

Research Article

Effect of pH, Temperature, Dissolved Oxygen, and Flow Rate of Overlying Water on Heavy Metals Release from Storm Sewer Sediments

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The concentrations and the amount of cumulative release of heavy metals (Zn, Cu, Cd, Cr, and Pb) in contaminated sediments collected from combined sewer system were investigated in order to provide a complete overview of the key variables affecting the heavy metals release from storm sewer sediments. The heavy metals release rates were affected to a much greater extent in the low pH (4–7) condition than in high pH (8–10) condition. At higher temperature (30–35°C) the release rates of metals were increased more rapidly than at low temperature. The release of Zn, Cu, Cr, and Pb appeared to increase under the aerobic condition during the first 150 min and then the concentration kept stable. Moreover, the adsorption of these metals and the release of Cd occurred in the anaerobic condition. The flow rate significantly affected the release amount of Zn, Pb, and Cr, while it slightly affected the concentration of Cu and Cd. This study reflects that any change of pH, temperature, dissolved oxygen, and flow rate of overlying water all cause the different variations of the concentrations of heavy metals.

1. Introduction

Combined sewer overflows (CSOs) are major sources of intermittent pollution affecting the receiving waters in the natural water areas as well as in many other urban areas serviced by combined sewers [1]. Nowadays, the importance of the CSOs pollution and their acute impacts on receiving water have been largely demonstrated and many authors have underlined their significant role as a source of pollution in CSOs discharges [2–5].

When overflow events occur, large volumes of water, charged with high concentrations of sediments, suspended solids, and dissolved and particulate-associated contaminants, such as metals, pesticides, polycyclic aromatic hydrocarbons, and nutrients, are released from the sewer system into the receiving water [6, 7]. However, unlike organic pollutants, natural processes of decomposition do not remove heavy metals. As a consequence, heavy metals originating from anthropogenic activities are frequently detected in the sewer sediments of combined sewers [8, 9] and water

columns of river/Lake [10], which cause the severe contamination of a considerable number of the world's water systems. Sediments often act as both carriers and potential sources for metals in the aquatic environment [11]. The physicochemical changes alter the bioavailability of metals in the pore waters [12] and the release rate (flux) of metals from the sediments [13]. The fate (speciation) of the metal released from sediments is affected by the overlying water condition, in particular the pH, salinity, dissolved oxygen concentration, and amount of suspended solids [14]. Moreover, it had been found that the physical disturbance of sediments released metals more rapidly than biological disturbance [15].

The objective of this study is to evaluate the influence of the pH, temperature, dissolved oxygen, and flow rate of overlying water on the release of heavy metals from storm sewer sediments. More precisely, metals including Pb, Zn, Cu, Cr, and Cd were investigated and the results were discussed in terms of how the factors may modify the release rate, the cumulative release amount, and the concentration of heavy metals.

TABLE 1: Concentration of heavy metals ($\text{mg}\cdot\text{Kg}^{-1}$) in test sediments.

Metal	Water-soluble metals	Acetic acid-extractable metals	Oxidizable metals	Reducible metals	Residual metals	Total metals
Pb	0.191	1.69	63.0	1.62	23.5	131
Cu	0.220	5.72	126	1.30	16.6	166
Zn	2.07	9.51	18.4	21.6	149	210
Cr	0.582	4.73	30.9	8.81	116	184
Cd	0.0139	0.564	0.603	0.120	0.246	2.18

2. Materials and Methods

2.1. Study Site and Sampling. Metal contaminated sediments were collected from an old neighborhood's sewer system of North Lishi Road, located in Xicheng District of Beijing, China. This area is poor that was surrounded by traders' stalls. The sediments were sampled using a shovel on August 3rd, 2011. The date was chosen because it had been kept dry for 5 days.

Sampling site (128 mm depth, 30–100 mm wide) was in the sewer (30 cm diameter), which was 0.3–0.5 m close to the sewer inspection pit. Once dredged, samples were immediately collected into the preparation bottles and then taken to the laboratory after the removal of large debris on-site. The water samples taken from the runoff were also collected by using the plastic containers. Then all sediments were kept in the plastic bags and stored at 4°C [15].

2.2. Experimental Procedures. All samples were transferred to the beakers (1000 mL) and soaked (6 cm depth) with the filtered runoff sewer water. To avoid photosynthesis, all experimental devices were wrapped with black plastic bags. Samples were run duplicates for each procedure. The concentrations of each heavy metal speciations and the main characteristics of the sediments are summarized in Table 1. The analyses were carried out by using the modified BCR procedure [16] and the microwave digestion-ICP-MS (Perkin Elmer Elan 6100, Perkin Elmer Inc., USA) was used to detect the metals of all the experimental samples. The contents of Pb, Cu, Zn, Cr, and Cd in all the water samples (acidified with 1% HNO_3 solution) released from the sewer sediments were determined by the atomic absorption spectrophotometer (Varian-AA240). To minimize the source of error, reagent blanks and duplicate samples were used in the analytical programs to assess the precision and bias. All the extractions and analysis were made with replicate samples ($n = 3$) for quality control assessment and the average results were reported. The reproducibility expressed as relative standard deviation (RSD) was found to be satisfactory and ranged between 0.83% and 2.92% and the recovery percentages of metal concentrations from the reference materials were as follows: Zn(103%), Cu(98%), Pb(96%), Cd(101%), and Cr(97%). Precision can be considered satisfactory for environmental analysis, as it was lower than 5% of the relative percentage difference (RPD).

The effect of overlying water pH on metal release from sediments was investigated at high (pH 10), mid (pH 7 ± 1), and low (pH 4) pH values (mean \pm standard deviation (SD)).

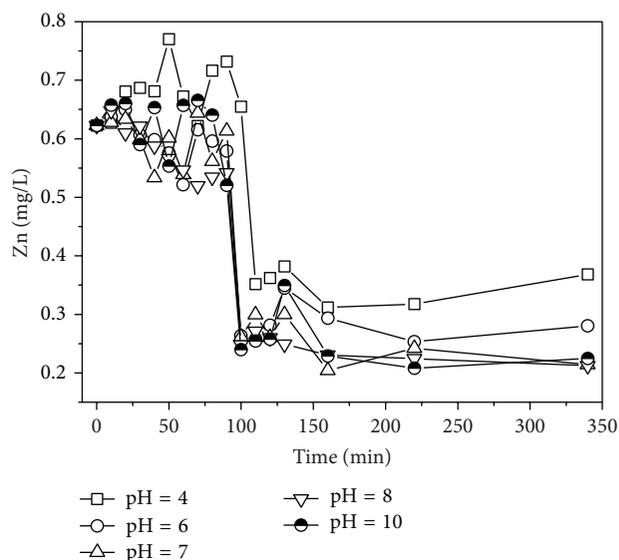


FIGURE 1: The concentration of Zn in the overlying water changed with times.

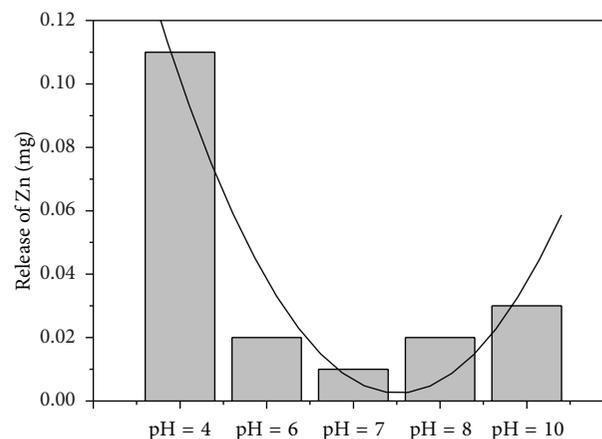


FIGURE 2: The relationship between the maximum amount of accumulated Zn release from sediment in experimental period and pH.

To investigate the effect of DO of overlying water, the DO was kept at high (9 mg DO/L), mid (7 ± 1 mg DO/l), and low (3 ± 2 mg DO/l) concentrations. The effect of flow rate of overlying water was investigated at 15, 20, 25, 30, and 35°C and to evaluate the effect of flow rate all the samples were investigated.

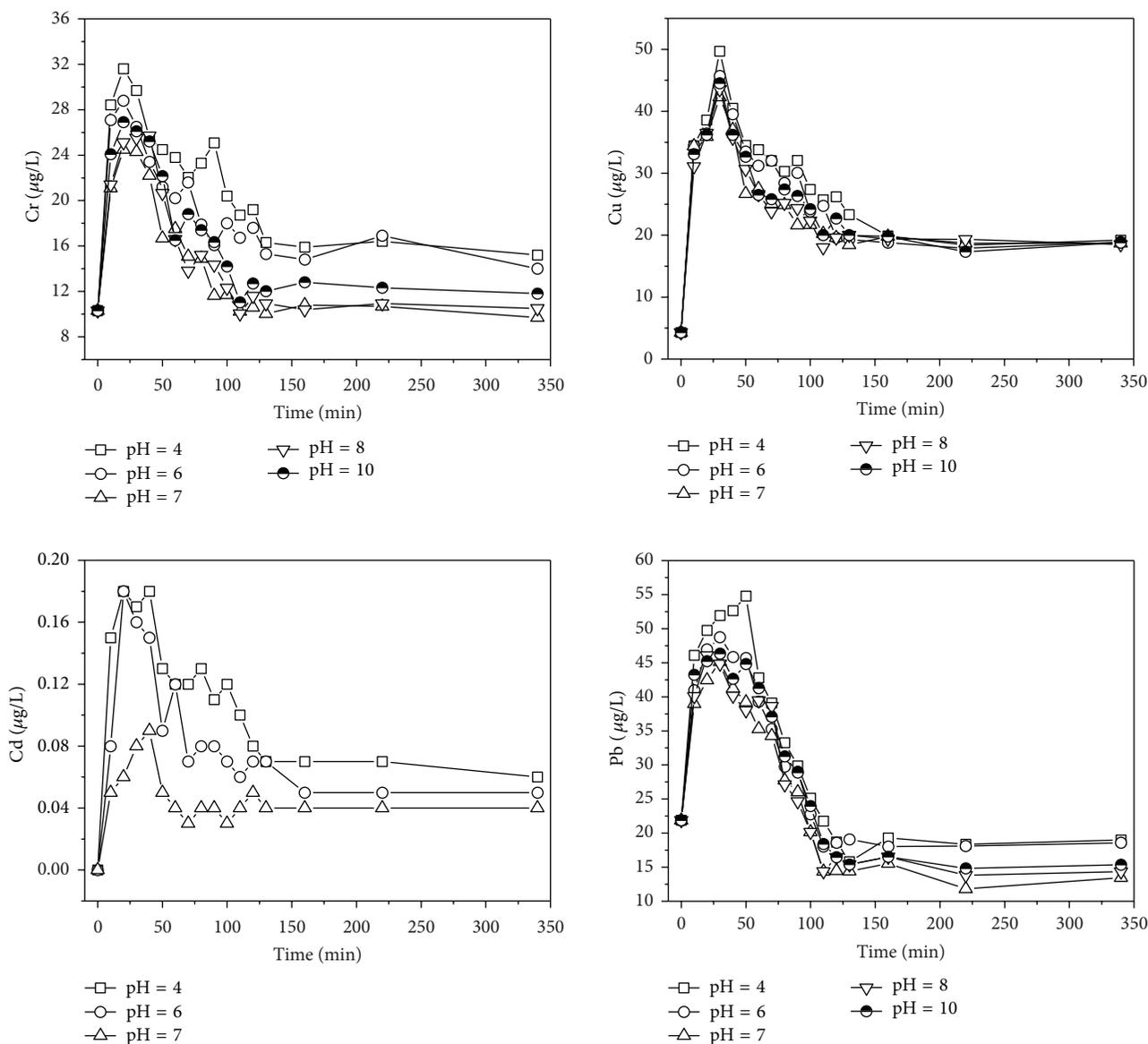


FIGURE 3: The concentration of Cr, Cu, Cd, and Pb in the overlying water changed with times.

3. Results and Discussion

3.1. Effect of pH on Metal Release from Sediments. Different studies based on combined sewer sediments have shown that heavy metals with different speciations are generally associated with sewer water [17, 18]. More specifically, Zn was frequently observed in natural water and sediments [8]. Normally, with pH decreasing in sediment, the competition between H^+ and the dissolved metals for ligands (e.g., OH^- , CO_3^{2-} , SO_4^{2-} , Cl^- , S^{2-} , and phosphates) becomes more and more significant. The adsorption abilities and bioavailabilities of the metals subsequently decrease and then increase the mobility of heavy metal [18]. Moreover, H^+ (or H_3O^+) occupies more adsorption sites at lower pH values [19], which results in soluble and carbonate-bound heavy metals precipitated more easily than at higher pH values. Both of

these processes result in faster heavy metal release rate with lower pH, and it can be found with the observed results (Figures 1 and 3).

At low-pH (pH = 4), the release rate of Zn in solution was much larger than at high-pH or mid-pH. As a matter of fact, the same discrepancy could be observed for the Zn concentrations in the overlying waters at pH 6, 7, and 8. These observations confirmed that the release of Zn to the overlying waters makes no significant difference under any pH conditions. The release of Zn remained low at pH 4 during the first 110 min, but the Zn concentration increased later. In contrast, under pH 6, 7, and 8 conditions, the release of Zn in the overlying waters was observed to be the easiest. Moreover, the lowest Zn concentration was found to appear at 100 min and then remained stable.

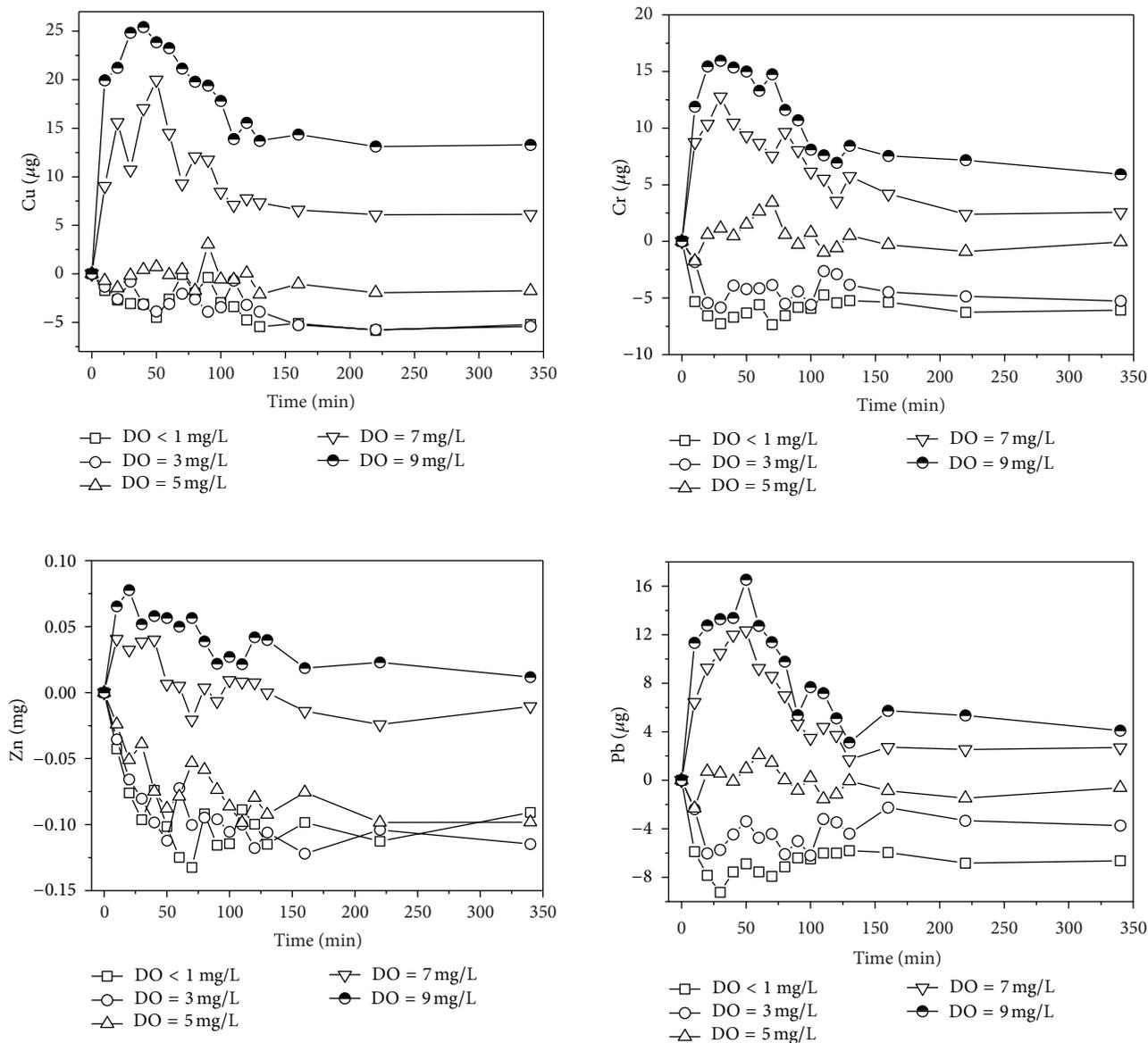


FIGURE 4: The amount of cumulative release of Cu, Cr, Zn, and Pb from sediments changed with times.

At all pH conditions, the Zn concentrations in the overlying waters changed significantly during 100–110 min and then remained stable, which indicated a good correlation between the maximum amount of accumulated Zn release from sediments and the pH (Figure 2). As a consequence, a parabolic equation (1) was deduced to estimate the effect between the maximum amount of accumulated Zn release and pH

$$y = 0.0149x^2 - 0.105x + 0.187. \quad (1)$$

Coefficient of determination: $R^2 = 0.903$.

Compared to the experiments of Zn, Figure 3 indicates that the releases of Cu, Pb, and Cr in the overlying waters were similar. The heavy metal concentrations changed more severely at pH 10 than pH 6. Moreover, little or no release of Cu, Pb, Cr was observed at pH 8 and the concentrations

changed as the same trend as pH 7. However, during the whole experiments, the higher release rate of Cd was observed in the low-pH than in the high-pH. The concentration reflected a higher release rate of metals in the sediment-water system at lower pH than higher pH. During the first 10 minutes all the heavy metal concentrations increased rapidly, which might result from the sediment disturbance with adding the original water. Then the release rate of the metal was gradually slow, which may be mainly due to pH, the main affecting factor of the metal release from the sediment during this period. Moreover, the decreases might result from the release of iron (and manganese) from anoxic sediments that becomes oxidized, precipitates, and carries metals with it.

3.2. Effect of Dissolved Oxygen on Metal Release from Sediments. There are many combined forms of heavy metals

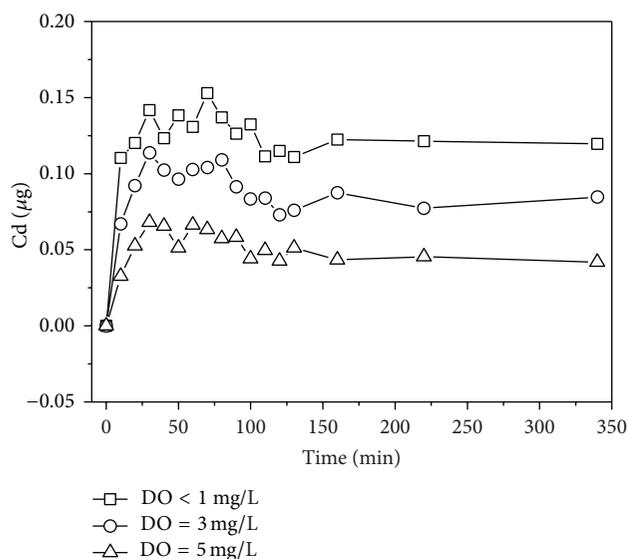


FIGURE 5: The amount of cumulative release of Cd from sediments changed with times.

in sediments, including organic matter bound, which could play a regulatory role in the process of heavy metal release from the sediment into the overlying water. While the bound metals release into the water when the decomposition of organic compound occurred [20]. Moreover, the dissolved oxygen content can affect the rate of oxidation of organic compound and then improve the release of the metal. Our results of DO effect on metal release from sediments are illustrated in Figure 4. Little or no release of Zn, Cu, Pb, and Cr was observed when $DO < 7 \text{ mg/L}$, but the amount of cumulative releases of Zn, Cu, Pb, and Cr were high when $DO = 7$ and 9 mg/L . As a consequence, adsorption frequently was observed in $DO = 3 \text{ mg/L}$ and $< 1 \text{ mg/L}$. Moreover, the adsorption of different metals was greater at lower DO concentration than at higher DO concentration, which was concluded by the variation of Zn and the larger release of Zn, Cu, Pb, and Cr with higher DO.

The results of all the heavy metal releases indicated that the higher DO in the overlying water facilitated more the release of the Zn, Cu, Pb, and Cr than the lower one. It could possibly be explained by the following reasons: (i) the samples were collected in the old neighborhood area and the organic matter contents of them were high; (ii) under the aerobic condition, the organic compound oxidation rate is higher than that under the anaerobic condition and then the release appeared to be higher with higher DO.

With $DO > 7 \text{ mg/L}$, there was a significant difference among the release amounts of Cu, Pb, and Cr (i.e., $26.4 \mu\text{g}$, $17.3 \mu\text{g}$, and $16.3 \mu\text{g}$, resp.). Compared with other metals, the effect of DO on the release of Zn in the overlying water appeared to be larger, and its maximum release amount was 80 mg . Moreover, the concentration of metal Cu, Pb, and Cr tended to be stable after 20 minutes in the low-DO conditions. On the contrary, the concentrations declined gradually (approximately 35–45% of total).

As a consequence, the release of Cu, Pb, and Cr was much faster at $DO > 5 \text{ mg/L}$ than $DO < 5 \text{ mg/L}$, especially at the first 50 minutes. Furthermore, the adsorptions of Cu, Pb, and Cr were easily measured under $DO < 5 \text{ mg/L}$, while the concentrations of these metals were kept at the steady level. These results indicated that $DO = 5 \text{ mg/L}$ might be the critical state. Contrary to Pb and Cr, the release of Cu was quite different under anaerobic condition. The small release rate of Cu was detected, which might be due to the different contents and percentages of the different forms of Cu. The results concerning Cd ($DO = 9 \text{ mg/L}$) are not shown, since there was no release activity detected during the whole experiments (Figure 5).

Under anoxic and anaerobic conditions, the adsorptions of Zn, Cu, Pb, and Cr might be due to ionic bond, formed by the reducible metals and iron and manganese oxides integrating, ruptured, the Fe^{2+} , Mn^{2+} were released and these ions formed into iron and manganese (hydr) oxide solid phases, which could absorb heavy metals in the overlying water under those conditions. Furthermore, the sulphides might compound with the heavy metals in the overlying water and might be deposited to the sewer sediment under anoxic and anaerobic conditions [15, 18]. These consequences could also cause the release amount declined after increasing, as the increases of the metal release amount resulted from that the decomposition of organic compound would facilitate the metal release.

As a matter of fact, there was little Cd in the overlying water at $DO = 7 \text{ mg/L}$. Compared to the experiments conducted at $DO < 7 \text{ mg/L}$, Figure 5 shows that lower DO results in a much higher release of Cd from sediments. The release amount was higher for Cu, Pb, and Cr at higher DO concentration. Moreover, the highest release rate of Cd was observed after 30 minutes and then reached equilibrium. Based on the results of the release of Zn, Cu, Pb, Cr, and Cd affected by DO, it was possible to conclude that the DO was the major factor to release the metals from sediments into the overlying water. Moreover, the differences in the release rate between the aerobic and anaerobic conditions suggested that different forms of these metals might exist under these conditions.

3.3. Effect of Temperature on Metal Release from Sediments.

Metals of sediments have been equilibrated at temperatures ranging from 4 to 25°C [21, 22] in previous studies. The results concerning the effect of temperature (15 – 35°C) on metal release from sediments are shown in Figure 6. The release rates of Zn, Cu, Pb, Cr, and Cd in the low- and high temperature experiments were observed and they were greater at high temperature than low-temperature. Normally, the residual speciation of the sediment metal exists most stably, the soluble speciation could release into the overlying water most easily, and other species might release by chemical reactions. With the temperature increasing, the reactions could be accelerated, and the DO concentration in the water and the dissolutions of the carbonates and hydroxides increased. Therefore, the metal release rate of the water-soluble fraction, carbonate fraction, and exchangeable fraction from the sediment into the overlying water increased.

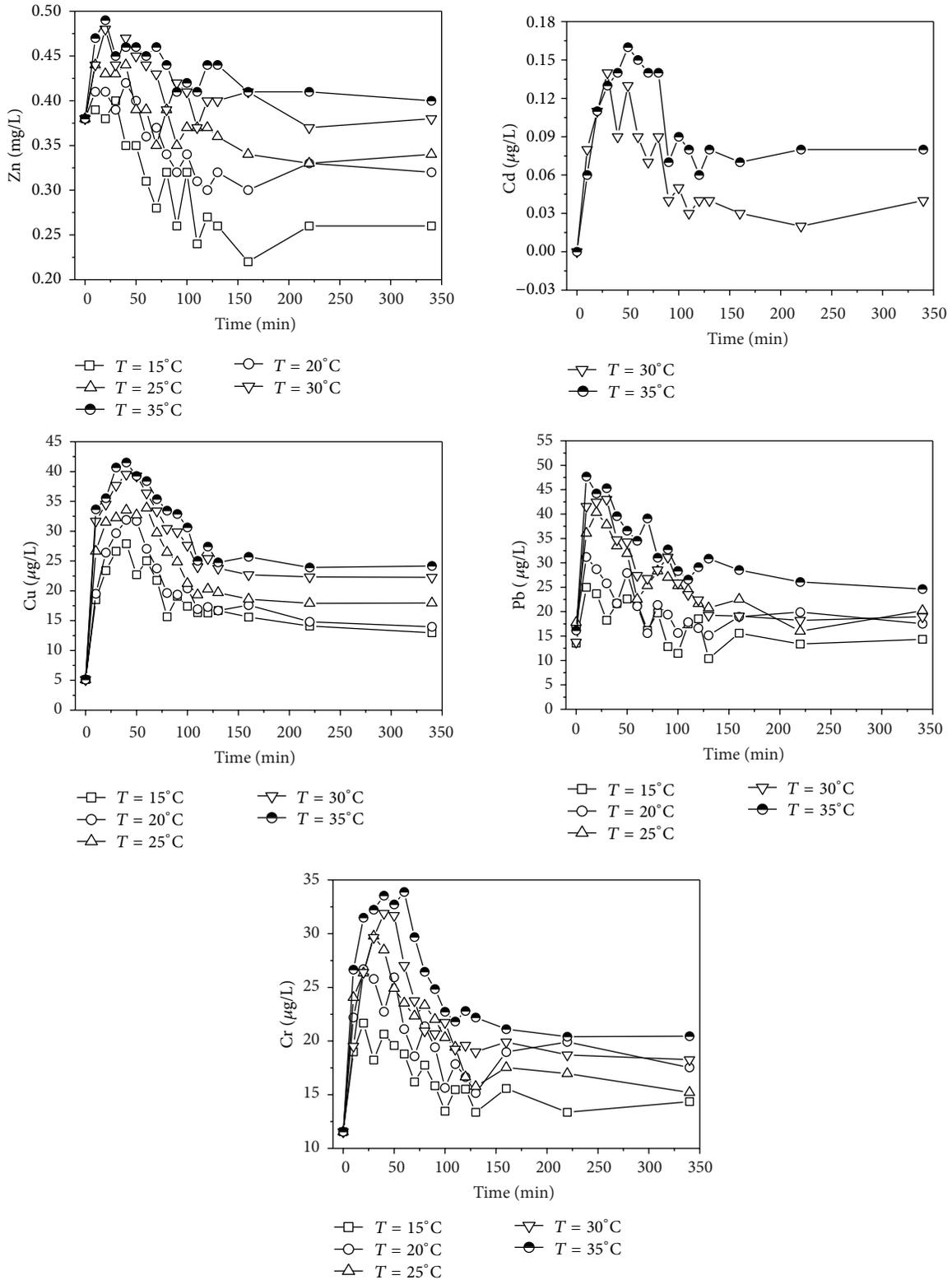


FIGURE 6: The concentration of Zn, Cd, Cu, Pb, and Cr from sediments changed with times.

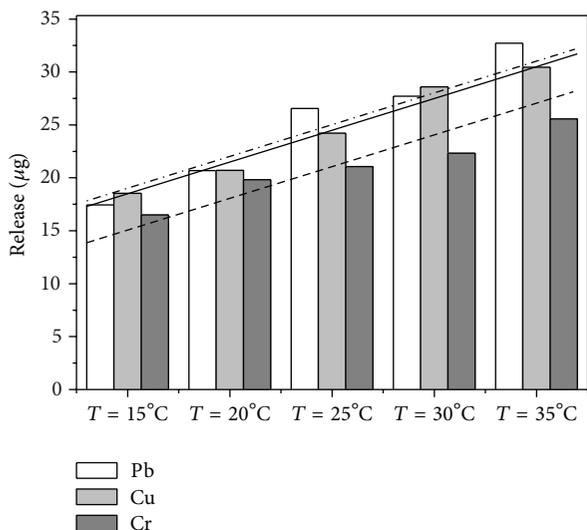


FIGURE 7: The relationship between the maximum amount of accumulated release of Cu, Pb, and Cr from sediment in experimental period and temperature.

The results of the temperature experiments were the good illustrations of the theory.

During the whole experiments, the release of Zn was significant at high-temperature with Zn concentrations of 0.381–0.483 ($T = 30^{\circ}\text{C}$) and 0.381–0.492 ($T = 35^{\circ}\text{C}$) mg/L measured at 20 minutes. Concerning Cu and Cr, a completely different behaviour compared with Zn can be observed in Figure 6. At different temperatures (15°C , 20°C , 25°C , 30°C , and 35°C), the highest release rates of Cu and Cr were observed with the maximum Cu concentration of $26.4\ \mu\text{g/L}$ and $25.6\ \mu\text{g/L}$ in the overlying water, respectively. The original concentration of Cr was only $11.6\ \mu\text{g/L}$.

The variations in concentrations of Pb were smaller than Cu and Cr at the same temperature. In addition, the changes in concentration of Pb were within $1\ \mu\text{g/L}$ between 15°C and 20°C . Compared to Zn, Cd, Cu, and Pb, Cd was not detected during the experiments at 15°C , 20°C , and 25°C . The release of Cd was only observed at 30°C and 35°C concentrations of ND ($<0.003\ \mu\text{g/L}$) to $0.130\ \mu\text{g/L}$ and ND ($<0.003\ \mu\text{g/L}$) to $0.162\ \mu\text{g/L}$ measured after 50 minutes, respectively. Comparing the release between Cd and Zn, it could be clearly seen that the effect of temperature on Cd was more obvious. As the proportion of the residual metal of Zn was more than 50%, the value of Cd was less than 12%, and the proportions of the oxidizable metals of Zn and Cd were 8.76% and 27.7% (Table 1). The residual metal was the stablest specie, the oxidizable fraction relatively migrated and transformed more easily, and the chemical reactions of oxidizable fraction also occurred more easily than the residual metal while the temperature increased. Therefore the release of Cd was more sensitive to the temperature than the release of Zn.

The concentrations Cu, Pb, Zn, Cr, and Cd increased rapidly during the first 10 mins and then the rate appeared to be gradually slow, which could be explained as the main effect factor affecting heavy metal release was the disturbance

and then was the temperature. Moreover, dissolved Cu, Pb, Cr, and Cd concentrations decreased dramatically after 50 minutes. The results are of great importance and may indicate that the release rate of different metals could be increased at high temperature. However, the final metal concentrations in the overlying water generally reached to the initial concentrations, indicating that the release of metals to the overlying water was only temporary. The present results are consistent with the conclusions drawn from the previous studies [14] suggesting a slower equilibration of sediments incubated at 4°C than room temperature ($18\text{--}25^{\circ}\text{C}$).

Moreover, during the whole experiments, the results indicated that the average concentrations of Pb, Cu, and Cr in the overlying water were very well correlated to the temperature evolution. According to Figure 7, linear relationship between the average concentrations and the temperature was clearly shown. Moreover, the association of the maximum release of metals (Pb, Cu, and Cr) from the the sewer sediments was also observed as linear relationship ($R_{\text{Cu}}^2 = 0.984$, $R_{\text{Pb}}^2 = 0.973$, $R_{\text{Cr}}^2 = 0.963$).

3.4. Effect of Flow Rate on the Release of Metal from Sediments.

The effect of flow rate was investigated by varying the rotating speed of agitator to simulate the different flow rates. With the flow of the sewer water increasing, the sediment could be exposed to the aerobic environment more easily and the oxidation rate of the organic compound and sulfide fraction might increase. Therefore the organic phase and sulfide fraction metals released more rapidly. Additionally, the flow rate also contributed to the physical disturbance of sediments and the disturbance may change the physical environment, such as pH and DO. The effects of DO and pH on the release of heavy metal mainly affected by the heavy metal speciation, and the contents of different species were different. Therefore the results of the metals release were different during different flow rate conditions and the results shown in Figures 9 and 10 emphasized the conclusion.

Figure 8 shows the concentration of Zn, Cr, and Pb measured in sewer sediments. With flow rate $<0.9\ \text{m/s}$, the concentration of these metals changed in a small scale. The maximum release amounts were $0.679\ \text{mg}$ ($V = 0.7\ \text{m/s}$) for Zn, $10.4\ \text{mg}$ ($V = 0.7\ \text{m/s}$) for Pb, and $8.03\ \text{mg}$ ($V = 0.7\ \text{m/s}$) for Cr. With a flow rate $1.1\ \text{m/s}$, the maximum release amount of Pb was $29.6\ \text{mg}$. In addition, the concentration was kept at a certain value ($20\ \text{mg/L}$) after reaching the maximum value at each flow rate. The concentration of dissolved Zn, Pb, and Cr remained low at low-flow rate, but the concentration increased at the high-flow rate. The results indicated that the flow rate significantly affects the release of Zn, Cr, and Pb, especially at the flow rate ($V \geq 0.9\ \text{m/s}$).

The effect of flow rate on the release of Cd was similar to Cu (Figure 9). The release amount of Cd from the sediments into the overlying water increased with the increase of flow rate, but it was lower than Cu. Although the concentration of Cd was very low, the final concentration was higher than the initial concentrations that reached to a stable value. Conversely to Zn, the concentration of Cr and Pb ($V \geq 0.9\ \text{m/s}$) decreased after reaching the highest value.

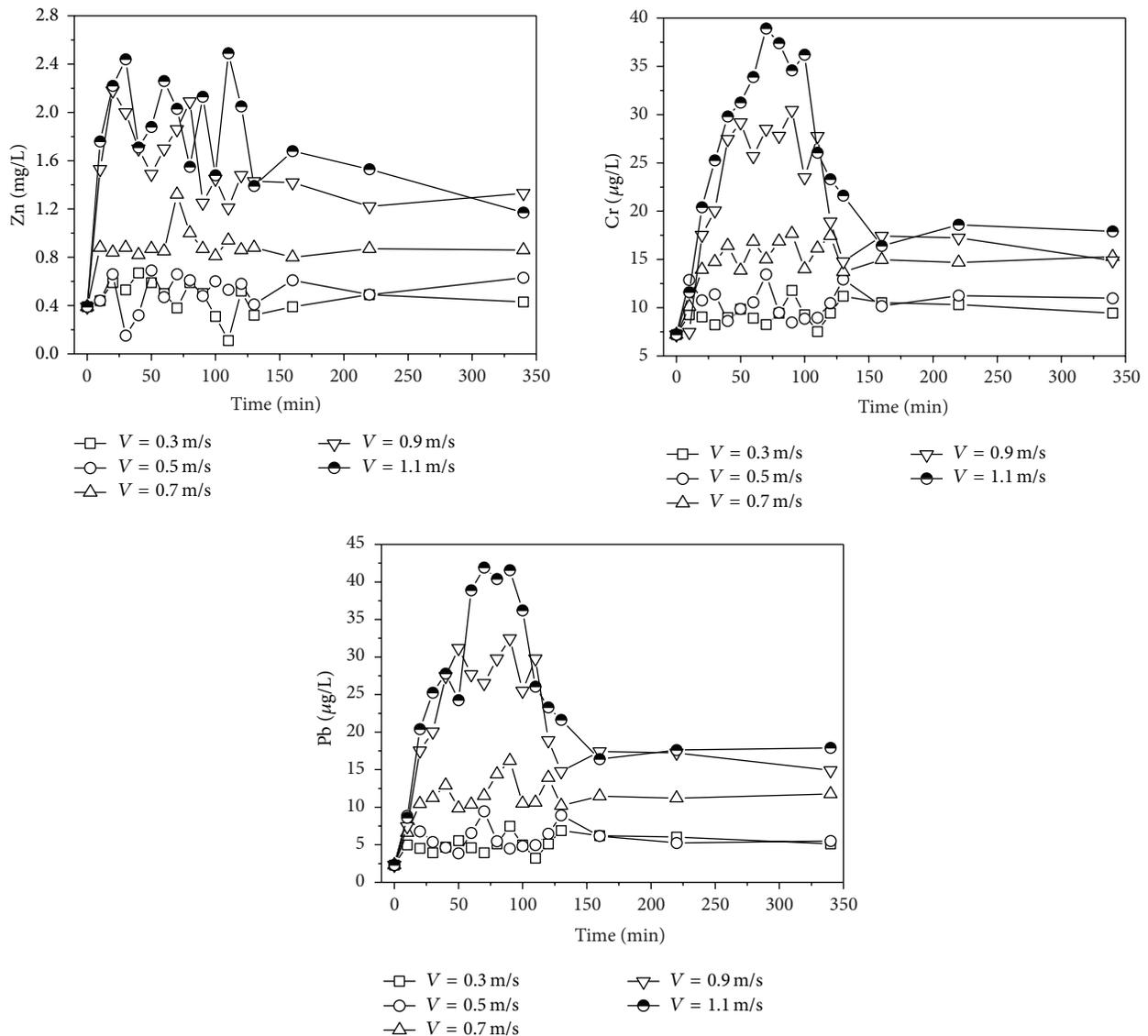


FIGURE 8: The concentration of Zn, Cr, and Pb in the overlying water changed with times.

The results indicated that flow rate was a significant factor affecting the release of heavy metals across sediment-water interface of rainwater pipes, which might be due to the resuspension of sediments at each flow rate.

All the results during different flow rates appeared to be the same diversification tendency that the concentrations of the metals increased firstly and then decreased again. The same reason could be summed up among the pH, DO, and temperature experiment results. Furthermore, the significant increase of the concentration at the first 10 mins also confirmed the previous research achievement [15] that the distance could obviously increase the metal release.

Preliminary results about the effect of flow rate may be obtained by deducing eventual correlations between the metal content in the overlying water and the flow rate. Therefore, the parabolic relationships between the average

concentration of Cu, Pb, Cr, and flow rates (Figure 10(a)) were deduced which suggested that the flow rate significantly affect release of the metals from sewer sediment.

A good correlation between Zn contents and flow rates was shown in Figure 10(b). The equation ($y = 0.288 + 0.062x + 0.05x^2$, y : maximum amount; x : flow rate) showed the good relationship between the maximum amount of accumulated release of Zn and the flow rate.

4. Conclusions

The present study focused on examining the effect of pH, temperature, DO, and flow rate of overlying water on the release of heavy metals and has indicated the major roles played by each factor. The effect of pH indicated that (i) the release of Zn, Cu, Cr, Cd, and Pb increased under both

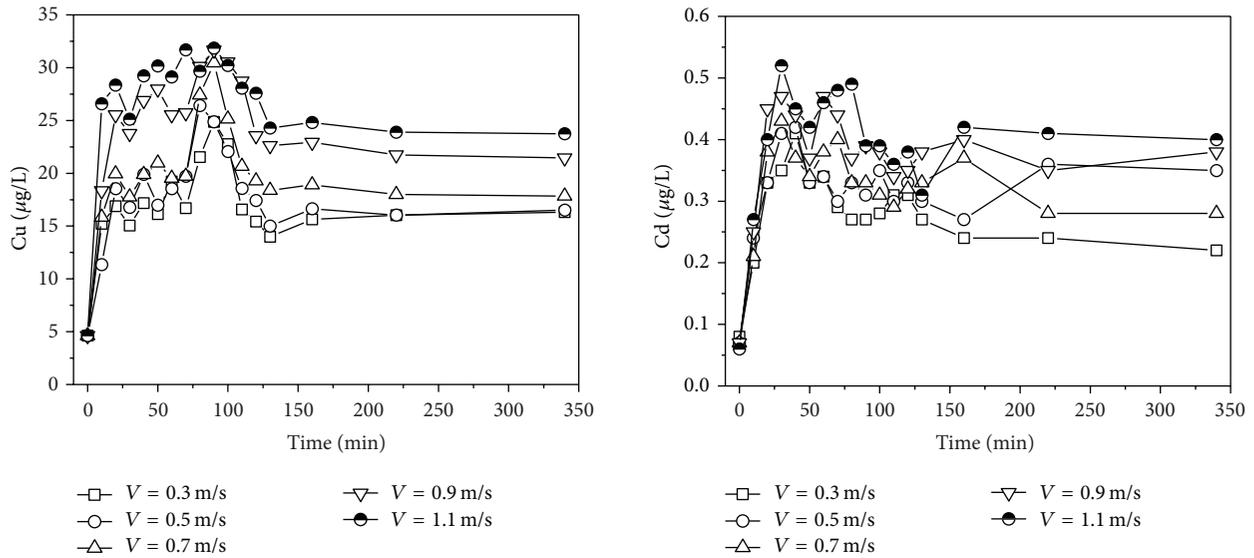


FIGURE 9: The concentration of Cu and Cd in the overlying water changed with times.

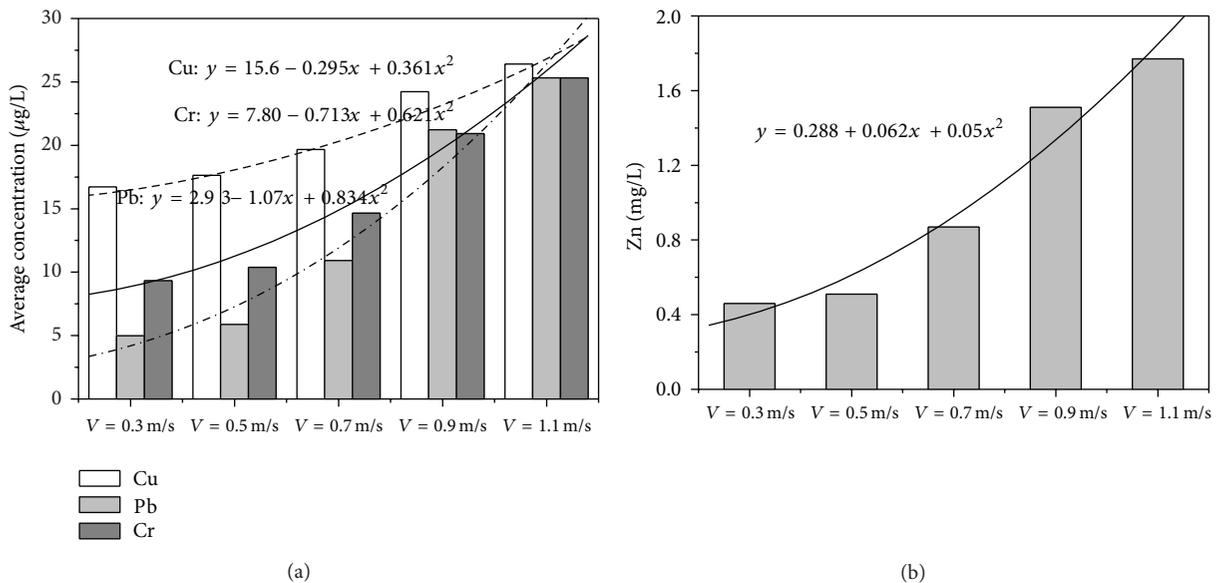


FIGURE 10: The relationship between the average concentration of metals in overlying water in experimental period and flow rates: (a) Cu, Cr, and Pb; (b) Zn.

the acidic and alkaline condition; (ii) the higher release rate occurred at lower pH; (iii) the maximum release amounts of the metals followed in an order: $Zn \geq Cu > Pb > Cr > Cd$ at the same pH; (iv) there was a good correlation between the average concentration/the maximum release amounts of each metal and the pH value.

The effect of temperature on the release of Zinc is not significant. Although the concentration of Cd was relatively low, the concentration varied considerably with the changing of the temperature. Actually, at higher temperature the release rates of metal increased with the temperature increasing. At

the same temperature, the release rate followed in the order: $Zn > Cu > Pb > Cr > Cd$. The linear relationship between the average concentration/the maximum release amount of metal and the temperature was deduced. Equilibration time was observed at about 100 min, which was likely extended since the temperature became higher.

The influence of DO on the release of metals was quite different from other factors. The effect of DO was varied from chemical species and proportions of heavy metals in the oxidizable and reducible level. As a matter of fact, the release behavior of Zn, Cu, Cr, and Pb was similar. The release of

the metal increased rapidly under the aerobic condition and the adsorption of the metal in the overlying water occurred under the anaerobic condition. In contrast, the release of Cd occurred in the anaerobic condition, and the release became more significant when the concentration of DO was low. The relationship between the average concentration of Zn, Cu, Pb and Cr and the concentration of DO was shown to be a parabolic curve. In addition, logarithmic relationship between the maximum release and the concentration of DO was observed. Nevertheless, the relationship between the average concentration of Cd and the concentration of DO followed an index correlation well.

High flow rate greatly promoted the release of metal from the sewer sediment, but it could also change the conditions such as pH, DO, and others. Therefore, the release behavior of the metals is different at different flow rates. The results suggested that (i) the flow rate significantly affected the release amount of Zn, Pb, and Cr, while it did not significantly affect the concentration of Cu and Cd; (ii) a good relationship between the average concentration/the maximum release amount of Zn, Cu, Pb, and Cr and the flow rate was deduced, and Cu was followed with a linear relationship. This research is a first step towards a better understanding of heavy metal pollution in the combined sewer system.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgments

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