Heavy Metal Removal and Remobilization under De-icing Salt Applications using Lab-scale Column Experiments

Remobilisation et élimination des métaux lourds avec les sels de déverglaçage ; Application sur des colonnes de laboratoire

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RÉSUMÉ

L'objectif de cette étude était de clarifier le comportement de cinq métaux lourds (le cadmium, le cuivre, le plomb, le nickel, et le zinc) qui avaient été simultanément fixés sur des matériaux drainants utilisés dans des systèmes décentralisés de traitement des eaux pluviales recevant le ruissellement d'une zone de trafic. Six matériaux drainants (charbon actif granulaire, un mélange d'alumine activée granulaire et de béton poreux, lignite activée granulaire, dolomite semi-calcinée, et deux hydroxydes ferriques granulaires) ont été examinés lors d'expériences par lot et en colonne pour étudier la remobilisation des métaux lourds lors de l'application de trois sels de déverglaçage différents. Dans les expériences en colonne, tous les matériaux drainants ont été préalablement chargés en métaux lourds. Ensuite, trois expériences avec des sels de déverglaçage ont été réalisées en double en utilisant du chlorure de sodium pur, un mélange de chlorure de sodium et de chlorure de calcium et un mélange de chlorure de sodium et de chlorure de magnésium. Trois couches de chaque colonne ont été séparées pour préciser l'atténuation des métaux lords en fonction de la profondeur. Les résultats de l'expérience ont montré que le cuivre et le plomb étaient mieux retenus par la plupart des matériaux drainants sélectionnés et que le cuivre était le métal le moins libéré par les trois sels de déverglaçage. Le mélange de chlorure de sodium et de chlorure de calcium est celui qui a eu l'effet le plus important sur la remobilisation, par rapport aux deux autres sels de déverglaçage. Les résultats de l'utilisation du mélange d'alumine activée granulaire et de béton poreux sont présentés en détail.

ABSTRACT

The objective of this research study was to elucidate the remobilization behavior of five heavy metals (i.e., cadmium, copper, lead, nickel, and zinc) that had been simultaneously fixed onto filter materials used in decentralized stormwater treatment systems receiving traffic area runoff. Six filter materials (i.e., granular activated carbon, a mixture of granular activated alumina and porous concrete, granular activated lignite, half-burnt dolomite, and two granular ferric hydroxides) were examined in batch and column experiments to study the remobilization of heavy metals during the application of three different de-icing salts. In the column experiments, all of the filter materials were preloaded with the heavy metals. Subsequently, three de-icing salt experiments were performed in duplicate using pure sodium chloride, a mixture of sodium chloride and calcium chloride, and a mixture of sodium chloride and magnesium chloride. Three layers of each column were separated to specify the attenuation of heavy metals as a function of depth. As a result of the experiments, copper and lead were retained best by most of the selected filter materials and copper was released the least of all the metals by the three de-icing salts. The mixture of sodium chloride and calcium chloride resulted in the strongest effect on remobilization compared to the other two de-icing salts. The results using the mixture of granular activated alumina and porous concrete are presented in detail.

KEYWORDS

Chloride, decentralized system, filter material, road maintenance, stormwater treatment

1   INTRODUCTION

The substances analyzed in traffic area runoff waters include organic parameters, heavy metals, and compounds of de-icing salts (Eriksson et al., 2007, Folkeson et al., 2009, Kayhanian et al., 2012). Heavy metals such as cadmium, copper, lead, nickel, and zinc are crucial pollutants because of their toxicity, non-degradability, and their increase in all environmental compartments in consequence of their industrial use (Roeva et al., 1996). In traffic area runoff waters, median total concentrations are approximately 1 µg/L (cadmium), 50 µg/L (copper), 35 µg/L (lead), 10 µg/L (nickel), and 230 µg/L (zinc) (Huber et al., 2016b). Therefore, these five elements are relevant in traffic area runoff and treatment of the runoff water is necessary to prevent negative effects on surface water and groundwater. Of these heavy metals, lead is mostly particle-bound, while cadmium, copper, nickel, and zinc occur at a higher fraction in the dissolved phase. Sodium chloride, calcium chloride, and magnesium chloride are widely used as de-icing salts (Stotz & Krauth, 1994, Marsalek, 2003, Bäckström et al., 2004, Nelson et al., 2009, LfU, 2013).

In Germany, dozens of technical decentralized stormwater treatment systems have been developed in recent years to treat traffic area runoff waters (Dierkes et al., 2015). All advanced systems consist of two treatment steps because dissolved substances cannot be removed in a mechanical step (e.g., by sedimentation and filtration). To reduce all metals, a second treatment step with a filter material is necessary (Hilliges et al., 2013). The German Centre of Competence for Construction (Deutsches Institut für Bautechnik, 2015) can approve these decentralized systems after conducting several tests (e.g., removal of fine particles, total petroleum hydrocarbons, and heavy metals; remobilization of copper and zinc under application of sodium chloride). However, there is no detailed method available to determine the impact of all relevant de-icing salts on the remobilization of heavy metals that were previously retained by filter materials. Consequently, there is no technical decentralized treatment system for road runoff available in Germany with proven filter stability against sodium chloride, calcium chloride, and magnesium chloride.

Concerning a constructed wetland as a technical decentralized treatment system, during applications of sodium chloride, an increase in cadmium, copper, nickel, and zinc concentrations was detected in the effluent (Tromp et al., 2012). Thereby, copper effluent concentrations were higher than the maximum influent concentration. Hence, the consideration of heavy metal releases provoked by de-icing salts is relevant for facilities treating traffic area runoff in cold and moderate climates.

Few studies examined the remobilization behavior of heavy metals bound onto soils, which are used to treat traffic area runoff waters (Amrhein & Strong, 1990, Amrhein et al., 1992, Amrhein et al., 1993, Norrström & Jacks, 1998, Bäckström et al., 2004). Nelson et al. (2009) tested two soils for the effects of sodium chloride and magnesium chloride on heavy metal mobility. Both soils showed different behaviors for each de-icing salt. The use of sodium chloride resulted in a large release of copper and lead. Magnesium chloride released high levels of cadmium for both soils and copper for one soil. Each soil showed different remobilization effects and all tested metals were released in different quantities. Furthermore, Huber et al. (2014) published results concerning the remobilization of copper and zinc by sodium chloride and calcium chloride for two soils and five filter materials used in decentralized stormwater treatment systems. In some cases, both heavy metals were released and in other cases only zinc showed higher mobility. These effects depended on the material and on the de-icing salt, e.g., some heavy metals were not released with pure sodium chloride but with a mixture of sodium chloride and calcium chloride. In addition, the pH of the effluent was often reduced by the use of de-icing salts. For a few materials, copper and zinc showed nearly no increased mobility during all de-icing salt experiments (Huber et al., 2014). However, both studies did experiments with only some of the five relevant heavy metals and the three de-icing salts.

The objective of this study is to elucidate the heavy metal removal and remobilization under de-icing salt applications for five heavy metals and three de-icing salts using lab-scale column experiments. As more and more technical decentralized treatment systems are used, the focus is set on six filter materials, which are all commercially supplied. The results can be helpful for the development and improvement of filter materials used in decentralized stormwater treatment systems.

2   MATERIAL AND METHODS

2.1   Sorbents and Chemicals

As sorbents, the following were used: granular activated carbon (Donau Carbon GmbH, Germany), a mixture (named alumina) of granular activated alumina (55%; Albemarle Corporation, USA) and porous concrete (45%; Xella Deutschland GmbH, Germany), granular activated lignite (Rheinbraun Brennstoff GmbH, Germany), half-burnt dolomite (Rheinkalk Akdolit GmbH & Co. KG, Germany), and two granular ferric hydroxides with different calcium contents (FerroSorp, HeGo Biotec GmbH, Germany; GFH, Wasserchemie GmbH & Co. KG, Germany). Before experiments, granular activated lignite and half-burnt dolomite were sieved to the size fraction of 0.5–2.0 mm to avoid the influence of different fractions of each sorbent on the retention of heavy metals (Smith, 1998). Subsequently, all materials were dried at 105±2 °C to achieve a constant weight, and finally stored in desiccators without additional treatment until usage. The heavy metal salts used were cadmium sulfate 8/3-hydrate p.a. (AppliChem GmbH, Germany), copper(II) nitrate trihydrate pure (AppliChem GmbH, Germany), nickel(II) nitrate hexahydrate p.a. (Merck KGaA, Germany), lead(II) nitrate pure (AppliChem GmbH, Germany), and zinc nitrate hexahydrate pure (AppliChem GmbH, Germany). pH adjustments and conservation of samples were performed with nitric acid 65% p.a. (Merck KGaA, Germany). As de-icing salts, sodium chloride >99.9% (AppliChem GmbH, Germany), calcium chloride dried, powdered pure (AppliChem GmbH, Germany), and magnesium chloride hexahydrate pure (AppliChem GmbH, Germany) were used.

2.2   Experimental Setup and Analyses

The experimental apparatus was constructed of glass and all pipes were of polyvinyl chloride. The glass column had an inner diameter of 2.0 cm and a length of 40 cm, providing a sorbent bed height of 14.4 cm. A glass screen was placed at the bottom of the column to support the filter materials. The influent went through the glass columns in up-flow mode (from bottom to top) at ambient temperature (21±2 °C). Lab-scale column experiments for the heavy metal removal of each sorbent were conducted with inflow concentrations of 0.625 mg/L (cadmium), 2.5 mg/L (copper, lead, and nickel), and 5.0 mg/L (zinc) at an initial pH of 4.9±0.3 to obtain dissolved metals. Deionized water (electric conductivity of <1 µS/cm) was used for influent preparation and the flow rate was adjusted to about 6.5 mL/min by a peristaltic pump (ECOLINE VC-MS/CA8-6, Ismatec SA, Switzerland). As a result, empty bed contact time (EBCT) was maintained at about 7 min. The experiments were performed parallel in eight columns per material from Monday to Friday. During weekends, the filter materials were kept saturated but without flow. The end of the preloading part was arbitrarily determined as a 50% breakthrough of zinc, which was not achieved by dolomite (reduced permeability because of precipitated metals). Subsequently, each column was flushed with deionized water for 60 min to remove the dissolved heavy metals, which were not fixed onto the filter material of the preloading part. Six of the eight columns containing filter materials with preloaded heavy metals were used for the experiments with de-icing salt solutions. All salt experiments were completed in duplicate. Thus, two columns were run with pure sodium chloride (10 g/L), two with a mixture of sodium chloride and calcium chloride (10 g/L and 2.5 g/L, respectively), and two with a mixture of sodium chloride and magnesium chloride hexahydrate (10 g/L and 2.5 g/L, respectively). All de-icing salt concentrations were taken from the results of a literature study (Huber et al., 2015) and the experiments simulated a typical rainfall (6.0 L/(s·ha) for 200 min). The ratio of the filter surface area to the drainage area was 1:1820. Thus, the flow rate was adjusted to about 20.5 mL/min.

For all experiments, one influent sample per stock solution and several effluent samples at designated times were collected in 50 mL polypropylene bottles. Parts of selected samples were filtered to determine the dissolved part of the heavy metals (0.45 µm pore size, VWR Collection, Germany). For all water samples, measurements of the pH value were done with a glass electrode (WTW Sentix 60) and electric conductivity was determined by using a WTW LF 340 EC conductivity meter. All samples were preserved with nitric acid at pH <2. At the end of every experiment, three layers of each column were separated to specify the attenuation of heavy metals after lithium metaborate fusion as a function of depth. Blank samples of all filter materials were also analyzed after lithium metaborate fusion. The concentrations of heavy metals were measured by flame atomic absorption spectrometry (AAS, Varian Spectrometer AA-240FS) with limits of quantification of 20 µg/L (cadmium), 50 µg/L (copper), 200 µg/L (lead), 50 µg/L (nickel), and 20 µg/L (zinc) according to Standard Methods (Eaton et al., 2005). Graphite furnace AAS (Varian Spectrometer AA-240Z with GTA 120) was used to reduce the limits of quantification for lead for all experiments and for all elements for the de-icing salt experiment samples (5 µg/L for all heavy metals).

3 RESULTS AND DISCUSSION

3.1 Batch Experiments

Equilibrium batch experiments (24 h to retain maximum capacities) and kinetic experiments (5 min to 120 min) were used to evaluate 14 filter materials for their retention of zinc, copper, and nickel. Selected results are published in Huber et al. (2016a). The six sorbents described in Section 2.1 were selected because of their heavy metal removal efficiencies. The focus of this study was also set on the examination of different removal mechanisms.

3.2 Lab-scale Column Experiments: Heavy Metal Removal

The results of the breakthrough curves obtained in the first part of the lab-scale column experiments showed wide differences in the removal efficiencies of all filter materials. In general, copper and lead were best retained. However, the removal of lead by half-burnt dolomite, which mainly consists of calcium carbonate, was less efficient. In addition, lead was mainly dissolved (about 70%) in the effluent compared to the partitioning of copper, nickel, and zinc. In waters of calcareous soil, the concentrations of lead are primarily attributed to the solubility of lead carbonates (Badawy et al., 2002). This process might also have occurred during the experiments with half-burnt dolomite, and, regarding the lead removal, it explains the different behavior of the half-burnt dolomite compared to the other filter materials. Lead removal at pH 10, which was the median prevailing effluent pH value during the experiment, seems to be less efficient because of competition with other heavy metals for carbonates. The amount of the inflow solution needed for the 50% breakthrough of zinc varied widely for all selected filter materials. The best material for heavy metal removal (FerroSorp) retained approximately six times more zinc compared to all other sorbents. The capacities of FerroSorp for zinc were comparable to the ones determined in the batch experiments. However, the capacities of all other filter materials were lower for column experiments in comparison to the batch experiments.

For each column that was not used for de-icing salt experiments, three layers were separated to specify the attenuation of heavy metals as a function of depth. The total amount of each heavy metal retained was always much higher than the corresponding solid contents of the blank materials. For example, the highest heavy metal content of the blank alumina sample was 18.2 mg/kg for copper. Despite different inflow concentrations, the removal capacities for copper, lead, and zinc were often similar because of the earlier increase of zinc effluent concentrations. The results of the preloaded alumina, a representative material for the heavy metal removal, are presented in Figure 1. More mobile elements (i.e., cadmium, nickel, and zinc) had an earlier 50% breakthrough and the highest contents in the top layer compared to copper and lead. The ratios of copper content to zinc content and lead content to zinc content were 82% and 89%, respectively. At the end of the preloading part, the dissolved part of copper in the effluent was approximately 86% and all other metals were at least 93% dissolved (pH 6.6).
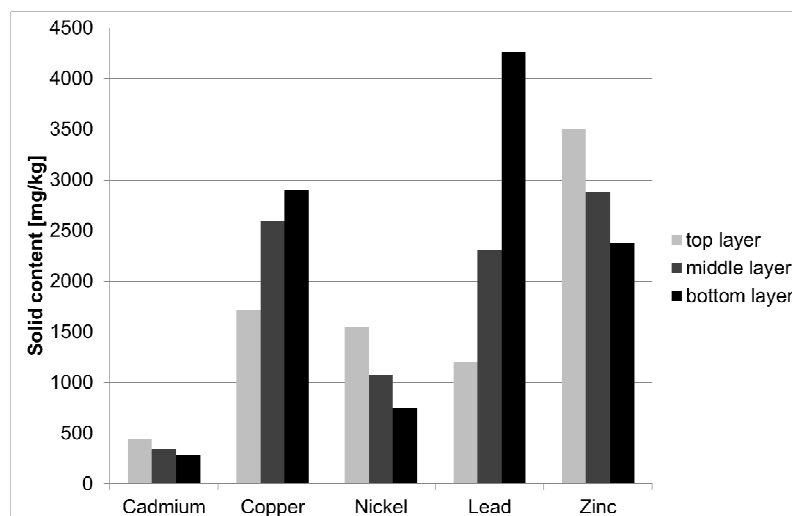

Figure 1: Alumina – heavy metal retention in three layers (preloading was conducted in upflow-mode)

3.3 Lab-scale Column Experiments: Heavy Metal Remobilization

The results were more heterogeneous for the de-icing salt experiments. The behavior of each filter material, each heavy metal, and each de-icing salt was different. In general, copper was released less from most of the selected filter materials by the three de-icing salts. Compared to the other two de-icing salts, the mixture of sodium chloride and calcium chloride resulted in the strongest effect on remobilization.

However, copper, nickel, and zinc that had sorbed onto granular activated carbon were released in highest amounts in the presence of magnesium chloride. For most heavy metals and filter materials, pure sodium chloride had a minor effect on the remobilization.

Half-burnt dolomite, which retained lead to a minor degree in the first part of the experiments, showed the highest remobilization of lead with effluent concentrations of up to 1380 µg/L. Copper was also released by calcium chloride and magnesium chloride in high concentrations. Furthermore, an average cadmium effluent concentration of 231 µg/L was measured for the experiments with a mixture of sodium chloride and calcium chloride (maximum at the beginning 552 µg/L). Boekhold et al. (1993) investigated the sorption of cadmium onto a sandy soil and found out that this metal was more mobile at high concentrations of calcium chloride and the formation of chloride complexes enhanced the remobilization. Acosta et al. (2011) described the effect of sodium chloride, calcium chloride and magnesium chloride on the mobility of cadmium, copper, lead, and zinc in alkaline soils with a calcium carbonate content of 12.2%–50.4%. One result was that the release of metals depended on the salt: For sodium chloride and calcium chloride, cadmium and lead were mobilized to a higher extent. In contrast, cadmium and copper were released the most by magnesium chloride. Thus, these results of the calcareous soils are comparable to the ones of half-burnt dolomite.

For alumina, the average effluent concentrations of each heavy metal are shown in Figure 2. Both twice positively charged de-icing salt cations (magnesium and calcium) had the strongest effect on the remobilization of heavy metals. Sodium chloride also released heavy metals in lower concentrations. For nickel, the differences between each de-icing salt were minimal. With the exception of zinc, all heavy metals were found in higher effluent concentrations compared to the influent concentration of each de-icing salt experiment. Zinc originating from the impurities of the salts was determined in all three influents of the de-icing salt experiments in concentrations of 86 µg/L, 131 µg/L, and 163 µg/L, respectively. Therefore, the averaged zinc effluent concentrations (39 µg/L, 84 µg/L, and 143 µg/L) were always lower than the corresponding influent concentrations. All other heavy metals in the influent were below the limits of quantification. With the exception of copper, all heavy metals measured in the effluent were mostly in the dissolved phase (88%–100%). In the tests with sodium chloride, the largest particulate copper fraction in the effluent was 26%, which correlates with the pH of 8.8. In the experiments with magnesium chloride and calcium chloride mixtures, the particulate fractions were only 13% (pH of 8.4) and 8% (pH of 8.1), respectively.
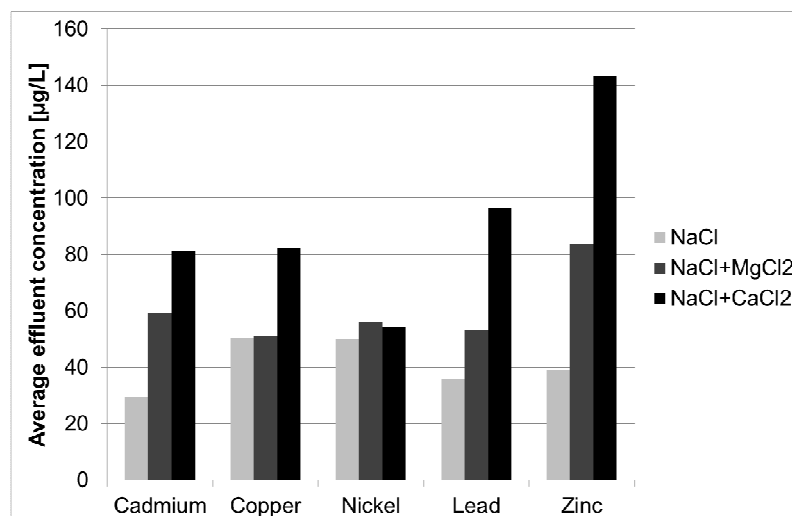

Figure 2: Alumina – results of the de-icing salt experiments (mean of duplicates)

Because of the small amounts of heavy metals being remobilized (e.g., a maximum of 12 mg/kg of lead during one experiment), a variation of the heavy metals retention in each of the three layers could not be detected for alumina.

Modelling will be performed to support the explanation of the different behavior of heavy metals, especially concerning the removal mechanisms of half-burnt dolomite. A special focus is set on the chloride concentrations since the concentrations of cadmium, copper, lead, and zinc in soil solutions are significantly correlated with the presence of chloride (Bäckström et al., 2004).

A further aspect is the correlation of the heavy metal solid content with the degree of remobilization that was found out by Acosta et al. (2011) for calcareous soils. Further experiments with low-preloaded sorbents and the same de-icing salt experiments are carried out to examine these effects. Thus, a comparison between high-preloaded and low-preloaded filter materials will be achieved. This comprises both the heavy metal attenuation as a function of depth and the degree of remobilization during de-icing salt experiments. Moreover, further analyses will be carried out to elucidate the removal and remobilization mechanisms of cadmium, copper, nickel, lead, and zinc.

4   CONCLUSION

The differentiated approach of this study made it possible to evaluate the risk of remobilization for five heavy metals and six filter materials, both against three different de-icing salts.

The removal efficiencies of each heavy metal varied for all filter materials in the lab-scale column experiments. Copper and lead were retained best with the exception of half-burnt dolomite. The bed volumes treated until a 50% breakthrough of zinc varied widely for all selected filter materials. The best material removed about six times more zinc compared to all other materials. The difference of removal capacities between the selected materials was much lower in the batch experiments. Therefore, the results of batch experiments can only show tendencies for the removal efficiencies.

Concerning the de-icing salt experiments, the results were more heterogeneous. The behavior of each filter material, each heavy metal, and each de-icing salt was different. In general, copper was released the least from most of the selected filter materials by the three de-icing salts. The mixture of sodium chloride and calcium chloride resulted in the strongest effect on remobilization compared to the other two de-icing salts. Pure sodium chloride had a minor effect on the remobilization for most heavy metals and filter materials.

After completion of further experiments regarding the correlation of the heavy metal solid content with the degree of remobilization and the advanced analyses, the final results will be presented elsewhere.

The results of this research can be implemented into the development and improvement of decentralized stormwater treatment systems using filter materials. By these findings, a more environmentally friendly operation of these systems for traffic areas can be ensured and a longer service time is also possible.

ACKNOWLEDGEMENT

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