INTRODUCTION

Evidence is accumulating that sorption of hydrophobic organic compounds (HOCs) to soils and sediments can be described by “dual-mode” sorption: Absorption in amorphous organic matter, and adsorption to carbonaceous geosorbents (CGs), such as soot, coke, coal, and lampblack [1,2]. Extensive sorption of HOCs to CGs can have major consequences for overall HOC binding to sediments and soils and, thus, for the fate of these compounds in the environment. An excellent review in this regard was provided by Cornelissen et al. [1].

Synthetic surfactants are widely used in household cleaning detergents, personal care products, textiles, paints, polymers, pesticide formulations, pharmaceuticals, mining, oil recovery, and pulp and paper industries. World production of synthetic surfactants amounts to 7.2 million tons annually [3,4]. Many surfactants are used to assist with the application of pesticides to crops or in urban uses. Because of their widespread use and high consumption, surfactants and their degradation products have been detected at various concentrations in surface waters, sediments, and soils. Ying [3] reviewed the fate and behavior of surfactants and their degradation in the environment.

Under subsaturation sorption (i.e., before sorption saturation is reached), surfactants have been found to significantly increase HOC sorption onto soils and sediments by forming an effective partitioning media on the soil and sediment surfaces [5–7]. The sorption of cationic surfactants onto the soils and sediment is so large, and the sorbed cationic surfactant is so efficient in partitioning HOCs, that the use of the sorbed cationic surfactant for in situ immobilization of HOCs has been proposed and tested [8–10]. To our knowledge, however, the effect of the surfactant on the sorption of HOC onto CGs has not been reported.

Unlike other CGs, which usually contain less than 90% carbon, lampblack consists of 99.5% elemental carbon [11,12], generating a very hydrophobic surface. Lampblack is a solid residue from high-temperature decomposition of crude oil or coal during gas manufacture, with a morphology and structure similar to that of char [1,11]. As a result, lampblack has been used by some researchers as a surrogate for hydrophobic surfaces [13,14]. It is believed that the adsorption of HOCs onto CG is mainly on hydrophobic surfaces via hydrophobic adsorption [1]. Thus, lampblack is a good candidate for studying the effect of surfactant on HOC sorption onto hydrophobic CGs.

The objectives of the present study were to investigate the adsorption behavior of three surfactants with different charge property (nonionic, cationic, and anionic) and HOCs (as represented by two of the most commonly used pesticides, atrazine and diuron) onto lampblack and to determine the effect of the presence of the surfactant on the HOC sorption onto hydrophobic environmental media. The hypotheses to be tested were that adsorption of surfactant onto hydrophobic CGs competes with HOC sorption, that the presence of surfactant in the environment tends to reduce the HOC sorption onto the hydrophobic CGs, and that the charge on the surfactant is an important factor in determining the overall effect. To test these hypotheses, batch equilibrium was used to investigate the HOC adsorption behavior as influenced by surfactant under different sorption sequences.
Table 1. Selected properties of the hydrophobic organic compounds

<table>
<thead>
<tr>
<th></th>
<th>Atrazine</th>
<th>Diuron</th>
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<tbody>
<tr>
<td>Molecular structure</td>
<td><img src="image" alt="Atrazine structure" /></td>
<td><img src="image" alt="Diuron structure" /></td>
</tr>
<tr>
<td>Molecular weight (mg/L)</td>
<td>217</td>
<td>233</td>
</tr>
<tr>
<td>Solubility (mg/L)</td>
<td>33</td>
<td>42</td>
</tr>
<tr>
<td>Octanol–water partition coefficient</td>
<td>398–562&lt;sup&gt;a&lt;/sup&gt;</td>
<td>648–747&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

<sup>a</sup> Department of Pesticide Regulation (http://www.cdpr.ca.gov/).

Table 2. Selected properties of the surfactants

<table>
<thead>
<tr>
<th></th>
<th>Triton&lt;sup&gt;a&lt;/sup&gt; X-100</th>
<th>Benzalkonium chloride</th>
<th>Linear alkylbenzene sulfonate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Molecular formula</td>
<td>C&lt;sub&gt;14&lt;/sub&gt;H&lt;sub&gt;22&lt;/sub&gt;O(C&lt;sub&gt;2&lt;/sub&gt;H&lt;sub&gt;4&lt;/sub&gt;O)&lt;sub&gt;10&lt;/sub&gt;</td>
<td>C&lt;sub&gt;14&lt;/sub&gt;H&lt;sub&gt;22&lt;/sub&gt;N(CH&lt;sub&gt;3&lt;/sub&gt;)&lt;sub&gt;3&lt;/sub&gt;</td>
<td>(CH&lt;sub&gt;3&lt;/sub&gt;(CH&lt;sub&gt;2&lt;/sub&gt;)&lt;sub&gt;2&lt;/sub&gt;CH(C&lt;sub&gt;6&lt;/sub&gt;H&lt;sub&gt;4&lt;/sub&gt;SO&lt;sub&gt;3&lt;/sub&gt;)Na)</td>
</tr>
<tr>
<td>Molecular weight</td>
<td>647</td>
<td>340</td>
<td>362</td>
</tr>
<tr>
<td>Critical micelle concentration (µM/L)</td>
<td>185&lt;sup&gt;b&lt;/sup&gt;</td>
<td>941&lt;sup&gt;+&lt;/sup&gt;</td>
<td>1,246&lt;sup&gt;c&lt;/sup&gt;</td>
</tr>
<tr>
<td>Octanol–water partition coefficient&lt;sup&gt;d&lt;/sup&gt;</td>
<td>1,386 ± 50&lt;sup&gt;d&lt;/sup&gt;</td>
<td>19 ± 3</td>
<td>40 ± 5</td>
</tr>
</tbody>
</table>

<sup>a</sup> Sigma-Aldrich, St. Louis, MO, USA.
<sup>b</sup> Wang and Keller [17].
<sup>c</sup> Rosen [18].
<sup>d</sup> Based on duplicate measurement, mean ± standard deviation.

**MATERIALS AND METHODS**

**Chemicals**

Atrazine (2-chloro-4-ethylamino-6-isopropylamino-1,3,5-triazine) was purchased from Supelco (Bellefonte, PA, USA), and diuron (3-(3,4-dichlorofenyl)-1,1-dimethylurea) was purchased from ChemService (West Chestnut, PA, USA). Triton<sup>a</sup> X-100 (a nonionic surfactant), linear alkylbenzene sulfonate (LAS; an anionic surfactant), and benzalkonium chloride (BC; a cationic surfactant) were purchased from Sigma-Aldrich (St. Louis, MO, USA). The chemicals were used as received. The selected physicochemical properties of the HOCs and surfactants are presented in Tables 1 and 2. Lampblack (specific gravity, 1.77) was obtained from Fisher Scientific (Pittsburgh, PA, USA). The BET (i.e., the standard method developed by Stephen Brunauer, Paul Emmett, and Edward Teller) surface area measurement was conducted on a TriStar 3000 gas adsorption analyzer (Micromeritics, Norcross, GA, USA) using N<sub>2</sub>. The surface area of this lampblack was determined to be 24.5 m<sup>2</sup>/g. A transmission-electron microscopic image of the lampblack is presented in Figure 1. Lampblack appears as aggregates of individual, spherical particles. The individual particle size of the lampblack was mostly in the range of tens of nanometers.

**Equilibration time determination**

The adsorption equilibration time of the surfactants and HOCs onto the lampblack was determined by batch experiments. The initial surfactant concentration was 0.20 g/L for all experiments, which corresponds to 1.67, 0.62, and 0.44 of the critical micelle concentration (CMC) of Triton X-100, BC, and LAS, respectively. The surfactant and HOC solutions were prepared in water containing 0.02% NaN<sub>3</sub>. The 0.02% NaN<sub>3</sub> was used as microbial growth inhibitor. For surfactant adsorption, 10 aliquots (200 mg) of the lampblack were mixed in 15-ml, glass centrifuge tubes with 10.0 ml of surfactant solution. The sorption of the HOC was conducted following the same procedure as used for the surfactant, except that given the much lower aqueous solubility of the HOCs, only 5.0 mg of lampblack were used. The initial HOC concentration was 8.40 mg/L for atrazine or 7.89 mg/L for diuron. The tubes were shaken at 60 rpm and 22 ± 2°C (mean ± standard deviation) continuously in an end-over-end shaker. At the end of every 3 d, one tube was taken out for HOC or surfactant analysis. Because of the small particle size and low density of lampblack, complete phase separation was achieved in two centrifugation steps. The tubes were first centrifuged at 5,000 g for 30 min at the same temperature; 1.5 ml of the supernatant were then carefully collected and transferred to a 1.5-ml microcentrifuge tube and centrifuged at 15,000 g for 10 min on an Eppendorf microcentrifuge (5415D; Westbury, NY, USA). Preliminary results showed that sorption of the HOC and surfactants onto the microcentrifuge tubes was negligible. Following the second centrifugation, 1.0 ml of the supernatant was taken for surfactant or HOC analysis by high-performance liquid chromatography (HPLC: Shimadzu, Kyoto, Japan). The amount of surfactant or HOC sorbed was calculated as the difference between the initial and final surfactant or HOC concentration in the aqueous phase.

**Adsorption isotherms**

The adsorption isotherms of the surfactants and HOCs onto the lampblack were determined in duplicate by the same procedure as used for the equilibration time determination, except that the initial surfactant concentration spanned a large range below and above the CMC of each surfactant studied (Triton X-100, 20–2,600 µM/L; BC, 50–3,500 µM/L; LAS, 50–5,500 µM/L), HOC concentrations ranged from 0.70 to 14.0 mg/L for atrazine and from 0.50 to 15.0 mg/L for diuron, and the
equilibration time was 10 d (240 h), which was determined to be enough for surfactant and HOC adsorption equilibrium to be reached. The pH of the suspensions was stable at approximately 6.6 and did not show significant change before or after adsorption.

**Adsorption of HOC in the presence of surfactant**

For the experiments with HOC and surfactants, the HOC concentrations used were 8.40 mg/L for atrazine and 7.90 mg/L for diuron. Aliquots (5.0 mg) of lampblack were treated with 10.0 ml of a solution containing the HOC and surfactant with varying concentrations in 15-ml glass centrifuge tubes. Thus, the HOC and surfactant were added to the lampblack/HOC/surfactant system at the same time. The concentrations of the surfactants were low enough to ensure that their saturation adsorption capacities on the lampblack were not reached. The mixing, separation, and analysis were the same as described for the adsorption isotherm experiments.

**Effect of adsorption sequence**

To study whether the adsorption sequence influenced the equilibrium conditions, three adsorption sequences were tested—namely, HOC adsorption first and surfactant second, surfactant adsorption first and HOC second, and HOC and surfactant adsorption at the same time (as described in the previous section). In all cases, the surfactant concentrations were 5.0 mg/L, and the HOC concentrations were 6.46 mg/L for atrazine and 6.06 mg/L for diuron, after mixing, to have the same initial mass of each compound in solution. Considering the sequence of HOC adsorption first and surfactant adsorption second as an example, 5.0 mg of lampblack were mixed with 10.0 ml of pesticide solution (atrazine, 8.40 mg/L; diuron, 7.89 mg/L) for 10 d following the same procedure as used for the adsorption isotherm measurement. At the end of the first treatment, 3.0 ml of surfactant solution, with a concentration of 21.7 mg/L of surfactant, were added to the system. The system was then mixed again for another 10 d to reach adsorption equilibrium. At the end of the second treatment, the HOC and surfactant in the aqueous phase were then analyzed using the same procedure as described earlier. In the case of HOC and surfactant adsorption at the same time, the mixture of 5.0 mg of lampblack and 13.0 ml of solution containing 5.0 mg/L of the surfactant and 6.46 mg/L of atrazine or 6.06 mg/L of diuron was mixed for 20 d. For the HOC and surfactant adsorption at the same time sequence, the equilibration time was doubled to have the same conditions in all cases.

**Surfactant K_{OW} measurement**

To measure the octanol–water partition coefficient ($K_{OW}$) of the surfactants, 4.0 ml of n-octanol were put into a 40-ml glass vial containing 20.0 ml of aqueous solution at a surfactant concentration of 1.50 mM, prepared with deionized water. The mixture was vortexed for 5 min and then allowed to stand for an additional 15 min before centrifugation at 1,000 g for 5 min [15]. The surfactant concentration in each phase was then measured by HPLC. The $K_{OW}$ of each surfactant was calculated as the ratio of the surfactant concentration in the n-octanol phase to the concentration in the water phase. The measurement was conducted twice, and the measured $K_{OW}$ is presented in Table 2. The pH of the aqueous phase was measured between 6.5 and 7.0 in all cases.

**HPLC analysis**

A Shimadzu HPLC system was equipped with two LC-10AT VP pumps, a Sil-10AF autosampler, a DGU-14A degasser, and a SPD-M10AVP diode-array detector. A Premier® C18 5µm reverse-phase column (length, 250 mm; inner diameter, 4.6 mm; Shimadzu) was used. The HPLC analyses were carried out using a gradient elution mode with a mobile phase comprised of 90% acetonitrile/10% water at 0.0 min and gradually changed to 50/50% at 8.5 min. The analyses were performed at a constant flow rate of 1.0 ml/min. The ultraviolet detector monitored the absorbance at 222 nm for atrazine, 247 nm for diuron, 225 nm for Triton X-100, 220 nm for LAS, and 209 nm for BC. Some samples were diluted as needed. The calibration was conducted daily, and $r^2$ was greater than 0.98 in all cases.

**Langmuir adsorption isotherm**

The adsorption considered in the present study was Langmuir isotherm, as described by Equation 1 [16]:

$$ C_s = \frac{C_{max} K_s C_m}{1 + K_s C_m} $$

where $C_s$ is the adsorbed surfactant concentration ($\mu$M/g), $C_{max}$ is the surfactant saturation adsorption capacity of the sorbent ($\mu$M/g), $C_m$ is the aqueous surfactant concentration ($\mu$M/L), and $K_s$ is the Langmuir constant (L/$\mu$g).

The linearized form of Equation 1 is as follows:

$$ \frac{1}{C_s} = \frac{1}{C_{max} K_s} + \frac{1}{C_{max}} $$

**RESULTS AND DISCUSSION**

**Adsorption isotherms**

Figure 2 presents the equilibration time versus the amount of HOC and surfactant adsorbed onto the lampblack. As can be seen, after the first 3 d, the change in the amount of HOC or surfactant adsorbed was not significant. Thus, the equilibration time (10 d) used for the isotherms measurement was more than sufficient.

The Langmuir isotherm (Eqn. 1) provided a reasonable fit to all the adsorption data; data fitting was conducted using...
Equation 2. Figure 3 presents the HOC and surfactant adsorption data (average of duplicate measurements) along with the fitted Langmuir isotherms, and Table 3 presents the corresponding Langmuir parameters ($C_{\text{max}}$ and $K_l$). The relative errors in all cases were within 10% of the averages.

Diuron showed higher adsorption capacities on the lampblack compared with those of atrazine (Fig. 3A). This is expected, because the lampblack surfaces are hydrophobic and diuron is more hydrophobic than atrazine, as denoted by its higher $K_{OW}$ (Table 1). At the saturation adsorption, the calculated areas occupied per atrazine and diuron molecule were 1.6 and 1.2 nm$^2$, respectively. The difference reflects lower affinity for atrazine compared with that for diuron.

The sorption capacity of surfactants onto soils and sediments usually follows the sequence cationic > nonionic > anionic. This results from the fact that soils and sediments surfaces are negatively charged and attract the cationic surfactant molecules, which can participate in cation-exchange interactions, whereas the anionic surfactants generally are repelled from the negatively charged soil and sediment surfaces. The sorption of Triton X-100 onto the soils and sediment has been found to be highly correlated with the soil cation-exchange capacity [17]. The same trend, however, was not observed for the surfactant adsorption onto the lampblack. Rather, the nonionic surfactant Triton X-100 was found to have the highest adsorption capacity among all the surfactants, followed by LAS and then by BC, which showed the least adsorption capacity among the surfactants (Fig. 3B).

It has been reported that at saturation adsorption, the adsorbed Triton X-100 on a lampblack surface results in a monolayer molecular orientation, which is characterized by a configuration of the molecules perpendicular to the surface [13,14]. The hydrophobic part of the surfactant chain is adsorbed on the lampblack surface, leaving the hydrophilic heads extending orthogonally into the bulk solution. At saturation adsorption, the calculated adsorption area per Triton X-100 molecule was 0.57 nm$^2$, which is very close to the results (0.58 nm$^2$) reported by Musselman and Chander [13]. If the same configuration is assumed for LAS and BC, then at saturation adsorption, the adsorption area per LAS and BC molecule would be 0.61 and 1.26 nm$^2$, respectively.

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Surfactant adsorption onto a hydrophobic surface is balanced by the hydrophobicity of its hydrophobic tail and the hydrophilicity of its hydrophilic head. The hydrophobic tail of the surfactant molecule tends to be attracted to the hydrophobic surface, whereas the hydrophilic head has higher affinity to the aqueous phase and tends to pull it away from the hydrophobic surface. For an ionic surfactant molecule, its ionic head is expected to be much less attracted by the hydrophobic surface compared with the head group of a nonionic surfactant. Thus, one would expect that LAS would exhibit much less adsorption onto the lampblack than Triton X-100 would. Ac-
tually, however, LAS showed an adsorption capacity (69.0 μM/g) similar to that of Triton X-100 (71.0 μM/g). In contrast, BC exhibited much lower adsorption capacity (33.2 μM/g) onto the lampblack compared with LAS or Triton X-100. Thus, the charge of a surfactant is not adequate for predicting its adsorption onto a hydrophobic medium. The results indicated that the $K_{OW}$ of these surfactants also was not a good predictor of their adsorption onto the hydrophobic surface, because Triton X-100 has a much higher $K_{OW}$ compared with LAS and BC (Table 2). Because $K_{OW}$ was measured with n-octanol, which serves as a medium into which the surfactant molecules absorb whereas lampblack serves as a surface onto which the surfactant molecules adsorb, the mechanisms involved in the two cases are different. In the latter case, the surfactant head groups do not get into the interior of the lampblack, but in the first case, LAS and BC have much more difficulty than Triton X-100 in getting into the octanol phase because of their charged head groups, leading to a much lower $K_{OW}$ than that of Triton X-100. The results did show that the $K_{OW}$ of LAS is twice as large as that of BC, suggesting LAS is more hydrophobic than BC. Although LAS and BC have similar molecular weights, the nature and location of the functional groups of LAS (benzene sulfonate) versus BC (benzene-quaternary ammonium), based on these findings, appear to dominate the hydrophobic interactions. The hydrophobic–hydrophilic balance of a surfactant could be a better indicator for predicting surfactant adsorption, because it considers both the head’s hydrophilicity and tail’s hydrophobicity [18]. Unfortunately, to our knowledge hydrophobic–hydrophilic balance has not been reported for ionic surfactants, such as LAS and BC, perhaps because of the complexity involved in measuring it.

The present results indicate that Triton X-100 and LAS reached their saturation adsorption capacities when the aqueous surfactant concentration was around their respective CMCs, which is consistent with the result of other studies [6,7], suggesting that surfactants adsorb only as monomers. Interestingly, the adsorption capacity of BC was reached at an aqueous concentration (240 μM/mL) much lower than its CMC (941 μM/mL), meaning that the lampblack surfaces were saturated with adsorbed BC before the CMC was reached in the aqueous phase. Because the loading of lampblack was constant for all experiments (200 mg), it is possible that a different behavior would be exhibited by BC under higher lampblack loading.

Effect of surfactant on HOC adsorption

Figure 4 presents the dependence of the amount of HOC sorbed on the amount of surfactant sorbed on the lampblack. Clearly, unlike surfactant sorption onto soils and sediment, the sorption of surfactant onto the lampblack reduces HOC sorption, with the reduction in HOC sorption increasing monotonically with the amount of surfactant sorbed, implying adsorption competition between HOC and surfactant. It is worth mentioning that for this range of amount of surfactant sorbed, the equilibrium aqueous surfactant concentrations were all determined to be less than their CMCs, so the effect of surfactant micelles does not need to be considered. The maximum equilibrium surfactant concentrations were 0.94, 1.95, and 1.27 μM/mL for Triton X-100, LAS, and BC respectively. Thus, as compared with Figure 3B, the adsorption saturation of any surfactant was not reached, and the observed reduction in HOC adsorption was caused, essentially, by surfactant adsorption onto the lampblack surfaces.

The results showed that the adsorbed Triton X-100 is most effective in reducing HOC adsorption, followed by LAS and then by BC. As the surfactant adsorption increases, the adsorbed surfactant gradually occupies more of the lampblack surface, decreasing the amount of adsorption sites available for the HOCs. In addition, as more and more surfactant is adsorbed, the surfaces of the lampblack become more and more hydrophilic, further reducing the approach of HOC molecules to the surfaces. This is somewhat counterintuitive, because the HOCs are much more hydrophobic than the surfactants, based on their $K_{OW}$. This highlights the difficulty in using $K_{OW}$ as a basis for such comparisons. The $K_{OW}$ measures the ability of a given molecule (HOC or surfactant) to partition between water and n-octanol. In this case, the n-octanol serves as a medium into which the molecules transfer, whereas the CG is a surface onto which the surfactant molecules adsorb. Thus, the mechanisms involved in the two cases are different. The ionic head groups significantly hinder the ability of cationic or anionic surfactants to transfer into n-octanol compared to Triton X-100 or the HOCs, even though their hydrophobic tails can interact strongly with the CG surface. This indicates that the tail groups of BC and LAS are significantly more hydrophobic than inferred based on their measured $K_{OW}$.

When comparing the percentage of HOC adsorbed onto the
Fig. 5. Percent hydrophobic organic compound adsorbed in the presence of the surfactant (considering the hydrophobic organic compound adsorbed in the absence of surfactant as 100%; □ = atrazine with Triton® X-100 [Sigma-Aldrich, St. Louis, MO, USA]; △ = atrazine with benzalkonium chloride [BC]; ◊ = atrazine with linear alkylbenzene sulfonate [LAS]; ★ = diuron with Triton X-100; ○ = diuron with BC; □ = diuron with LAS).

Fig. 6. Hydrophobic organic compound (HOC) adsorption onto the lampblack under different adsorption sequences for (A) atrazine and (B) diuron. BC = benzalkonium chloride, LAS = linear alkylbenzene sulfonate.

Effect of adsorption sequence

Figure 6 presents the amount of HOC sorbed onto the lampblack under three adsorption sequence scenarios as described in Materials and Methods. As can be seen, the amount of HOC adsorbed under these scenarios did not show significant differences, which suggests the following: The almost identical adsorption of the HOCs under the three scenarios implies that the HOC adsorption equilibrium was achieved in all three cases, but unlike the presorbed HOC onto soil and sediment, the presorbed HOC onto the lampblack could be easily desorbed by the surfactant solution. Minimal desorption hysteresis was observed, suggesting that the HOC adsorption takes place mainly via hydrophobic adsorption and that the contribution from site-specific interactions was minimal.

CONCLUSION

The adsorption of two HOCs, atrazine and diuron, onto lampblack in the presence of surfactant was studied. The results showed that the hydrophobicity of the HOC determines the adsorption capacity onto the lampblack. Linear alkylbenzene sulfonate showed an adsorption capacity higher than that of BC but similar to that of Triton X-100. Under subsaturation adsorption conditions, surfactant adsorption was found to reduce HOC adsorption to a significant extent, with the reduction increasing monotonically with the amount of surfactant adsorbed. Among the three surfactants, Triton X-100 was the most effective in reducing HOC adsorption, whereas BC and LAS showed similar effectiveness in this regard. For the same amount of the surfactant sorbed, the reduction in atrazine adsorption was consistently more than that for diuron because of atrazine’s lower hydrophobicity. No significant difference was observed in the amount of the HOC adsorbed under different HOC and surfactant adsorption sequences. The presence of surfactant can significantly decrease HOC adsorption onto hydrophobic CGs. Thus, understanding it is important in predicting HOC fate and transport in the environment, because HOC sorption significantly affects the HOC retardation and the degradation rate.

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REFERENCES


Surfactant affects HOC sorption onto hydrophobic CG


