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RESEARCH ARTICLE

Bioturbation effects on heavy metals fluxes from sediment treated with activated carbon

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Abstract Adding activated carbon (AC) to sediment has been proposed as an in situ sediment remediation technique. To date, it is not clear whether this technique is effective in the treatment of heavy metal-contaminated sediment in the presence of bioturbators. In the present study, we compare the ability of granular-activated carbon (GAC) and powder-activated carbon (PAC) to reduce Cu, Zn, and Pb pore water concentrations at environmentally relevant concentrations in the absence and presence of *Chironomid* larvae. Compared to untreated sediment, PAC and GAC addition in the absence of *Chironomid* larvae resulted in reductions of free Cu concentrations of 78 and 66 % just below the sediment–water interface after 28 days, respectively. While for Pb and Zn these concentration reductions were only 40 and 38, 19 and 25 %, respectively. The presence of *Chironomid* larvae in untreated, and GAC sediment generally increased the free heavy metals concentrations in pore water, especially in the deeper layers. In comparison with untreated sediment, the coexistence of AC enhanced the accumulation of heavy metals, especially for PAC. This increased bioaccumulation may decrease the survival of *Chironomid* larvae. The result indicated that ACs may not be suitable for the remediation of heavy metal-contaminated sediments.

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Introduction

Sediment is a huge storage reservoir for natural and anthropogenic pollutants in aquatic systems (Huguenot et al. 2015; Koelmans and Jonker 2011; Xu et al. 2012). These sediment-bound pollutants including heavy metals act as pollutants sources to aquatic ecosystems. However, remediation of contaminated sediments remains a technological challenge. Recently, in situ sediment remediation technology introducing sorbent amendments into contaminated sediments effectively reduces organic contaminant bioavailability and has provided a new direction in contaminated sediment management (Ghosh 2011). Adsorbents most frequently used for this purpose include activated carbon (AC) (Beckingham and Ghosh 2011, 2013; Kupryianchyk et al. 2012; Tomaszewski et al. 2007). Adding activated carbon (AC) to sediment has been proposed as a remediation technique to reduce HOC release to the overlying water (Cho et al. 2007; Oen et al. 2011; Sun and Ghosh 2008; Zimmerman et al. 2004). In fact, with high surface areas, the sorption capabilities of activated carbon are very high not only for organic contaminants, but also for metals (Kongsuwan et al. 2009; Weber and Van Vliet 1981). For example, ACs can be effective in reducing pore water concentrations of Hg and MeHg in sediments (Gomez-Eyles et al. 2013). Therefore, ACs are hypothesized to be effective to enhance heavy metals binding to sediments, and thus to reduce the bioavailability of heavy metals. However, most earlier studies addressed the effectiveness of AC on remediation of HOCs contaminated sediment (Cho et al. 2009; Kupryianchyk et al. 2013), whereas the effectiveness on heavy metals has been studied less frequently.

Bioturbation can influence the fate, transport, and bioavailability of sediment-bound heavy metals (Ciutat and Boudou 2003; Schaller 2014), it may be responsible for a major fraction of the pollutants released from sediments to the water column (Cardoso et al. 2008; Josefsson et al. 2010; Thibodeaux and Bierman 2003). In addition, bioturbation may affect sediment remediation processes because it is a main process controlling mobilization of elements including dissolved organic carbon (DOC) from sediments (Schaller 2014). For example, the presence of *Asellus aquaticus* could lead to increased turbulence and HOCs fluxes, and therefore decreased efficiency of sediment remediation with AC (Kupryianchyk et al. 2013). Therefore, if AC amendment was proposed as an in situ treatment of several heavy metal impacted sediment, bioturbation may change the efficiency of remediation.

Addition of 1 % AC, which can reduce water exposure concentration of organisms, was observed to increase the survival of *Gammarus pulex* and by 30 % in 8 days and 5 % after 28 days exposure, increase that of *A. aquaticus* by 100 % in 8 days and 50 % after 28 days exposure, respectively (Kupryianchyk et al. 2011). Janssen and Beckingham reviewed that AC amendments can reduce bioavailability of sediment-associated HOCs by more than 60–90 % (Janssen and Beckingham 2013). However, several studies have reported that addition of AC may have negative impacts on the habitat quality of the benthic organisms, reducing their activity (Janssen and Beckingham 2013; Kupryianchyk et al. 2013). On the other hand, many invertebrate species associated with metals and metalloids polluted sediments in aquatic systems and some of them accumulated metals through diet (Geffard et al. 2010; King et al. 2006; Schaller et al. 2011) Therefore, addition of AC and other sorbents to sediment may affect the metal accumulation in organisms. Shen et al. (2012) reported that BSAF values for polycyclic aromatic hydrocarbons (PAHs) were elevated when MWNT-2 addition to sediment was greater than 1.5 %. It needs to investigate the ecotoxicity response to AC exposure when AC addition to sediment used as an in situ sediment remediation technology.

This work aimed to assess if AC was suitable for the remediation of several heavy metals impacted sediment, the effect of bioturbation on AC amendments was also investigated. Furthermore, the accumulation of heavy metals within the bioturbators was assessed.

Materials and methods

Sediment collection and preparation

Sediments were taken from Ming Tombs Reservoir in north China in May 2013. Copper, zinc, and lead were added into sediment as $CuSO_4 \cdot 5H_2O$, Zn $SO_4 \cdot 7H_2O$, $Pb(NO_3)_2$ in aqueous solution to create sediments with 881.13 mg Cu/kg, 1132.84 mg Zn/kg, and 875.81 mg Pb/kg (dw). After 12 months (kept at 4 °C), the sediment were diluted with Milli-Q water to obtain 20 % d.w. and homogenized. Then, the sediment was amended with powder-activated carbon (PAC) and granular-activated carbon (GAC) to obtain 4 % d.w. and homogenized with an electrical stirrer for 10 min, respectively. The sediments were settled for 1 day before sampling. The sediments were then introduced into acid-washed glass container of 2 L (∅: 13 cm) and 600 mL reconstituted fresh water was carefully added in order to avoid disturbances at the sediment surface. For this indoor experiment, reconstituted fresh water was prepared followed EPA procedures. *Chironomid* larvae (*Chironomus plumosus* larvae) were selected as the test organisms because of their high bioturbation potential and high abundance in aquatic ecosystems.

Experimental setup

The following six experimental conditions were studied: [No bioturbation + untreated], untreated sediment in the absence of *Chironomid* larvae; [No bioturbation + PAC], PAC sediment in the absence of *Chironomid* larvae; [No bioturbation + GAC], GAC sediment in the absence of *Chironomid* larvae; [With bioturbation + untreated], untreated sediment in the presence of *Chironomid* larvae; [With bioturbation + PAC], PAC sediment in the presence of *Chironomid* larvae; and [With bioturbation + GAC], GAC sediment in the presence of *Chironomid* larvae. 100 ± 5 individuals of *Chironomid* larvae were added to the three [With bioturbation] treatments. Four replicates were done for each condition. One subsample was used for the survival experiment and one for a determination of heavy metal concentration in the sediment and pore water.

The experiment systems were placed in an artificial climate chamber (RXZ intelligent, Ningbo Jiangnan Instrument Factory). Temperature was maintained at 23 °C, humidity remained 50 %, and the daily period of light at 16:24-h throughout the entire experiment. Air was continuously bubbled into each unit from a diffuser in the upper layer of the water column and supplied with an air pump. Water depth was kept constant during the 28-day experiment by water additions, which compensated losses due to evaporation and sampling.

Sampling, sample preparation, and analysis

At the exposure time 1, 3, 5, 7, 9, 11, 14, 19, and 28 days, 10 mL of unfiltered water were sampled from each experiment beaker with a polypropylene syringe, then microwave digestion (MARS, China Everbest Machinery Industry Co., Ltd.) with aqua regia, stored at 4 °C and analyzed for total heavy metals within 3 days. Ten milliliter of filtered water was also sampled from each experiment beaker, filtered through 0.45-μm mixed cellulose ester membrane filter (0.45 μm, $d = 25$ mm, Millipore, USA), then acidified with nitric acid and analyzed for dissolved heavy metals. After 28 days of exposure in the microcosms, survival was determined by gently transferring the beaker content to a tray and counting living organisms. Then, the *Chironomid* larvae were depurated in clean, aerated, synthetic freshwater for 6 h, and then placed in glass vials and frozen at −15 °C. Samples were lyophilized and then extracted by microwaved digestion with aqua regia, and analyzed for heavy metals. Then, measured the pH and turbidity of the overlying water for each unit every day. Copper, zinc, and lead were determined by inductively coupled plasma mass spectrometry (ICP-MS) (7500a, Agilent Technologies Co. Ltd., America).

Freely dissolved heavy metal concentrations in sediment pore water were determined using R-LSPM diffusive gradients in thin films (DGT). Device used were obtained from DGT Research Ltd (Lancaster LA20QJ, UK.). DGT samplers were deployed in the water column on the 28th day at the end of sampling in overlying water, following established procedures (Zhang et al. 1998). The DGT probe was pushed gently and smoothly into the sediment. Due to the probe shape, DGT devices were made at sediment depth of about 6 cm and at water depth about 4 cm. After 24 h of deployment (DGT equilibration), DGT probes were gently removed from the beakers, and the position of the sediment–water interface and overlying water depth were recorded. The probes were immediately rinsed with Milli-Q water to remove sediment particles, and then held in clean plastic bags at 4 °C until disassembly. DGT devices were disassembled and resin gel slices cut using a Teflon-coated blade to obtain the desired vertical profile. The upper 4 cm and the lower 6 cm of the gels were cut into twelve 1-cm sections. Each slice was eluted with 1 M HNO_3 for at least 24 h before analysis by ICP-MS.

Results

Effects of AC amendments and bioturbation on pH and turbidity

A significant difference in pH in the treatments with and without *Chironomid* larvae was found. From 0 to 5 days, pH values increased for all of the six treatments. The pH values remained unchanged after 9 days in the absence of *Chironomid* larvae. In treatments with *Chironomid* larvae, the pH values increased sharply after 19 days then decreased sharply at the end of the experiment. The pH values in no bioturbation group are 0.15–0.42 higher than those in the bioturbation groups during the first 20 days. At the end of the experiment, average values were 7.95 and 7.54 for [No bioturbation + untreated] and [With bioturbation + untreated] conditions, respectively, 7.95 and 7.57 for [No bioturbation + PAC] and [With bioturbation + PAC] conditions, respectively, and 7.95 and 7.70 for [No bioturbation + GAC] and [With bioturbation + GAC] conditions, respectively (Fig. 1).

Figure 2 shows the turbidity data in the overlying water during the 28 days. Turbidity in water column was a direct indicator of bioturbation. Without *Chironomid* larvae, average turbidity was low. With *Chironomid* larvae, turbidity was enhanced in all systems, which is attributed to bioturbation. In addition, with AC particles in the sediment, turbidity was less than that of the systems without AC particles after 9 days, due to the reduction in bioturbation activity. After 28 days, average values were 1.15 and 6.90 NTU for [No bioturbation + untreated] and [With bioturbation + untreated] conditions, respectively, 0.89 and 2.91 NTU for [No bioturbation + PAC] and [With bioturbation + PAC] conditions, respectively, and 0.90 and 3.53 NTU for [No bioturbation + GAC] and [With bioturbation + GAC] conditions, respectively.

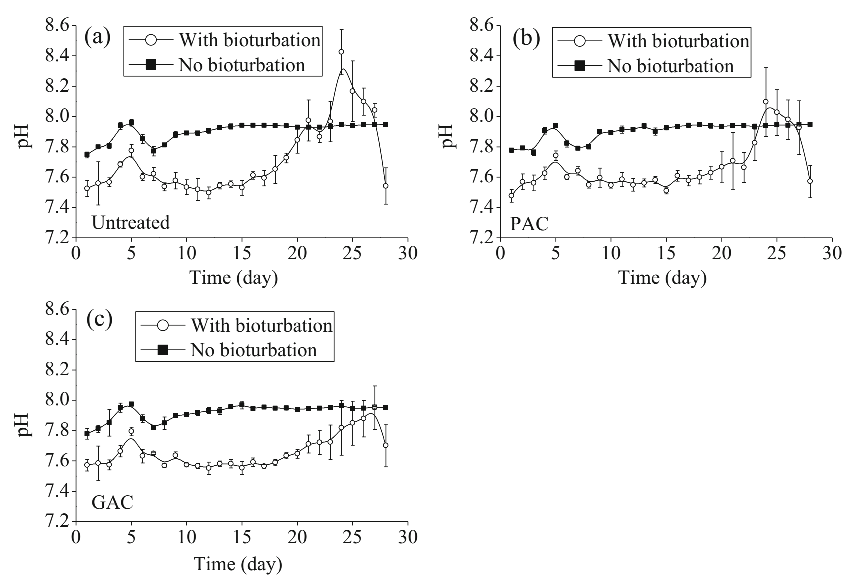

Fig. 1 Comparative study of water column pH during the 28 days experiment for the six experimental conditions studied: in untreated sediment in the absence and presence of *Chironomid* larvae (**a**), in PAC sediment in the absence and presence of *Chironomid* larvae (**b**), and in GAC sediment in the absence and presence of *Chironomid* larvae (**c**)

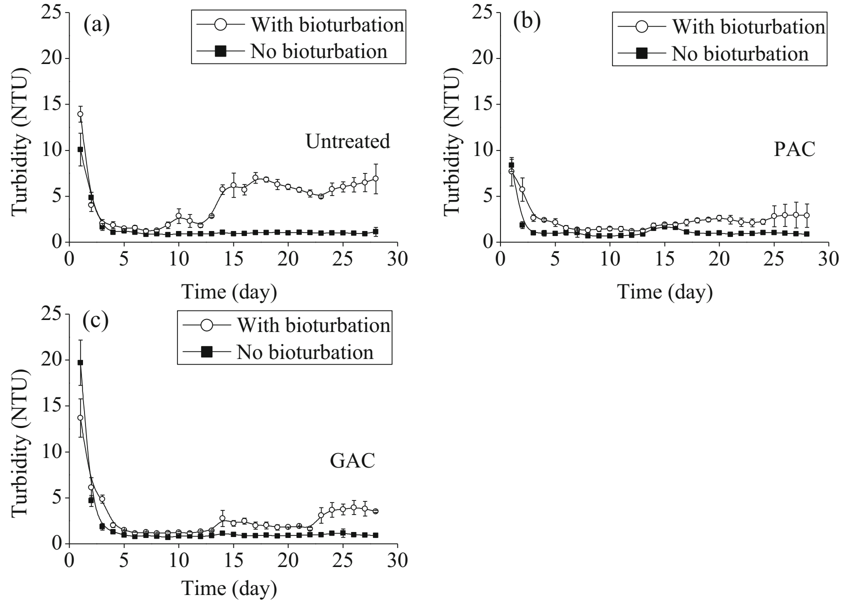

Fig. 2 Comparative study of water column turbidity during the 28 days experiment for the six experimental conditions studied: in untreated sediment in the absence and presence of *Chironomid* larvae (**a**), in PAC sediment in the absence and presence of *Chironomid* larvae (**b**), and in GAC sediment in the absence and presence of *Chironomid* larvae (**c**)

Effects of AC amendments and bioturbation on free heavy metals concentrations in pore water

Treated sediments were equilibrated for 28 days after which free concentrations were determined. In the systems without bioturbators, free concentrations in pore water followed the order Zn > Cu > Pb. In the GAC and PAC-treated sediment, the free heavy metal concentrations were always lower than in untreated sediment. As shown in Fig. 3, sediment treatments with PAC and GAC in the absence of *Chironomid* larvae resulted in reductions of free Cu concentrations of 78 and 66 % in the 0 to 1 cm just below the sediment–water interface, respectively. For Pb and Zn, these concentration reductions were 40 and 38, 19 and 25 %, respectively.

In systems with bioturbators, free heavy metal concentrations just below the sediment–water interface were much lower than the treatment without bioturbators. However, the presence of *Chironomid* larvae generally increased the free heavy metals concentrations in pore water, especially in the deeper layers. For example, the DGT concentration of Cu in the 5 to 6 cm below the sediment–water interface were 73.27 and 80.71 μg/L for [No bioturbation + untreated] and [With bioturbation + untreated] conditions, respectively, 20.61 and 15.17 μg/L for [No bioturbation + PAC] and [With bioturbation + PAC] conditions, respectively, and 32.06 and 37.08 μg/L for [No bioturbation + GAC] and [With bioturbation + GAC] conditions, respectively.

Effects of AC amendments and bioturbation on heavy metals sediment–water partitioning

In the absence of *Chironomid* larvae, PAC addition, GAC addition treatments resulted in reductions of dissolved Cu concentrations of 93 and 68 %, respectively, as compared to untreated sediment after 28 days (Fig. 4). For Pb, these concentration reductions were 37 and 17 %, respectively. No distinct differences were noted among the three different treatments in reducing the aqueous Zn concentration after 28 days. However, PAC addition, GAC addition treatments resulted in reductions of dissolved Zn concentrations of 35 and 47 %, respectively, as compared to untreated sediment after 19 days. In addition, PAC reduces the aqueous Cu concentrations more efficiently than GAC. These results were similar to that of free heavy metals in pore water.

The bioturbation process significantly affected such a sediment–water system. In the first few days, the dissolved heavy metal concentrations in bioturbation groups were significantly higher than those in the no bioturbation groups. After 7 days, the dissolved Cu concentration were 41.01 and 50.85 μg/L for [No bioturbation + untreated] and [With bioturbation + untreated] conditions, respectively, 2.84 and 5.88 μg/L for [No bioturbation + PAC] and [With bioturbation + PAC] conditions, respectively, and 13.94 and 20.40 μg/L for [No bioturbation + GAC] and [With bioturbation + GAC] conditions, respectively. After 28 days, the presence of *Chironomid* larvae in untreated, PAC, and GAC sediment generally decreased the aqueous concentrations, especially for Zn and Pb. In addition, in the presence of bioturbators, there is no significant difference among the dissolved concentrations for Zn and Pb in untreated, PAC, and GAC sediment after 28 days.

Effects of AC on bioaccumulation of heavy metals

In Fig. 5, the bioaccumulation of heavy metals in *Chironomid* larvae after a 28-day exposure to the three treatments are plotted. In comparison with untreated sediment, the coexistence of AC enhanced the accumulation of heavy metals, especially for PAC. The heavy metal concentrations in PAC addition sediments were

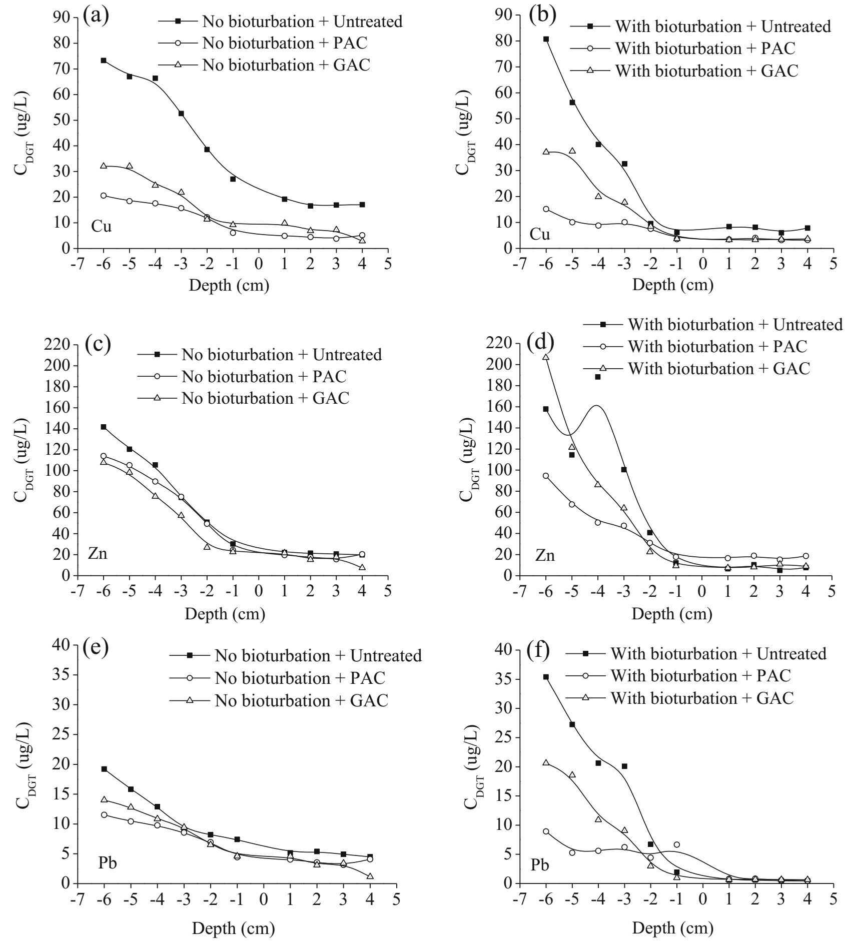

Fig. 3 Vertical distribution of Cu in the absence of *Chironomid* larvae (**a**), Cu in the presence of *Chironomid* larvae (**b**), Zn in the absence of *Chironomid* larvae (**c**), Zn in the presence of *Chironomid* larvae (**d**), Pb in the absence of *Chironomid* larvae (**e**), and Pb in the presence of *Chironomid* larvae (**f**) in sediment pore water

increased by 1.5–3.7-fold upon the untreated sediment exposure for 28 days. The 28-day test duration of the bioassays revealed that survival rates in untreated sediment was higher than in all AC-treated sediments (untreated sediment (68 %) > GAC sediment (54 %) > PAC sediment (49 %)).

Discussion

Effects of AC amendments and bioturbation on pH and turbidity

Oxygen can penetrate deep into the sediment as a result of bioturbation. With *Chironomid* larvae, the lower pH values in the first 20 days and the significantly decreased pH found in the water column after 28 days can be mainly due to the oxygenation of the sediment and the increased mineralization process of organic matter by bacteria, producing quantities of carbon dioxide, which can bring down pH values (Ciutat and Boudou 2003). However, the pH in the water column increased to a relatively high value 8.43 ± 0.15 for the treatments [With bioturbation + untreated], which can be explained by the photosynthesis processes of microbial and phytoplankton increased by the bioturbation, tends to increase the pH levels.

The turbidity in the overlying water in the presence of *Chironomid* larvae is higher than that in the absence of *Chironomid* larvae. In addition, the lack of turbidity enhancements in the PAC and GAC treated sediments in the presence of *Chironomid* larvae was observed in this study. After 9 days, the *Chironomid* larvae began to move to the sediment surface to pupate, the turbidity showed the AC addition had strong inhibitory effects on the eclosion for the bioturbator. Mortality of *Chironomid* larvae may be an important reason for the reduction of turbidity. However, (Kupryianchyk et al. 2013) reported that the bioturbation activity of *Lumbriculus variegatus* was reduced in AC amended sediments, and the survival rate was 100 % for this species, which showed that the reduction was not caused by mortality. Therefore, adverse effects of AC amendments on organisms' behavior were another reason for the reduction of turbidity.

Effects of AC amendments and bioturbation on heavy metals fluxes

In the systems without bioturbators, free concentrations in pore water followed the order Zn > Cu > Pb, which is explained by the higher sorption of Pb to sediment. Generally, treatment effectiveness for Cu was higher than for Pb and Zn,

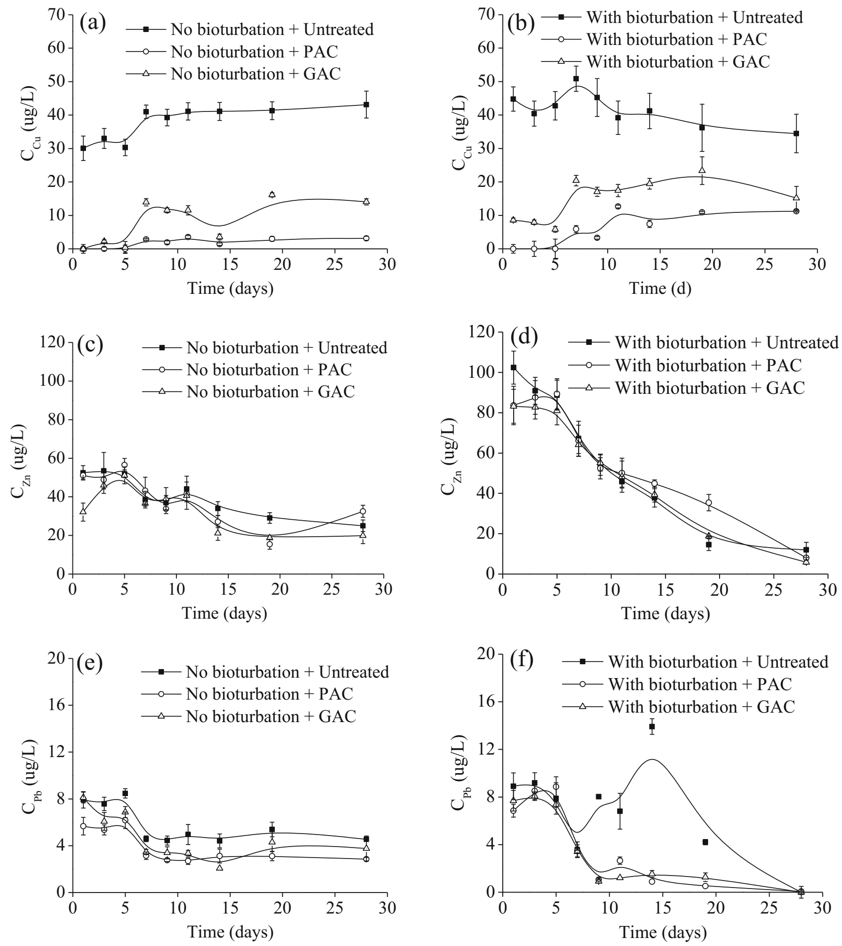

Fig. 4 Evolution of dissolved Cu concentrations in the absence of *Chironomid larvae* (**a**), dissolved Cu concentrations in the presence of *Chironomid* larvae (**b**), dissolved Zn concentrations in the absence of *Chironomid* larvae (**c**), dissolved Zn concentrations in the presence of *Chironomid* larvae (**d**), dissolved Pb concentrations in the absence of *Chironomid* larvae (**e**), and dissolved Pb concentrations in the presence of *Chironomid* larvae (**f**) in the overlying water during the 28 days experiment for the six experimental conditions studied

which can be attributed by the higher affinity of Cu to AC. PAC is more efficient than GAC in reducing the free pore water Cu and Zn concentrations at equal dosage due to the larger external surface area. The results for overlying water heavy metal concentrations were similar to that of free heavy metals in pore water, which showed that decreasing particle

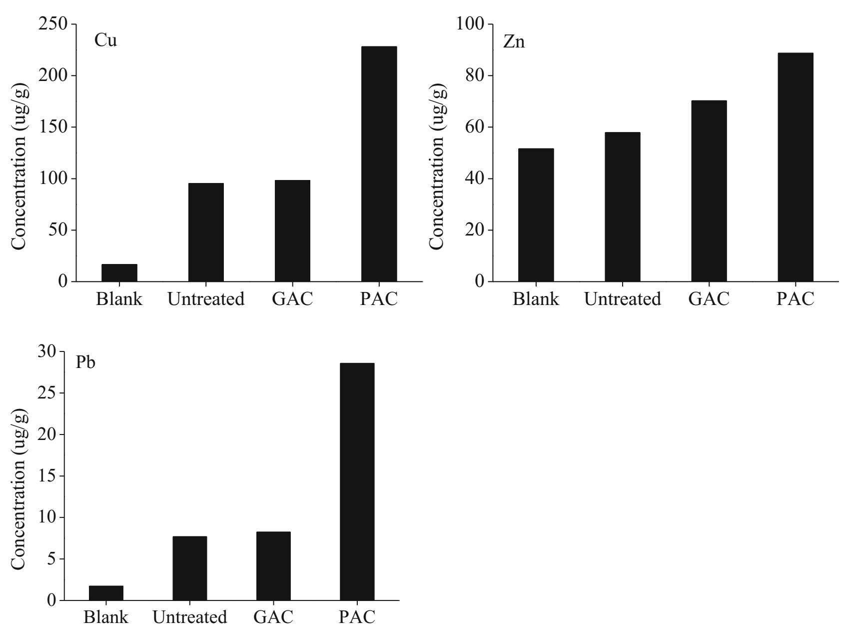

Fig. 5 Cu, Zn, and Pb average concentrations at 28 days in the whole body for the six experimental conditions studied

size increases its effectiveness in reduction of aqueous concentrations of Cu. AC addition treatments resulted in reductions of dissolved Cu and Pb concentrations as compared to untreated sediment. It can be attributed by the lower pore water concentrations in PAC and GAC addition treatments, causing lower heavy metal concentrations for transport to the water column.

It seems that bioturbation is a very important factor affecting the AC remediation of heavy metals impacted sediment. In systems with bioturbators, free heavy metal concentration in pore water just below the sediment–water interface were much lower than the treatment without bioturbators, probably because the formation process of iron and manganese hydrous oxides enhanced by bioturbation, tends to sorb or coprecipitated heavy metals (Ciutat and Boudou 2003). Therefore, bioturbation in sediment are important factors influencing chemical-diffusion fluxes across the sediment–water interface (Josefsson et al. 2010; Thibodeaux 2005). However, the presence of *Chironomid* larvae generally increased the free heavy metals concentrations in pore water, especially in the deeper layers, indicating that organisms in sediment increase the chemical-diffusion flux through bioturbation.

The dissolved heavy metals in overlying water in bioturbation groups was significantly higher than the no bioturbation group at the first few days, which was probably because the resuspension of sediment resulted in variable desorption rates of metals absorbed to sulfides (Ciutat and Boudou 2003). After 28 days, the presence of *Chironomid* larvae generally decreased the aqueous concentrations, especially for Zn and Pb. It is likely that these decreased amounts can be explained by the resuspension of sediment resulted in the formation of iron and manganese hydrous oxides, tends to sorb or coprecipitated heavy metals (Caetano et al. 2003). The concentration of particulate manganese confirmed this hypothesis. In addition, the impact of bioturbation on the remobilization of elements, from sediment into the water body, depends on the chemistry of the elements and conditions within the sediments (Schaller 2014). It may be another reason that the remobilization of Cu, Zn, and Pb in the presence of *Chironomid* larvae from sediment into overlying water is different.

Effects of AC on bioaccumulation of heavy metals

It seemed that the DGT technique could not provide a useful measure of the bioavailability of the heavy metals for *Chironomid* larvae in this study. The DGT accumulated heavy metal concentration in pore water were following the order: PAC sediment < GAC sediment < untreated sediment, which were totally different from the bioaccumulation concentrations of heavy metals in *Chironomid* larvae. The *Chironomid* larvae are linked with both the organic debris via digestive system, and overlying water or pore water via respiratory surfaces and skin (Mackay and Fraser 2000). It can directly take up, metabolize, and eliminate the soluble heavy metals in overlying water and porewater, whereas for the AC and sediment-associated heavy metals, it would ingest and eliminate the ACs and sediment. Therefore, the coexistence of ACs might enhance the accumulation of heavy metals in comparison with untreated sediment. We may get the conclusion that the presence of ACs could increase the bioaccumulation of heavy metal in *Chironomid* larvae due to the retention of AC-associated heavy metals in the digestive tract. This increased bioaccumulation may decrease the survival rates of *Chironomid* larvae.

Janssen and Beckingham (2013) found that for a given dose of AC, the bioaccumulation of contaminants also decreases with decreasing AC particle size which was different from what this study got. At a constant AC dose, smaller particle size offering more surface area and shorter diffusional distances can increase faster sorption kinetics (Lehmann et al. 2011). The effect of particle size can also be found in decreasing bioaccumulation of HOCs for smaller particle size AC treatment. For example, the reduction in total PCB bioaccumulation was 70 % for 75–300 μm size carbon, and 92 % for the 45–180 μm size carbon for *L. variegatus* with 2.6 % GAC amendments (Sun and Ghosh 2007), indicating the bioaccumulation decreased with decreased AC particle sizes. However, negative responses to AC amendments including changes in growth, lipid content, behavior, and survival on some benthic invertebrate species have been observed by (Janssen and Beckingham 2013). In addition, the AC-associated heavy metals may be bioavailable to benthic organisms as shown in this study. Therefore, the AC amendments in sediments for heavy metals remediation may not be suitable. The possible effect of AC on bioaccumulation should be taken into account before the application.

Conclusion

Our results showed that sediment treatment with AC decreased pore water concentrations and release of heavy metals into overlying water, especially for Cu. PAC reduces the aqueous Cu concentrations more efficiently than GAC. The presence of *Chironomid* larvae decreased the free heavy metals in pore water just below the sediment–water interface. Although GAC and PAC were both efficient in adsorbing heavy metals, the potential for negative effects has been investigated in this study. Negative effects were observed for changes in bioaccumulation. The coexistence of PAC might enhance the accumulation of heavy metals in the digestive track of *Chironomid* larvae. The increased bioaccumulation may decrease the survival of *Chironomid* larvae. Therefore, this negative effects need to be taken into account when assessing the effectiveness of AC treatment and the risk in further study.

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