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Resilience of the sorption capacity of soil organic matter during drying-wetting cycle

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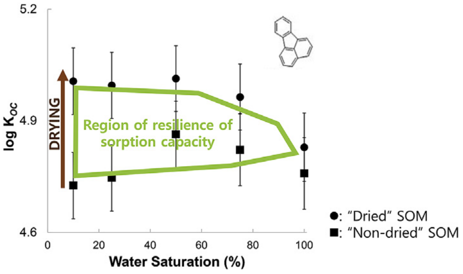
HIGHLIGHTS

- Effects of drying-wetting cycle on K_{OC} were evaluated.
- Oven-drying of SOM increased K_{OC}, however, it was lowered by re-wetting.
- K_{OC} at 100% water saturation were not different during drying-wetting cycle.
- The range of K_{OC} shows the resilient margin under environmental drying-wetting cycle.

GRAPHICAL ABSTRACT



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ABSTRACT

The sorption capacity of soil organic matter (SOM) for hydrophobic organic chemicals (HOCs) is affected by various environmental factors, such as soil water saturation and drying. In this study, we used passive sampling to investigate the changes in the sorption capacity of SOM during a drying-wetting cycle using batch sorption experiments. Dried and non-dried peat mosses were used to observe the effect of the drying process on the sorption capacity of SOM at various levels of water saturation in soil pores. At soil with non-dried peat moss, the partition coefficient between the sampler and the soil ($K_{sampler/soil}$) slightly increased with decreasing water saturation. At soil with dried peat moss, however, there were almost no differences in the $K_{sampler/soil}$ among different water saturations except for 100%. The soil organic carbon-water distribution coefficients (K_{OC}) for dried peat moss were consistently larger than those for non-dried peat moss at all water saturation levels. However, the K_{OC} values obtained at 100% water saturation for both non-dried and dried peat mosses differed only by 18–29%. For fluoranthene, there was only an 18% difference between the two K_{OC} values at 100% water saturation, whereas it was 91% at 10% water saturation. This finding suggests that wetting SOM returns mostly its sorption capacity for HOCs after the increase in K_{OC} caused by extreme drying. The range in sorption capacity obtained in this study showed the resilient margin of the sorption capacity of SOM for HOCs according to microclimatic changes that would occur constantly under environmental conditions.

1. Introduction

Hydrophobic organic chemicals (HOCs), such as polycyclic aromatic hydrocarbons (PAHs), are pervasive contaminants in soils. The sorption of HOCs to soil organic matter (SOM) is one of the most important processes determining the fate of HOCs in soils

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(Chiou et al., 1984; Chefetz et al., 2000; Onken and Traina, 1997; Pignatello and Xing, 1996). It is crucial to understand the bioavailable fraction of HOCs for the prioritization, monitoring, and remediation of HOC-contaminated soils (Alexander, 2000; Lamoureux and Brownawell, 1999; Loonen et al., 1997).

It is well acknowledged that the sorption capacity of SOM for HOCs may vary significantly among types of SOM (Gao et al., 1998; Hawthorne et al., 2006; Kim and Kwon, 2010) and also be affected by various environmental factors, such as soil water saturation (Bartolomé et al., 2018; Chiou and Shoup, 1985; Kim et al., 2017; Roy et al., 2000; Rutherford and Chiou, 1992). For example, the measured soil organic carbon-water distribution coefficients (K_{OC}) of phenanthrene vary among different types of SOM (Gao et al., 1998; Hawthorne et al., 2006; Kim and Kwon, 2010). Even for the same organic matter, the K_{OC} values change with the level of water saturation in pore spaces. In our previous study, the K_{OC} increased with increasing soil water saturation for relatively low molecular weight PAHs (i.e., phenanthrene, anthracene, fluoranthene, and pyrene), whereas the trend was the opposite for higher molecular weight PAHs (i.e., chrysene, benzo[*a*]pyrene, and dibenzo[*a,h*]anthracene) (Kim et al., 2017). In addition, drying is another important environmental factor that affects the sorption capacity of SOM. Liu and Lee (2006) suggested that drying sedimentary organic matter makes it more hydrophobic by exposing hydrophobic functional groups on the surface. Conformational changes due to drying would be responsible for the changes in the sorption properties of organic matter (Wang et al., 2016). Thus, the variability of K_{OC} could be attributed to the differences in the molecular-level structure of organic matter and the conformational changes due to environmental factors such as pH, water saturation, and drying.

Although sorption experiments are usually conducted under controlled conditions in a laboratory such as 100% saturation of pores, organic matter in soil might undergo continuous changes owing to the microclimate conditions of soils (Hong and Luthy, 2008; Scherr et al., 2009; Stefaniuk and Oleszczuk, 2016). These conformational changes in organic matter would occur within relatively shorter time, whereas longer time is required for changes in the covalent bonding structures of natural organic matter (Dziadowiec, 1987; Zech et al., 1992). Therefore, it is helpful to understand the margin of K_{OC} by determining the band of sorption capacity of organic matter due to the microclimatic changes and whether the conformational changes in SOM are resilient.

In this study, we aimed to evaluate whether the changes in the sorption capacity of SOM during a drying-wetting cycle are resilient. Soil peat moss was chosen as the model SOM because it is widely used for agricultural purposes and sorption studies (e.g., Kim et al., 2017; Chefetz et al., 2000) and recommended for preparing artificial soil in soil bioaccumulation tests (OECD, 2010), and K_{OC} values for phenanthrene, anthracene, fluoranthene, and pyrene were measured under various drying-wetting conditions. A passive sampling technique was applied using polydimethylsiloxane (PDMS) as the sampling material. Artificial soil was used for the test. Dried and non-dried peat mosses were used to observe the effect of the drying process on the sorption capacity of SOM at various levels of water saturation in the soil pores.

2. Materials and methods

2.1. Chemicals

Phenanthrene (98%), anthracene (99%), fluoranthene (98%), and pyrene (99%) were selected as model HOCs. Model PAHs and solvents (ACS grade), whose purities were greater than 98%, were purchased from Sigma-Aldrich (St. Louis, MO, USA), Supelco (Bellefonte, PA, USA), or Fluka (Buch, Switzerland). The relevant chemical properties of the PAHs are summarized in Table 1.

2.2. Passive sampler

A custom-cut medical-grade PDMS sheet (10 mm × 40 mm; 125 μm; Specialty Silicone Products, Inc., Ballston Spa, NY, USA)[14] was inserted in an aluminum sampler holder (see Fig. S1, Supplementary Material) and used as a passive sampler.

2.3. Artificial soil

Artificial soil composed of 75% sand (SiO_2), 20% kaolin clay ($H_2Al_2Si_2O_8 \cdot H_2O$), and 5% peat moss was used for passive sampling and determination of K_{OC}, following the composition of artificial soil in the OECD's test guideline (OECD, 2010). Sand and kaolinite were purchased from Fisher Scientific (Loughborough, UK), and peat moss was purchased from Premier (Rivière-du-Loup, Canada). For homogenization, peat moss was grinded using a blender and sieved through a 2 mm sieve. This was used as the non-dried peat moss. To prepare the dried peat moss, sieved non-dried peat moss was placed in a drying oven at 105 °C for 1 week. After drying, the mass was reduced by 38.8% upon the loss of combined water. Sand and kaolinite clay were mixed without any further treatment with non-dried or dried peat moss to make artificial soils.

2.4. Passive sampling experiments

Three hundred grams of artificial soil was placed in a glass box and spiked with 50 mL of methanol solution containing 5 mg of each PAH. The initial concentrations of PAHs were rather higher than typical natural levels, but less than reported concentrations in heavily polluted sites (Bhatt et al., 2002; Eom et al., 2007). After spiking, the soil samples were mixed thoroughly by hand, followed by air-drying in a fume hood for 3 d to completely evaporate the methanol. The levels of water saturation of the soil samples were adjusted to 0%, 10%, 25%, 50%, 75%, and 100%, as conducted in our

Table 1
Chemical properties of the selected PAHs in this study.

Chemicals	log K_{ow}[a]	log K_{PDMSw}[b]	log K_{oc}[c]	H (Pa m^3 mol^{-1})[d]	D_a (10^{-6} m^2 s^{-1})[e]	D_w (10^{-10} m^2 s^{-1})[e]
Phenanthrene	4.52	3.87	4.04	5.61	5.93	6.70
Anthracene	4.50	3.98	4.11	6.33	5.93	6.70
Fluoranthene	5.20	4.32	4.90	2.98	5.73	6.36
Pyrene	5.00	4.36	4.79	2.44	5.73	6.42

[a] Sangster Research Laboratory, LOGKOW-A databank of evaluated octanol water partition coefficient (log P). (http://logkow.cisti.nrc.ca/logkow/index.jsp expired on October 3, 2014).
[b] Measured using the ABL permeation method reported by Kwon et al. (2007).
[c] Measured log K_{OC} values by Gao et al. (1998).
[d] Determined using the diffusion in air and water boundary layers determined by Lee et al. (2012).
[e] Calculated values from Lee et al. (2012).

previous study (Kim et al., 2017). To prepare 100% saturated soils, 300 g of water was poured into the glass box containing 300 g artificial soil. All soil solids including peat moss were submerged in water. Before deploying passive samplers, soils were prewetted for one day. To meet desired levels of water saturation, calculated volume of water based on the porosity of artificial soil was added and then mixed with soil thoroughly by hands. Five passive samplers were completely submerged vertically per glass box and all the glass boxes were placed in an incubator at 25 °C in the dark. Gravimetric analysis of three samples of 10 g soil after the experiments revealed that the water saturation of the soil and the concentrations of the spiked PAHs did not change during the experiments (Table S1, Supplementary Material). The passive samplers were removed from the boxes 7, 14, 28, 35, and 42 d after deployment, and the concentrations of PAHs in the samplers were measured. Each PDMS sheet was submerged in 4 mL of acetonitrile, and PAHs were extracted for 1 d at 25 °C in a shaking incubator at 150 rpm.

2.5. Determination of partition coefficients, uptake rate constants, and organic carbon-water partition coefficients

Because the PAH concentrations in the soils remained constant during the experiments, the changes in the concentration of a PAH in a PDMS sampler ($C_{sampler}$) is described by a differential equation:

$$\frac{dC_{sampler}}{dt} = k_u C_{soil} - k_e C_{sampler} \tag{1}$$

The analytical solution for $C_{sampler}$ is

$$\begin{aligned} C_{sampler} &= C_{soil} k_{sampler/soil}[1 - e^{-ket}] \\ &= C_{soil} k_{sampler/soil}[1 - e \frac{k_u}{k_{sampler/soil} t}] \end{aligned} \tag{2}$$

where C_{soil} is the concentration of a PAH in the soil, $K_{sampler/soil}$ is the partition coefficient between the sampler and the soil [kg L^{-1}], k_u is the uptake rate constant [kg L^{-1} d^{-1}], and k_e is the elimination rate constant from the sampler [d^{-1}] (Kim et al., 2017). The concentration of PAHs in the PDMS was measured using an high-performance liquid chromatography system (Waters, Milford, MA, USA). Time-course measurements of $C_{sampler}$ were used to determine both $K_{sampler/soil}$ and k_u using the nlsfit function in R software (R Development Core Team, 2019).

The distribution coefficient between soil and water (K_d) was calculated using the determined $K_{sampler/soil}$ value and the value of the partition coefficient between PDMS and water ($K_{PDMS/w}$) in the literature (Table S2, Supplementary Material) (Kwon et al., 2007). The organic carbon-water partition coefficient (K_{OC}) was then calculated by dividing K_d by the mass fraction of the soil organic carbon in artificial soil because sorption of neutral HOCs is dominated by sorption to SOM (e.g., Chefetz et al., 2000; Onken and Traina, 1997) although clay mineral components are also important for sorption of especially more polar chemicals (e.g., Cox et al., 1997, Shi et al., 2010).

2.6. Instrumental analyses

The selected PAHs were analyzed using an HPLC system equipped with a quaternary gradient pump (Waters 600 E, Milford, MA, USA), an auto sampler (Waters 717+), and a multiwavelength fluorescence detector (Waters 2475). The flow rate of the eluent was 1 mL min^{-1}. The wavelengths of excitation/emission were 260/352 nm for phenanthrene; 260/420 nm for anthracene, pyrene; and 260/460 nm for fluoranthene. Concentration in the passive sampler extracts was quantified using a calibration curve obtained from independently prepared external standards at the range of 0.0625–1.0 mg L^{-1} (Kim et al., 2017). The coefficients of determination (r^2) were greater than 0.99 and the relative standard deviations for a control standard (0.20 mg L^{-1} each) were 1.1–7.3% ($n = 5$).

The fractions of organic carbon in the non-dried and dried peat mosses were measured using an elemental analyzer (Elementar Analysensysteme GmbH, Vario-Micro Cube, Germany), and they were 34.3% and 49.6% on total weight basis, respectively. In addition, pore volume, pore size, and surface area of the non-dried and dried peat mosses were measured using a surface and pore size analyzer (BET; Quantachrome, Autosorb-iQ 2ST/MP, Austria). The results of the BET measurements are shown in Table 2.

3. Results and discussion

3.1. Kinetic experiments

The uptake of PAHs by the PDMS passive sampler is shown in Figs. S2 and S3 (Supplementary Material). Corresponding to our previous study, for 0% water-saturated soils, both the $K_{sampler/soil}$ and k_u values for all the selected PAHs were much lower than those obtained from soils containing at least some pore water (Kim et al., 2017). This could have been explained by the fact that the presence of at least a certain level of pore water that is sufficient to wet mineral surfaces sharply reduces the adsorption capacity of mineral surfaces for organic compounds (Chiou and Shoup, 1985; Goss et al., 2004; Rutherford and Chiou, 1992). For the soils with dried peat moss, obtained mass by passive sampling per time was higher than those for soils with non-dried peat moss at the same soil water saturation.

3.2. Effects of drying-wetting cycle on distribution coefficients and uptake rate constants

The effects of water saturation on $K_{sampler/soil}$ and k_u were similar with our previous study, although the peat moss was obtained from a different supplier in this study (Figs. 1 and 2). All the $K_{sampler/soil}$ and k_u values are listed in Tables S1 and S2, Supplementary Material. For the soils with non-dried peat moss, $K_{sampler/soil}$ and k_u slightly increased with decreasing water saturation. The water saturating process leads to a conformational change in organic matter and swells the inner void volume of organic matter (Liu and Lee, 2006). The expansion of organic matter may create inner sorption sites that can host relatively smaller molecules. Kopinke and Stottmeister (1996) suggested that the steric restrictions could be important for sorbates greater than 1.0 nm in wet SOM. The minimal cross-section of low molecular weight PAHs in this study are less than 1.0 nm, sufficiently smaller than pore diameters (Table 2). Thus, wet expansion of SOM might increase the observed K_{OC} for lower molecular weight PAHs.

However, for the soils with dried peat moss, the $K_{sampler/soil}$ and k_u values were lower than those for soils with non-dried peat moss at the same soil water saturation level (Figs. 1 and 2). Because the drying process makes organic matter more hydrophobic (Chiou and

Table 2
Specific surface areas, pore diameters, and volumes of dried and non-dried peat mosses.

	Dried peat moss	Non-dried peat moss
Surface area (m^2 g^{-1})	36.06	17.04
Pore diameter (nm)	2.6	2.8
Pore volume (cm^3 g^{-1})	0.033	0.017

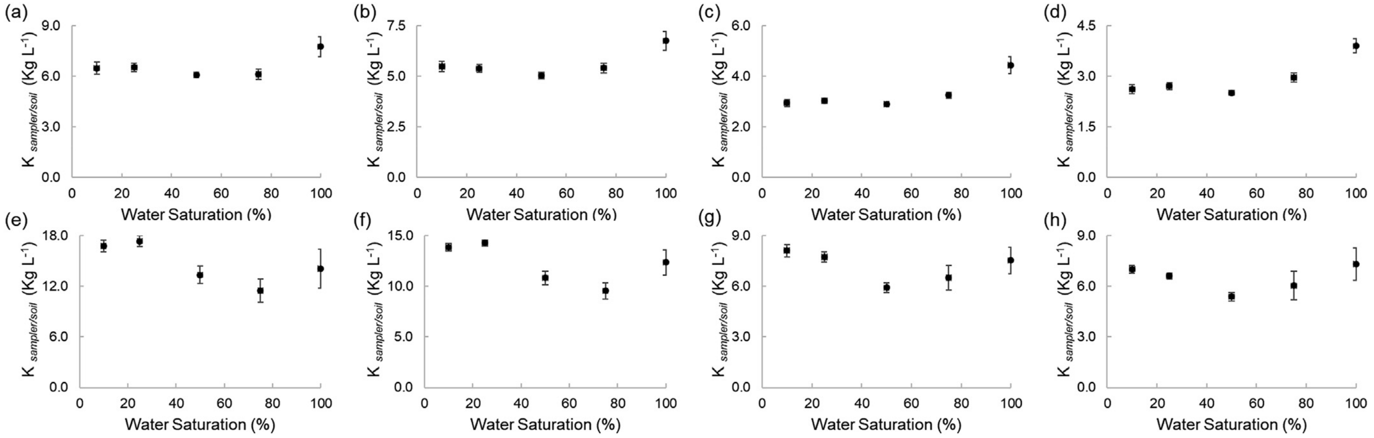

Fig. 1. Partition coefficient between sampler and soil ($K_{sampler/soil}$) at various soil water saturations for phenanthrene (a), anthracene (b), fluoranthene (c), pyrene (d) in soil with "dried" peat moss, and for phenanthrene (e), anthracene (f), fluoranthene (g), pyrene (h) in soil with "non-dried" peat moss. Error bars represent the standard errors of regression using eq (2).

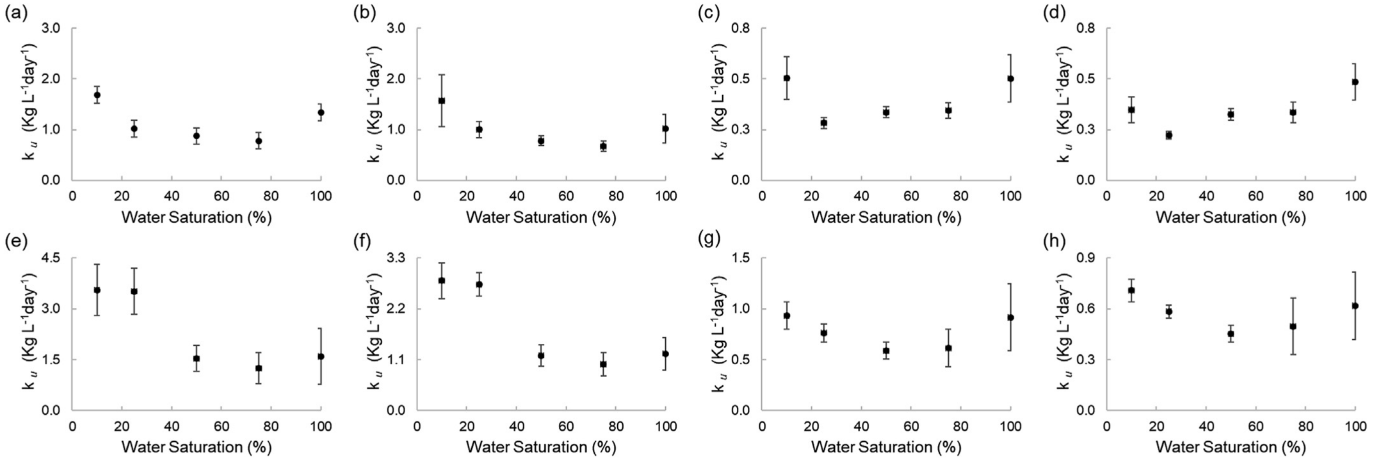

Fig. 2. Uptake rate constant (k_u) at various soil water saturations for phenanthrene (a), anthracene (b), fluoranthene (c), pyrene (d) in soil with "dried" peat moss, and for phenanthrene (e), anthracene (f), fluoranthene (g), pyrene (h) in soil with "non-dried" peat moss. Error bars represent the standard errors of regression using eq (2).

Shoup, 1985; Goss et al., 2004; Liu and Lee, 2006; Roy et al., 2000; Rutherford and Chiou, 1992), the sorption capacity of SOM would increase. In addition, the surface area and total pore volume were increased after drying, as demonstrated by the BET areas in Table 2. The pore diameter did not change significantly, whereas the total pore volume and surface area were almost doubled. In Fig. 1a–d, except for 100% saturated soil, the differences between the $K_{sampler/soil}$ values at various water saturation levels were small. For 100% saturated soil, the $K_{sampler/soil}$ value was relatively larger than those of the other saturation levels, which was likely because the complete wetting of soil pores mitigated the conformational changes caused by the drying process (Liu and Lee, 2006). The effects of water saturation on $K_{sampler/soil}$ and k_u shown in soils with non-dried peat moss were different with soils with dried peat moss.

The changes in K_{OC} values at various soil water saturation levels are depicted in Fig. 3, and the values of K_{OC} are also presented in Table 3. The K_{OC} values for soil with non-dried peat moss under 100% saturated soil were similar to those reported in a previous study (Gao et al., 1998), as most of the earlier studies measured K_{OC} values using saturated soils or sediments (Gao et al., 1998; Hawthorne et al., 2006; Kim and Kwon, 2010). However, the K_{OC} values for dried peat moss were consistently larger than those for non-dried peat moss at all other water saturation levels. As mentioned previously, the drying process makes organic matter more hydrophobic (Chiou and Shoup, 1985; Goss et al., 2004; Liu and Lee, 2006; Roy et al., 2000; Rutherford and Chiou, 1992) and makes the surface area and total pore volume increased, as demonstrated by the BET areas in Table 2, explaining the increased sorption capacity of SOM.

3.3. *Resilience in sorption capacity of soil organic matter during drying-wetting cycle*

The K_{OC} values obtained at 100% water saturation for both the non-dried and dried peat mosses differed by only 18–29% from each other, unlike other pairs of values at different water saturation levels (Fig. 3). For all test chemicals, as soil water saturation increases, difference of K_{OC} between soil with non-dried and dried peat moss tends to decrease (values are shown in Table 3). For example, for fluoranthene, the difference between the two K_{OC} values was 91% at 10% water saturation, whereas it was only 18% at 100% water saturation. Because the experiments were conducted for 42 d, the wetting of organic matter was likely to recover the sorption capacity for HOCs at least to a certain extent. Natural organic matter forms from biological materials over a long period of time (Dziadowiec, 1987; Zech et al., 1992) and is likely to experience drying-wetting cycles owing to the fluctuation in temperature and the level of pore water. After chemically inert organic matter forms,

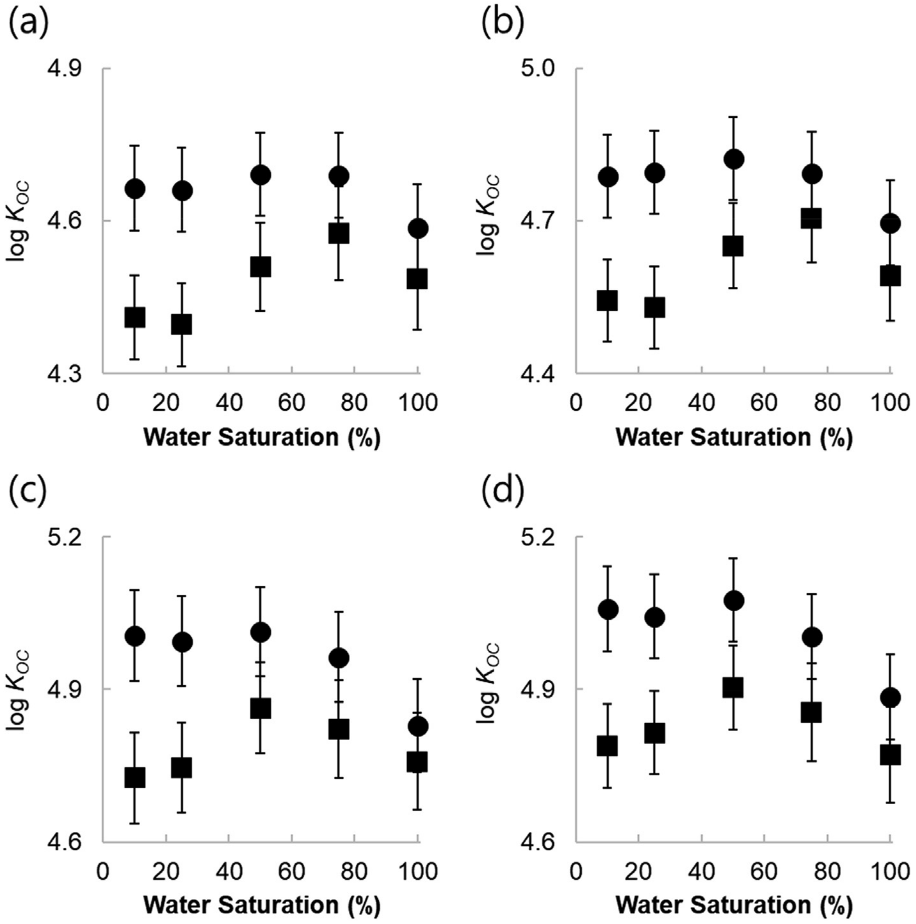

Fig. 3. Logarithmic organic carbon-water partition coefficients (log K_{oc}) at various soil water saturation levels for soil with non-dried peat moss (■) and dried peat moss (●) for (a) phenanthrene, (b) anthracene, (c) fluoranthene, and (d) pyrene. Error bars represent the standard errors.

Table 3
Values of log K_{OC} at various soil water saturation levels (Φ) for four polycyclic aromatic hydrocarbons.

Φ (%)	log K_{OC} for soil with dried peat moss				log K_{OC} for soil with non-dried peat moss			
	Phenanthrene	Anthracene	Fluoranthene	Pyrene	Phenanthrene	Anthracene	Fluoranthene	Pyrene
100	4.59 (4.48, 4.67)	4.65 (4.54, 4.73)	4.83 (4.71, 4.92)	4.88 (4.78, 4.97)	4.49 (4.35, 4.59)	4.54 (4.43, 4.63)	4.76 (4.64, 4.85)	4.77 (4.65, 4.87)
75	4.69 (4.59, 4.77)	4.74 (4.64, 4.82)	4.96 (4.85, 5.05)	5.00 (4.9, 5.09)	4.58 (4.46, 4.67)	4.66 (4.55, 4.74)	4.82 (4.70, 4.92)	4.85 (4.73, 4.95)
50	4.69 (4.59, 4.77)	4.77 (4.67, 4.85)	5.01 (4.90, 5.10)	5.08 (4.98, 5.16)	4.51 (4.40, 4.60)	4.60 (4.50, 4.68)	4.86 (4.75, 4.95)	4.90 (4.80, 4.99)
25	4.66 (4.56, 4.74)	4.75 (4.65, 4.83)	5.00 (4.88, 5.08)	5.04 (4.94, 5.13)	4.40 (4.29, 4.48)	4.48 (4.38, 4.56)	4.75 (4.63, 4.84))	4.81 (4.71, 4.90)
10	4.66 (4.56, 4.75)	4.74 (4.64, 4.82)	5.01 (4.89, 5.10)	5.06 (4.96, 5.14)	4.41 (4.31, 4.49)	4.49 (4.39, 4.57)	4.73 (4.61, 4.82)	4.79 (4.69, 4.87)

95% confidence limits were calculated using error propagation, and are shown in parentheses.

it still undergoes continuous conformational changes owing to such environmental factors. Thus, the sorption capacity of organic matter could be understood as a dynamic property. The extreme drying condition in this study might have exaggerated real environmental drying-wetting cycles. However, the sorption capacity of organic matter for selected PAHs seemed to be almost recovered within a few months, and the results provided the resilient range of the sorption capacity of organic matter via conformational changes induced by water in soil pores.

The dynamic range of K_{OC} for a given organic matter was up to a factor of two in this study. Under typical drying-wetting conditions and the fluctuation of water saturation levels of unsaturated soils, the variation might be neglected for practical purposes. However, the dynamic range of K_{OC} were greater for larger PAHs (Kim et al., 2017) and the steric effects become more important on sorption of larger hydrophobic organic molecules (Kopinke and Stottmeister, 1996). Thus, further research is needed to know the dynamic ranges of K_{OC} due to water saturation and drying-wetting cycles for HOCs especially having higher molecular weights.

4. Conclusions

Effects of drying-wetting cycle on the sorption capacity of SOM were evaluated by using passive sampling to observe the changes in K_{OC} at various soil water saturations. Although different types of peat moss were used, the same effects of water saturation on K_{OC} shown in the previous study (Kim et al., 2017) were observed on soil with non-dried peat moss. Drying makes SOM more hydrophobic and increases the surface area and total pore volume of SOM, leading to an increase in the sorption capacity. As soil water saturation increased, however, difference of K_{OC} between soil with non-dried and dried peat moss decreased. This means that the sorption capacity of SOM is likely to change within a resilient range during natural drying-wetting cycle. Although other environmental factors, such as pH, ionic strength, and pore water composition, could also be responsible for conformational changes in SOM, the range of sorption capacity obtained in this study by changing extreme drying-wetting cycle extends our understanding of the dynamic nature of the sorption processes of HOCs in soils.

Declaration of competing interest

The authors claim no conflicts of interest.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.chemosphere.2019.125238.

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