



# Sorption and desorption of fluorene on five tropical soils from different climes



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## ABSTRACT

The main processes controlling soil–polycyclic aromatic hydrocarbon interaction is sorption–desorption as influenced by the soil physicochemical conditions. Sorption–desorption phenomena can influence translocation, persistence and bioavailability. Hence, laboratory batch experiments were undertaken to investigate the sorption characteristics of fluorene on five tropical soils from varying tropical agro-ecological zones having different physicochemical properties. Fluorene concentrations used for the study ranged from 20 to 100 µg/L, and results showed that fluorene sorption equilibria were attained usually within 24 h. Increase in pH had a negative effect on fluorene sorption. Fluorene sorption was concentration dependent and exothermic. The sorbed fluorene molecules were distributed between surface adsorption sites and phases in soil. Sorption was controlled by weak hydrophobic forces, such as the  $\pi$ – $\pi$  interactions and Van der Waal's forces. Desorption hysteresis were most pronounced in soils with high organic matter contents than lower ones. The sorption was characterized as multiple reaction phenomena composed of several linear and non-linear isotherms. The physicochemical conditions of the soil must be understood in order to predict the fate and bioavailability of fluorene in soil.

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## 1. Introduction

Persistent organic pollutants are introduced into the soil environment via numerous anthropogenic activities (Diagboya et al., 2014; Okoli et al., 2014; USEPA, 2007). Most organic pollutants, especially the polycyclic aromatic hydrocarbons (PAHs), are known to be toxic, some being mutagenic and/or carcinogenic to biota; and thus they have been classified as priority pollutants (USEPA, 2007; ATSDR, 1995). Their effect on biota can be amplified by bioaccumulation in the food chain because PAHs like most organic pollutants are very soluble in fat. Once in the environment, they are subjected to various physical and/or chemical processes that determine their environmental fate (Okoli et al., 2014; Teixeira et al., 2011; Ramirez et al., 2001). Information on the bioavailability of pollutants in the environment is important because bioavailable pollutants are linked to the negative health disorders experienced by biota.

Among the various processes that determine the environmental fate of PAHs, sorption is the relatively most studied (Teixeira et al., 2011; Ramirez et al., 2001) because data obtained can give valuable information on suitable remediation strategy for the polluted soil. Sorption experiments can be used to determine the quantity of PAHs retained by

a soil and, hence, the leachable or bioavailable quantity (Sparks, 2003). Some studies have reported the sorption characteristics of some PAHs in some soils (An et al., 2010; Cottin and Merlin, 2007; Hwang and Cutright, 2002; Guo et al., 2010; Sun and Yan, 2007; Haftka et al., 2010; Teixeira et al., 2011; Ramirez et al., 2001), however there is a dearth of information on the sorption characteristics of these pollutants in several soils especially soils from tropical Africa. Moreover, this kind of information is vital because data obtained from other parts of the world may not be useful for prediction of PAHs' sorption characteristics/remediation strategies for African soils since these soils are unique; formed from different parent materials under different environmental conditions (temperature, moisture, and biota).

The objective of this work was to study the sorption and desorption of fluorene on representative soils obtained from five major sub-Saharan agro-ecological zones (AEZs) varying extensively in physical and chemical properties. The effects of time, pH, and temperature were evaluated and the data obtained were used to predict the mechanism(s) of the sorption process.

## 2. Materials and methods

### 2.1. Sampling and physicochemical characterization

Five representative surface soils (0–30 cm) were obtained from different sub-Saharan AEZs (Table 1a) by compositing randomly collected samples from locations which are relatively free from human activities

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**Table 1a**  
Physico-chemical properties of the soil samples.

Soil	*AEZ	Sampling coordinate	pH water	pH 1 N KCl	EA	*OM (%)	Particle size analysis (%)			Oxides (wt.%)	
							*Sand	*Silt	*Clay	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>
Agbor (AG)	Fresh water swamp	6°11'36" E; 6°15'06" N	6.29	5.39	0.10	5.26	83.80	15.44	0.76	1.54	2.78
Port Harcourt (PH)	Mangrove swamp	6° 59' 55" E; 4° 47' 21" N	7.66	7.24	0.06	5.29	68.04	30.50	1.45	1.09	2.96
Ibadan (IB)	Moist-lowland/Rainforest	3° 54' 00" E; 7° 26' 30" N	6.57	5.88	0.08	4.68	69.94	16.44	13.63	3.71	3.18
Maiduguri (MA)	Sudan SV*	13° 09'42" E; 11°50'36" N	6.67	5.21	0.02	1.67	70.44	28.89	0.67	0.99	2.23
Monguno (MG)	Sahel SV*	13°36'51" E; 12°40'14" N	8.40	7.59	0.12	0.18	82.24	17.25	0.50	0.93	1.50

\*AEZ – Agroecological zone; SV\* – savanna; \*OM – Organic matter; EA – Exchange acidity; % sand (>20 µm); % silt (2–20 µm); % clay (<2 µm).

or impacts, such as locations far from road traffic sites, wastes dumps, places where little or no fertilizers have been applied, etc. The samples were air-dried, crushed gently and sieved through a 230-mesh size sieve and the fine fractions retained for the sorption study. The soils' pH values were determined in Millipore ultra-pure water and 1.0 M of KCl (ratio 1:1) (Benton, 2001). Particle size determination was done by the laser particle analyzer (Mastersizer-2000) after removal of carbonates by washing with hydrochloric acid, treatment with 30% H<sub>2</sub>O<sub>2</sub> to remove organic matter, and dispersing in 0.1 M of sodium hexametaphosphate (Breuning-Madsen and Awadzi, 2005). Organic matter was determined by the Walkley and Black (1934) wet digestion method. Metals (oxides, exchangeable and trace) were determined using inductively coupled plasma optical emission spectrometry (ICP-OES, Optima 5300DV), except for Cd which was done by inductively coupled plasma mass spectrometry (ICP-MS, Agilent 7500). The soil mineralogy was obtained by a Bruker phaser diffractometer system using the whole sample procedure (randomly oriented mounts) and the spectra were analyzed using PANalytical X'Pert HighScore software.

## 2.2. Fluorene sorption

Fluorene solution was prepared in 10% v/v methanol/Millipore ultra-pure water using 0.005 M of CaCl<sub>2</sub> and 0.01 M of NaN<sub>3</sub> as background electrolyte and biocide to eliminate microbial degradation, respectively. Sorption experiments were carried out by adding 10 mL of 100 µg/L (except for the effect of concentration experiments) fluorene solution into brown vials containing 0.50 g of the soil sample and then tightly sealed with Teflon lined screw caps. Aluminum foil was used to wrap the vials to minimize possible losses by photochemical decomposition. The vials were equilibrated in the dark by shaking at 100 rpm in a temperature (25 °C) controlled shaker. For the effect of time, the vials were incubated from 30 to 4320 min. Equilibrium studies and effect of temperature were investigated in the concentration range of 20–100 µg/L at 25 and 40 °C. After the equilibrium experiments fluorene desorption was carried out immediately by thoroughly decanting the incubated solution and replacing it with a fresh background solution of methanol/Millipore ultra-pure water solution containing the same concentration of CaCl<sub>2</sub> and NaN<sub>3</sub> but no fluorene. These were incubated in similar conditions as above, centrifuged after 4320 min, and fluorene concentration determined. All experiments were done at the ambient soil pH except the effect of pH which was done from pH 3 to 9 using 100 µg/L fluorene concentrations at 25 °C. Control experiments without soil samples were done to account for possible losses due to volatilization and sorption onto the vial walls (and these were found to be negligible). After incubation, the vials were centrifuged at 4000 rpm for 20 min, and the concentration of fluorene in solution determined by Perkin Elmer fluorescence spectroscopy (model LS 55) using the synchronized scan at a wavelength difference (Δλ) of 45 nm and scanning from 200 to 350 nm. The synchronous peak at 264 nm was used to monitor the sorption. All experiments were done in triplicate and the averages of the data have been used for computation.

## 2.3. Data treatment

The amounts of fluorene removed from solution were calculated from the initial ( $C_0$ ) and final ( $C_e$ ) concentrations in solutions after the incubation using the Eq. (1):

$$q_e = \frac{(C_0 - C_e)V}{M} \quad (1)$$

where  $q_e$ ,  $V$  and  $M$  are the amount of fluorene sorbed (µg/g), volume of the solution (mL) and mass (g) of sample, respectively.

The sorption data was explained using four kinetics models (Lagergren (1898) pseudo-first and -second order, Elovich (Olu-Owolabi et al., 2012) and Weber and Morris (1963) intra-particle diffusion kinetics models); three adsorption isotherm models (Langmuir (1916), Freundlich (1906), and the distributed reactivity model (Weber et al., 1992)); as well as the thermodynamic parameters – standard free energy ( $\Delta G^\circ$ ), enthalpy change ( $\Delta H^\circ$ ), and entropy change ( $\Delta S^\circ$ ) (see supporting materials SM1–3 for further data treatments).

## 3. Results and discussion

### 3.1. Soil properties

The physicochemical characteristics of the soils are shown in Tables 1a and 1b. The pH values were mainly neutral (6.0–7.5) except for MG which was alkaline (8.0–9.0). Soil organic matter (SOM) values were high except for MA and MG soils which have been described as medium and very low, respectively (Fagbami and Shogunle, 1995). AG and PH had the highest SOM contents. The mineralogical compositions (Table 1b) indicated that the major minerals in the soils are aragonite, calcite, dolomite, gibbsite, hematite, kaolinite, quartz, and siderite. Quartz was the most common mineral followed by the Al oxide mineral – gibbsite. Kaolinite and the carbonate minerals (aragonite, calcite, siderite, and dolomite) are common features of these soils. The 2:1 expansive clay minerals, montmorillonite and illite, were observed in MA and MG, respectively. The textural measurements (Table 1c) showed that the soils are majorly sandy loam (PH, IB, and MA) and loamy sand (AG and MG) (soil survey staff, 2006).

### 3.2. Sorption studies

#### 3.2.1. Sorption time and kinetics mechanism

Result (Fig. 1a) of the effect of incubation time on sorption showed that equilibrium fluorene sorption on the whole soils were attained usually within 1440 min for these soils, except for PH soil which attained equilibrium in 2880 min. At equilibrium, fluorene sorption was constant and no significant desorption was recorded. Literature has shown that equilibrium attainment for fluorene sorption on soils does not occur at a specific time but is dependent on soil type (Kohl and Rice, 1999; Chilom et al., 2005). The variation in attainment of equilibria on these soils was attributed to the sorption characteristics of the soils' constituents. It was observed that soils with higher organic matter

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