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Stabilization of nickel and chromium in sewage sludge during aerobic composting

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Abstract

The speciation of heavy metals can play a major role in the toxicity of those metals in sewage sludge following land application. Tessier sequential extraction was used to investigate the changes in Ni and Cr speciation in sewage sludge treated by forced-aeration composting. Ni and Cr concentrations increased 30.4% and 36.0%, respectively, during the composting process, with H₂O and CO₂ volatilization being a major contributor to the change. It was found that the exchangeable, carbonate-bound, Fe–Mn oxide-bound, and organic matter-bound Ni and Cr were transformed to residual fractions. For Cr, the carbonate-bound, Fe–Mn oxide-bound, and organic matter-bound fractions were major contributors, while organic matter-bound Ni was a major contributor to the residual fraction. Composting appeared to reduce Ni and Cr availability by stabilizing the two metals and making them more stable and less mobile.

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1. Introduction

Rapidly expanding urban areas throughout the world are straining existing waste water treatment systems, many of which are decades old and rely on inefficient, out-of-date technology. To deal with growing populations and increasing water use and disposal, more waste water treatment plants will need to be built. Since a critical component of waste water treatment is solids removal, it is important that efficient and environmentally-sound options be developed for handling the resulting sewage sludge. Approximately 4.5×10^6 tonnes of sewage sludge were produced in China in 2001 [1] which, if not collected and treated, can pose a significant risk to human health as well as the environment. Both safe and economical methods must be utilized to dispose of, or use, the sludge materials.

Landfill, incineration and land application are the three primary methods of sludge handling. Landfill has the potential for groundwater contamination due to leaching and it is often difficult to find suitable, stable locations for landfills. Incineration

0304-3894/\$ - see front matter © 2006 Elsevier B.V. All rights reserved. doi:10.1016/j.jhazmat.2006.08.003 can contribute to air pollution and therefore may require expensive treatment systems to control emissions [2]. Compared to landfill and incineration, land application is a preferable and practical option for disposal of sewage sludge in China. Sewage sludge can be applied to arable land to improve soil fertility and soil physical properties, and this practice is inexpensive and easy to operate [3–5].

However, raw sewage sludge is being used less and less because it releases nitrogen and other chemical stressors that can reach ground water, resulting in phytotoxicity, and/or contributing to unpleasant odour problems [6]. To reduce these problems to acceptable levels, sewage sludge can be composted. Heavy metals (e.g. Cu, Zn) that may occur in sewage sludge, can affect waste treatment and processing, and limits the use of these waste materials as soil amendments [7–11]. However, most sewage sludge contains low levels of pollutants and therefore meets the strict regulations of many countries [12,13].

Current legislation in several countries limits the use of sewage sludge in agriculture. Legislation uses heavy metals concentrations in wastes and soils to establish limits, and recommends that soil pH be maintained at 6 or higher [14]. Some methods, such as acid treatment, electrodialytic remediation and bioleaching, when used prior to land application, can result in the

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dissolution and removal of a significant fraction of heavy metals from sludge [2,15–18]. Nevertheless, many of these approaches are marginally cost-effective or difficult to implement in developing countries. Those processes may also result in a significant loss of the nutritional value of sewage sludge [19].

The potential environmental hazard of a heavy metal is related not only to its total concentration in sewage sludge, but also to its chemical speciation [20-23]. Therefore, toxicity cannot be evaluated by determination of total metal concentration alone [24]. Qiao and Ho [25] found that the addition of red mud prior to composting not only reduced leachability and plant availability of heavy metals in the compost, but also significantly reduced the levels of metals extracted by acid digestion. Li et al. [26] reported that the addition of coal fly ash and phosphate rock to sewage sludge was effective for heavy metal stabilization during composting. Several researchers have addressed the issue of heavy metal availability in sewage sludge, and quantitatively evaluated changes in metal concentrations during composting. Nevertheless, microorganisms break down organic matter and produce carbon dioxide, water, heat, and humus, the relatively stable organic end product in the process of composting. Due to the decrease in volume and weight of the raw material after composting, changes in phytotoxicity of the metals cannot be assessed solely by concentration. And the humification of organic matter will have a significant effect on the physicochemical form of existence of heavy metals during composting [26]. There is a need for more quantitative data on heavy metal speciation in sewage sludge during composting.

In this study, a large-scale composting experiment was conducted to evaluate Ni and Cr speciation changes. The study focused mainly on the influence of composting on the distribution of Ni and Cr available and nonavailable fractions in order to determine if composting may reduce or enhance the phytotoxicity of the metals.

2. Materials and methods

Dehydrated digested sewage sludge was collected from the Beixiaohe Wastewater Treatment Plant, Beijing. The moisture content of sewage sludge in the reactor had been decreased to about 60% through mixing with a bulking agent at a 1:1 v/v ratio [27]. As an inorganic and porous material, the bulking agent is ideal for large scale composting because of its strong water absorbability, ability of deodorizing, and the feature that it can be recycled. When mixing the sludge with bulking agent at the ratio 40-60%, the moisture content of sludge could be modified to be appropriate for compost. The pile dimensions were $2.5 \text{ m} \times 1.5 \text{ m} \times 1.4 \text{ m}$ (length \times width \times height). The treatment process was controlled by a forced-aeration static pile system (temperature feedback control system used to monitor the temperature of compost pile) and the sewage sludge was composted for 31 days. Temperature was automatically monitored with a real-time and on-line monitoring system everyday. The three phases of composting were: the mesophilic phase (27.3–50 °C, 0-5 days), the thermophilic phase (50-61.2 °C, 5-25 days), and the cooling phase (25–31 days). Samples were taken from the pile at days 0, 2, 5, 9, 14, 19, 25 and 31 for chemical testing.

The samples were air-dried, crushed and passed through plastic sieves (0.075 mm mesh for pH, DOC, and 1.0 mm mesh for heavy metals) for chemical testing.

Tessier sequential extraction has been used previously to determine the metal fractions in soil samples [28-30]. The method yields five different solutions: exchangeable (1 mol L^{-1} MgCl₂, pH 7); bound to carbonates (1 mol L^{-1} NaOAc/HOAc, pH 5); bound to Fe–Mn oxides (0.04 mol L^{-1} NH₂OH·HCl in 25% HOAc); bound to organic matter (0.02 mol L^{-1} HNO₃ in 30% H₂O₂, pH 2; $3.2 \text{ mol } \text{L}^{-1}$ NH₄OAc in 20% HNO₃); residual (digested with concentrated HNO₃ + HClO₄). The concentrations of Ni and Cr were determined using flame atomic absorption spectrophotometry (Analytik Jena AG, Germany; wavelength: Cr 357.87 nm, Ni 232.00 nm). The compost samples were extracted with purified water at a solid:water ratio (w/v) of 1:10 by shaking in a 25 °C bath for 1 h. The suspensions were centrifuged at 10,000 rpm and the supernatants were filtered through 0.45 µm membrane filter papers. The pH was measured with a calibrated pH meter (REX PHSJ-4A, China). Dissolved organic carbon (DOC) concentration was determined with a total organic carbon analyzer (Apollo 9000, USA). All measurements were made in triplicate for each sample and results are presented as the mean of the three replicates. Correlation analysis was conducted with Statistical Analysis System software.

3. Results and discussion

3.1. Concentrations and percentages of Ni fractions in compost

The concentration of total Ni increased throughout the composting period (Table 1). There was a moderate increase in total Ni during the mesophilic phase (2.29 mg/kg). The greatest increase in Ni (19.53–23.34 mg/kg) occurred during the thermophilic phase. At the end of the composting process, the total concentration of Ni was 23.45 mg/kg.

The concentrations of exchangeable, carbonate, organic, and residual Ni increased during the thermophilic phase of compositing (Table 1). Although there was a little drop in the concentration of exchangeable Ni, it increased during the thermophilic phase, reaching the maximum concentration (3.95 mg/kg) at the end of the phase (day 25). Carbonate and organic Ni concentrations did not increase steadily, but dropped somewhat before rising again to reach a maximum on day 19; these Ni components then decreased through the end of the thermophilic and cooling phases. The final concentration of carbonate Ni was actually lower than the initial concentration. Residual Ni continued to increase throughout the composting period, with a maximum concentration on day 31. The concentration of Fe-Mn oxide-bound Ni fluctuated during composting, ending at a lower level (relative to the initial concentration) at the end of the thermophilic period and reaching the lowest concentration (2.71 mg/kg) at the end of composting.

The relative percent of total Ni accounted for by the five Ni fractions shifted during composting process. The greatest change occurred for residual Ni, which increased from 9.45% to

Table 1
Concentration and percent of each Ni fraction during composting

Day	Total (mg/kg)	Exchangeable		Carbonate		Fe–Mn oxide		Organic		Residual	
		mg/kg	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg	%
0	17.24	2.67	15.5	3.82	22.2	3.47	20.1	5.65	32.8	1.63	9.45
2	17.36	2.83	16.3	3.39	19.5	3.61	20.8	5.43	31.3	2.10	12.1
5	19.53	2.96	15.2	3.77	19.3	3.09	15.8	6.00	30.7	3.71	19.0
9	20.55	3.24	15.8	3.89	18.9	3.18	15.5	5.87	28.6	4.37	21.3
14	21.77	3.43	15.8	3.93	18.1	3.32	15.3	6.02	27.7	5.07	23.3
19	23.06	3.36	14.6	4.04	17.5	3.44	14.9	6.24	27.1	5.98	25.9
25	23.34	3.95	16.9	3.88	16.6	3.09	13.2	6.17	26.4	6.25	26.8
31	23.45	3.55	15.1	3.59	15.3	2.71	11.6	6.08	25.9	7.52	32.1

%, percent of each fraction with respect to the total Ni content.

Table 2

Concentrations and percent of each Cr fraction during composting

Day	Total (mg/kg)	Exchangeable		Carbonate		Fe-Mn oxide		Organic		Residual	
		mg/kg	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg	%
0	33.76	1.95	5.8	0.98	2.9	0.40	1.2	20.5	60.7	9.93	29.4
2	33.77	1.99	5.9	1.12	3.3	0.46	1.4	19.7	58.3	10.5	31.1
5	34.52	1.63	4.7	1.07	3.1	0.52	1.5	19.6	56.8	11.7	33.9
9	36.66	1.37	3.7	1.03	2.8	0.46	1.3	20.1	54.8	13.7	37.4
14	37.56	1.33	3.5	0.90	2.4	0.33	0.9	19.3	51.4	15.7	41.8
19	39.91	1.38	3.5	0.86	2.2	0.27	0.7	19.9	49.9	17.5	43.8
25	40.29	1.25	3.1	0.85	2.1	0.29	0.7	16.7	41.4	21.2	52.6
31	44.03	1.50	3.4	0.88	2.0	0.35	0.8	18.9	42.9	22.4	50.9

%, percent of each fraction with respect to the total Cr content.

32.1%. The relative percent of exchangeable Ni essentially did not change, while the percent of the other fractions all decreased during composting. Actual proportions fluctuated during the 31day composting period.

3.2. Concentrations and percentages of Cr fractions in compost

There was only a slight increase in total Cr during the mesophilic period (Table 2). During the thermophilic period, the Cr concentration increased from 34.52 to 40.29 mg/kg. Whereas the total Ni concentration changed little during the cooling phase, total Cr climbed to 44.03 mg/kg at the end of composting process. Changes in exchangeable, carbonate, Fe-Mn oxide, and organic Ni concentrations were not substantial during composting, although levels tended to go up and down and the final concentrations of all were lower. As with Ni, residual Cr increased substantially throughout the composting period. Initial residual Cr (9.93 mg/kg) accounted for 29.4% of initial total Cr, while final residual Cr (22.4 mg/kg) accounted for 50.9% of total Cr. The relative percent of other fractions proportionally decreased. The majority of total Cr at both the beginning and end of the composting period was comprised of organic-bound Cr and residual Cr.

3.3. Changes of pH and DOC during composting

Compost pH decreased from 6.2 to 5.9 early during the mesophilic phase, but began to rise on day 2 (Fig. 1). The



Fig. 1. Variation of pH in the pile during composting of sewage sludge.

pH reached a plateau and remained relatively steady (between approximately 6.5 and 6.7) during the thermophilic phase and began to increase again on day 19. At the latter stage of the composting process, pH rose to a maximum value of 7.5. To determine if the partitioning of Cr and Ni into different fractions is dependent upon the pH of the compost, linear regression analysis was conducted (Table 3). The distributions

Table 3

Results of regression analysis between pH and the percentage of Cr and Ni fractions in the compost (n = 8)

Metal	Correlation coefficients derived using each fraction (R)								
	Exchangeable	Carbonate	Fe-Mn oxide	Organic	Residual				
Ni	+0.01	-0.84^{**}	-0.94**	-0.86**	+0.90**				
Cr	-0.84^{**}	-0.87^{**}	-0.71^{*}	-0.94^{**}	+0.94**				

P*<0.05; *P*<0.01.



Fig. 2. Variation of dissolved organic matter (DOC) in the pile during composting of sewage sludge.

of carbonate-bound, Fe–Mn oxide-bound, and organic matterbound Ni and Cr were negatively correlated with pH, and the distributions of residual Ni and Cr were positively correlated with pH. The pH was significantly negatively correlated with the distribution of exchangeable Cr. However, no correlation was found between pH and the distribution of exchangeable Ni.

The DOC generally decreased during the composting process, although there was a slight increase during the second half of the mesophilic phase (Fig. 2). By day 19, DOC had fallen to 5000 mg/kg, and remained at that level for the remainder of the composting period. To determine if the partitioning of Cr and Ni into different fractions is dependent upon the DOC of the compost, linear regression analysis was conducted (Table 4). The distributions of carbonate-bound, Fe–Mn oxide-bound, and organic matter-bound Ni and Cr were positively correlated with DOC, and the distributions of residual Ni and Cr were negatively correlated with DOC. The DOC was significantly positively correlated with the distribution of exchangeable Cr. However, no correlation was found between DOC and the distribution of exchangeable Ni.

Bolton and Evans [31] reported that dissolved organic matter in material leached from municipal wastes was an important factor in controlling solubility of trace metals. DOC can form stable, soluble complexes with heavy metals [32,33]. Persistence of soluble organo-metal complexes in compost added to soil could increase potential toxicity since soluble material will tend to be more bioavailable to receptor organisms. The hazards of heavy metals in compost may be reduced by decreasing the DOC concentrations.

The exchangeable fractions of heavy metals tend to be the most bioavailable and phytotoxic. Exchangeable fractions can only be released by increasing the ionic strength of the medium. Carbonate-bound metals are very sensitive to pH fluctuations and tend to leach at an increasing rate as the pH decreases. Metals

Table 4

Results of regression analysis between DOC concentration and the percentage of Cr and Ni fraction in the compost (n=8)

Metal	Correlation coefficients derived using each fraction (R)								
	Exchangeable	Carbonate	Fe-Mn oxide	Organic	Residual				
Ni	-0.01	+0.91**	+0.82*	+0.98**	-0.91**				
Cr	+0.92**	+0.86**	+0.83*	+0.89**	-0.91**				

 $^{*}P < 0.05; ^{**}P < 0.01.$

bound to Fe–Mn oxides are unstable under reducing conditions [22]. Ho and Qiao [34] reported a close relationship between availability to plants and metals in exchangeable, carbonate and Fe–Mn oxide-bound fractions. It is likely, therefore, that the heavy metals present in these fractions are also the most available to plants.

3.4. Transformation of metal fractions during sludge composting

The results of these studies indicated total Ni and Cr concentrations rose steadily during composting, resulting in final levels approximately 130.4% and 136.0%, respectively, in raw sludge concentrations. During composting, the microbial community converts easily-degradable organic matter to more stable and hydrated fractions and where possible to inorganic products, producing heat in the process [35]. Since the heavy metals are non-biodegradable, the proportion of inorganic matter tended to increase as organic matter decomposed. Based on data collected during this study, it appears that the total concentrations of heavy metals in sewage sludge increased due to the volatilization of the gases such as H_2O , CO_2 when the organic matter decomposed.

In order to assess the potential hazard posed by the application of sewage sludge to land, the heavy metal concentrations are usually determined. While this may be appropriate to estimate long-term risk, it may result in an inaccurate picture of short- and medium-term risks because concentrations alone do not reflect potential mobility and availability of the metals under field conditions. Sequential extraction can provide more information about the chemical speciation of a metal [11,30].

By comparing the percentages of the five fractions of heavy metals in the raw sewage sludge to the percentages in the final product, relative proportional changes during composing are apparent (Fig. 3) [23,36]. Studies on the chemical fractions of Ni in sewage sludge have indicated that organic matter-bound fractions comprised the majority of Ni in raw material, followed by carbonate and Fe–Mn oxide-bound fractions. However, after composting, the residual fraction contained the greatest amount of Ni. Results of the Cr composting study were similar.

By composting, the proportions of exchangeable, carbonatebound, Fe-Mn oxide-bound and organic matter-bound Ni and Cr decreased while the proportion of residual Ni and Cr increased (Fig. 3). The results demonstrate that the four former fractions of Ni and Cr are transformed during composting to the residual fraction. While the increased quantity of residual Ni likely comes from the carbonate, Fe-Mn oxide, and organic matterbound Ni, the increased amount of residual Cr comes primarily from the organic matter-bound Cr. There is a close relationship between the metal fraction available to plants and its exchangeable, carbonate and oxide-bound fractions. The metal present in those fractions is considered to be the more available and thus potentially more phytotoxic. Ni and Cr bound to organic matter and residual Ni and Cr are not considered to be available to plants. The concentrations of non-available fractions decreased during the composting stage (Fig. 3). During composting, the stabilization of Cr and Ni in sewage sludge offers the potential to reduce the risks of the heavy metals in the compost.



Fig. 3. Percent change of total Ni and Cr accounted for by Cr and Ni fractions after composting of sewage sludge: (F1) exchangeable; (F2) carbonate-bound; (F3) Fe–Mn oxide-bound; (F4) organic matter-bound; (F5) residual. Percent change was determined by subtracting percentages in the raw sludge from percentages in the final product (compost).

4. Conclusions

During the composting process, the distributions of carbonate-bound, Fe–Mn oxide-bound, and organic matterbound Ni and Cr were negatively correlated with pH, and the distributions of residual Ni and Cr were positively correlated with pH, while the DOC exhibited an opposite phenomenon. After the composting process, Cr and Ni levels rose in sewage sludge; their total concentrations increased by 30.4% and 36.0%, respectively. The distribution of exchangeable, carbonate-bound, Fe–Mn oxide-bound, and organic matterbound Cr and Ni decreased while distribution of residual fractions increased substantially. This transformation results in a reduction of available Cr and Ni, and increases the stability of both metals in the sewage sludge.

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