

Scavenging of Cd through Fe/Mn oxides within natural surface coatings

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Abstract: The dynamics of Cd scavenging from solutions by Fe/Mn oxides in natural surface coatings (NSCs) was investigated under laboratory conditions. Selective extraction methods were employed to estimate the contributions of Fe/Mn oxides, where hydroxylamine hydrochloride (0.01 mol/L $\text{NH}_2\text{OH}\cdot\text{HCl}$ + 0.01 mol/L HNO_3), sodium dithionite (0.4 mol/L $\text{Na}_2\text{S}_2\text{O}_4$) and nitric acid (10% HNO_3) were used as extraction reagents. The Cd scavenging was accomplished with developing periods of the NSCs (totally 21 data sets). The resulting process dynamics fitted well to the Elovich equation, demonstrating that the amount of Cd scavenged was proportional to the increments of Fe/Mn oxides that were accumulated in the NSCs. The amount of Cd bound to Fe oxides (M_{CdFe}) and Mn oxides (M_{CdMn}) could be quantified by solving two equations based on the properties of two extraction reagents. The amount of Cd scavenged by Fe/Mn oxides could also be estimated using M_{CdFe} and M_{CdMn} divided by the total amounts of Fe and Mn oxides in the NSCs, respectively. The results indicated that the Cd scavenging by Fe/Mn oxides was dominated by Fe oxides, with less roles attributed to Mn oxides. The estimated levels of Cd scavenging through Fe and Mn oxides agreed well with those predicted through additive-adsorption and linear-regression models.

Keywords: cadmium; scavenging; natural surface coatings; selective extraction; iron oxides; manganese oxides

Introduction

Understanding the mechanism of scavenging for heavy metals through the natural surface coatings (NSCs) is crucial for providing meaningful information about environmental behaviors of the metals in aquatic environments. This is particularly significant for pollution control, ecological assessment, environmental planning, and risk management. With the awareness of the toxicity and non-biodegradability of heavy metals, a number of studies were dedicated to their mitigation, transport and scavenging in natural waters. Scavenging (or adsorption) was suggested as the main mechanism for controlling heavy metals; this could be accomplished through various adsorbents such as soils (Kinniburgh and Jackson, 1981), suspended solids in natural water (Sigg, 1985), sediments (Lion *et al.*, 1982; Tessier and Campbell, 1987), periphytons (Newman *et al.*, 1985; Gray *et al.*, 2001), and NSCs (Nelson *et al.*, 1999a, b; Dong *et al.*, 2000). Among these adsorbents, the NSCs can grow on surfaces of all materials in rivers, lakes and wetlands, habituated with a wide range of microbial organisms (Costerton *et al.*, 1987). As a valuable adsorbent, the NSCs play a critical role in controlling transport and fate of heavy metals in aquatic environment (Headley *et al.*, 1998; Nelson *et al.*, 1999a, b; Dong *et al.*, 2000, 2001a). Dissolved substances will be adsorbed on the NSCs till they reach the surface of carrying materials. The sorption properties of this layer are therefore of importance for the sorption dynamics (Flemming, 1995).

In the perspective of biology, the NSCs are composed of algae, bacteria, protozoa and fungi, which are embedded in a matrix of polysaccharides and other polymers (Lock *et al.*, 1984); from an environmental chemistry point of view, the main components of the NSCs are metal oxides (Fe, Mn and Al oxides), organic materials and some mineral materials (Dong *et al.*, 2001a). It is then desired to identify the component that contributes the most to the adsorption of heavy metals. In addition to the well-established effects of solution chemistry (e.g. pH, ionic strength, and metal speciation), trace metal adsorption to the NSCs is expected to be governed by Mn and Fe oxides, with smaller roles attributed to the adsorption by organic materials and Al oxides in the NSCs (Nelson *et al.*, 1995, 1999a, b; Dong *et al.*, 2000, 2001a). As to different heavy metals, the contributions of Fe and Mn oxides to metal adsorption are significant different.

Previously, Dong *et al.* (2000, 2001a, 2003b) found that the adsorption of Pb to Mn oxides and that of Cd to Fe oxides were statistically higher than those of Pb and Cd to other substances using additive-adsorption and linear-regression models; the adsorption of Cd and Pb was conducted under laboratory conditions after the NSCs had been developed in fields. Dong *et al.* (2003a) employed selective extraction techniques to estimate aquatic Pb binding by solid phases of heterogeneous NSCs materials in the Cayuga Lake, USA. The results showed that Pb adsorption capacity of Mn oxides exceeded that of Fe oxides on a molar basis by

approximately an order of magnitude, with Pb adsorption finished during the development of the NSCs; this information provides more reliable mechanism of heavy metals adsorption to the NSCs than those predicted through additive-adsorption and linear-regression models (Dong *et al.*, 2000, 2001a).

However, for Cd that is another most toxic metal affecting the aquatic environment, there has been no report concerning on its scavenging by Fe and Mn oxides in the NSCs development or the dynamics of Cd scavenging. Hence, the aim of the present work is to estimate the Cd scavenging by Fe, Mn oxides in the NSCs developed in the water of Nanhu Lake with added Cd solution in lab. Selective extraction methods were used to investigate the contributions of Fe and Mn oxides in the NSCs to remove trace metal. These results will help us to further understand the scavenging/adsorption mechanisms of Cd by the NSCs in aquatic environments and compare with those previously obtained.

1 Materials and methods

1.1 Site description

Water samples, in which the NSCs were developed, were obtained from the Nanhu Lake, a eutrophic lake with significant Mn deposition (Xu *et al.*, 1999). The Nanhu Lake is in the southern part of Changchun, China. It is an artificial semi-closed shallow continental lake with a water surface area of 0.85 km²; its deepest depth is 6.6 m, with an average of 2.8 m and a storage capacity of 2.5×10^6 m³. The water comes from three sources: surface runoff, rainfall and sewage. It is light yellow-green in color with a pH level of 7.3–8.9, and contains plentiful algae and planktons. Levels of heavy metals in the lake water are 0.025 µmol/L for Cd and 0.187 µmol/L for Cu respectively (Dong *et al.*, 2002).

1.2 Development of heterogeneous NSCs

To estimate scavenging of Cd through Fe and Mn oxides, the NSCs were developed on glass slides (48 × 75 × 10 mm) in two glass containers (119 × 33 × 31 cm). The lake water in the containers was vigorously stirred with added Cd solution (approximately 0.01 µmol/L). Then 96 pieces of glass slides were held in four polypropylene racks, and then submerged in the water at a depth of about 25 cm for a period of eight weeks in August 2004.

Prior to the placement into the lake water, glass microscope slides and polypropylene racks were pre-cleaned with detergent, soaked for 24 h in soap solution, acid washed for 24 h in 6:1 (v/v) of H₂O:HNO₃; then rinsed in distilled-deionized water (dd H₂O), followed by a second 24-h acid wash and final rinse in ddH₂O. The 0.01 µmol/L Cd adsorption solution was prepared by dilution of 1000 µmol/L Cd (NO₃)₂ reference solution using the retrieved water,

which was poured into the glass containers in laboratory. The water in the containers was almost static, except that air was pumped into the system for 30 min every day to keep adequate air-phase composition. The water temperature was kept constant (23 ± 0.5)°C at each observation time point. Four racks of glass microscope slides (racks 1 to 4) were placed in parallel within the two glass containers (two racks in each glass container) with racks 1 and 2 (as duplicates) to be used for studying the process dynamics, and the other two (as duplicates) for examining the contributions of Fe/Mn oxides. After the desired periods (totally 52 d) of development, the glass slides with visible NSCs were taken out of the polypropylene racks from the containers.

1.3 Dynamics of Cd scavenging

Duplicate glass slides with NSCs attached (one from rack 1, and the other from rack 2) were collected at multiple periods of time (days 0.5, 1, 2, 4, 7, 10, 13, 16, 19, 22, 25, 28, 31, 34, 37, 37, 40, 43, 46, 49 and 52) for studying the dynamics of Cd scavenging by Fe/Mn oxides while the NSCs were being developed. After the development periods, glass slides with the NSCs were taken out from the racks; then each slide was extracted in 25 ml of extraction reagent in 150 mm plastic petri dishes using 10% HNO₃ (trace metal free) for 24 h. Acid extracts were then analyzed for Cd (as scavenged by Fe/Mn oxides, expressed in the unit of µmol Cd/m²) and Fe/Mn oxides (as accumulated on the NSCs, also expressed in the unit of µmol Fe or Mn per m²) by flame atomic absorption spectrometry (WYX-9004, Shenyang, China) equipped with a SML-III graphite furnace. One of the most useful models for dynamic studies of such activated adsorption is the Elovich equation (Taylor and Thon, 1952; Aharoni and Tompkins, 1970; Klusacek *et al.*, 1989), which was employed to analyze the generated data as follows:

$$C = k + n \times \ln t \quad (1)$$

where C is the amount of Cd scavenged from solutions by Fe/Mn oxides (µmol/m²); k is the amount of Cd scavenged during initial equilibrium phase (fast phase reaction, µmol/m²); t is the developing time of NSCs (d); n is the scavenging rate as a function of time during the slow phase of the reaction (µmol/(m·d)).

1.4 Selective extraction techniques of the NSCs

For each slide with NSCs from racks 3 and 4, the estimated Cd scavenging by each oxide was compared to the total observed Cd removed under controlled laboratory conditions. Three extraction reagents were employed, including hydroxylamine hydrochloride (0.01 mol/L NH₂OH·HCl + 0.01 mol/L HNO₃) used for solubilization of Mn oxides and associated Cd, and sodium dithionite (0.4 mol/L Na₂S₂O₄) used to dissolve Mn plus Fe oxides and associated Cd (Dong *et al.*,

2003a, b). Sodium dithionite was prepared immediately before use to avoid any reduction of $S_2O_4^{2-}$. All of the extractions were performed in 100 ml glass petri dishes with 25 ml of extractants. For each extraction technique, four slides from each rack and two controls (the clean glass slides without NSCs attached) were used. After exposure to the selective extraction reagent, the residual unextracted metals in the NSCs were removed by extraction with 10% HNO_3 . Extraction with 10% HNO_3 for 24 h was also used to remove extractable Fe/Mn oxides and associated Cd, and the amount of Fe, Mn oxides and the associated Cd extracted with 10% HNO_3 is defined as 100% of the total extractable. The levels of Fe, Mn and associated Cd in 10% HNO_3 and other specific extractants were analyzed through flame atomic absorption spectrometry (WYX-9004) equipped with a SML-III graphite furnace.

The selective extraction techniques (extraction with hydroxylamine hydrochloride and sodium dithionite) could help avoid substantial damage to microorganism cells. This has been demonstrated by microscopic examination of the extracted NSCs; in comparison, significant cell lyses was observed after extraction in the study of Lead *et al.* (1998).

2 Results and discussion

2.1 Dynamics scavenging of Cd

The dynamics of Cd scavenged from solutions by Fe/Mn oxides is shown in Fig.1. The data of Cd scavenging dynamics fitted well to the Elovich equation ($C = 0.0145 + 0.4415 \times \ln t$, $R^2 \geq 0.68$). This indicated that the scavenging reaction for Cd could occur in two distinct phases: an initial fast phase followed by a subsequent slow one. The fast phase occurred before the day 28 with the slow one being after the day 28. The two phases were similar with the accumulation of Fe/Mn oxides on the NSCs (Fig.2). These meant that Cd scavenged increased with increasing of Fe/Mn oxides during the NSCs' development. The relationships between the scavenged Cd and the Fe/Mn oxides that accumulated on the NSCs could be quantified based on a linear regression analysis, with $R^2 > 0.88$ for Fe oxides vs. Cd and $R^2 > 0.97$ for Mn oxides vs. Cd (Table 1).

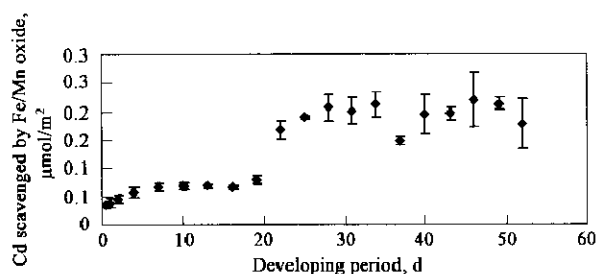


Fig.1 Dynamics of Cd scavenging by Fe/Mn oxides
Error bars indicate mean \pm one relative deviation

These highlighted the importance of both Fe oxides and Mn oxides for Cd scavenging during the development of the NSCs. The results also agreed well with the previous prediction through the additive-adsorption and linear-regression models (Dong *et al.*, 2000, 2001a), where the NSCs were developed in fields and used to estimate the mechanism of trace-metal adsorption on the NSCs.

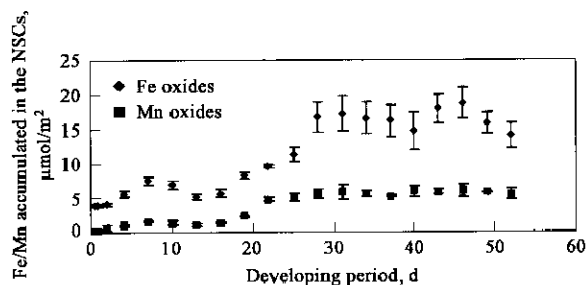


Fig.2 Dynamics of Fe/Mn oxides accumulating on the NSCs
Error bars indicate mean \pm one relative deviation

Table 1 Relationship between Cd scavenged and Fe/Mn accumulated on the NSCs

	Fe oxides vs. Cd	Mn oxides vs. Cd
Linear regress model	$Y = 71.818x + 1.5411$	$Y = 32.714x - 0.7339$
R^2	> 0.88	> 0.97
Significant level	$p < 0.001$	$p < 0.001$
Sample numbers	$n = 20$	$n = 20$

Notes: Y means Cd scavenged by Fe/Mn oxides on the NSCs; x means Fe/Mn accumulated on the NSCs

2.2 Contributions of Fe/Mn oxides

The Cd in the extract reagent plus the residual Cd remaining on the NSCs after extraction with $NH_2OH \cdot HCl + HNO_3$ reached 95% and 98% of the total extractable Cd in racks 3 and 4, respectively. The Cd in the extract reagent and the residual Cd remaining on the NSCs after extraction with 0.4 mol/L $Na_2S_2O_4$ reached 107% and 109% of the total extractable Cd in racks 3 and 4, respectively. These are considered as acceptable confirmations of mass balance, given that the total extractable Cd (extracted by 10% HNO_3 for 24 h) and the Cd in the extract (extracted by the selective extraction reagent, including 0.01 mol/L $NH_2OH \cdot HCl + 0.01$ mol/L HNO_3 and 0.4 mol/L $Na_2S_2O_4$) plus residual (extracted by 10% HNO_3 after extracted by the selective extraction reagents) were based on analyses of different slides in each sampling rack.

The results about the extracted amount of Fe/Mn oxides and the associated Cd are summarized in Table 2. Hydroxylamine hydrochloride could remove most of Mn oxides (75%–88%), part of Fe oxides (37%–43%) and the associated Cd (51%–52%); sodium dithionite could remove most of Fe oxides (76%–77%), Mn oxides (98%), and the associated Cd (83%–86%).

Assume that each extraction reagent has extracted

a total amount of Cd that is bound to the extracted Fe/Mn oxides. This means that there will be no readsorption of the liberated metals on the residual solid phases after the extraction, and that each extraction reagent can remove Fe/Mn oxides selectively. Thus, each extraction process could be expressed as follows (Dong *et al.*, 2003a):

$$F = a \times M_{CdFe} + b \times M_{CdMn} \tag{2}$$

where *F* represents total amount of Cd removed by the individual extraction (μmol/m² slide surface); *M*_{CdFe} and *M*_{CdMn} are amounts of Cd bound to the extracted Fe and Mn oxides (μmol/m²), respectively; *a* and *b* are percentages of total extractable Fe and Mn removed by individual extractions. It is noted that scavenging is

expressed as the nominal surface area of glass slide per unit containing the NSCs, rather than the total surface area of the adsorbing phase. On the basis of data in Table 2, *M*_{CdFe} and *M*_{CdMn} were obtained by solving two equations based on results of the two extractions for each rack. The 10% HNO₃ can help remove Fe/Mn oxides, part of Al oxides and organic materials (Dong *et al.*, 2001b) which can also adsorb Cd; therefore, an equation group based on the results or extractions using hydroxylamine hydrochloride and sodium dithionite was established to solve the problem. The amounts of Cd scavenged by Fe oxides (*F*_{CdFe}) and Mn oxides(*F*_{CdMn}) were also estimated using *M*_{CdFe} and *M*_{CdMn} divided by the total of Fe and Mn

Table 2 Assessment of removal of Fe, Mn and Cd from the NSCs by selective extractions

Extractant ^b	Fe oxides		Mn oxides		Cd	
	Surface concentration ^a , μmol Fe/m ²	Removal, %	Surface concentration ^a , μmol Mn/m ²	Removal, %	Surface concentration ^a , μmol Cd/m ²	Removal, %
Rack 3						
10% HNO ₃ 24 h	12.0 ± 0.44	100	10.31 ± 0.86	100	1.02 ± 0.05	100
0.01mol/L NH ₂ OH·HCl + 0.01 mol/L HNO ₃ 30 min	4.40 ± 1.19	37	7.70 ± 1.17	75	0.52 ± 0.00	52
0.4 mol/L Na ₂ S ₂ O ₄ 40 min	9.10 ± 0.18	76	10.09 ± 1.20	98	0.87 ± 0.17	86
Rack 4						
10% HNO ₃ 24 h	10.57 ± 0.25	100	5.60 ± 0.54	100	0.90 ± 0.12	100
0.01mol/L NH ₂ OH·HCl + 0.01 mol/L HNO ₃ 30 min	4.50 ± 0.35	43	4.91 ± 0.44	88	0.46 ± 0.07	51
0.4 mol/L Na ₂ S ₂ O ₄ 40 min	8.11 ± 0.22	77	5.37 ± 1.85	98	0.75 ± 0.07	83

Notes: ^a Data are mean ± SD; ^b the extracted amount per m² of slide surface area for NH₂OH·HCl + HNO₃ and Na₂S₂O₄, based on analysis of the residual metals (remaining after exposure to the extract reagent) as extracted with 10% HNO₃

Table 3 Estimation of Cd scavenging through Fe, Mn oxides during development of the NSCs

	<i>M</i> _{CdFe} , μmol/m ²	<i>M</i> _{CdMn} , μmol/m ²	<i>F</i> _{CdFe}	<i>F</i> _{CdMn}
Rack 3	0.679	0.365	5.7 × 10 ⁴	3.5 × 10 ⁴
Rack 4	0.813	0.129	7.7 × 10 ⁴	2.3 × 10 ⁴

oxides in the NSCs (μmol/m²), respectively (Table 3). The results showed that the amount of Cd scavenging by Fe oxides (*F*_{CdFe}) exceeded that by Mn oxides (*F*_{CdMn}). In other words, Fe oxides contribute more than Mn oxides to Cd scavenging during the development of the NSCs. This result agrees closely with the independent studies of Dong *et al.* (2000, 2001a) who estimated Langmuir parameters for Pb and Cd adsorption to organic materials and metal oxides in lake NSCs based on the additive-adsorption and linear-regression models. An obvious difference was that the current research revealed that the Cd with lower concentration levels would be scavenged in a relatively consistent way under both conditions in the laboratory and the Nanhu Lake. Moreover, the results

conformed that the Cd's association with solid Fe phase exceeds that to Mn surface in the study lake water. The reactivity as well as the abundance of Mn oxides contributed much to Cd scavenging. This is due to the fact that the water selected to carry out the experiment was eutrophic, and thus had a relative low oxygen concentration which might affect the oxidization process and lower the reactivity of Mn oxides (Dong *et al.*, 2001a). Moreover, the scavenging property of Mn oxides in the NSCs that were developed in a eutrophic aquatic environment would be similar to that of abiotic Mn oxides, which was proved to have lower adsorption capacities than biogenetic Mn oxides that were developed in oligotrophic and mesotrophic environments (Lion *et al.*, 1982; Nelson *et al.*, 1995). Collectively, the above results confirmed predictions based on laboratory studies (Dong *et al.*, 2000, 2001a, 2003b); meanwhile, the results also provided a useful insight into the scavenging/adsorption mechanisms of Cd by the NSCs in aquatic environments.

3 Conclusions

Dynamic scavenging of Cd by Fe/Mn oxides on the NSCs was conducted during the development of the NSCs in an aquatic environment in laboratory conditions. The data fitted adequately well to the Elovich equation with $R^2 \geq 0.68$ ($n=20$). Linear regression analysis revealed that both Fe and Mn oxides contribute significantly to Cd scavenging while the NSCs were being developed. This demonstrated the importance of Fe/Mn oxides for trace metal transportation in aquatic environments.

To estimate the contribution of Fe/Mn oxides on the NSCs in controlling Cd scavenging from solutions, selective extraction techniques were employed to analyze the related mechanisms (Dong *et al.*, 2003a, b). The results showed that Fe oxides contribute the most to Cd scavenging by the NSCs, while the role of Mn oxides was relatively low. These results also revealed a strong influence of Fe oxides on Cd scavenging by the NSCs in natural environments, especially at low Cd concentrations. The estimated Cd scavenging mechanisms were in significant agreement with the previous studies based on the additive-adsorption and linear-regression models.

Selective extraction procedures were confirmed to be effective for investigating transport mechanism of trace-level heavy metals in water systems. Further research efforts would be focused on the detailed characteristics associated with Fe/Mn oxides.

Acknowledgements: The authors are grateful for valuable comments on the manuscript from the anonymous reviewers.

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(Received for review April 25, 2006. Accepted July 31, 2006)