



## Performance of retrievable activated carbons to treat sediment contaminated with polycyclic aromatic hydrocarbons



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### HIGHLIGHTS

- Magnetic ACs show PAH sorption coefficients comparable to non-magnetic AC in water.
- PAH sorption coefficients for magnetic ACs are slightly reduced in sediment slurry.
- Magnetic ACs show reasonable performance to extract PAHs from sediment.
- Low collection efficiency of magnetic ACs is a practical constraint for application.

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### ABSTRACT

The feasibility of sediment treatment by magnetized and textile forms of activated carbon (AC) is evaluated in a laboratory well-mixed condition, targeting removal of polycyclic aromatic hydrocarbons (PAHs) from contaminated sediments by amendment of the AC and subsequent AC retrieval. In water, the apparent PAH sorption coefficients for magnetized ACs are comparable to those for a non-magnetized AC, while the textile form of AC exhibits smaller values, which is likely due to the slow PAH sorption kinetics resulting from its thickness. When the magnetized ACs are added in a sediment slurry, the apparent PAH sorption coefficients generally become somewhat smaller than those determined in water, suggesting the sorption attenuation effect by dissolved and/or colloidal organic matter for the ACs. Still, treatment of a PAH-contaminated sediment by 5 dry wt% of the magnetized ACs substantially reduces polyethylene sampler uptakes (by 88–89%). By analyzing PAHs after magnetic separation of the AC particles and sediment, it is shown that significant PAH mass removal can be achieved by the AC-sediment contact followed by AC retrieval. The feasibility of the sediment treatment is challenged by low magnetic particle collection efficiency (50–60%), suggesting that the durability of magnetic susceptibility of the magnetized ACs should be improved.

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### 1. Introduction

Activated carbon (AC) is one of the most efficient sorbents for organic contaminants, widely used for the purification of water [1]. Exploiting its excellent sorption capacity and affinity for hydrophobic organic contaminants (HOCs), an *in-situ* sediment remediation technique using AC as sediment amendment

was developed and is being practically applied in several sites around the world [2,3]. The effectiveness of *in-situ* AC amendment to control the (bio)availability of HOCs in sediment has been demonstrated using aqueous concentration measurements [4,5], passive sampler uptakes [5–7], and uptakes in sediment biota [5,7] as indicators.

Retrieval of AC from sediment after deployment would achieve HOC mass removal and would be attractive in some situations [8]. For example, in field application the technique is used to control the risk and bioavailability of HOCs, and this may fail to comply with local regulations or treatment goals, which are often established as sediment HOC concentration determined by exhaustive extraction procedures [8].

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A limited number of studies have been recently reported in the literature for the application of the AC amendment technique with complete removal of HOCs from sediment by physical separation of the AC from sediment [8,9]. One approach is to contact fine-grained sediment with granular activated carbon (GAC) and subsequently separate the GAC from the sediment by sieving and centrifugation [8]. The applicability of this technique to extract HOCs from sediment was demonstrated by Rakowska et al. [8]. Another approach suggested is to use magnetic AC produced by a wet precipitation of iron oxide to AC, and magnetic separation of the particles from sediment after contact [9]. Han et al. [9] reported that the effectiveness of magnetic ACs to reduce aqueous HOC concentrations in sediment slurry is comparable to that of non-magnetized ACs.

In this study, magnetic ACs were produced in the laboratory using AC from two different feedstocks, coal and coconut. A commercially available AC in textile form, which can also be easily retrieved after contacting with sediment, was obtained. Polycyclic aromatic hydrocarbon (PAH) sorption characteristics of the three types of retrievable ACs were compared with that of a non-magnetized AC both in clean water and a sediment slurry. The potential of PAH mass removal from contaminated sediment by contacting with the coal-based magnetic AC and sediment and then selectively collecting the magnetic particles was experimentally demonstrated.

## 2. Materials and Methods

### 2.1. Materials

Two types of commercially available AC, one derived from bituminous coal by Calgon Corporation (Type TOG; Pittsburgh, PA) and the other derived from coconut shells by Cabot Norit Nederland B.V. (Amersfoort, the Netherlands), were obtained. The coal-derived AC (henceforth referred to as *coal-AC*) was ground and sieved to obtain 75–150  $\mu\text{m}$  grain size range. The coconut shell-derived AC (henceforth referred to as *coconut-AC*) was pulverized to obtain a grain size of <65  $\mu\text{m}$ . The geometric mean particle size of the coconut-AC was determined to be 7.3  $\mu\text{m}$  by Han et al. [10].

The coal- and coconut-ACs were magnetized by a chemical precipitation technique suggested in Han et al. [10] with the details of procedure provided therein. In brief, 25 g coal- or coconut-AC, 18.3 g of  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ , and 33.3 g  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  were added to a 2 L beaker filled with 1 L de-ionized water, and placed on a heater. After heating the mixture up to 65 °C, it was cooled down to 40 °C while stirring. The pH of the mixture was adjusted to 11 by adding 5 M NaOH solution to precipitate the iron hydroxides, and then the stirring was continued for another hour. After resting overnight, the supernatants were removed from the mixture. The precipitants were then washed and rinsed with de-ionized water into the filter paper followed by rinsing with ethanol. The magnetized product was ready for use after drying at 80 °C in an oven overnight. The magnetic carbons produced from coal- and coconut-ACs were referred to as *mag-coal-AC* and *mag-coconut-AC*, respectively.

In addition to the magnetized ACs, a commercially-available, textile form of AC (Type FM30 K; Chemviron Carbon Cloth Division, Houghton le Spring, U.K.) was used as a candidate for retrievable AC. This material is hereafter referred to as *AC cloth*. According to the manufacturer, the AC cloth was produced from viscous rayon. The source material underwent a series of chemical and heating processes to reduce the rayon and activate the carbon-rich product, resulting in a textile with a thickness of 0.4 mm made entirely of AC.

Low-density polyethylene (PE) with 51  $\mu\text{m}$  thickness was obtained from Brentwood Plastics (St. Louis, MO, U.S.). The PE was pre-cleaned by rolling at 2 rpm for 24 h with clean hexane, 30 min

**Table 1**

Physicochemical properties and PAH concentrations for the sediment sample used in this study. All values are obtained from Choi et al. [11]. Values in parentheses represent standard deviations for triplicate measurements.

Properties	Values
Total organic carbon content (%)	4.0 ( $\pm 0.1$ )
Black carbon content (%)	0.73 ( $\pm 0.07$ )
Total petroleum hydrocarbons ( $\text{mg kg}^{-1}$ )	3740
Oil and grease content ( $\text{mg kg}^{-1}$ )	10800
Total parent-PAHs <sup>a</sup> ( $\text{mg kg}^{-1}$ )	11.6 ( $\pm 4.3$ )
Total alkylated-PAHs <sup>b</sup> ( $\text{mg kg}^{-1}$ )	114 ( $\pm 16$ )
Total PAHs <sup>c</sup> ( $\text{mg kg}^{-1}$ )	125 ( $\pm 17$ )

<sup>a,b</sup>List of the parent- and alkylated-PAHs is found in Fig. 1.

<sup>a</sup> Sum of 18 parent-PAHs.

<sup>b</sup> Sum of 20 groups of alkylated-PAH isomers.

<sup>c</sup> Sum of total parent PAHs and total alkylated PAHs.

with clean acetone, and then 30 min with deionized water. The PE was then collected, dried at 60 °C, and stored at 4 °C until use.

A sediment sample collected from a petroleum-impacted site was used for the current study. The physicochemical properties and the PAH concentration of the sediment analyzed in our previous study [11] are provided in Table 1 and Fig. 1.

### 2.2. Sorbent characterization

Specific surface area, pore volume, and average pore size were determined by gas ( $\text{N}_2$ ) adsorption/desorption using the BET method with the Micrometrics TriStar 3000 Analyzer system as described in Han et al. [10]. The material density was measured by a pycnometer (Accupyc 1330, Micrometrics). The surface element distribution was analyzed by a Scanning Electron Microscope (SEM) equipped with an Everhart Thornley secondary electron detector, and an Energy-Dispersive X-ray spectroscopy (EDX) detector (Leo 1450 VP, Oxford INCA Energy 200 Premium Si (Li) SATW-Detector). An acceleration voltage of 17 kV was used. The SEM-EDX analysis was conducted for one of the magnetized ACs, mag-coconut-AC, to verify the formation of the iron oxide composite and observe the surface coverage of the AC by the iron deposits.

### 2.3. Sorption coefficient measurements in clean water

Sorption coefficients for PAHs between water and the retrievable ACs, mag-coal-AC, mag-coconut-AC, and AC cloth, were determined in clean water following a procedure described previously [11,12]. The sorption coefficients for coal-AC were also determined for comparison. Firstly, clean 2.5 cm x 25 cm PE strips were contacted with the sediment for 20 weeks to preload the PEs with a mixture of PAHs. The preloaded PEs were collected, wiped clean, and stored at 4 °C in a capped vial until use. Triplicate 2.5 cm x 2.5 cm PE pieces were taken from the batch to analyze preloaded PAH concentration. The PAH concentration in the preloaded PE followed the characteristic PAH distribution in the original sediment as shown in Fig. A.1 in the Supplementary Data.

The preloaded PEs were equilibrated with each of the sorbents studied to determine sorption coefficients. After adding 50 mg preloaded PE and 200 mg sorbent in a 40 mL vial, the vial was filled with deionized water containing 1  $\text{mg L}^{-1}$  sodium azide as a microbial inhibitor. The vials were capped and shaken at 100 rpm for 3 months to achieve equilibrium. The preloaded PEs were then retrieved, wiped clean, and analyzed for PAH concentrations. The apparent sorption coefficients in clean water,  $K_{s(app)-clean}$  ( $\text{cm}^3$

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