Selective adsorption and partitioning of nonionic surfactants onto solids via liquid chromatograph mass spectra analysis

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Abstract

The sorption characteristics of three Triton series surfactants (Triton-100, Triton-305, and Triton-405) from aqueous solution onto four different solids with a wide range of organic matter (OM) content were studied through the liquid chromatograph mass spectrometry (LC–MS) analysis. The examined surfactant concentrations ranged from below to above the critical micelle concentration (CMC) of the selected surfactants. A parameter, Φ, defined as the ratio of the average ethylene oxide (EO) number of surfactant on the adsorbed phase to that in the aqueous solution, was used to distinguish the controlling mechanism (adsorption or partitioning) of surfactants from aqueous solution onto the solids. For solids with very low OM content, adsorption was the primary mechanism and the Φ values were found to be larger than 1.0 and might reduce to 1.0 with the increasing surfactant concentration. On the other hand, the Φ values for solids with very high content of OM were equal to or less than 1.0 and remained constant as the surfactant concentrations varied, in which partitioning was the most likely dominant mechanism. For solids with an intermediate content of OM, adsorption and partitioning mechanisms coexisted and the Φ values could be larger or less than 1.0 and decreased with the increasing surfactant concentration.
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1. Introduction

Surfactants are commonly used for various industrial and household activities. They may contaminate the soil and groundwater and have a negative impact on human beings and the environment. Aside from the potential pollution problems, surfactants are also considered for the remediation of contaminated soils [1–5]. Therefore, in order to evaluate the possible contamination or application, a better understanding of surfactant sorption by soils is of great importance.

The capacity of the surfactant uptake by the solids is mainly determined by the type of surfactants and the composition of solid [6–9]. The adsorption of cationic surfactant onto solids is dominated by electrostatic forces [10,11] and the uptake capacity is determined via the cation exchange capacity and surface area of the solids [12]. The adsorption uptake of nonionic surfactants by solids is more complicated than for cationic surfactants. The surfactant properties including ethylene oxide (EO) number and critical micelle concentration (CMC) value as well as the solid's surface area, mineral phase composition, and organic matter (OM) contents are key factors in the determination of the uptake capacity [13–17]. Generally, on the basis of adsorptive mass, surfactants with a higher EO number would have a relatively higher adsorption to a given solid [18] as well as solids with a higher surface area may possess higher uptake capacity for a given surfactant. However, most of the commercially available nonionic surfactants consist of a mixture of different components and the adsorptive competition resulting from the difference in the affinity of the individual component of surfactants to the solid leads to the possibility of an estimation error for the overall surfactant adsorption. Although a number of studies have described the adsorption characteristics of individual nonionic surfactant components onto the low OM content solids [15,16], the information of surfactant adsorption by solids with

high OM content is still hardly found. One major reason for this fact is that the dissolved OM in the solid may release from the solid into the aqueous solution, which may interfere with the surfactant analysis by UV or TOC [4]. However, since the high OM content solids are often found in nature, it is important to correctly determine the uptake capacity and adsorption mechanism of nonionic surfactants onto such adsorbents.

In this study, three Triton series commercial surfactants with different EO numbers (Triton-100, Triton-305, and Triton-405) and four types of solids with the OM content ranging from ≈0 to 86% were used to examine the effects of both surfactant properties and OM content on the surfactant sorption onto the solids. In order to avoid being influenced by the release of organic compounds from the solids with high OM content, a liquid chromatograph mass spectrometry (LC–MS) equipped with an electrospray ionization interface was adopted to quantitatively determine the surfactant components in the aqueous solution, from which the amounts of individual component of surfactant adsorbed on the solids could be calculated by the mass balance.

2. Materials and methods

2.1. Adsorbents

Four kinds of solids with different OM content were selected as adsorbents. They included a clay, Ca-montmorillonite (Ca-Mon, purchased from the University of Missouri-Columbia, Source Clay Minerals Repository), a sandy soil from Taichung County, Taiwan (TCS), an organic-rich top soil from Shamao Mountain in Taipei County, Taiwan (SMS), and a type of peat from the Everglades, Florida, USA (FP). Before the adsorption experiments, the natural soil samples, TCS and SMS, were air-dried and then sieved to obtain the particles of less than 2.0 mm. The Ca-Mon and FP were used directly without pretreatment. The physicochemical characteristics, including the texture, BET surface area, and OM contents, of the above adsorbents are given in Table 1. The BET surface area was evaluated from the nitrogen adsorption isotherms measured at 77 K with a Micromeritics ASAP-2000. One to two grams of solid samples were outgassed with He for 16 h at 135 °C prior to the adsorption measurement. The OM content for the selected solids was measured with the Walkley-Black method [19]. As can be seen from Table 1, because the OM content covered a broad range of values (from ≈0.0 to 86%), the selected adsorbents might be the suitable candidates for examining the effects

Table 1
Properties of the studied solid samples

Solid	Abbreviation	Texture	BET surface area (m²/g)	SOM (%)
Ca-montmorillonite	Ca-Mon	Clay	76.04	<0.03
Taichung soil	TCS	Sandy loam	10.2	2.4
Shamou mountain soil	SMS	Loam	6.85	27.3
Florida peat	FP	Peat	1.3	86.4

Table 2
Properties of the selected surfactants: molecular formula, molecular weight (MW), and CMC

Surfactants	Molecular formula	MW (g/mole)	CMC (mg/L)
TX-100	$C_8H_{17}C_6H_4O(CH_2CH_2O)_{9.5}H$	624	130
T X-305	$C_8H_{17}C_6H_4O(CH_2CH_2O)_{30}H$	1526	1068
T X-405	$C_8H_{17}C_6H_4O(CH_2CH_2O)_{40}H$	1966	1592

of OM content on the sorption of nonionic surfactants onto solids.

2.2. Surfactants

Three commercial nonionic surfactants, Triton-100 (TX-100), Triton-305 (TX-305), and Triton-405 (TX-405), supplied by the Riedel de Haën Company (Germany) were adopted as adsorbates. They had a similar chemical formula only with difference in the average EO number, which were assumed to be 9.5, 30, and 40 for TX-100, TX-305, and TX-405, respectively. The molecular structures, molecular weights, and CMCs of the selected surfactants are given in Table 2.

2.3. Adsorption measurements and surfactant composition analysis

In this study, both the overall adsorption isotherm and the uptake of individual component of surfactants from aqueous solution onto solids were measured. For the overall adsorption isotherms, solids with 0.15–1.5 g were added to a Corex centrifuge tube containing 25 mL of surfactant solution with a desired concentration ranging from below to above the CMC (66–1600 mg/L for TX-100, 326–3686 mg/L for TX-305, and 436–5455 mg/L for TX-405, respectively). Equilibrium was reached by shaking the tubes for 24 h in a reciprocating shaker at 25 °C. The solution and solid phases were separated by a high-speed centrifuge (Sorvall Co., Model RC-5C) operated at 8000 rpm (7649 × g) for 25 min. A 1 mL aliquot sample of the supernatant was taken and analyzed for TX-100, TX-305, and TX-405 by UV at a wave-length of 275 nm. Since the three surfactants had a similar chemical structure with only the difference in the EO number, the optimal wave-length for their identification was set to be 275 nm. The adsorptive amount of the examined surfactants was calculated by the relation $Q = V\Delta C/m$, where V is the volume of the liquid phase, m is the mass of the solid, and ΔC was the difference between the initial and final concentration of surfactants at aqueous solutions, which could be computed simply from the initial and final UV readings. For the sorption uptake of individual component of surfactants, the procedures were similar to the overall adsorption isotherm experiments. After the solution and solid phases were separated by the high-speed centrifuge, the concentration of the individual components of surfactant in the aqueous solution was determined with the LC–MS (Waters-600-MS). The surfactants were chromatographed on a C18 column. The mobile phase included 50% acetonitrile and 50% deionized water and was acidified with 0.1% formic acid in order to increase the surfactant ioniza-

Table 3
Initial and equilibrium concentrations of the three surfactants in different adsorption experiments (mg/L)

Surfactant	Ionized types	Ca-Mon	TCS	SMS	FP
TX-100	[a]M(EO)n(H$_3$O)$^+$	500[b](99)	100(48)	100(47)	500(65)
		800(122)	300(115)	200(112)	800(96)
		1599(451)	400(241)	400(237)	100(199)
TX-305	M(EO)n(2H$_3$O)$^{+2}$	2143(710)	1000(498)	800(558)	1000(501)
		3000(937)	1500(813)	1500(1046)	2000(1122)
		3500(1419)	2000(1484)	2143(1625)	3500(2420)
TX-405	M(EO)n(3H$_3$O)$^{+3}$	2063(316)	988(473)	617(314)	617(239)
		3087(1612)	2058(1683)	2058(1737)	2058(1752)
		4939(2921)	3087(2676)	2355(2355)	3087(2524)

[a] M is C$_8$H$_{17}$C$_6$H$_4$O and EO is CH$_2$CH$_2$O.
[b] The value in the bracket represents the equilibrium concentration.

tion ability. The total flow rate was 0.2 mL min^{-1}. A quadrupole was used for detecting and quantifying surfactant components with different EO number. The MS was operated in positive-ion mode. Probe energy of 3.5 KV was applied. The source energy temperature was 100 °C. The cone voltage was set at 30 V.

The ionized types of the selected surfactants were important for determining the surfactant concentration by LC–MS. The initial and equilibrium concentration and the ionized types that could determine the mass-ion pattern for the examined surfactants are given in Table 3. The obtained mass-ion ratio was considered to determine the surfactant components with different EO number in the aqueous solution. When the surfactants with known concentrations were injected into the LC–MS, the calibration curve for the surfactant could be established based on the response of the specific mass-ion ratio. The uptake of individual components of the examined surfactants by the solids can be obtained with their concentration changes in the aqueous phase during the sorption process.

Since the individual components of the examined surfactants can be quantitatively identified with the LC–MS, the average molecular weight (AMW) of the surfactant on the adsorbed phase and in the aqueous solution can be defined as

$$AMW = \sum(MW \text{ of individual component} \times x) \qquad (1)$$

where x is the mole percentage of individual component. Moreover, the average EO number of the surfactants can be calculated as follows:

$$\text{Average EO number} = \frac{(AMW\text{-}206)}{44} \qquad (2)$$

where 206 is the molecular weight of the surfactant when the EO chain is neglected and 44 is the molecular weight of EO (i.e., CH$_2$CH$_2$O). In the LC–MS analysis, the average EO number in the solution can be directly estimated by the components distribution patterns of the examined surfactants. The average EO number in the adsorbed phase is then calculated by the difference between the initial and final concentration of individual components of surfactants in the aqueous solution.

In this study, a characteristic parameter, Φ, was used in order to distinguish the controlling mechanism (adsorption or parti-

tioning) of surfactants from aqueous solution onto the solids. It could be defined as

$$\Phi = \frac{EO_S}{EO_W} \qquad (3)$$

where EO$_S$ and EO$_W$ were the average EO number of surfactants on the adsorbed phase and in the aqueous solution, respectively.

3. Results and discussion

3.1. Overall adsorption isotherms

In a solid-aqueous solution system, the solid may possibly act as a dual adsorbent for a nonionic surfactant, where the mineral phase is thought of as a conventional solid adsorbent and the organic matter phase is a partitioned medium. Therefore, the OM content in the solids plays a key role on the determination of the sorption characteristics of nonionic surfactant onto the solids. For solid with a very low OM content (<3%), the surfactants will more likely be adsorbed on the mineral surface, while for solid with a high OM content, the partition process may be the dominating mechanism, because the surfactant molecules with the hydrophobic moieties may result in a possible partitioning interaction force with the OM in the solids. However, it should be noteworthy that although the partitioning interaction between the surfactant and the high OM content solids is well known, the experimental results for this topic are hardly found in the literature due to the interference of OM released from the solids in the accurate analysis of surfactant concentration in the aqueous solution.

In follows, we first demonstrate the overall adsorption isotherms of surfactants on the examined solids. As can be seen from Figs. 1 and 2, the overall isotherms can be divided into two groups based on the OM content of the solids. For Ca-Mon and TCS, the adsorption isotherms are L-type (see Fig. 1) and can be fitted with the Langmuir equation. The adsorption capacity of surfactants on the two solids has a decreasing order: TX-405 > TX-305 > TX-100, namely, the adsorption capacity of surfactants is proportional to their average EO number. This result is similar to other reports in the literature [4,14,18,20]. On the other hand, although several studies have reported that surfactants may aggregate on the solid surface to form a admi-
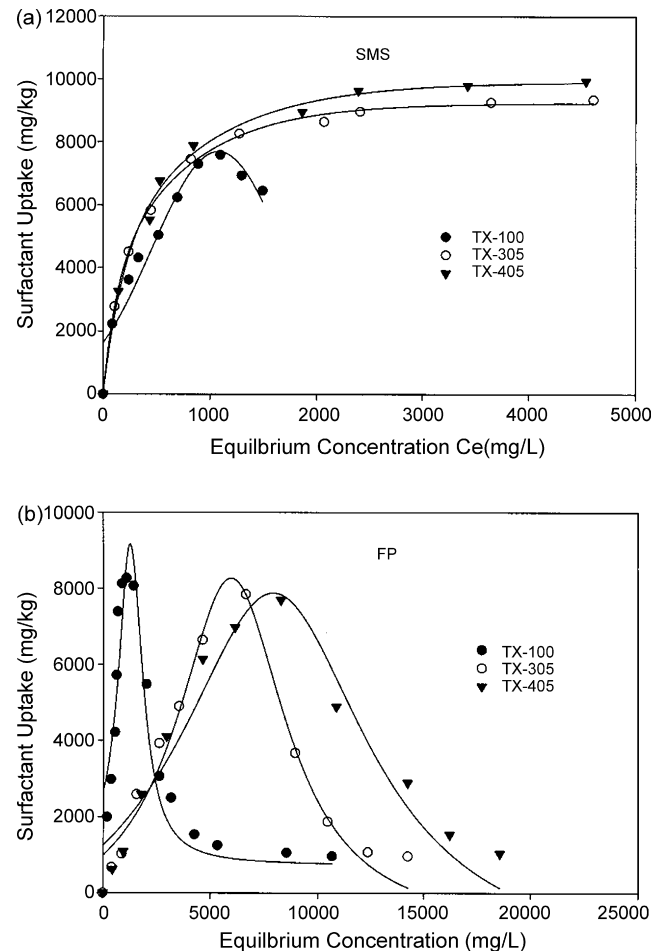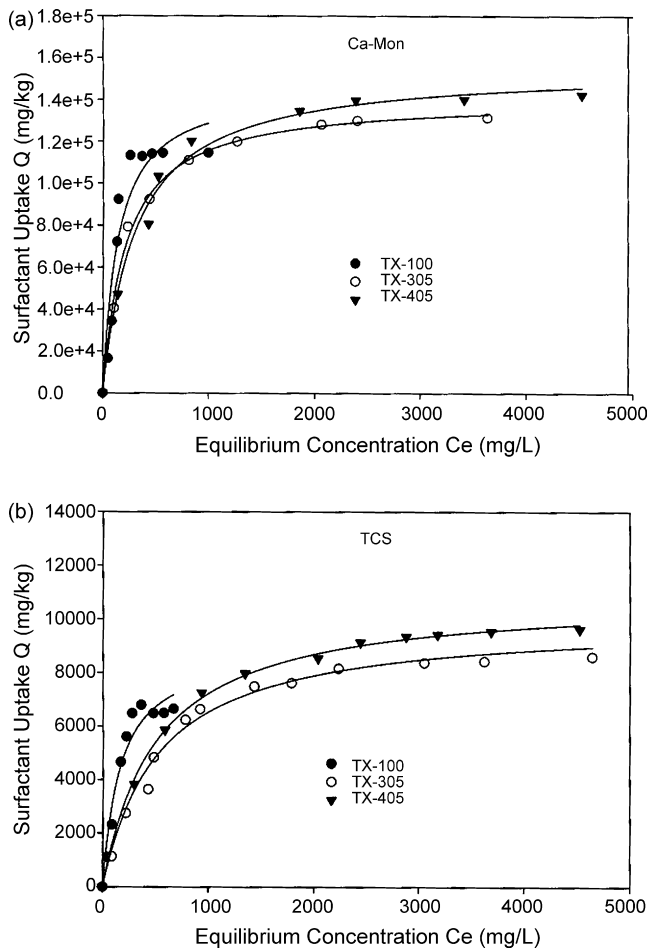
Fig. 1. The overall adsorption isotherms for the selected surfactants on the low OM content solids. The experimental data are fitted with the Langmuir equation.

Fig. 2. The overall adsorption isotherms for the selected surfactants on the high OM content solids. The experimental data are fitted with the Langmuir equation or skewed-Gaussian distribution.

celle [11,21], the isotherms shown in Fig. 1 certainly indicate that the adsorption capacity of the surfactants on the examined soils reach to a limited level.

For both SMS and FP that have a high OM content, the isotherm type of surfactants is strikingly different from that of Ca-Mon and TCS. As shown in Fig. 2, the isotherms exhibit a skewed-Gaussian shape, with a maximum uptake at a specific equilibrium concentration and then sharply dropping to the bottom. This result may be ascribed to the release of dissolved OM from the solids into the aqueous solution, which interferes with the surfactant concentration analysis with UV. This shortcoming may be overcome with the aid of LC–MS analysis, as shown in next.

For all examined surfactants, if the top of the isotherms for the high OM solids and the plateau of the isotherms for the low OM solids are set as maximum uptakes, the maximum uptake of the selected solids has the order: Ca-Mon > FP > SMS > TCS. The high adsorption capacity of Ca-Mon may be ascribed to its high surface area and the dominating mechanism is the adsorption process. However, the high adsorption capacity of FP, which has a very low surface area, indicates that the primary mechanism is the partitioning process. In order to make a further comparison for the adsorption capacity of different solids, the maximum uptakes of per surface area (mg m^{-2}), i.e., specific uptake, for

the selected surfactants on the tested solids are listed in Table 4. As expected, FP possesses the highest specific uptake among the examined solids. The high specific uptake of FP for the nonionic surfactants indicates that the dominating mechanism is indeed the surfactant partitioning into the OM of FP. Since the isotherm types for the low and high OM solids are L-type and skewed-Gaussian shape, respectively, the cause inducing the isotherm type for TX-100 on SMS may also ascribed to the release of OM from the SMS into the aqueous solution and interferes with the TX-100 concentration measurement. However, for both TX-305 and TX-405, the surfactant partitioning into the OM may be neglected and adsorption is still the dominant mechanism.

Table 4
Specific adsorption uptake of the surfactants onto the examined solids (mg/m^2)

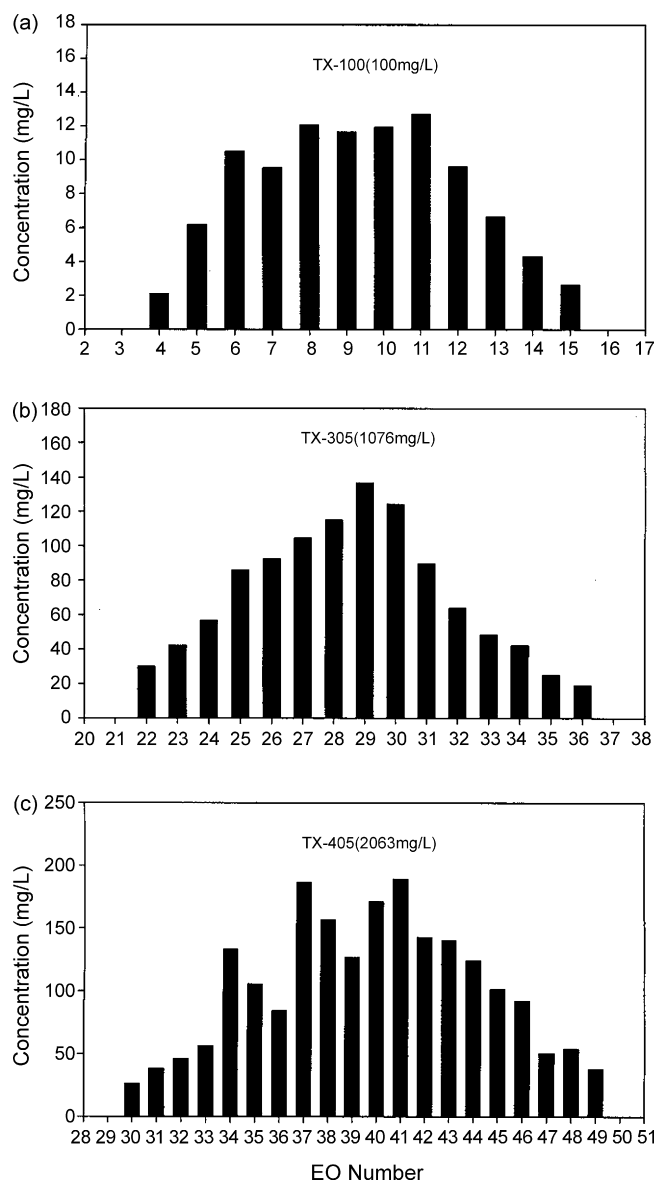| Solid	TX-100	TX-305	TX-405
FP	6615	6231	6077
Ca-Mon	1421	1724	1868
TCS	1269	1635	1846
SM	1162	1353	1456

Fig. 3. The distribution patterns of the components of TX-100, TX-305, and TX-405 at the specific concentrations.

Table 5
Average Φ values (standard deviation) for the three surfactants in relation to the tested solids

Equilibrium concentration	Ca-Mon	TCS	SMS	FP
TX-100				
Below CMC	1.24 (0.08)	1.09 (0.04)	0.94 (0.04)	0.84 (0.04)
Near CMC	1.17 (0.06)	1.06 (0.03)	0.90 (0.04)	0.84 (0.03)
Above CMC	1.08 (0.05)	1.04 (0.04)	0.88 (0.06)	0.84 (0.05)
TX-305				
Below CMC	1.07 (0.06)	1.05 (0.06)	1.07 (0.04)	0.95 (0.03)
Near CMC	1.04 (0.05)	1.02 (0.04)	1.03 (0.04)	0.95 (0.03)
Above CMC	1.02 (0.03)	1.01 (0.04)	1.02 (0.04)	0.95 (0.04)
TX-405				
Below CMC	1.06 (0.05)	1.05 (0.05)	1.07 (0.06)	0.98 (0.03)
Near CMC	1.04 (0.03)	1.04 (0.04)	1.04 (0.06)	0.97 (0.03)
Above CMC	1.02 (0.03)	1.02 (0.05)	1.02 (0.03)	0.97 (0.03)

are measured. This also indicates that the mass-ion ratios listed in Table 3 are accurate. The estimated AMW for TX-100, TX-305 and TX-405 are 621, 1520 and 1959, respectively, which is similar to the molecular weights listed in Table 2. The average EO numbers obtained for the tested surfactants are also similar to those listed in Table 2. In follows, the mass-ion ratios listed in Table 3 and the distribution patterns of the components of the three Triton surfactants will be used to further examine the sorption characteristics of surfactants onto solids.

3.3. Sorption of the individual components of surfactants on the solids

In the above sections, the overall isotherms of surfactants on the examined solids have been discussed. Moreover, the accuracy of the LC–MS analysis was also confirmed. Next, the sorption characteristics of the nonionic surfactants on the soils are further discussed. Fig. 4 indicates the typical concentration variation patterns of individual component of surfactant (TX-100) during the sorption on the four selected solids. As shown in Fig. 4, the concentration variation patterns are closely related to both the components of surfactant and the composition of solids. Moreover, it is difficult to give a clear description for these results. To overcome this shortcoming, the parameter, Φ, defined in Eq. (3) is applied.

The average Φ values and the measured standard deviation at three surfactant equilibrium concentrations (above, below, and near the CMC) are listed Table 5. As can be seen from Table 5, all of the Φ values for the three surfactants on the low OM content solids (i.e., Ca-Mon and TCS) exceed 1.0. It can be found from Fig. 1 that the high EO number surfactants favorably adsorb on the solid's mineral surface based on the adsorptive mass (mg/kg). This leads to the relatively higher Φ values. The Φ value exceeding 1.0 indicates that the primary mechanism of surfactants onto the solids is the adsorption on mineral surface. Moreover, the Φ values of the TX-100 are higher than that of either TX-305 or TX-405. The result can be ascribed to the TX-100 being composed of low EO number (EO4 to EO15) surfactants. The percentages of relative differences for low EO
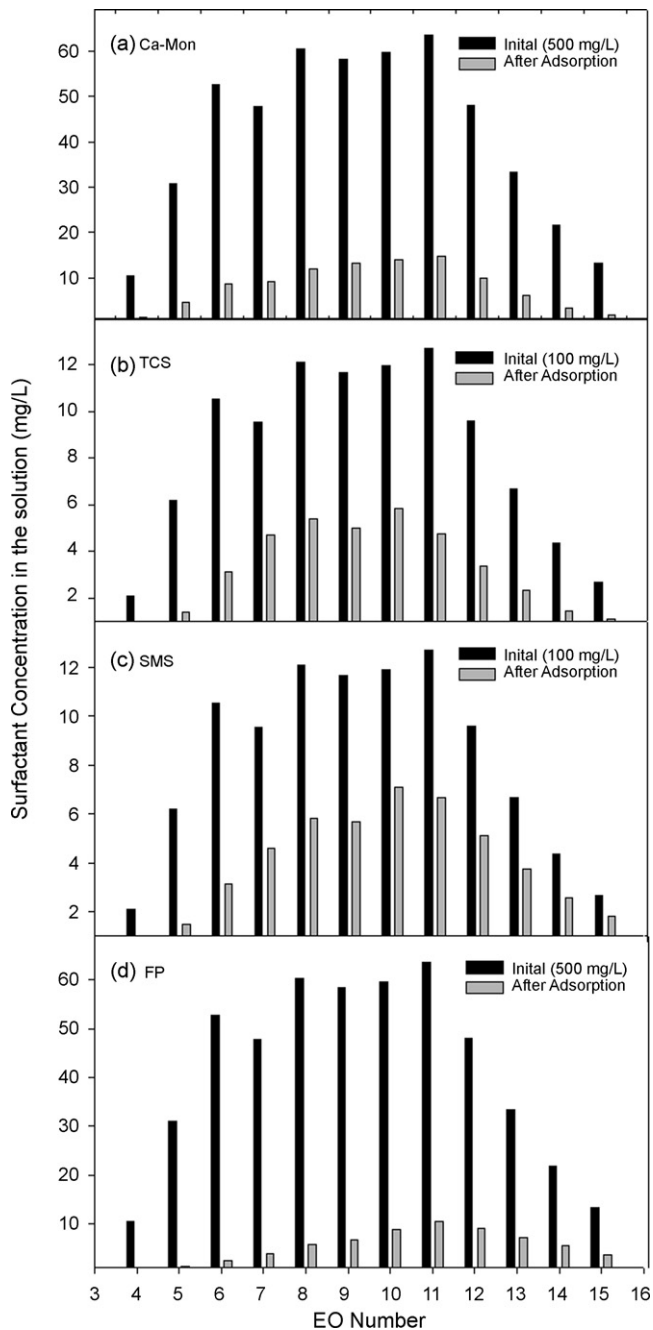
3.2. Composition of surfactants

As mentioned earlier, the OM in soils may be released from the soil into the solution, which would interfere with the measurement of the surfactants. To avoid the afore-mentioned interference, an LC–MS was used to analyze the components of the surfactants. The composition of the selected surfactants was first analyzed to examine the validity of the LC–MS analysis. The distribution patterns of the components of the three Triton surfactants at concentrations near the CMC are shown in Fig. 3. As demonstrated in Fig. 3, the EO numbers of TX-100, TX-305 and TX-405 are concentrated between 4 and 15, 22 and 36, and 30 and 49, respectively. The accuracy of Fig. 3 is supported by the fact that the sum of all the recovered components relative to the whole mass of the tested surfactants is more than 95%, implying the principal components of the surfactants

Fig. 4. The adsorption uptake of the components of TX-100 onto to the examined solids.

solution) will first decrease (increase) and then approach to a constant value with the increasing surfactant concentration.

For the high OM content solid, FP, all of the Φ values are less than or equal to 1.0, indicating that the low EO number components have larger tendency to be adsorbed on the FP than the high EO number components. Moreover, the Φ values remain constant as the surfactant concentration increases, implying the very weak competition for the adsorptive sites. These results are consistent with the characteristics of partitioning process, namely, the major mechanism for the transformation of surfactants from the aqueous solution onto the solids with a high OM content is a partitioning process and the adsorption competition among the surfactant components do not occur. For natural soil, SMS, the Φ values may be larger than 1.0 for TX-305 and TX-405 or less than 1.0 for TX-100. Moreover, the Φ values decrease with the increasing surfactant concentration. As listed in Table 1, SMS has an intermediate OM content and both adsorption and partitioning mechanisms might occur simultaneously. The variation of the Φ values with the surfactant concentration indicates the presence of adsorptive competition.

4. Conclusions

In this study, we elucidated the adsorption and/or partitioning process of nonionic surfactants from aqueous solution onto the solids with various OM contents. The overall adsorption isotherms indicated that for solids with a very low OM content (such as Ca-Mon and TCS), adsorption was the primary mechanism, while for solids with a very high OM content (such as FP), partitioning was the most likely dominant mechanism. LC–MS analysis and the parameter Φ were used to give a microscopic picture for the sorption characteristics of nonionic surfactants onto the solids. It was found that if adsorption was the primary mechanism, the Φ values were found to be larger than 1.0 and might reduce to 1.0 with the increasing surfactant concentration. On the other hand, if partitioning was the most likely dominant mechanism, the Φ values were equal to or less than 1.0 and remained constant as the surfactant concentrations varied. For solids with an intermediate content of OM, adsorption and partitioning mechanisms coexisted and the Φ values could be larger or less than 1.0 and decreased with the increasing surfactant concentration.

number surfactants are higher than those for high EO number surfactants. Because Φ is ratio of the average EO numbers, the relatively higher differences in percentage of EO number lead to obviously higher Φ values. On the other hand, the Φ values of Ca-Mon are larger than that of TCS, which may be ascribed to the fact that Ca-Mon has higher surface area and can offers more adsorption sites on which high EO number surfactants can be adsorbed. Another important feature in Table 5 is that the Φ values of both Ca-Mon and TCS decrease as the surfactant concentrations increase. This tendency is consistent with the results of Kibbey and Hayes [15,16], which indicates that the average EO number of surfactant on the adsorbed phase (in the aqueous

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