



Sorption of Triton X-100 on soil organic matter fractions: Kinetics and isotherms

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Abstract

Kinetics and isotherms of Triton X-100 sorption on soil, base-extracted soil (BE), humic acid (HA) and humin (HM) were investigated respectively to get better understanding on characteristics of the surfactant sorption onto different soil organic matters (SOMs). It was demonstrated that the kinetics results could be satisfactorily described by the pseudo-second order model. The half of the time to reach equilibrium ($t_{1/2}$) for different sorbents followed the sequence of soil > HA > BE > HM. Furthermore, the calculated equilibrium sorption capacity (C_{eq}) was found in the sequence of HA > BE > HM > soil, which agreed well with the experimental results. The isotherms of Triton X-100 sorption on soil and HA could be well described by the S-type isotherm, but BE and HM by the L-type. The isotherms of all the four sorbents were found reasonably fitted to the Langmuir equation. The K_d value, defined as the ratio of Triton X-100 in sorbent and in the equilibrium solution for given concentrations, generally followed the order of HM > HA > soil > BE. Separated HM and HA showed high affinity for Triton X-100, but the HA and HM in soil and BE were tightly bounded by the minerals. Thus, the HA on the soil surface might dominate the sorption, whereas the bounded HM would play a key role upon the surfactants being penetrated inside the soil.

Key words: Triton X-100; sorption; soil organic matters; nonionic surfactant; kinetics; isotherms

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Introduction

The contamination of soils by hydrophobic organic compounds (HOCs), such as polycyclic aromatic hydrocarbons and pesticides is a worldwide environmental problem. To eliminate these pollutants from the contaminated sites, soil washing, phytoremediation, microbial remediation and electrokinetic remediation have been developed for years (Brusseu *et al.*, 1995; Alkorta and Garbisu, 2001; Aislable *et al.*, 2006; Saichek and Reddy, 2005). However, the low solubility, mass transfer and desorption difficulty of HOCs in the soils have greatly limited the wide application of these remediation methods. Surfactants, a type of amphiphilic chemicals with polar and non-polar regions, can facilitate these processes by promoting the solubility, desorption and mobility of HOCs in soils. Surfactant-enhanced remediation has been suggested to be a promising technology for the rapid removal of HOCs from contaminated soils (Pastewski *et al.*, 2006; Mulligan *et al.*, 2001).

During the remediation, however, surfactant sorption onto soils is a major constraint. The structure of the soils can be changed and thus will inhibit the natural

transport of aqueous phases (Mingorance *et al.*, 2007; Wiel-Shafran *et al.*, 2006). The partition of HOCs into the sorbed surfactants lead to more HOCs sorption (Zhu and Zhou, 2008). Besides, surfactants loss can increase the remediation cost; the residual surfactants and metabolites may pose potential risks to the environment (Scott and Jones, 2000; Ying, 2006).

It has been found that the sorption depended greatly on the type of surfactants and the properties of soils (Sánchez-Martín *et al.*, 2008; Brownawell *et al.*, 1997). Many researchers have studied the relationship between surfactants sorption and different soil components through kinetics and isotherms. Some indicated that the sorption was related to the soil clay content, and the soil organic matters (SOMs) were not important in the process (Shen, 2000; Cano and Dorn, 1996). However, Fytianos *et al.* (1998) pointed the importance of SOMs during the surfactant sorption. Yeh and Lin (2003) found that the sorption rate of Triton X-100 increased linearly with SOM content when initial surfactant concentrations were high. Rodríguez-Cruz *et al.* (2005) and Zhu *et al.* (2003) employed a multi-component statistic analysis to investigate the sorption of surfactants on various soils/sediments, and confirmed the importance of SOM and clay fraction during the surfactants sorption process. To further understand the

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mechanisms, many surfactants sorption experiments have been conducted using separate clays and metal oxides such as montmorillonite, illite, muscovite, kaolinite, sepiolite, palygorskite, hematite (Fe_2O_3), manganese dioxide (MnO_2) and gibbsite ($\text{Al}(\text{OH})_3$) (Ochoa-Loza *et al.*, 2007; Sonon and Thompson, 2005; Yang *et al.*, 2007). For soil organic matter fractions, the sorption of Triton X-100 on humic acid has been studied to compare with clays (Zhu *et al.*, 2003). However, the investigations focused on the role of different SOMs during the surfactants sorption were scarcely conducted.

The objective of our work was to clarify the contributions of different SOMs separated from soils using a humic separation procedure to surfactants sorption. Triton X-100 was chosen as a model surfactant, because it was widely used in soil remediation and had a potential application foreground. The concentration levels studied in our research were below the critical micelle concentration (CMC) to avoid abrupt change of the surfactant solution properties at CMC. Although the results might somewhat deviate from the practical situation, due to the separation procedure might change the interior properties of the soils, this study was expected to show a direct and new angle to understand the relationship between surfactants sorption and SOMs.

1 Materials and methods

1.1 Reagents and chemicals

The model nonionic surfactant used in this study was Triton X-100 (TX100, $\text{C}_8\text{H}_{17}\text{C}_6\text{H}_4\text{O}(\text{CH}_2\text{CH}_2\text{O})_{9.5}\text{H}$) with a CMC of 0.15 g/L (0.24 mmol/L). It has an average of 9.5 ethylene oxide units per molecule with a molecular weight of 625 g/mol. All the reagents and chemicals used were purchased from Beijing Chemical Reagents Company and were of analytical reagent grade.

1.2 Soil and soil organic matters

The surface agricultural soil sample was taken from the northeast of China. The soil was air dried and sieved through a 1-mm mesh. The organic carbon content was 1.59%, which was determined using a Multi N/C 3000 TOC analyser (Analytik Jena AG, Germany). The pH was 5.41 at soil/water ratio 1:1 (W/V).

Base-extracted soil (BE), humic acid (HA), and humin (HM) were isolated from the sample soil as the following procedures (Rice and Maccarthy, 1989; Wen *et al.*, 2007; Chen *et al.*, 2007). Soil sample was mixed with deionized water (1:10, W/V) and adjusted to pH range 1–2 with 6 mol/L HCl. After shaken for 1 h, the mixture was centrifuged at 4000 r/min for 10 min and the supernatant fluid was decanted. The residue was neutralized to pH 7 with 1 mol/L NaOH, then mixed with 0.1 mol/L NaOH (1:10, W/V) under the atmosphere of N_2 . The mixture was immediately sealed, shaken overnight, and centrifuged. The supernatant fluid was collected, acidified to pH 1 with 6 mol/L HCl and stood for 24 h. After centrifugation, the precipitated HA fraction was rinsed and freeze dried.

Base-extracted soil was prepared by repeated extraction of soil with 0.1 mol/L NaOH. Sample soil was equilibrated overnight with 0.1 mol/L NaOH at a mass volumetric ratio of 1:10. Then centrifuge the mixture and discard the supernatant fluid. This procedure was repeated eight times. The residue was named as base-extracted soil. One part of BE was rinsed and freeze dried for sorption experiment; the other was deashed twice to get HM with HCl (6 mol/L) and hydrofulvic acid (HF) (22 mol/L) mixture (1:2, V/V) at 60°C for 10 h. The residue (humin) was rinsed and freeze dried.

1.3 Sorption experiments

The kinetics studies were conducted by agitating 500 mL Triton X-100 solution at initial concentrations 60, 80, and 100 mg/L with 2.5 g solid samples in glass flasks on a reciprocating shaker at $27 \pm 2^\circ\text{C}$. Samples were taken every few minutes and measured after centrifuged at 4000 r/min for 15 min.

Batch equilibrium tests were conducted to obtain sorption isotherms of Triton X-100 on soil and different SOM fractions. A 0.1-g solid sample and 10 mL Triton X-100 aqueous solution at concentrations of 20, 40, 60, 80, 100, and 120 mg/L were mixed in 10 mL glass centrifuge tubes. The centrifuge tubes were then sealed and shaken to reach equilibrium on a reciprocating shaker for 24 h at $27 \pm 2^\circ\text{C}$. Subsequently, the suspensions were centrifuged at 4000 r/min for 15 min and analyzed.

All experiments were carried out in duplicates against a blank to correct the possible interferences. The sorbed Triton X-100 amounts were calculated from the difference between the initial and the final concentration after reach equilibrium.

1.4 Chemical analysis

Triton X-100 concentrations were determined by a Agilent 1100 HPLC (USA) fitted with UV detector at 223 nm and a Agilent Zorbax SB-C18 column (150 mm \times 4.6 mm, particle size 5 μm) with methanol/water (85:15, V/V) as the mobile phase at a flow rate of 1 mL/min. The linear range of Triton X-100 concentrations was 0–140 mg/L with $r^2 = 0.9995$.

2 Results and discussion

2.1 Sorption kinetics

Kinetics investigation is a good approach to determine the sorption rate and equilibrium time, and to reveal the surfactant sorption process and mechanism. Figure 1 shows the change of Triton X-100 concentration in solution with time during the sorption on soil, BE, HA, and HM at initial concentrations of 60, 80, and 100 mg/L.

Triton X-100 sorbed on BE, HM and HA increased continuously until equilibrium. However, the sorption on soil decreased a little at the initial 30 min. This indicated that the sorption on soil was weaker, and desorption might occur during the interaction. The complex structure and heterogeneity of the soil might be the dominant reason.

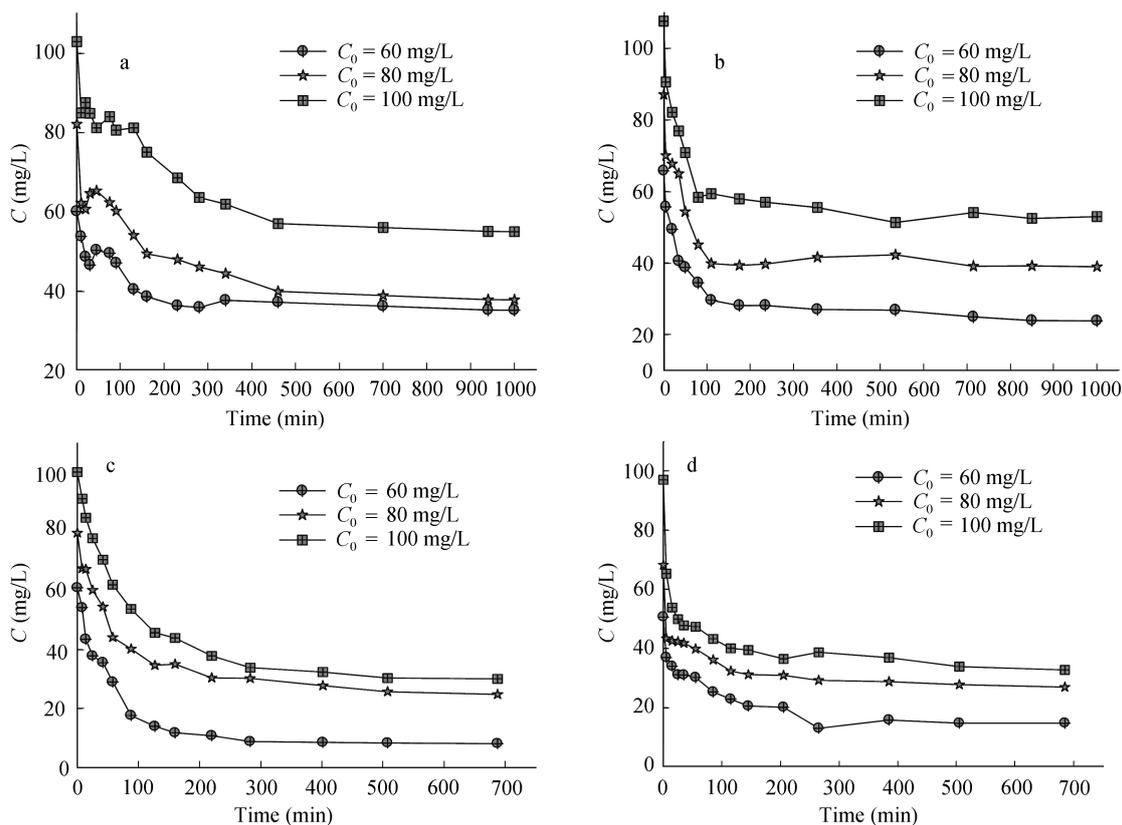


Fig. 1 Sorption kinetics of Triton X-100 on soil (a), BE (b), HA (c), and HM (d) at different initial concentrations (C_0).

Soil was composed of HA, FA (fulvic acid), HM, and minerals etc., and competitions between the different parts were inevitable. Pan *et al.* (2006) have confirmed the competitions between HM and FA/HA for phenanthrene and pyrene.

The results could be satisfactorily described by the pseudo-second order model with $R^2 > 0.98$. The pseudo-second order equation was based on the assumption that the rate limiting step might be chemical sorption. The kinetics rate equation could be expressed as Eq. (1).

$$\frac{t}{C_s} = \frac{1}{kC_{eq}^2} + \frac{t}{C_{eq}} \quad (1)$$

where, C_s (mg/g) was the mass of Triton X-100 sorbed, C_{eq} (mg/g) was the equilibrium Triton X-100 uptake, t (min) was the contact time, and k (g/(mg·min)) was rate constant. When this model was applicable, the plot of t/C_s against t should give a linear relationship, from which k and calculated C_{eq} could be determined from the intercept and slope of the plot, respectively. The average sorption

rate was determined from $t_{1/2}$ (half equilibrium time), which was calculated from Eq. (2).

$$t_{1/2} = \frac{1}{kC_{eq}} \quad (2)$$

The sorption kinetics parameters calculated from pseudo-second order model are shown in Table 1. The parameter $t_{1/2}$ generally followed the order of soil > HA > BE > HM. The sorption on HM and BE was much faster than soil and HA. For HM, approximately 40% was sorbed within 5 min at all three different initial concentrations. BE and HM needed similar time to reach equilibrium. In addition, the calculated equilibrium sorption capacity C_{eq} was in the sequence of HA > BE > HM > soil, which agreed well with the experimental results ($r = 0.999$).

2.2 Sorption isotherms

The sorption isotherms of Triton X-100 on the soil and soil organic matter fractions at the initial concentrations below the CMC are shown in Fig. 2.

Table 1 Sorption kinetics parameters calculated from pseudo-second order model

Sorbent	$C_0 = 60$ mg/L				$C_0 = 80$ mg/L				$C_0 = 100$ mg/L			
	C_{eq} (mg/g)	$t_{1/2}$ (min)	k (g/(mg·min))	R^2	C_{eq} (mg/g)	$t_{1/2}$ (min)	k (g/(mg·min))	R^2	C_{eq} (mg/g)	$t_{1/2}$ (min)	k (g/(mg·min))	R^2
Soil	2.61	50.81	0.00754	0.994	4.73	69.90	0.00303	0.994	5.59	77.37	0.00231	0.989
BE	8.54	27.87	0.00420	0.999	9.71	21.83	0.00472	0.995	11.21	20.25	0.00441	0.999
HA	61.7	45.49	0.00036	0.992	58.1	45.20	0.00038	0.998	78.74	60.99	0.00021	0.999
HM	7.49	30.51	0.00438	0.997	8.40	18.42	0.00647	0.999	10.89	16.76	0.00548	0.999

BE: base-extracted soil; HA: humic acid; HM: humin; C_{eq} : the equilibrium Triton X-100 uptake; $t_{1/2}$: half equilibrium time; k : rate constant.

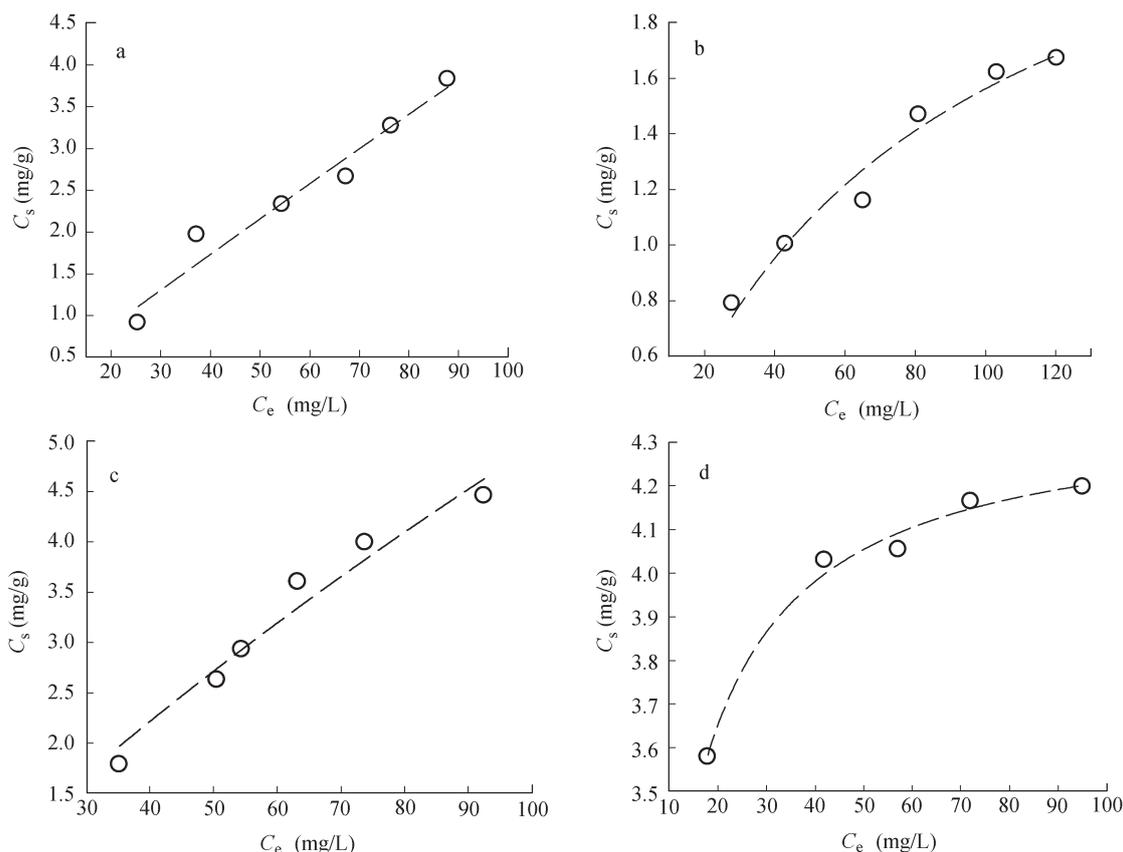


Fig. 2 Sorption isotherms of Triton X-100 on soil (a), BE (b), HA (c), and HM (d). C_s : mass of Triton X-100 sorbed, C_e : equilibrium concentration in solution.

According to the isotherms, soil and HA corresponded to the S-type, while BE and HM belonged to the L-type which was stated by Giles *et al.*, (1960). The initial curvatures of the soil and HA isotherms were low. With the increase of Triton X-100 concentration, the sorption increased, which implied the sorbed molecules would promote additional fix of surfactant through surfactants interaction. BE and HM showed a rapid increase at the beginning of the sorption isotherms, which indicated the high affinity of the sorbent for the sorbate. However, as the active sites of the sorbent were filled, the sorption would be difficult.

Rodríguez-Cruz *et al.* (2005) found that the sorption of Triton X-100 on 18 soils belonged to different types. The soils with OM content above 5% were S-type, and the soils with medium or low OM content and very low clay content were L-type. In our study, the sorption of Triton X-100 on the sample soil was S-type with the SOM content being below 5%. Thus, we considered except SOM content, the structure of the SOM might also influence the sorption.

HA was mainly on the surface of the soil, and the results showed that it had the same S-type isotherm as soil did, which indicating the HA on the soil surface might contribute mainly to the sorption. It has been reported that HA had both hydrophilic and hydrophobic properties, which presented surfactant properties like other amphiphilic molecules (Wu *et al.*, 2002). Therefore, the S-type isotherm might be due to the interaction between HA and Triton X-100 as the interaction between surfactant

monomers.

BE was the SOM residue, as mainly humin tightly attached to minerals after base extraction and HA and FA removed from the soil surface. BE and HM belonging to the L-type, confirmed the importance of HM and the little effect of minerals. However, Sánchez-Martín *et al.* (2008) found that the individual minerals showed high affinity for surfactants. The reason was the interactions between the polar parts of the surfactants and the high specific surface or the interlayer space of the minerals through ion-dipole-type interaction or hydrogen bond. The sorption of Triton X-100 on HM might be mainly the interaction between the hydrophobic moieties of surfactant and the HM, which was different from the minerals. The hydrophilic fraction pointed to the bulk solution, and the surface was changed from hydrophobic to hydrophilic, thereby the other Triton X-100 monomers could not aggregate through hydrophobic moiety interaction onto the surface to form admicelles.

The isotherms of soil, BE, HA, and HM were fitted to the Langmuir equation (Eq. (3)) with $R^2 > 0.95$.

$$C_s = \frac{C_{\max} b C_e}{1 + b C_e} \quad (3)$$

where, C_s (mg/g) is the mass of Triton X-100 sorbed, C_e (mg/L) represents the equilibrium concentration in solution, C_{\max} (mg/g) is the maximum Triton X-100 uptake and b (L/mol) is Langmuir constant. Langmuir isotherm fitting parameters are shown in Table 2, and the regression curves are presented in Fig. 2.

Table 2 Langmuir isotherm fitting parameters for soil, BE, HA, and HM

Sorbent	b (L/mol)	C_{\max} (mg/g)	R^2
Soil	0.4	110	0.95
BE	13.5	2.72	0.97
HA	2.3	26.3	0.96
HM	251.7	4.38	0.99

In order to better describe the sorption capacity, we used the distribution coefficient, K_d , which was defined as the ratio of Triton X-100 in sorbent and in the equilibrium solution for given equilibrium concentrations. K_d values calculated for equilibrium concentration 30, 50, 70, and 90 mg/L are shown in Table 3. The K_d values followed the sequence of $HM > HA > soil > BE$, with the exception $C_e = 90$ mg/L. The K_d values again showed that SOM played an important role in the surfactant sorption. HM had the biggest sorption capacity. This was consistent with the report of Pan *et al.* (2006), who have found the importance of HM for the sorption of phenanthrene and pyrene.

Table 3 K_d values for soil, BE, HA, and HM at different equilibrium concentrations

C_e (mg/L)	Soil	BE	HA	HM
30	0.0310	0.0261	0.0564	0.1322
50	0.0355	0.0219	0.0543	0.0811
70	0.0415	0.0189	0.0521	0.0592
90	0.0499	0.0166	0.0501	0.0466

HA also showed high affinity for Triton X-100 due to the amphiphilic molecule structure. However, Ochoa-Loza *et al.* (2007) have used HA-coated clay to sorb the surfactants so as to elucidate the SOM effects. Compared with the clay alone, HA-coated clay did not show obvious increase or was not critical to surfactant sorption. Although the sorbed HA might execute the sorption, the effect of clay could be weakened by HA. The reason is that HA could occupy the active sites used to sorb Triton X-100 on clays surface. Therefore, the sorption characteristics changed very little because of the dual interaction. The sorption capacity of BE was significantly lower than the other sorbents, which further indicated that HA played important roles during the sorption. The inner HM might be tightly bounded to minerals in BE and soil, so the HM effect was somewhat blocked.

3 Conclusions

Sorption of nonionic surfactant Triton X-100 on soil, base-extracted soil (BE), humic acid (HA), and humin (HM) were investigated in terms of kinetics and isotherms. The sorption kinetics could be reasonably described by pseudo-second order model. The sorption on HM and BE was significantly faster than on soil and HA. The calculated equilibrium sorption capacities was in the sequence of $HA > BE > HM > soil$, which was in good agreement with experimental results. Triton X-100 sorbed on BE, HM, and HA increased continuously until equilibrium; however, the sorption on soil decreased a little at the initial 30 min.

According to the types of isotherms for different sorbents, the S-type was appropriate for description of soil and HA, and the L-type for BE and HM. The data fitting showed that the sorption on those four sorbents could be well described by the Langmuir equation with $R^2 > 0.96$. Furthermore, separated HM and HA had high affinity for Triton X-100 through surfactant monomers-like structure and hydrogen bonds between hydrophobic moiety of the surfactant and HM, respectively. Nevertheless, HA and HM in soil and BE were tightly bounded to the minerals during sorption. Therefore, the sorption mechanism was dominated by HA bounded on the surface of the soil, but the HM bounded became significant to sorption as the surfactant penetrated inside the soil. This study is of particular importance to optimize the application of surfactants in soil remediation.

Acknowledgments

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