Sorption of Polycyclic Musks on Soil Components of Different Aggregate Sizes: The Effect of Organic Matter–Mineral Interactions

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Abstract
Polycyclic musks (PCMs) in soils have been of increasing concern because of their potential characteristics of persistence, bioaccumulation, and ecological risk. However, little is known about their fate process in soil environment. Here, two PCMs namely galaxolide (HHCB) and tonalide (AHTN) were selected as sorbates to explore their sorption process in soils. Sorption batch experiments with six soils and their different aggregate fractions were carried out to elucidate the effect of organic matter–mineral interactions in different aggregate fractions on sorption of these two PCMs. The possible causes of variation in the organic carbon-normalized partition coefficient ($K_{oc}$) for HHCB and AHTN have been investigated. The strong influence of organic matter–mineral interactions on $K_{oc}$ was evidenced by the large variation in $K_{oc}$ on HF-treatment for both bulk soils and their different aggregate fractions. This study verified the dual effect of organic matter–mineral interactions among selected soils, and in promoting or inhibiting sorption may be related to the types of organic matter–mineral interactions. There were also interactions between soil components with different aggregate sizes, which affected the variation of $K_{oc}$ in the bulk soil. This study represents a valuable contribution to the understanding of the fate processes and behaviors of HHCB and AHTN in soils and its implication on the risk assessment.

Keywords Organic matter–mineral interactions · PCMs · Sorption · $K_{oc}$ · Different aggregate fractions

Polycyclic musks (PCMs) were used as fragrance ingredients in numerous consumer products such as cleaning agents and personal care products (Kannan et al. 2005). It was found that PCMs could have strong potentials to be persistent (Balk and Ford 1999), toxic to various aquatic organisms (Gooding et al. 2006; Pedersen et al. 2009; Schnell et al. 2009; Yamauchi et al. 2008) and accumulated in human blood, adipose tissue and even breast milk (Kannan et al. 2005; Luckenbach and Epel 2005; Reiner et al. 2007). Studies have reported the wide distribution of some PCMs in environmental compartments (Azaroff et al. 2020; Lange et al. 2015; Lyu et al. 2021; Sumner et al. 2010; Wang and Kelly 2017; Yang and Metcalfe 2006; Zhang et al. 2008, 2020). Given to the wide distribution, toxic effect, and ecological risk, PCMs have raised significant concerns. Due to the hydrophobicity ($\log K_{ow}>5$) and poor removal efficiencies of sewage treatment plants (Horii et al. 2007), PCMs released from wastewater effluents were most likely to accumulate in sewage sludge or biosolids and finally discharged to soils by sewage irrigation and sludge application (Kannan et al. 2005; Yang and Metcalfe 2006). Therefore, soils were always considered as main receiver of PCMs. Based on this circumstance, studying the fate process of PCMs in soils is of great significance for understanding their environmental occurrence, risk management and control.

Sorption of organic pollutants (OPs) to soils was an environmentally important fate process that would affect the bioavailability, ecotoxicity, rate of degradation, and their mobility of OPs (He et al. 2014; Smernik and Kookana 2015). For hydrophobic compounds, the sorption process was considered as a partitioning between the solution and the soil organic matter (SOM), and such sorption was generally quantified by the carbon-normalized partition coefficient ($K_{oc}$) (Ahmad et al. 2001). The $K_{oc}$ values were used to estimate the mobility potential of OPs in soils.
However, the wide variations of reported $K_{oc}$ values were common in the scientific literature both between soils (Ahangar et al. 2008; Savini et al. 2017; Smernik and Kookana 2015) and between different isolated aggregate fractions (Bonfleur et al. 2016; Bonin and Simpson 2007). Many studies were devoted to elucidate the relative contribution of soil minerals and SOM to $K_{oc}$ variation with different model contaminants. The chemical composition of SOM was known to have a substantial influence on the sorption of OPs. Smernik and Kookana (2015) reported that the $K_{oc}$ of diuron increased with the aromatic C content and decreased with O-alkyl C and alkyl C content. While Ahangar et al. (2008) found that $K_{oc}$ was positively correlated with aryl C for diuron. Some authors hold that variations in herbicide $K_{oc}$ values were induced by changes in SOM aromaticity (Ahmad et al. 2006; Farenhorst et al. 2010). Besides the chemical properties of SOM, sorption of OPs by SOM were also influenced by minerals. The ratio of clay content to total organic carbon content (RCO) was used to explore the relative importance of minerals in influencing OP behavior in different soils by some investigators (He et al. 2014, 2011; Liu et al. 2008).

As a multi-component and structurally organized combination, soil matrix contained organic matter–mineral complexes of different aggregates sizes (Zhou et al. 2004). Some studies have shown that the interaction between the SOM and minerals may block adsorbable functional groups on SOM surfaces, which changed configuration of SOM when associated with minerals and resulted in a negative contribution of minerals to OP sorption by soils (Bonfleur et al. 2016; Savini et al. 2017; Smernik and Kookana 2015; Wang and Xing 2005a, b). However, minerals may also positively contribute to OP sorption in specific soils as organic matter–mineral complexes highly exposed (He et al. 2011). Besides, a dual function of organic matter–mineral interactions for OPs sorption in soils was also found (He et al. 2014; Liu et al. 2010). He et al. (2014) proposed that soil weathering might affect the contribution of minerals for OP sorption, but there has no any direct evidence to demonstrate the effect on soil weathering.

Not only the bulk soil components, but also the components in different particle sizes could interact with each other. Because the chemical nature of SOM varied with different aggregate sizes (Amelung et al. 1998), the mechanisms of OP sorption by organic matter–mineral interactions could vary as a function of particle-size distribution as well as organic matter chemistry (Liu et al. 2010). Many studies attributed the $K_{oc}$ variation for a specific OP to organic matter–mineral interactions in bulk soil. However, little information was obtained for organic matter–mineral interactions in different aggregate fractions that could influence sorption of OPs in soil. The aim of this study is to further explore the sorption behavior of OPs, using PCMs as model contaminants, in bulk soils, and in particular, the effect of organic matter–mineral interactions in different particle size was studied and reported.

**Materials and Methods**

In this study, we hypothesized that the effect of organic matter–mineral interactions in different particle size to sorption of bulk soils was different. Two PCMs, i.e., galaxolide (1,3,4,6,7,8-hexahydro-4,6,6,7,8,8-hexamethyl-cyclopenta-(g)-2-benzopyran, HHCB) and tonalide (7-acetyl-1,1,3,4,4,6-hexa-methyltetraline, AHTN) were selected as model contaminants because of their high production and consumption (Rimkus 1999). The physicochemical properties of the two PCMs have been given in Table S1 of Supporting information (SI). Our objective was to explore how organic matter–mineral interactions in different particle size influence $K_{oc}$ variation of the two model PCMs among soils. To achieve it, we measured the sorption to soils and their different aggregate fractions using a batch equilibration method. The effect of organic matter–mineral interactions in different aggregate fractions were evaluated by comparing $K_{oc}$ variation before and after HF-treated soils. This research is a step forward toward a better understanding of the effect of organic matter–mineral interactions in different aggregate fractions to PCMs sorption.

HHCB (purity > 85%), AHTN (purity > 98.5%) and hexamethylbenzene (HMB), were purchased from Dr. Ehrenstorfer (Germany). HMB was the internal standard. The reagents (methanol, dichloromethane, acetone and n-hexane) were of HPLC grade and obtained from Fisher (USA). Uncultivated soils were collected from the surface horizon (0–20 cm) in the Hunan, Gansu, Zhejiang, Jiangxi, Jilin and Guangdong provinces of China. All soil samples were air dried, ground and passed through 2- and 0.25-mm sieves. The pH of all soil samples was measured in a 1:1000 or 1:4000 soil/water suspension, total organic carbon (TOC) was determined by the oxidation method with K$_2$Cr$_2$O$_7$–H$_2$SO$_4$. Basic properties of the soil samples were provided in Table S2. Fourier transform infrared spectrometer analysis was described in SI and characterization of different bulk soils has been given in Fig. S1.

Aggregate size fractionation of the bulk soil was conducted for soils 1–6 based on mechanical dispersion. The detailed fractionation procedures are described in Table S3. After fractionation, 50–200 μm, 5–50 μm, 1–5 μm and < 1 μm fraction were freeze-dried and then weighed.

Hydrofluoric acid can effectively remove paramagnetic species and also concentrate SOM by removing most soil minerals (Ahmad et al. 2001). Briefly, the HF treatment consisted of 12 successive treatments with 2% HF solution. Each treatment involved shaking 5 g soils (the bulk soil and...
their different aggregate fractions respectively) in 50 mL of 2% HF solution and filtered through 0.45 μm Teflon filters, end-over-end, for periods of 0.5 h (two times), 1 h (two times), 2 h (seven times) and 16 h (once). After the final extraction, the residue was washed three times with deionized Millipore water for shaking 10 min and the supernatant solution was discarded and then freeze-dried. The sorption studies were performed using the batch method and then samples were determined using an Agilent 6890 N-5975B gas chromatography-mass selective detector (GC-MSD, Agilent Technologies, USA), details were given in the SI.

### Results and Discussion

The result shown that the $K_{oc}$ values varied greatly among all bulk soils (Fig. 1). It ranged from 185 to 815 L g$^{-1}$ for HHCB and 152 to 822 L g$^{-1}$ for AHTN among six bulk soils. The bulk soil 5 had the highest $K_{oc}$ values and the lowest appeared in bulk soil 2. Additionally, the $K_{oc}$ variations were also large in different aggregate fractions of soils for both two PCMs. The obtained $K_{oc}$ values in < 1 μm aggregate fraction spread out in a wide range: 227 to 895 L g$^{-1}$ for HHCB (Fig. 1A), and 258 L g$^{-1}$ to 819 L g$^{-1}$ for AHTN (Fig. 1B). In 1–5 μm aggregate fraction, the $K_{oc}$ values ranged from 147 to 450 L g$^{-1}$ for HHCB; and 148 to 280 L g$^{-1}$ for AHTN. The $K_{oc}$ values ranged from 126 to 291 L g$^{-1}$ for HHCB and 148 to 311 L g$^{-1}$ for AHTN in 5–50 μm aggregate fraction. In 50–200 μm aggregate fraction, the $K_{oc}$ values ranged from 53 to 343 L g$^{-1}$ for HHCB and 51 to 236 L g$^{-1}$ for AHTN.

Some studies found that sorption was highest at the smaller soil fractions (< 2 μm) due to their higher specific surface area and the presence of more humified organic materials (De Jonge et al. 2000; Liu et al. 2010; Wang and Keller 2009). However, in the present study, the greatest $K_{oc}$ values did not always occur in the smaller soil fractions, such as < 1 μm aggregate fraction. The bulk soil and the 5–50 μm aggregate fraction also yielded the largest $K_{oc}$ values under specific soil conditions (e.g., with bulk soil in soil 3, soil 5 and soil 6, and the 5–50 μm aggregate fraction in soil 2 for both HHCB and AHTN). The 50–200 μm aggregate fraction in all soils had the lowest $K_{oc}$ values except soil 5. There was a significant difference in the $K_{oc}$ values of the same soil with different aggregate fractions. Some studies explained this phenomenon that different humic fractions from the same soil may resulted in different $K_{oc}$ values (Gunasekara and Xing 2003; Kang and Xing 2005; Mao et al. 2002).

Tables S6 and S7 shown the TOC content of SOM ($f_{oc}$) in different aggregate fractions and bulk soil before and after HF-treatment, respectively. It could be found that the HF-treatment increased the TOC content of the bulk soils and their different aggregate fractions by a factor of 1.13–27.0 (average 4.1), which indicated that organic matter–mineral complexes were distributed in different aggregate fractions and bulk soils. From Fig. S2, it was obviously that corresponding $\Delta K_{oc}$ values for whole soils and $\Delta f_{oc}$ content was only weakly correlated, a fact confirmed by linear regression ($R^2 = 0.230$ for HHCB, $R^2 = 0.224$ for AHTN). It was interesting that the greater $\Delta K_{oc}$ values were appeared at soils with lower $\Delta$ TOC, such as soil 1. The $K_{oc}$ values were lower for all the HF-treated bulk soils than the untreated bulk soils (Fig. 2 and Fig. S3). This finding was consistent with a previous study (He et al. 2014), which reported a much tighter range of increase in $K_{oc}$ on H$_2$O$_2$-treatment. The results indicated that the organic matter–mineral interactions may directly contribute to the sorption of PCMs on bulk soils. He et al. (2014) hold that the newly formed sorption sites on the soil minerals increased the $K_{oc}$ values in those aggregate fractions after the chemical removal of SOM.

However, the $K_{oc}$ values in different aggregate fractions were not always decreased after HF-treatment. Instead, the $K_{oc}$ increased in 50–200 μm fraction or 5–50 μm fraction of all soils except soil 1. For example, with the depletion

![Fig. 1](image-url) The organic carbon content normalized partition coefficient for HHCB (A) and AHTN (B) in bulk soil and different sized soil aggregate fractions.
Fig. 2 $K_{oc}$ variation of HHCB before and after HF-treatment among selected bulk soils and their different aggregate fractions. The gray bar indicates the change in $K_{oc}$ before and after HF-treatment. $\Delta K_{oc} = K_{oc \text{ (after the HF-treatment)}} - K_{oc \text{ (before the HF-treatment)}}$. $\Delta K_{oc} > 0$ after HF-treatment indicates an increase of $K_{oc}$; and $\Delta K_{oc} < 0$ after HF-treatment indicates a decrease of $K_{oc}$.
of minerals, TOC ranging from 10.40 to 14.62 mg g\(^{-1}\) in 50–200 \(\mu\)m fraction of soil 5 (Tables S6 and S7), the \(K_{oc}\) increased consistently from 243 to 564 L g\(^{-1}\) for HHCB and from 236 to 520 L g\(^{-1}\) for AHTN, respectively (Fig. 2 and Fig. S3). This indicated that, the organic matter–mineral interactions blocked sorption in these aggregate fractions. An increase in \(K_{oc}\) values on HF-treatment or a decrease in \(K_{oc}\) of the soils after SOM removal were also previously reported (Ahangar et al. 2008; Bonin and Simpson 2007; Smernik and Kookana 2015). Smernik and Kookana (2015) proposed that it was the liberation of SOM sorption sites blocked by interactions with minerals in the whole soils. Bonin and Simpson (2007) believed that physical fractionation may expose sorption sites within SOM that are closely associated with minerals and typically not accessible in bulk soils. Other investigators explained these results with the hypotheses of chemical alteration of the SOM components (Rumpel et al. 2006; Zegougah et al. 2004) and conformational changes (Salloum et al. 2001). However, the \(K_{oc}\) values in <1 \(\mu\)m fraction were decreased among all soils after HF-treatment. This suggested that the organic matter–mineral interactions promoted sorption in this fraction. Therefore, we proposed that maybe it was not sorption site of SOM but minerals were blocked in <1 \(\mu\)m fraction lead to \(K_{oc}\) decreased. In soil 1 and soil 6, there was little change on \(K_{oc}\) after the HF-treatment for the 50–200 \(\mu\)m fraction and the 1–5 \(\mu\)m fraction. Even though the \(\Delta\)TOC content increased from 6.12 to 153.1 mg g\(^{-1}\) (Tables S6 and S7), but there was little contribution to sorption. This shown that maybe the accessible SOM governed the content of target sorption.

However, even for components with the same aggregate size, the effect and strength of matter–mineral interactions to sorption were also different in soils. The organic matter–mineral interactions in 5–50 \(\mu\)m fraction promoted sorption in soil 1, soil 3, soil 4 and soil 5 but blocked sorption in soil 2 and soil 6. The organic matter–mineral interactions in 1–5 \(\mu\)m fraction blocked sorption in all selected soils except soil 5. This could be explained that distinct organic matter–mineral complexes differed significantly in structure and composition were distributed in different aggregate fractions and bulk soils due to the SOM bonded to minerals by various forms (Garbarini and Lion 1986), which in turn influenced the organic matter–mineral interactions strengthen on both the bulk soil and different aggregate fractions to sorption (He et al. 2008).

In order to explore the existence of interactions between different aggregate fractions of soils, we calculated the overlay values of \(\Delta K_{oc}\) which was the sum of \(\Delta K_{oc}\) in different aggregate fractions. The details of calculation were described in SI. If there is no interaction between different aggregate fractions, the measured \(\Delta K_{oc}\) values in bulk soils should match the overlay \(\Delta K_{oc}\) values. Comparing the overlay \(\Delta K_{oc}\) values with the measured \(\Delta K_{oc}\) values, it was obviously unmatched each other (Fig. 2 and Fig. S3). For example, in soil 1, soil 3 and soil 4, both the overlay and the measured \(\Delta K_{oc}\) values indicated that the organic matter–mineral interactions promoted sorption, but the strength of promotion was different. While in the rest bulk soils, the measured \(\Delta K_{oc}\) values suggested organic matter–mineral interactions were positive to sorption while the overlay \(\Delta K_{oc}\) values shown that organic matter–mineral interactions blocked the sorption. Due to the components in different aggregate sizes were not dispersed and independent, but interacted with each other, such as wrapping, covering and so on. As a result, there was a significant difference between the overlay and the measured \(\Delta K_{oc}\) values, which was manifested in the effect and strength of organic matter–mineral interactions on the sorption. Due to the considerable variability in organic matter chemistry in different aggregate sizes, the effect of organic matter–mineral interactions to bulk soil sorption was also different. Therefore, the interactions between different aggregate fractions of soils may also have influence on \(K_{oc}\) variation. Litz et al. (2007) concluded from their experiment that HHCB and AHTN may remain in the soil horizons due to their strong sorption, weak degradability and their low leaching behavior. It can be inferred that HHCB and AHTN are more likely to accumulate in the soil surface. The contribution of the accessible SOM and minerals attraction sites to the two PCMs sorption should be further clarified and quantified to control the ecological risk to soils.

The effect of organic matter–mineral interactions on two PCMs sorption was quantified in six natural soils and their different aggregate fractions. The result shown that wide variations in \(K_{oc}\) values were found in bulk soils and their different aggregate fractions. Organic matter–mineral interactions have important effect on \(K_{oc}\) variety in all soils with different physical and chemical properties. The dual impact of organic matter–mineral interactions was determined on PCMs sorption in different aggregate fractions. Furthermore, there were also associations between components with different particle sizes, which influenced the \(K_{oc}\) variation to some extent. Findings of this work may help understand the effect of organic matter–mineral interactions on PCMs and their fate process in soils.

Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s00128-022-03531-5.

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Data Availability  The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Conflict of interest  The authors declare that they have no competing interests.

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