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Effect of compost aging on zinc adsorption characteristics

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ABSTRACT

One approach to improving the performance of biofiltration based treatment systems is to amend filtration media with compost to improve metal sorption. The effect of aging these amended compost particles on zinc sorption is investigated using batch and column tests. Batch tests showed that aged compost had a lower rate of zinc sorption but a similar zinc sorption capacity compared with fresh compost. Pseudo-second-order kinetics was found to best fit the batch experimental data. The intraparticle diffusion plot shows that there are differences due to the effect of aging of compost and that intra-particle diffusion is the dominant rate controlling mechanism. Column studies show that aging the compost decreased the sorption capacity by 22% (from 16 mg g⁻¹ to 13 mg g⁻¹). It is evident that aging compost media have an effect on the magnitude and kinetics of zinc adsorption. This was also evidenced in the decreased CEC and peak intensities of relevant spectra in the FTIR of the aged compost.

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Introduction

Water Sensitive Urban Design (WSUD) emphasises a more decentralised approach of stormwater treatment systems and often utilise biofiltration media amended with compost. Sorption of heavy metals by woody biofiltration materials (e.g. compost, mulch) wastewater and stormwater treatment has been reported in the literature [1,2].

Woody materials are naturally rich in organic matter and it is well documented that organic matter in soil plays a key role in heavy metal adsorption [3,4]. Structurally the organic matter contains different organic functional groups including carboxyl, carbonyl, phenyl hydroxyl, amino, imidazole, sulfhydryl, and sulfonic groups. These groups provide a wide range of reactive surfaces that are able to bond with heavy metals.

Aging is a natural process where woody amended materials within an operating biofiltration system is subject to a performance change through time or with use, as a result of the degradation processes of the woody amendments. Biofilters are designed to operate over long time periods using woody materials as an amendment. However these woody materials are dynamic and it is a well-known phenomenon [5–7] that the physicochemical properties of woody materials will change with time under different operational conditions. Aging of woody biofiltration

E-mail addresses: othman.mashaqbeh@rss.jo (O.A. Al-Mashaqbeh), robert.mclaughlan@uts.edu.au (R.G. McLaughlan). materials may cause leaching of organic matter and therefore change their physicochemical properties and consequently affect their performance for the removal of heavy metals. The effect of aging these materials on metal sorption is not taken into account in the design of field-based treatment systems and has not been widely studied. Sciban and Klasnja [8] found that leaching organic matter does not impact the adsorption of heavy metal ions by woody adsorbents using batch experiments. Another study under high flow conditions using different sizes of compost media have shown the leaching DOC from columns does not stop the metal sorption [9]. Conversely pesticide sorption was found to be affected by aging [10]. It is clear from these studies that there is a crucial need to understand the effect of the aging process (such as long term organic leaching) of woody biofiltration materials on heavy metal sorption.

The goals of this paper are to determine the impact of accelerated aging of compost on zinc sorption from an aqueous solution using batch and column experiments. Various kinetic models, namely the pseudo-first-order, pseudo-second-order and intra-particle diffusion were used to evaluate the adsorption kinetics. It is expected this study will be useful to other researchers and engineers seeking knowledge about the influence of aging on the treatment performance of compost biofiltration amendments.

Experimental methodology

Materials

The compost (Composted Leaf mulch, Australian Native Landscapes) was compliant with AS4454 and consisted of a screened

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composted mulch (nominally 10–35 mm) sourced from garden organics. Garden organics includes putrescible garden organics (grass clippings); non-woody garden organics; woody garden organics; trees and limbs; stumps and root balls. The compost was dried at 45 °C for 48 h and then ground using a hammer mill until the required particle size range was obtained. The particle size of compost is 1.18 mm which passed through a 2.36 mm sieve but was retained over a 1.18 mm mesh sieve.

Aging of the compost

To simulate and accelerate the aging process, the compost samples were leached with Milli-Q water using 24 h sequential batch leaching as described in McLaughlan and Al-Mashaqbeh [11]. This sequential batch leaching procedure was repeated every 24 h for 19 days until the DOC of the leachate water had reached a steady-state of DOC release with concentrations below 20 mg L⁻¹. Sample leaching beyond 19 days was expected to have minimal benefit in terms of reducing the leachable DOC content of the compost. All the leached compost samples were subsequently dried at 45 °C for 48 h prior to use (referred to as aged compost).

Analytical methods

All water samples were measured for electrical conductivity (EC) and pH (TPS WP-81). Samples were filtered with a 0.45 µm cellulose membrane filter and preserved by acidifying to pH < 2using 1 N HNO₃ [12]. The preserved samples were analysed for zinc concentrations using Atomic absorption spectrometry (GBC AAS932, air/acetylene flame). All plastic and glassware in contact with samples were acid washed for 24 h with 0.1 M HNO₃ and rinsed with Milli-Q water, unless otherwise noted. Cation Exchange Capacity (CEC) of different adsorbents was measured using saturated ammonium extractants at pH 7 [13]. Compost particle structure characteristics (specific surface areas, pore volume and size) were measured by Particle Analysis Service, CSIRO using BET/N₂ (Micromeritics Tristar 3000). Lignin, cellulose, and hemicellulose were determined also in the Wagga Wagga Feed Quality Testing Laboratory (Department of Primary Industries, NSW). Total carbon and nitrogen for all solid materials were determined in duplicate using a TruSpec Carbon Nitrogen Determinator. Ash content was measured using the method for Total Volatile Solids at 550 °C [12].

Fourier transforms infrared analysis (FTIR)

In order to determine which functional groups were responsible for metal sorption, the FTIR spectra of the compost and aged samples were recorded using a KBr disk in conjunction with a Perkin-Elmer infrared spectrophotometer having a resolution of 4 cm^{-1} and range between 4000 and 400 cm⁻¹. Duplicate KBr disks were prepared by mixing 0.003 g of compost and 0.005 g of aged compost dried sample with 0.25 g KBr crystals, the resulting mixture being ground to a fine powder. Finally, the mixture was pressed into a KBr disk and used as such for FTIR studies.

Sequential batch sorption test

The kinetics experiments used a sequential batch extraction methodology [14]. One gram of the specified size of compost was added to 2 L glass bottles filled with a solution of zinc (5 mg L⁻¹). The vessels were shaken on an orbital shaker at 200 rpm for 120 h. Samples (10 mL) were withdrawn at predetermined time intervals and analysed for zinc. The total volume of all withdrawn samples was 60 mL which had minimal effect on the liquid–solid ratio (decreased from 2000:1 to 1940:1). The initial pH for each vessel

was initially set to 5.5 but was not adjusted during the sorption period and was measured immediately prior to each sampling event. Blank experiments with zinc solution without sorbent were carried out to check possible losses of the examined metal. All the sorption experiments were carried out at laboratory temperature 20 ± 1 °C.

All the batch experiments were carried out in duplicate and the results given as an average. Metal sorption at different times was calculated using the mass balance between the solid and the solution as follows:

$$q_t = (C_0 - C_t) \frac{V}{M} \tag{1}$$

where q_t the amount of metal ion adsorbed at time t (mg g⁻¹), C_t is the liquid phase metal ion concentration at time t (mg L⁻¹), C_0 the initial liquid phase metal ion concentration (mg L⁻¹), V the initial solution volume (L) and M is the mass of sorbent (g). All sorption tests were performed in duplicates and the standard deviation values of duplicate samples were less than 1.

Column specification and sorption/desorption test

The columns were constructed in PVC class 18 with an inner diameter of 5.3 cm and a length of 58 cm, giving a cross-sectional area for filtration of 22 cm². Since the ratio of column diameter to median particle diameter is high, the effects of channelling are expected to be negligible [15]. Columns were constructed by sealing a PVC cap at the end of the column using Silastic. A layer of plastic mesh and then a 2-3 cm depth of 3 mm diameter glass beads was placed upon the end cap at the influent end of the column. A plastic mesh was placed over the glass beads to support the filtration media. Both columns had a mix of 68 g of 1.18 mm compost and aged compost with 1663 g of 0.6 mm glass beads (\approx 20–25%, v/v compost). The mix was wetted and then added in increments using continuous column vibration without any compaction over the filtration media surface [16]. The final depth of filtration media in the columns was approximately 55 cm with a total bulk density of 1.5 g cm^{-3} . The sorption columns were initially saturated with distilled water for 2 h, then zinc solution $(\sim 5 \text{ mg L}^{-1})$ was pumped using peristaltic pump (Masterflex Model 7553-85) from a 45 L PVC container into the column in up flow mode. The average flow rate was approximately \sim 12 mL min⁻¹. Influent and effluent samples were taken at different time intervals and analysed for EC and pH. Samples (40 mL) were filtered with a 0.45 µm cellulose membrane filter and preserved by acidifying to pH < 2 using 1 N HNO₃ [12]. All glassware in contact with samples was acid washed for 24 h with 0.1 M HNO₃ and rinsed with Milli-Q water. Zinc chloride was added to distilled water to form the zinc effluent with pH adjustment using dilute HCl and NaOH solutions. Metal salt and other reagents used were all of analytical grade.

To determine the reversibility of the sorbed metal to clean water during actual operation in the field, once the zinc effluent concentration matched the influent concentration (5 mg L⁻¹), a desorption cycle was started using distilled water (pH of 5.5, EC < 3.5) at a similar flow rate (~12 mL min⁻¹). The column sorption experiments were continuously carried out for three weeks with an influent (5 mg L⁻¹) of zinc solution with a pH ~ 5.0 to prevent zinc hydroxide precipitation. The performance of a filtration based treatment system for removing dissolved contaminants is often evaluated through the use of a breakthrough curve. The zinc breakthrough curve for a column is determined by plotting as the ratio of effluent zinc concentration (*C*) to inlet zinc concentration (*C*₀) against either elapsed time, effluent volume or pore volume for a given filtration bed height. The maximum sorption capacity of zinc onto compost and aged compost in the

columns was estimated using "Simpson's Rule" based on the mass calculated from the area over the curve divided by the mass of compost (68 g) in the column. Total amounts of zinc desorbed during water flushing were also calculated using Simpson rule.

Modelling of kinetic batch test data

Three different kinetic models were used for correlation of the sorption data. These were the pseudo-first order, pseudo-second-order and intra-particle (parabolic) diffusion.

The pseudo first-order rate expression can be expressed as follows [17]:

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \tag{2}$$

where q_e and q_t are the amounts of zinc ions adsorbed onto the sorbent (mg g⁻¹), at equilibrium and at time *t*, respectively, and k_1 is the rate constant of first-order (h⁻¹).

A pseudo-second-order equation based on the sorption capacity of solid phase was also used. This model is based on the assumption that adsorption follows second order chemisorption and is expressed as [18]:

$$\frac{dq_t}{dt} = k_2 (q_e - q_t)^2 \tag{3}$$

where k_2 is the rate constant of second-order sorption (g mg⁻¹ h⁻¹) and other terms as previously described. The fitted parameters for the first and second order equations were calculated using least squares regression (Microsoft Solver). This approach was used rather than linearisation of the equations since linearisation may result in improperly weighted data points during the analysis [19]. The mathematical models were tested for best fit using regression co-efficient (R^2), standard error (SE) as well as visually plotting the data.

An intra-particle diffusion model describes the relation between sorption and the square root of time. The root time dependence is known as a Weber Morris plot with the intraparticle diffusion rate equation expressed as [20]:

$$q_t = k_{ID} t^{0.5} + c \tag{4}$$

where k_{ID} is a diffusion rate parameter and c gives information about the boundary layer.

Results and discussion

Physicochemical properties

The properties of the compost and aged compost particles and their leaching characteristics are detailed in Table 1. Since the compost was a relatively woody mixture it would be expected to have characteristics closer to wood. The compost particles had a high ash content (10.9%) and low C:N ratio which reflects the high mineral content (e.g. Si, Ca) often found in leaves [21]. The C:N ratio increased after the aging process from 50 to 70. This suggests the percent of leached N is higher than the percent leached C from the compost.

It is very clear from Table 1 that the aging process has leached part of the CEC of the compost as it dropped by more than 50%. However, the lignin, cellulose and hemicellulose increased as a percentage in response to the residue fraction which decreased by 33%. Also, the BET surface area for compost and aged compost did not change while the pore size and volume slightly decreased. This suggests that the aging of compost may only have a minor effect on the internal pores structure of compost.

Table 1

Characteristics of adsorbents.

Item	Compost	Aged compost
Sorbent treatment	Primary washing	Long term leaching
Particle size (mm)	1.18	1.18
C/N ratio	50	70
BET (m ² /g)	0.98	0.98
Pore volume (cm ³ /g)	0.0036	0.0031
Pore size (nm)	14.8	12.7
CEC (cmol kg $^{-1}$)	85.6	33.6
Ash (%)	10.9	ND
Lignin (%)	22	28
Cellulose (%)	27	35
Hemicellulose (%)	6	7
Residue (%)	45	30

Fourier transform infrared analysis (FTIR)

The main components in a wood cell are cellulose, hemicellulose and lignin [17] and some pectin and extractives (mainly fat, fatty acids, fatty alcohols, phenols, terpenes, steroids, resin acids, rosin and waxes). Sorption of heavy metal ions is believed to occur by bonding the metal ion with the hydroxylic, carboxylic and phenolic groups on the adsorbent. Most woody material contains these functional groups such as carboxylic (primarily present in pectin and hemicellulose), phenolic (lignin and extractives) and to some extent hydroxylic (cellulose, hemicellulose, lignin and pectin) and carbonyl groups (lignin) [7,21].

The spectra for the compost and aged compost showed a number of different peaks, indicating the complex nature of the sorbent (Fig. 1) and were similar to other FTIR images of different composts and woody fibre materials in the literature [22–27].

A key difference between the compost and aged compost was the increased peak intensity in the compost sample evident in the FTIR spectra. The peaks represent specific functional groups and structural elements on the compost surface with higher peak intensity due to the higher concentration of molecules in the sample undergoing FTIR [24]. The major areas where peak intensity differs correlate with functional groups such as C–N (1230 cm⁻¹), C=O (1675 cm⁻¹), –OH (3426 cm⁻¹) which have been documented to play a role in heavy metal sorption in composts [22–27]. This suggests that aging compost decreased the prevalence of some functional groups on the compost surface related to metal sorption.

Influence of aging and particle size

The zinc sorption for both the compost and aged compost are shown (Fig. 2).



Wavenumber cm⁻¹





Fig. 2. Zinc sorption onto compost and aged compost particles.

The final equilibrium sorption of compost and aged compost are relatively similar at equilibrium (7.2 mg g^{-1} , 7.1 mg g^{-1}). The magnitude of these results for zinc sorption are lower than those reported by Jang et al. [28], who obtained the sorption capacity by mulch, at pH 5.0 of 185 mmol/g (12.1 mg g^{-1}) and at pH 6 of 0.187 mmol/g (12.2 mg g^{-1}), but higher than those reported by Gibert et al. [29] for municipal compost for zinc of 3.9 mg g^{-1} at pH 6.5. The relatively small differences in our final equilibrium zinc sorption are consistent with findings by Sciban and Klasnia [8]. who examined the effect of leaching of organic matters from wood sawdust (0.5-1 mm). They found that soaking and radical washing changed the copper sorption from 0.022 mmol g⁻¹ untreated to 0.020 mmol g^{-1} soaked and 0.023 mmol g^{-1} washed. They concluded that leached organic matters do not prevent the adsorption of heavy metal ions by woody adsorbents. The equilibrium zinc sorption after aging shows relatively small differences (\sim 10%) to the compost samples.

Sorption kinetics

The general trend evident in the kinetics data is similar to other studies with a faster initial sorption rate and a slower rate for the rest of the sorption time. These final slow sorption rates have been observed on many different sorption studies and generally three possible causes are applied: (1) diffusion to internal sites, (2) surface precipitation, or (3) adsorption to sites that have a slower rate due to low affinity [30,31].

The sorption kinetic curves of the compost and aged compost differed in the rate of adsorption (Fig. 2). In order to better



Fig. 3. Zinc sorption onto compost with observed and predicted data.



Fig. 4. Zinc sorption onto aged compost with observed and predicted data.

understand and quantify the sorption kinetics mechanisms of aging on zinc sorption onto compost and aged compost various kinetic models were fitted to the data (Figs. 3 and 4; Table 2).

A pseudo-second-order kinetic model was found to be a better overall fit for the data than the pseudo-first-order-model with higher R^2 and lower SE, however the equilibrium sorption capacity (q_e) was better predicted by the pseudo-first-order model due to the slight desorption at the later stage of the test. This is consistent with a review study [18] which showed that in most sorbentsorbate published kinetic data the pseudo-second-order model is more suitable than a pseudo-first order model using linear regression method. Importantly, the rate constants in both models were lower for the aged compost. This shows that aging has decreased the rate of zinc sorption.

Intra-particle diffusion equation

The adsorption of zinc onto compost follows four main steps (i) migration of the zinc from the bulk solution to the surface of boundary layer surrounding the sorbent (ii) diffusion of the zinc across the boundary layer surrounding the sorbent particle (film diffusion), (iii) diffusion of zinc within the pores of the sorbent particle (intra-particle diffusion), and (iv) adsorption of the zinc on the sorbent surface (internal pores) [32].

A linear relation of q_t vs. $t^{0.5}$ is obtained if the sorption process is considered to be influenced by diffusion in the spherical particles and convective diffusion in the solution [33]. These linear relations may have different slopes due to separate stages of diffusion occurring in the adsorption processes [34–36]. These studies indicate that the first sharper line is the external surface adsorption or instantaneous adsorption, where external diffusion is ratecontrolled. The second line is the gradual adsorption stage, where intra-particle diffusion is rate-controlled. The third portion is the final equilibrium stage, where intra-particle diffusion starts to

Table 2	
Fitted and estimated parameters for the various kinetic models.	

Parameters	Compost	Aged compost
Pseudo-first-order model		
$k_1 (h^{-1})$	0.11	0.11
$q_e (\mathrm{mg/g})$	2	2.2
R^2	0.934	0.998
Pseudo-second-order model		
$k_2 (g m g^{-1} h^{-1})$	0.41	0.06
$q_e (mg/g)$	7.31	7.35
R^2	0.998	0.999
Intra-particle diffusion equation		
$k_{ID} (\text{mgg}^{-1} \text{h}^{-1(0.5)})$	1.73	1.50
Intercept	1.04	0.45
R^2	0.976	0.996



Fig. 5. Intra-particle diffusion model kinetics of compost and aged compost.

slow down due to the extremely low solute concentration in solution. Using Eq. (4), q_t was plotted against $t^{0.5}$ for compost and aged compost, and intra-particle diffusion rate constants (k_{ID}) were determined from the slope of the plots (Fig. 5 and Table 2).

The first stage, film diffusion, does not appear because the initial sorption happened very quickly (within 30 min) however the second stage (gradual adsorption due to intra-particle diffusion) is apparent and is followed by the final equilibrium stage. The calculated intra-particle diffusion rate (k_{ID}) value slightly decreased from 1.73 for compost to 1.50 (mg $g^{-1} h^{-(0.5)}$) for aged compost (Table 2 and Fig. 5). This is might be due to the slight decrease in the pore size and volume resulting from aging the compost. Furthermore, according to Weber and Morris, if the rate-controlling step is solely intra-particle diffusion, then a plot of q_t vs. $t^{0.5}$ should yield a straight line with the intercept (C) passing through the origin with any deviation of the intercept from the origin indicating that different types of diffusion (e.g. film diffusion and intra-particle diffusion) may be rate controlling. The higher the value of C, the thicker is the boundary layer [37]. The intercept value (C) for compost is slightly higher than aged compost (Table 2) but still a relatively small fraction of the total sorption (10-15%). This shows that the process controlling sorption in both compost and aged compost involves both film diffusion and intra-particle diffusion but that intra-particle diffusion dominates.

Column sorption/desorption

Sorption batch experiments have traditionally been used to provide preliminary results about the sorption capacity and kinetics of solute sorption by any adsorbents. However, the batch results cannot give accurate scale-up data to be used for continuous flow columns which best represent treatment processes used in the field. Therefore, two column sorption experiments with a sorption/desorption cycle were carried out with a column packed with compost and aged compost to delineate the effect of compost aging on zinc adsorption characteristics.

The breakthrough curves (BTC) for sorption of zinc onto the compost and aged compost from $C/C_0 = 0$ to 1 are shown in Fig. 6. The breakthrough curve for zinc in the compost and aged compost column showed a sharp front with a sigmoidal (S-shaped) breakthrough curve, however the BTC of aged column exhibit slight tailing.

The breakpoint ($C/C_0 = 0.1$) for aged compost column occurs at 89 L while in the compost column it is delayed until 135 L. This suggests the performance of the aged compost column, in terms of time till the breakpoint is reached, is lower than compost column. The BTC also showed that the sorption capacity of zinc decreased



Fig. 6. Breakthrough curves for sorption and desorption of zinc onto the compost and aged compost.

from 16.9 mg g⁻¹ for the compost column to 13.1 mg g⁻¹ for the aged compost column (by 22% drop) which is slightly greater than the drop in batch tests (10%).

After reaching a saturation point $(C/C_0 = 1)$ for zinc sorption, distilled water was pumped through columns and zinc desorption behaviour was monitored. The desorption curves of zinc from compost and aged compost showed a very similar shape, with a rapid decline show that low amounts of zinc was desorbed followed by a flatter diminution (Fig. 6). The result suggests that Zn sorption is generally irreversible, owing to strong interaction between Zn and the surface active sites of the compost and aged compost. However the equilibrium desorption zinc concentration in the effluent from the compost (0.36 mg L^{-1}) is a little bit higher than the aged compost (0.17 mg L^{-1}) and this may be due to the higher initial zinc mass sorbed on the compost (16.9 mg g^{-1}) compared with the aged compost (13 mg g^{-1}). The mass of zinc desorbed was 0.75 mg g⁻¹ of compost and 0.63 mg g⁻¹ of aged compost. The loss, however, in both case, did not exceeded more than 5% of the sorbed amount. Losses of the sorbed heavy metals during desorption are probably due to the different types of sorption bonds of the metals with the functional groups (sorption sites) of compost and aged compost.

In summary, the batch and column results indicated that the zinc capacity of compost is higher than aged compost. Sorption kinetic data was better predicted by a pseudo-second-order model. The modelled rate constants showed that aging of the compost decreased the rate of zinc sorption. The intra-particle diffusion plot shows that both film diffusion and intra-particle diffusion are rate controlling. Column results showed the sorption capacity of compost column (16 mg g⁻¹) is higher than the aged compost column (13 mg g⁻¹).

Conclusions

The physiochemical properties of compost relevant to metal sorption changed during the aging process. This was evident in the FTIR spectra with decreased peak intensity for functional groups relevant to metal sorption and was expressed in the particle characterisation as decreased CEC and in the batch and column studies as lower metal sorption capacities and decreased rates of metal sorption after aging. This suggests that the nature of the DOC involved in metal bonding needs to be better understood and in particular its stability with regards to leaching. The stability of these organic biosorbents should be taken into account in the design of these systems. The effects of aging upon other types of woody materials also need to be investigated.

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