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SPATIAL AND TEMPORAL BEHAVIOR OF PB, CD, AND ZN RELEASE DURING SHORT TERM LOW INTENSITY RESUSPENSION EVENTS

M. T. Mahamod[a], W. H. M. Wan Mohtar[a*], S. F. M. Yusoff[b]

[a]Department of Civil Engineering, Faculty of Engineering and Built Environment, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia
[b]School of Chemical Sciences and Food Technology, Faculty of Science and Technology, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia

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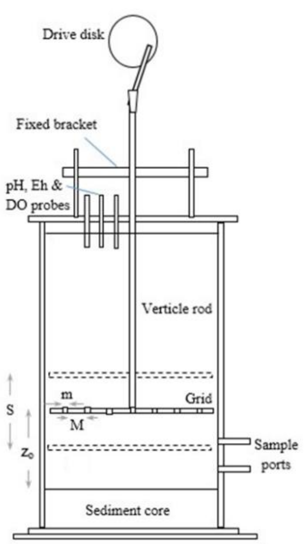
*Corresponding author
hanna@ukm.edu.my

Graphical abstract



Abstract

Sediment as the largest storage and resources of heavy metal plays an important role in the metal remobilisation and release. Continuous particulate matter in suspension increased the level and mobility of metals, of which poses a serious problem particularly to aquatic organisms. The purpose of this research is to investigate the fate and amount of contaminant released through sediment resuspension by using particle entrainment simulator (PES) with low turbulence intensity. The heavy metal release from sediment mixture of sand/silt/clay with percentage of 90/5/5, respectively was studied. In particular, the research paid attention to the contaminant release at the early resuspension stage (i.e. within 3 hr resuspension) by reviewing the metal releases of Zn, Cd and Pb in water column at five elevations from the bed of PES. Results showed that the concentration of remobilised heavy metal during resuspension event for Cd, Zn and Pb was in ranged of 0.034 to 0.139 mg/L, 0.014 to 0.071 mg/L and 0.007 to 0.029 mg/L respectively. The affinities of heavy metal release discussed in this study was Cd > Zn > Pb. Concentrations of the heavy metal fluctuated within the water column and have the lowest values at near bed. The highest concentration observed for Cd and Zn was at one integral length scale from the sediment bed and at *t* = 120 mins. Even at low turbulence intensity, the heavy metal release was still observed and is significant to the water quality.

Keywords: Heavy metal contaminated sediment, resuspension, Pb, Cd and Zn release, spatial and temporal release

Abstrak

Sedimen sebagai penyimpan logam berat terbesar memainkan peranan yang penting di dalam mobilisasi dan pelepasan semula logam. Bahan partikel yang berterusan berada di dalam keadaan ampaian meningkatkan tahap dan pergerakan logam, yang mana boleh menimbulkan masalah yang serius terutama kepada organisma akuatik. Tujuan kajian ini adalah untuk menyiasat jumlah bahan cemar dimobilisasi semula melalui sedimen diampai semula dengan kematan pergolakan rendah di dalam Kebuk Iringan Partikel (PES). Pembebasan semula logam berat daripada campuran sedimen pasir / kelodak / tanah liat dengan peratusan 90/5/5 telah dikaji. Kajian ini memberi perhatian khusus kepada pelepasan bahan cemar di peringkat awal ampaian semula (iaitu dalam tempoh 3 jam) dengan mengkaji semula pelepasan logam Zn, Cd dan Pb di dalam air pada lima aras ketinggian dari dasar PES. Hasil kajian menunjukkan bahawa kepekatan logam berat yang dimobilisasi semula semasa proses ampaian semula untuk Cd, Zn dan Pb adalah masing-masing di antara 0.034 kepada 0.139 mg / L, 0.014 kepada 0.071 mg / L dan 0.007-0.029 mg / L. Kecendurangan untuk pelepasan semula logam berat yang dibincangkan di dalam kajian ini adalah Cd > Zn > Pb. Kepekatan logam berat dilihat turun naik mengikut aras ketinggian dan mempunyai nilai yang terendah berdekatan dasar. Kepekatan tertinggi telah diperolehi bagi logam Cd dan Zn adalah pada ketinggian satu skala panjang integral dari dasar dan pada t = 120 minit. Walaupun pada keamatan pergolakan rendah, pelepasan semula logam berat dari sedimen tercemar masih berlaku dan mampu memberi kesan signifikan kepada kualiti air.

Kata kunci: Sedimen logam berat tercemar, ampaian semula, pelepasan semula Pb, Cd dan Zn, pelepasan semula mengikut spatial dan tempoh masa.

1.0 INTRODUCTION

In waters, heavy metal contaminated sediment has potential to have effects on the aquatic organisms and contribute to the degradation of ecosystem functions. These metals (in dissolved phase) are indestructible and most of them have toxic effects on living organisms, particularly when the minimum concentration of metal is exceeded (Banu *et al.*, 2013). Coastal and estuarine waters for example receive contaminants via local anthropogenic activities and through riverine inputs. Due to rapid industrialization and uncontrolled urbanization around many cities and coastal areas, an alarming level of pollutants has contaminated these aquatic environments. More than 99% of heavy metal entering into river can be stored in river sediments in various forms (Salomons and Stigliani, 1995). These significant quantities can be accumulated within the water column, bound to sediment as well as aquatic food chain and posed disturbance on the local fish population, and possibly to human health (Ankley *et al.*, 1996; Yi *et al*., 2011).

Remobilisation of contaminants from contaminated sediment can occur during natural events, such as tidal movement and storms, or during human activities such as dredging, dredge disposal and fishing (Eggleton and Thomas, 2004). The release and distribution of contaminants are highly regulated by hydrodynamics, biogeochemical processes and environmental conditions (redox, pH, salinity and temperature) of the individual system (Cantwell *et al.*, 2008). However, dredging and dumping operations mobilise large quantities of contaminants to the water column in short periods of time where they become eventually available to the biota.

Upon sediment resuspension, the high kinetic energy from the turbulence is transferred to sediments. The driving (fluid) force for mass transfer is favourable for desorption of contaminant into the water. The extent of desorption determines the availability of heavy metal compounds in the overlying water. Theoretically, higher turbulence imposed on the sediments increased the level of contaminants desorption (Borglin *et al.*, 1996). Once entrained from the sediment bed, fine sediments, particularly are not immediately settle but are kept in suspension. These suspended particulate matters would physically affect the aquatic organisms due to its fine size. Furthermore, they have been found to contain higher concentration of heavy metal load on an mg/g basis than coarse sediments, whereby the release of contaminants would be mostly from the fine particles (Reible *et al.*, 2002).

Although lots of researches has focused on the contribution of dredging activities as the main event in the mobilisation and release of contaminants, Van Den Berg *et al.*, (1987) indicated that dissolved metal concentrations in the water column were not significantly influenced by it. Instead, the increased level and mobility of metals is in the suspended particulate matter, which present a more serious problem. Suspended sediments are kept in suspension by turbulence and thus the effect of turbulence on the remobilisation of contaminant is crucial.

Reible *et al*. (2002) found out that heavy metal release is in multiphasic, which is one set of process controlling the early resuspension and a completely different set of processes in a long-term resuspension. Thus, this research proposed to focus on the short-term (i.e. early stages) sediment resuspension contribution to the mobilisation and release of heavy metal contaminants. We also paid attention to the low turbulence intensity effect on the release of heavy metal.

The outcome of this study is foreseen to provide a preliminary base to quantify the potential impact on the aquatic environment, and assists the decision makers to propose a suitable remediation plan. Fine sand was used to investigate the impact of low turbulence intensity and the spatial heavy metal profile was examined up to five different elevations from the sediment bed. The use of fine sandy sediment is beneficial and reflected to the size distribution found in the upstream contaminated sediment, where mostly coarser sizes and less fine sediment could be observed. Three heavy metals (i.e. Cd, Pb and Zn) were examined, prepared as formulated contaminated sediment to determine their releases in a controlled environment. These metals were chosen as the main focus in this study as they are widely distributed in rivers and coastal area, and posed significant health hazards to human, particularly through food chain (Yap et. al., 2004).

2.0 METHODOLOGY

2.1 Particle Entrainment Simulator (PES) Designs

The PES chamber has an area of 12.9 x 12.9 cm^2, and 25 cm high with a tight-fitting lid. The oscillating grid inside the chamber consists of 5 x 5 mesh made out of 0.50 cm rectangular aluminium bars, with a centre-to-centre spacing of 2.50 cm. The grid stroke length is adjustable within the range of 2 to 6 cm by changing the pin location of a connecting rod between the vertical shaft and eccentric drive.

Flow generated by oscillating grid gives a representative of second order turbulence statistics, often described as root mean square (*r.m.s.*) horizontal velocity u_{rms}. The horizontal velocity decays with the distance from virtual origin (i.e. mid-plane of oscillation stroke) based on the relation

$$u_{rms} = CfS^{3/2}M^{1/2}z_o^{-1}, \quad (1)$$

developed by Hopfinger and Toly(1976). Thus, the variation for turbulence intensity can be made by adjusting the values of oscillation frequency (*f*), stroke length (*S*), mesh spacing of the grid (*M*), and the distance between the mid-point of the grid and the interface (z). Values of coefficient *C* depend on the grid characteristics which are the mesh size and solidity of the grid bars. The coefficient of *C* is commonly adopted as 0.25 as suggested by Hopfinger and Toly (1976). The relation of r.m.s horizontal velocity (u_{rms}) and shear stress (θ) can be represented by

$$\theta = \frac{{u_{rms}}^2}{(s-1)dg}, \quad (2)$$

where u_{rms} = r.m.s horizontal velocity, *s* = relative density, *d* = median particle of bed, and *g* = specific gravity. Incorporating Equation (1) into Equation (2), the shear stress imposed on the sediment bed is expressed as

$$\theta = \frac{C^2 f^2 S^3 M}{{z_o}^2(s-1)dg}. \quad (3)$$

The physical fluid forces acted on the contaminated sediment were simulated by using a Particle Entrainment Simulator (PES) as shown in Figure 1. The symmetrical grid plane was placed at near-bottom of the tank and was vertically oscillated to generate quasi-isotropic homogeneous turbulence. The movement of particle was observed when the turbulence force applied is significantly larger than the critical sediment motion of the particle θ_{cr}. The critical threshold force with respect to the median particle size is estimated as $\theta_{cr} = 0.14\ (D_*)^{-0.64}$ and $D_* = d_{50}\ [(s-1)g/\nu^2]^{1/3}$ developed by Miller *et al.* (1977), where d_{50} = median particle of bed; $s = \rho_s/\rho_w$ = relative density; ρ_s= sediment density; and ρ_w= fluid density.

The experiments were performed at small turbulence intensity of 1.1 dynes cm^{-2} (equivalent to *S* = 6 cm and *f* = 2.58 Hz). Thus, the force impacted upon the sediment layer was $\theta/\ \theta_{cr} \approx 2.84$, ensuring a sediment movement and the initiating of resuspension event. At this intensity, the turbulence was sufficient to keep the particles in suspension throughout the experimental period. The flow is fixed and adhered to $\theta \gg \theta_{cr}$, where the sediments are continuously and visibly being entrained from the bed. We note that the generated turbulence is slightly small compared to the work Tsai and Lick (1986), who used similar apparatus and applied 2 to 5 dynes cm^{-2}.

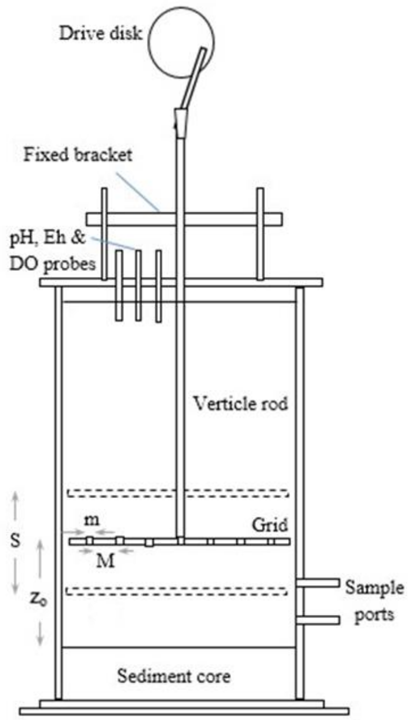


Figure 1 Schematic diagram of PES. There are five sample ports in experiment, however in the schematic diagram, only two sample ports are shown for ecstatic values. Symbol *S* = stroke length, *M* = mesh spacing of the grid, *m* = grid bar size, z_o = height of oscillating grid from sediment surface

2.2 Sediment

The sediment mixture was prepared using sand/silt/clay percent distributions of 90/5/5. Such distribution was chosen to simulate the fluvial sediment bed, where the material is not often to be homogeneously sand, and usually comprises a certain fraction of finer sediment size. A 5 % volume of each silt and clay were used in the mixture, where the median grain size d_{50} of both silt and clay were 63 μm and 8 μm, respectively. Note that the sand used is homogeneous and has particle size of d_{50} = 300 μm. The sediment mixture was formulated and spiked with Pb, Zn and Cd sulphides, respectively to yield a sediment concentration of 200 mg kg^{-1} dry weight of each metal. All the manipulation to the formulated sediment was performed in a glove box purged with nitrogen gas to avoid oxidation of the sediments (Simpson *et al.*, 2004). Equilibrium of spiked sediment with metals sulphide required oxygen free condition whereby all apparatus and the fluid used are deoxygenated prior use. Deoxygenated waters were prepared by bubbling solutions with high purity oxygen-free nitrogen gas up to 8 hours (Simpson *et al.*, 2004). A ratio of sediment: water of 4:1 (*w:w*) were used for the metal-spiked sediment. A small layer of water formed over the sediments due to settling of mixed sediments during the equilibration period. The metal-spiked sediments were prepared with uncontrolled pH (that is allowing the solution to either decline to near pH 6 or incline to pH 7). The metal-spiked sediments were equilibrated in Teflon bottles. Within the equilibrium period, the overlying water in the bottle samples was replaced with deoxygenated water on the days of 3, 13, 15 and 24 prior to use as proposed by Atkinson *et al.*, (2007).

2.3 Sediment Resuspension

The formulated sediment mixture was placed at 1 cm from the bottom of chamber. The grid/lid/frame assembly was removed prior placement and the sediment was carefully put in and the surface was levelled using a bed leveller. The filtered fresh water sample was added slowly through a small tube placed parallel to the chamber wall, in a manner not to resuspend any of the sediment. The height of water column was set at 20 cm from the bottom of chamber. The probe of pH, dissolve oxygen (DO) and temperature was placed at the top of unit where continuous measurement was permitted through two openings. The sediment sample was allowed to settle in the PES chamber for a day prior experimental work.

One set of water sample was collected prior experimental work as the control sample and taken as the initial condition $t = t_0$. Water samples were taken out via sampling ports at height of $\bar{z} = z/z_o$ = 0.05, 0.1, 0.2, 0.3 and 0.4, where z = distance between sediment surface to sampling ports (in cm), and z_o = height of oscillating grid from the sediment surface. Within the 3 hour of experiments, 50 mL water column samples were withdrawn at time *t* = 5, 15, 45, 90, 120, and 180 minutes for every sampling ports, and the values of pH, DO, and temperature were also measured at every 30 minutes of resuspension.

2.3 Analytical Method

All collected samples were isolated by filtration through a 1 µm Nuclepore filter. The water samples were analysed using Inductively Coupled Plasma Mass Spectrometry (ICP-MS), giving focus to the concentration of Cd, Zn and Pb. The value of pH was determined using a Eutech model CyberScan pH 11 meter with pH electrode FC7252201B, and ATC probe with dissolved oxygen was quantified with a Eutech model DO 6+ meter with a probe. The pH meter and probe was calibrated up to five points of pH buffer options USA, NIST and up to two points; 100% in air and/or 0% in known solution for DO meter. The DO probe was calibrated following the manufacturer's instructions.

3.0 RESULTS AND DISCUSSION

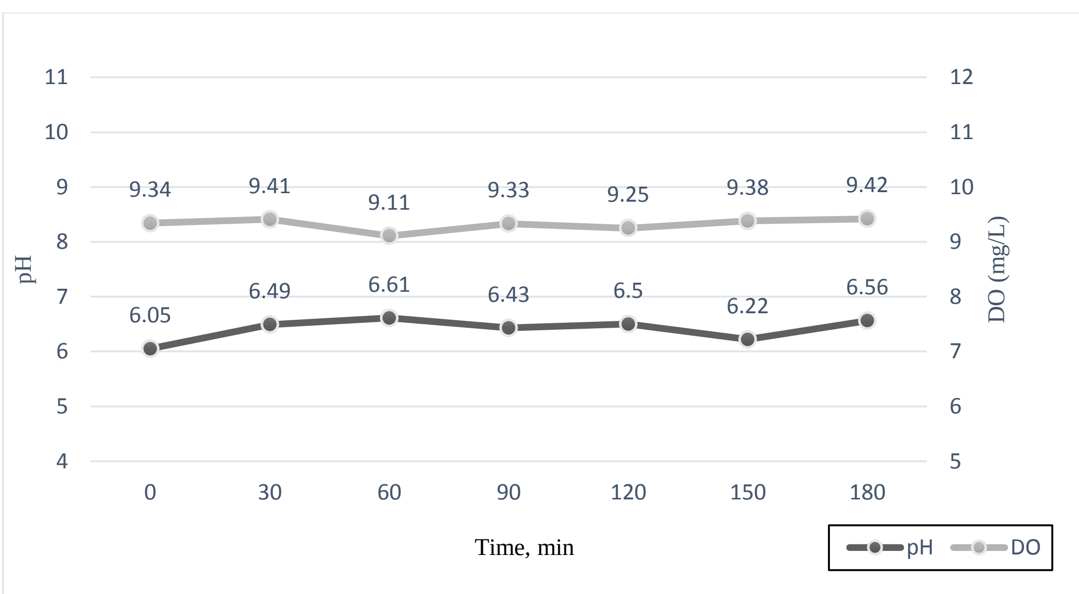
The small θ applied in the experiment resulted in suspension, particularly from fine particles of silt and clay within the sediment mixture. It was observed that a suspension layer was visible due to the water column turned cloudy. Coarser sand particles, in particular the top layer, were observed to consistently in motion and resuspend above the sediment bed.

3.1 Properties of Formulated Sediment and the Water Column

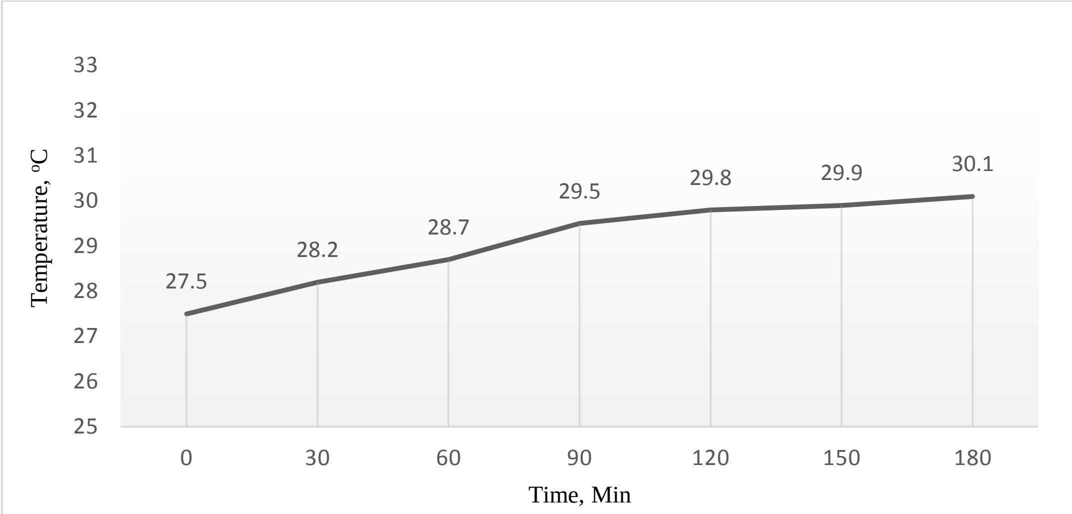
As have been discussed before, three physical variables were monitored throughout the short term resuspension which are the DO, pH and temperature. During experiment, values of pH ranged from 6.05 to 6.61 for the 3 hour resuspension as shown in Figure 2a. pH was expected to be declined corresponding to the free hydrogen ions present in water columns (Simpson *et al.*, 2000; Breu *et al.*, 2008). Thus the decreasing of pH values observed can be attributed to the oxidation of metal sulphide and heavy metal releases are taking place (Cappuyns *et al.*, 2004; Simpson *et al.*, 2004; Eggleston and Thomas, 2004). However, in these experiments, the pH variation was not evident. The pH does not necessarily have to be decreased and may found event of increment when the fresh water and sediment buffering capacity is sufficient to counter any pH declination (Cantwell *et al.*, 2008).

The recorded DO values were observed between 9.11 to 9.42 mg/L. Obviously, within the short term resuspension, the variation of DO is not significant. The DO is influenced by the changes of total suspended solid (TSS), redox state and the grain size of the sediment, where TSS is low in sandy sediment mixture compared to finer particles (Cantwell and Burgess, 2004).

With two parameters of pH and DO showed insignificant behaviour, the temperature, however, showed a consistent increment from 27.5 to 30.1 °C (Figure 2b). Temperature was found to increase when metal activity in the water column as well as the oxidation of metals sulphide became more active and has faster oxidation rates.



(a)



(b)

Figure 2 Variations of a) pH and DO, and b) temperature changed within the 3 hour sediment resuspension event

3.2 Particulate Metals

Figure 3 shows the concentration of dissolved Zn, Pb and Cd during the 3 hour resuspension taken at five elevations, $\bar{z}$ = 0.05, 0.1, 0.2, 0.3 and 0.4. In general, the heavy metals have been remobilised and released back to the overlying water, even at low turbulence intensity. Out of the three heavy metals discussed here, Cd showed the highest release and therefore will be first discussed.

For all elevations, the released Cd has the concentration ranges from 0.034 to 0.139 mg/L. The highest concentration of dissolved Cd was recorded at $\bar{z}$ = 0.1, whereas the lowest Cd concentration was found at $\bar{z}$ = 0.05. Generally, there has been a sharp increase for the first 5 minute of resuspension, continued by a steadily decline in concentration within 40 minutes for the release of Cd. Then, the Cd concentration experienced a second rises up at *t* = 120 min, before marked a level off to the end of experiment. The most consistent release with high concentration of Cd was obtained at $\bar{z}$ = 0.1. Elevation $\bar{z}$ = 0.3 gives the most linear readings Cd concentration with consistent range between 0.071 to 0.089 mg/L. Throughout the experiment, the lowest elevation i.e. $\bar{z}$ = 0.05 was consistently has the lowest concentration.

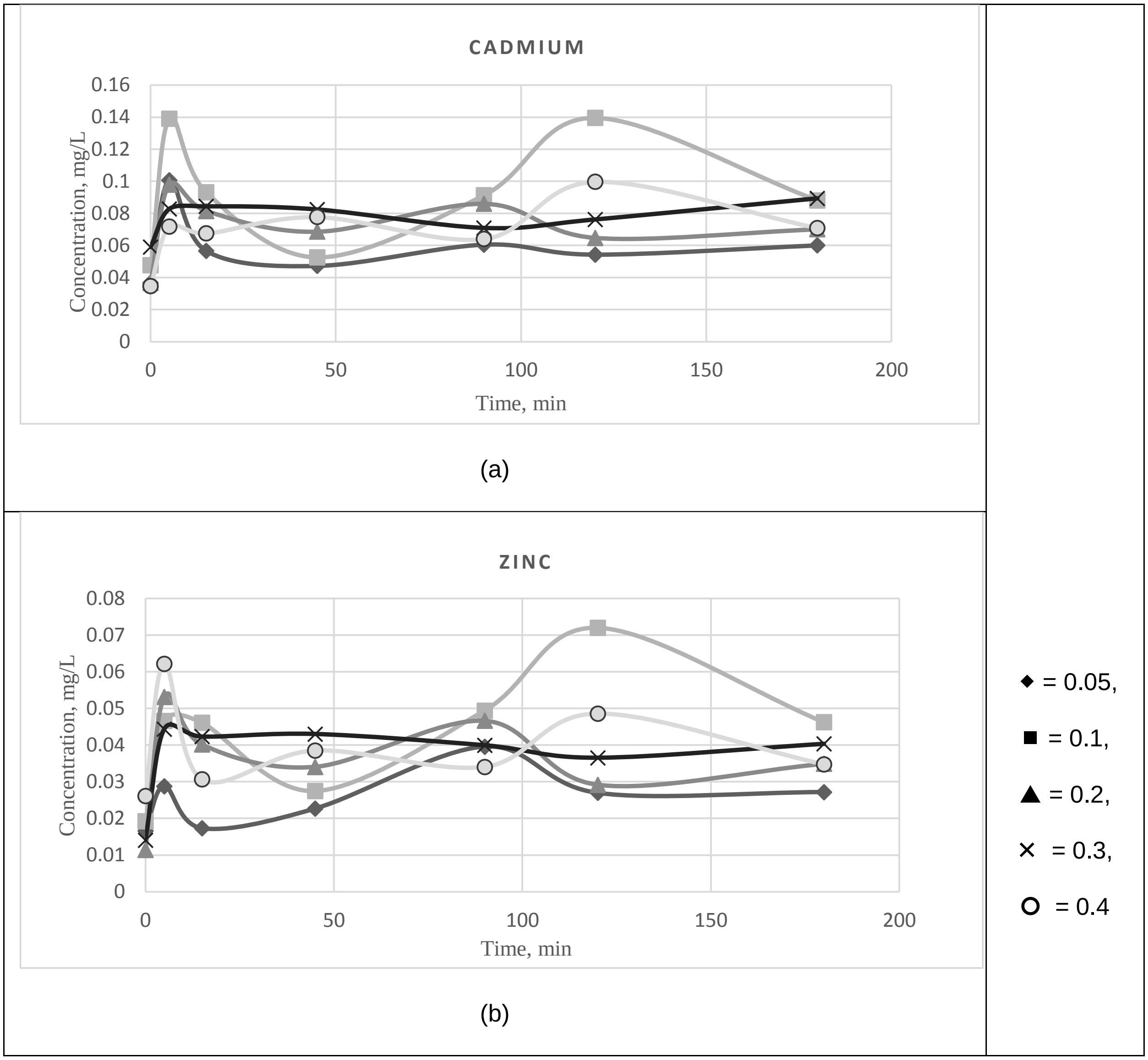
Next, we will discuss on the Zn release where data shows the concentration ranged between 0.014 to 0.071 mg/L. At $\bar{z}$ = 0.05, lower set of Zn readings compared to others was observed and only a bit higher value at *t* = 90 min. The pattern of readings at elevation $\bar{z}$ = 0.05 and 0.2 showed the similar trend where both elevations experienced a decrease up to the first 45 minute of resuspension, but had immediate increase and peaked at *t* = 90 min. The peak of Zn release was only temporary as the concentration was continuously dropped after *t* = 90 min. The maximum Zn release was detected at *t* = 120 minutes, where the peak concentration at $\bar{z}$ = 0.1 was too observed at the same time. Interestingly, the linear trend of Zn release was observed at $\bar{z}$ = 0.3, as shown by the Cd profile, except at lower concentration ranged between 0.014 to 0.044 mg/L.

The concentration of Pb remobilisation was found to be within the range of 0.007 to 0.029 mg/L. The smaller Pb readings was recorded at $\bar{z}$ = 0.05, and the highest set of readings was measured at $\bar{z}$ = 0.1. The readings of Pb at $\bar{z}$ = 0.3 showed the most linear concentration recorded throughout the resuspension event.

Within the same amount of initial heavy metals concentration, Cd shows the highest concentration release. It shows that Cd was the most mobile element compared to Zn and Pb which was also found by Cantwell *et al.* (2008). However, in natural surroundings, Cd is one of the lowest element recorded in the contaminated sediment layer (Nguyen *et al.*, 2005). Resuspension event due to naturally occurred turbulence provided limited Cd release compared to some other element such as Zn, Mn and Fe with the same surrounding parameter (Ghrefat and Yusuf, 2006). The impact of Cd toxicity in water would more significant when the sediment contain higher Cd concentration since the element is easier to release or remobilise.

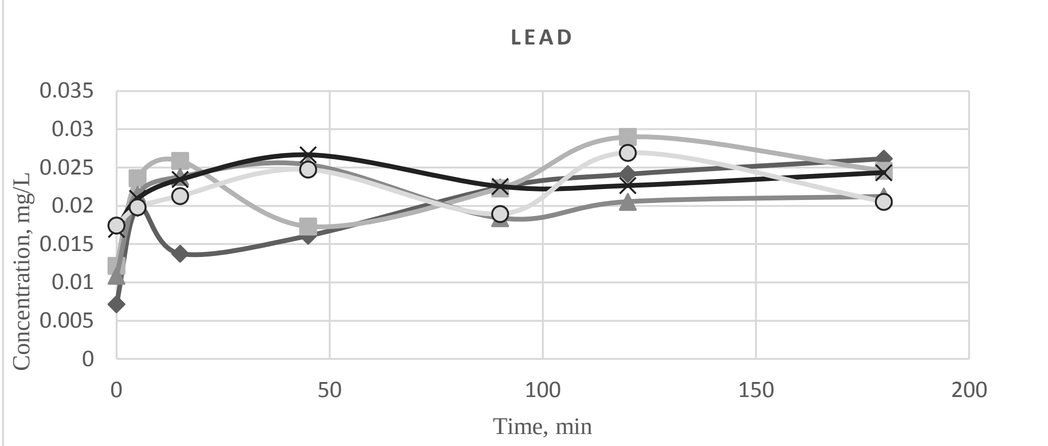
The release of Zn element was recorded at slightly lower values than Cd. Besides, the Cd and Zn release profiles showed relatively similar pattern across the experiment, indicated that Zn has the same responses (particularly related to metal-sediment bound and the oxidation of metal sulphide) as Cd, and when the similar disturbance strength was applied within the same surrounding parameters. The metal Zn also was expected to have more release due to its flexibility and independent to any acidic phase (probably as lower as pH 2) (Cappuyns *et al.*, 2004). The low release of Zn in this study, however, was believed contributed by the increasing pH observed.

It is rather evident that the element Pb showed the lowest affinity of release compared to Cd and Zn. We suspect that such condition was obtained due to low turbulence intensity, where a higher Pb release than a few others element such as Ni, Cu and Cr was observed in natural fresh water condition (Huang *et al.*, 2012). Thus, the mobility of Pb is anticipated to be greater should higher turbulence forces were enforced.



(a)

(b)



(c)

Figure 3 The concentration of (a) Zn, (b) Pb, and (c) Cd, with respected to values of Ƶ. The variation of Ƶ values are represented as ◆ = 0.05, ■ = 0.1, ▲ = 0.2, ✕ = 0.3, O = 0.4, respectively

The effect of spatial within the water column obviously is rather eminent for the remobilisation of Cd and Zn. Lowest concentration was consistently observed at the lowest elevation of Ƶ = 0.05, whereas the highest concentration can be found at Ƶ = 0.1. The remobilised heavy metal from sediment and available in the water column following the characteristics of the largest eddy. The largest size of energy containing eddies is 1 cm (that is when l/z = 0.1), which corresponds to the elevation where the highest concentration is observed (for both Cd and Zn). Note that the value 0.1 is obtained from calculating the integral length scale $l = 0.1z$ (as described by Hopfinger & Toly (1976)).

Higher elevations Ƶ > 0.1 have considerably lower concentration values and were consistent throughout the 180 minutes. Even so, there were some fluctuations from the beginning of disturbance except at Ƶ = 0.3 where steadily linear values were observed for the experimental duration.

4.0 CONCLUSION

The presence of fine-grained sediment in sediment mixture is crucial in the release of heavy metal pollutants due to resuspension events. Here, the experiments were conducted using cohesive-like sediment by adding more percentages in fine-grained sediment (i.e. silt and clay) in the sediment mixture with a distribution of sand/silt/clay is 90/5/5. This study also focused on the release behaviour of three heavy metals i.e. Pb, Zn and Cd at low turbulence intensity. Even though at small disturbance forces, the release of studied heavy metal was detected. The general order of affinities of metals release was Cd > Zn > Pb.

The concentration of Pb, Zn and Cd were fluctuating within the short term of resuspension and the highest concentration observed was 0.029, 0.071, 0.139 mg/L respectively. Note that however, the peak concentration was occurred simultaneously, at Ƶ = 0.1 within *t* = 120 min for each Pb, Cd and Zn elements.

The effect of spatial distance from the sediment bed was also evident, where the most consistent high readings was obtained at Ƶ = 0.1 and the lowest is at Ƶ = 0.05. The results also showed the concentration varied with distance from the bed. Although changes with time, the closest distance from the bed i.e. at Ƶ = 0.05 did not necessary exhibited higher concentration. Besides, the random concentration readings at different elevations shows that the evident of circulation flow occurred within the chamber due to oscillating grid forces.

This study showed that Pb is less mobile compared to Cd and Zn corresponded to Pb release recorded relatively small concentration and independent after 15 minute of resuspension. Moreover, Cd and Zn shows the same pattern of releases over time by having the peak reached at *t* = 120 min.

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