sup>-1</sup> )	pH <sup>b</sup>	SSA <sup>c</sup> (m <sup>2</sup> ·g <sup>-1</sup> )	Specific pore volume (cm <sup>3</sup> ·g <sup>-1</sup> )	Pore diameter (nm)	Composition <sup>d</sup>
Taihu Lake	10.42	7.08	16.9964	0.0519	12.21	SiO <sub>2</sub> ,CaAl <sub>2</sub> Si <sub>2</sub> O <sub>8</sub>

<sup>a</sup> Organic-carbon weight in the whole sediment, potassium dichromate-outside heating method.

<sup>b</sup> Sediment: water 1:2.5 (W/V).

<sup>c</sup> Specific surface area, determined by N<sub>2</sub>-BET method

<sup>d</sup> Chemical composition came from XRD analysis results.

was less than 5%, which was neglected in the experiment. The aqueous solution used in adsorption experiments contained 0.1 mmol·L<sup>-1</sup> CaCl<sub>2</sub> to mimic background surface water ionic strength and to reduce dispersion of fines. An adsorption kinetic study was conducted with sampling at 0, 4, 8, 12, 24, 48, and 96 h, respectively, which showed that nearly 98% of adsorption was achieved within 24 h. On this basis, 24 h was chosen as the equilibration time for all adsorption experiments.

Duplicate sets of PE tubes were filled with 2.00 g of sediment and 50 mL of 0.1 mmol·L<sup>-1</sup> of CaCl<sub>2</sub> solution containing SMX at concentration ranging from 0.5 mg·L<sup>-1</sup> to 10 mg·L<sup>-1</sup>. In order to test the effect of the surfactants on the adsorption behavior of SMX on the sediment, CTAB or SDBS was added to the solutions at initial concentrations of 3.61, 18.1, and 36.1 mg·L<sup>-1</sup> for CTAB, and 4.34, 21.7, and 43.4 mg·L<sup>-1</sup> for SDBS. Concentration levels for the surfactants was far below the critical micelle concentration (CMC), which was 361 mg·L<sup>-1</sup> for CTAB and 433.5 mg·L<sup>-1</sup> for SDBS, respectively. The sediment samples were equilibrated on a mechanical shaker for 24 h at 200 rpm at 25±1°C. During the experiment, the solution pH was kept at 7.0±0.1 by intermittent adjustment with 0.02 mol·L<sup>-1</sup> HCl or NaOH solutions. After equilibration, samples were centrifuged at 8,000 rpm for 10 min. One mL of each sample supernatant was decanted to a 2 mL GC vial, and analyzed for SMX remaining in the aqueous phase. The adsorbed amount was calculated from the difference between the initial and final solute concentrations in the solution.

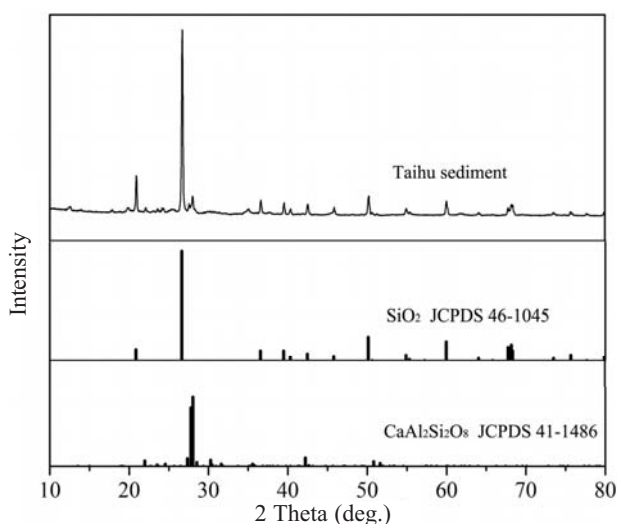


Fig. 1. X-ray diffraction spectrum of Taihu sediment.

Desorption and kinetics experiments were performed on the sediments that reached adsorption equilibrium. 45 mL of the centrifuged supernatant was removed and the tubes were refilled to the original volume by adding 0.1 mmol·L<sup>-1</sup> of CaCl<sub>2</sub> solution containing no SMX. The mixtures were then re-equilibrated for 24 h under the same conditions (pH, temperature, and rotating velocity) as for the adsorption experiments. Upon equilibrium, the distribution of SMX between the sediment and solution was analyzed following the same procedures described above. The dilution and re-equilibration procedures were repeated successively to yield the desorption isotherms.

### SMX Solubility in Surfactant Solutions

Batch experiments were performed to quantify the changes of aqueous solubility of SMX in the presence of CTAB and SDBS. In each test, 50 mg of SMX was added in 50 mL of the solution that contained 0.1 mmol·L<sup>-1</sup> of CaCl<sub>2</sub> and 0-50 mg·L<sup>-1</sup> of CTAB or SDBS in 100 mL centrifuge tubes. The tubes were shaken at 200 rpm in darkness for 48 h at 25±1°C, and then centrifuged at 8000 rpm for 15 min to separate the undissolved SMX. The supernatant was diluted and analyzed for SMX. Duplicate tests in 0.1 mmol·L<sup>-1</sup> CaCl<sub>2</sub> without surfactants were conducted under the same conditions.

### Isotherm Models

Effective adsorption at the given pH was modeled in two different manners, with both linear partition coefficients ( $K_D$ ) and Freundlich isotherm parameters ( $K_F$  and  $n$ ) being developed, but only the Freundlich isotherm was used here. The nonlinear form of the Freundlich adsorption model is represented by:

$$C_s = K_F C_e^n$$

...where  $C_s$  is the sorbed concentration (mg·kg<sup>-1</sup>),  $C_e$  is the aqueous-phase concentration (mg·L<sup>-1</sup>),  $K_F$  is a measure of the degree of sorption strength, and  $n$  reflects the curvature in the isotherm and maybe is taken to represent the energy distribution of adsorption sites.

### Chemical Analyses

The concentration of SMX was determined by high performance liquid chromatography (HPLC, Agilent 1200)

Table 3. Freundlich adsorption and desorption isotherm parameters, and thermodynamic index of irreversibility (*TII*).

Surfactants (mg·L <sup>-1</sup> )		Adsorption				Desorption				<i>TII</i>
		K <sub>F</sub> <sup>a</sup>	n	R <sup>2</sup>	N <sup>b</sup>	K <sub>F</sub>	n	R <sup>2</sup>	N	
Blank		8.14	1.27	0.943	8	53.07	0.41	0.922	5	0.58
CTAB	3.61	20.42	0.91	0.972	8					
	18.1	23.44	0.91	0.947	7	114.82	0.73	0.963	4	0.78
	36.1	33.11	0.83	0.946	8	123.03	0.74	0.986	5	0.85
SDBS	4.34	8.12	1.13	0.981	8	138.04	0.72	0.925	5	0.58
	21.7	7.08	1.04	0.993	8	117.49	0.73	0.966	5	0.49
	43.4	6.03	1.02	0.991	8	93.33	0.76	0.954	5	0.40

<sup>a</sup> K<sub>F</sub> (mg<sup>1-n</sup> L<sup>n</sup> kg<sup>-1</sup>)<sup>b</sup> N is sample numbers

with a diode array detector detection at 254 nm. The separation was performed with an Agilent Zorbax Eclipse XDB-C18 column (2.1×100 mm, 3.5 μm) and an Agilent Zorbax Eclipse Plus C18 guard column (2.1×12.5mm, 5 micron). The column was maintained at 30°C during sample analysis. The measurement for SMX was performed in an isocratic elution program with 35% methanol and 65% water (0.1 % formic acid) as mobile phase. Flow rate was kept at 0.2 mL·min<sup>-1</sup>, and the injection volume was 10 μL. The retention time for SMX was 3.61 min. The limit of detection for SMX in this study was determined at 25 ng·mL<sup>-1</sup>.

## Results and Discussion

### Influence of Surfactants on the Adsorption of SMX on Sediment

The experimental data were plotted in Figs. 2 and 3, and the model-fitted adsorption and desorption parameters are given in Table 3. Fig. 2 shows that the adsorption of SMX on sediment was increased by cationic surfactant CTAB. When the CTAB concentration increased from 0 mg·L<sup>-1</sup> to 3.6, 18.1, and 36.1 mg·L<sup>-1</sup>, the K<sub>F</sub> values increased from 8.14 to 20.42, 23.44, and 33.11, respectively (Table 3). A good linear correlation existed between the K<sub>F</sub> values and the CTAB concentration ( $y = 0.3964x + 18.018$ ,  $r^2 = 0.95$ ), revealing that the addition of CTAB at concentrations below CMC (361 mg·L<sup>-1</sup>) undoubtedly increased the adsorption of SMX on sediments. The effect of anionic surfactant SDBS on SMX adsorption on sediments was opposite compared to CTAB, as shown in Fig. 3. When the concentration of SDBS was lower than 4.34 mg·L<sup>-1</sup>, SDBS slightly changed the adsorption of SMX. However, when SDBS concentration increased to 21.7 and 43.4 mg·L<sup>-1</sup>, the adsorption of SMX reduced remarkably, with K<sub>F</sub> values decreasing from 8.14 (no surfactant) to 7.08 and 6.03, respectively.

The above results indicated that CTAB enhanced the adsorption of SMX on sediment. Generally, sediments offered a much greater adsorption amount for CTAB than SDBS. Pan et al. [9] reported that the equilibrium distribution coefficient for CTAB was 2 times greater than that of SDBS, and they attributed the greater adsorption amount for CTAB on sediments to the electrostatic interactions between the positively charged ammonium groups ( $[(CH_3)_3NR]^+$ ) and the overall negatively charged surface of the sediment. The SMX sorption in soils was remarkably influenced by the physicochemical properties of SMX and soils, which are especially influenced by pH, because pH value could significantly influence their speciation in solution, thus affecting their environmental behaviors [10, 17, 18]. In this study, at pH 7.0 around 96% of SMX in anionic form, resulting in repulsion from negatively charged surface of the sediment. However, the cationic CTAB was eas-

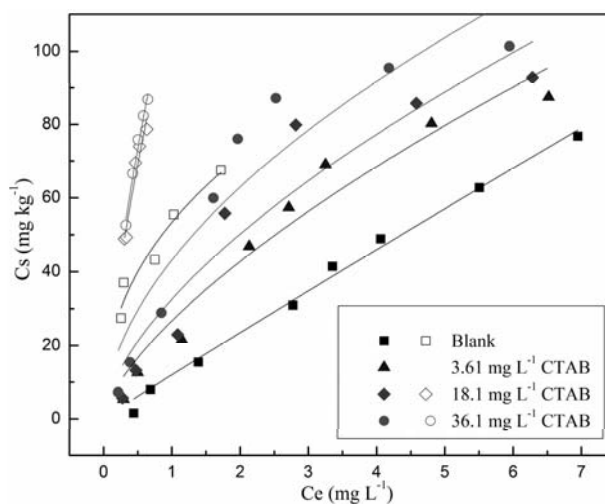


Fig. 2. Fit of the Freundlich model to the influence of the CTAB on the adsorption and desorption of SMX on the sediment. Solid symbols: adsorption data; hollow symbols: desorption data. Data given were the mean of duplicates.



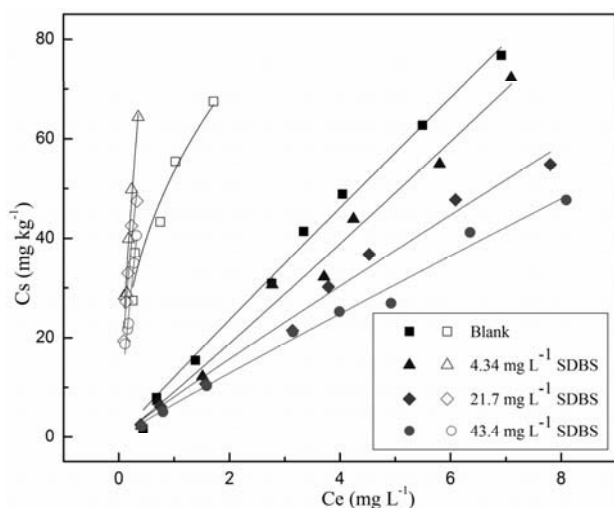


Fig. 3. Fit of the Freundlich model to the influence of SDBS on the adsorption and desorption of SMX on the sediment. Solid symbols: adsorption data; hollow symbols: desorption data. Data given were the mean of duplicates.

ily sorbed to the sediment via electrostatic interactions and specific sorption (e.g. hydrogen bonding between surfactants and sediment surface). Besides, the sorbed cationic surfactant was dispersive on sediment colloid, resulting in the natural sediment organic matter, sediment area increasing. Subsequently, the adsorption of SMX on sediment was increased dramatically through hydrophobic partitioning and weak physical forces such as van der Waals force, which also improved the adsorption amount for other organic sorbates [19-21]. The above results also showed that the adsorption of SMX became linear with the increase of CTAB concentration, implying that the increased adsorption of SMX may be due to the additional partitioning of SMX into the adsorbed CTAB. Cao et al. [22] demonstrated that the presence of CTAB was able to remarkably accelerate and enhance the sorption of DDT. Zohra et al. [23] reported that the adsorption amount of Benzopurpurin 4B distinctly increased on clay treated with CTAB.

In this study, adsorption of SMX by sediment was inhibited in the presence of SDBS. Although adsorbed SDBS on sediment/soil surfaces could increase the adsorption of chemicals, the proportion of SDBS adsorbed by sediment was small because both SDBS and sediments were negatively charged. One possible reason for SDBS inhibition on SMX adsorption on sediment was the competition of the limited adsorption sites by SDBS. Torn et al. [19] found SDBS competing with vinylpyrrolidone in adsorption sites on kaolinite, resulting in the reduced adsorption of vinylpyrrolidone. The other explanation was due to the characteristics of the anionic surfactant. SDBS has the property to enhance the solubility of organic chemicals in water [10, 24, 25]. Solubility of SMX in solutions in the presence of CTAB or SDBS was determined in this study and shown in Fig. 4. It indicated that the presence of CTAB at the tested concentration range did not affect the solubility of SMX significantly. In contrast, the aqueous solubility

of SMX was increased from 266 to 341  $\text{mg}\cdot\text{L}^{-1}$  when SDBS concentrations increased from 0 to 50  $\text{mg}\cdot\text{L}^{-1}$ , thereby reducing the adsorption of SMX on sediment. Sun et al. [26] reported that the anionic surfactant SDBS reduced the sorption of phenanthrene and showed a higher soil washing effectiveness in a saline system.

### Adsorption Reversibility

Sander et al. [27] proposed the thermodynamic index of irreversibility (*III*) for quantifying hysteresis in soils where natural organic matter dominates the sorption process. *III* is based on the difference in free energy between the real desorption state and the hypothetical fully reversible state, and defined as the ratio of the observed to upper limit loss of free energy due to irreversibility, which is given by:

$$III = \frac{\ln C_e^r - \ln C_e^D}{\ln C_e^S - \ln C_e^D}$$

...where  $C_e^S$  is the solution concentration of SMX at adsorption equilibrium,  $C_e^D$  is the solution concentration of SMX at desorption equilibrium, and  $C_e^r$  is the solution concentration of the hypothetical reversible desorption state.  $C_e^S$  and  $C_e^D$  are obtained from the adsorption and desorption isotherms.  $C_e^r$  corresponds to the aqueous phase concentration on the adsorption isotherm where the solid phase concentration of SMX ( $C_s^r$ ) is equal to  $C_s^D$ . *III* is based on the difference between the real desorption state and the hypothetical fully reversible state. The *III* value lies between 0 and 1, with 0 indicating a completely reversible system and 1 complete irreversibility.

The calculated *III* values are listed in Table 3. It can be seen that the adsorption irreversibility increased with the increase of CTAB concentration. When the CTAB concentration increased from 0 to 36.1  $\text{mg}\cdot\text{L}^{-1}$ , the *III* values increased from 0.58 to 0.85, indicating that CTAB not only enhanced the adsorption amount for SMX, but also made

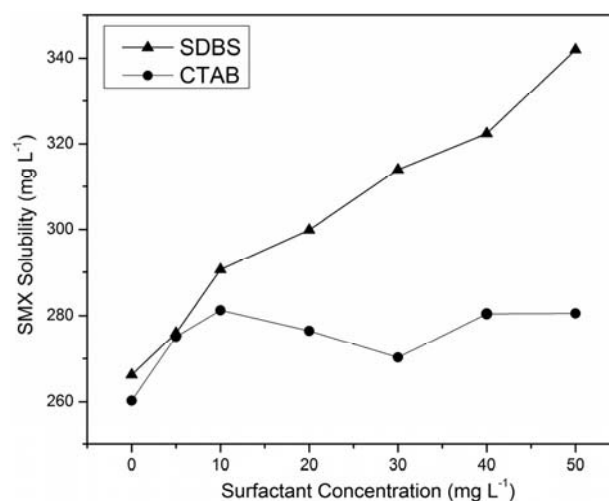


Fig. 4. Solubility of SMX as a function of CTAB or SDBS concentrations.

the adsorption of SMX highly irreversible, which was consistent with the results by Pan et al. [9]. The surfactants adsorbed on the sediment provide a more hydrophobic and homogeneous sink than natural sediment organic matter, which typically consists of mixtures of hydrophilic and hydrophobic chemical units [28]. Unlike CTAB, with the increase of SDBS concentration (from 4.34 to 43.4 mg·L<sup>-1</sup>), the *K<sub>d</sub>* values decreased obviously, suggesting that SDBS can remarkably remove the adsorbed SMX from sediment to solution.

### Implications for Transport of SMX in Water/Sediment System

The results show that different surfactants (cationic CTAB and anionic SDBS) have different effects on the adsorption of SMX on sediment. The CTAB remarkably increase the adsorption of SMX and immobilized SMX, but the SDBS may decrease adsorption and mobilize SMX in contaminated sediments. From the adsorption and desorption isotherms in this study it can be concluded that about 85% of SMX adsorbed on the sediment remained non-desorbable in the presence of high concentrations of CTAB at 36.1 mg·L<sup>-1</sup>. This finding may be helpful to better understand the distribution of SMX in the sediment/water environment.

Surfactants are particularly attractive for soil and sediment remediation due to their low toxicity and favorable biodegradability [13], and can be used as an additive to prevent some organic contaminants transferring in soil or sediment. SMX is a widely useful antimicrobial and has been detected in sediments, soils, and some biosomes. Therefore, in inhibiting the SMX from transport in the subsurface environment, the cationic surfactants may play a critical role. For this purpose, our finding may be of important practical value. Cationic surfactants can be used to immobilize the contaminant, serving as a simple and cost-effective approach for SMX sequestration.

### Conclusions

The cationic surfactant CTAB increased the adsorption amount of SMX on sediment, while the anionic surfactant SDBS exerted an opposite impact. The CTAB strengthened the adsorption of SMX on sediment, which was mainly dominated by two factors. One is electrostatic interaction between the positively charged CTAB and the overall negatively charged surface of the sediment. The other is the adsorbed cationic surfactant disperse on sediment colloid, resulting in the sediment area and adsorption sites increasing, and then improved the adsorption amount for SMX. Besides, hydrophobicity partitioning to the adsorbed CTAB may be responsible for the increased adsorption of SMX. The influence of SDBS on the adsorption of SMX was negatively correlated with SDBS concentration. These findings may be useful for providing possible strategies for sulfonamide-contaminated soil or sediment remediation.

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