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# Influences of natural zeolite on speciation of heavy metals during rotary drum composting of green waste

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## ABSTRACT

The presence of heavy metals in water hyacinth compost is the main limiting factor for its land application. Water hyacinth can accumulate heavy metals in its tissues. Application of natural zeolites (clinoptilolite) during water hyacinth composting may be helpful to immobilise heavy metals. The present study was carried out on the speciation of heavy metals (Zn, Cu, Mn, Fe, Pb, Ni, Cd and Cr) during rotary drum composting of water hyacinth mixed with cattle manure, sawdust and natural zeolite. The Tessier sequential extraction method was used for heavy metal speciation determination. The water hyacinth, cattle manure and sawdust at a ratio of 6:3:1 ratio was mixed with 5, 10 and 15% natural zeolite by weight. Influences of physicochemical parameters such as temperature, pH and organic matter degradation on speciation of heavy metals were studied during the process. A rotary drum composter has been shown to be highly efficient for organic matter degradation. The most bioavailable fractions (exchangeable and carbonate fractions) were reduced significantly due to the combined effects of zeolite addition and metal complexing with stabilised organic matter as a result of the high degradation of organic matter in a rotary drum. Ni, Pb and Cd were not found in the reducible and oxidisable fractions. Overall, the addition of an optimum quantity of natural zeolite significantly reduced the bioavailability fractions of heavy metals during rotary drum composting of the water hyacinth.

**Keywords:** water hyacinth, composting, heavy metals, speciation, bioavailability, natural zeolite

## 1 INTRODUCTION

The water hyacinth (*Eichhornia crassipes*) is a free floating aquatic weed originating in the Amazon in South America and has been considered as the most noxious plant species in the world (Gunnarsson and Petersen, 2007). It has become as the world's worst invasive aquatic plant due to its extremely rapid propagation and density of growth (Gupta *et al.*, 2007). However, water hyacinth has been used in phytoremediation for the removal of pollutants such as heavy metals from water (Malik, 2007). Composting is one of the most cost-effective and easy techniques for the handling and final disposal of water hyacinth after phytoremediation as it combines material recycling and biomass disposal (Villasenor *et al.*, 2011; Singh and Kalamdhad, 2012). On the other hand, the major drawback of water hyacinth composting is the high content of heavy metals in the mature compost (Singh and Kalamdhad, 2012, 2013a and 2013b). Metal uptake by plants and its accumulation in the food chain is a potential hazard to animals and human

health if the compost contaminated with heavy metals is used as a soil conditioner (Wong and Selvam, 2006).

The availability of heavy metals for plants depends on their specific chemical forms rather than the total concentration obtained after strong acid digestion (Nair *et al.*, 2008). Sequential extraction of heavy metals during the composting process is a useful technique for determining the chemical speciation (Walter *et al.*, 2006). Estimation of the chemical speciation of heavy metals in compost helps to evaluate their bioavailability and suitability for land application (Wong and Selvam, 2006). According to Tessier *et al.* (1979) a sequential extraction procedure can determine heavy metals bound in five fractions:

- Exchangeable fraction (F1): likely to be affected by changes in water ionic composition as well as sorption-desorption processes.
- Carbonate fraction or acid extractable (F2): susceptible to changes in pH and can become soluble and mobilised in acidic conditions.

- Reducible fraction (F3): likely to be associated with precipitated iron fractions and may therefore be released under anoxic conditions.
- Oxidisable fraction or fraction bound to organic matter (F4): can be released and solubilised under oxidising conditions.
- Residual fraction (F5): is permanently bound in the crystal lattice of the mineral components of the compost and soil. This fraction never will be available for plants under natural conditions.

Natural zeolite has generally been used for the improvement of the physical and chemical properties of the compost and immobilisation of heavy metals during sewage sludge composting (Zorpas *et al.*, 2000; Sprynsky *et al.*, 2007; Villasenor *et al.*, 2011). Zeolite improves the composting process due to its ability to increase the porosity of the substrate. It has the ability to readily uptake almost all heavy metals that are bound to the exchangeable and the carbonate fractions (Zorpas *et al.*, 2000). Cations such as  $\text{Na}^+$ ,  $\text{K}^+$  and  $\text{Ca}^{2+}$  are weakly held in the pore structure of zeolite where they can be replaced by competing ions (metals) depending on complex interactions between the aluminosilicate frameworks (Leggo, 2000). The addition of the natural zeolite during water hyacinth composting may therefore change the chemical speciation of the heavy metals in composts and decrease their mobility and bioavailability.

Limited literature data are available on speciation of heavy metals during water hyacinth composting (Singh and Kalamdhad, 2012, 2013c). However, there is no literature available on the speciation of heavy metals during rotary drum composting of the water hyacinth with natural zeolite. Therefore, the objective of the present study was to assess the reduction in bioavailable forms of Zn, Cu, Mn, Fe, Ni, Pb, Cd and Cr by the addition of natural zeolite (clinoptilolite) during rotary drum composting of water hyacinth mixed with cattle manure and sawdust.

## 2 MATERIALS AND METHODS

### 2.1 Feedstock materials

Water hyacinth, cattle manure (cow dung) and sawdust were used for the preparation of different compost mixtures. Water hyacinth was collected from the Amingaoan industrial area near the Indian Institute of Technology Guwahati campus, India. Cattle manure was obtained from a dairy farm near the campus. Sawdust was purchased from a nearby saw mill. Natural zeolite (powder form) was purchased from G. M. Chemicals Pvt. Ltd (Gujarat, India). Singh and Kalamdhad (2012, 2013a and 2013b) suggested that a mixture of 90 kg water hyacinth, 45 kg cattle manure and 15 kg sawdust was the best combination for the reduction of bioavailability of heavy metals during water hyacinth composting, therefore in this study the same combination was used with 5, 10 and 15% zeolite. Prior to composting, the maximum particle size of the waste mixture

was restricted to 1 cm in order to provide better aeration and moisture control. The waste composition of different treatments are given as follow: control (water hyacinth 90 kg + cattle manure 45 kg + sawdust 15 kg), zeolite 1 (control + 5% zeolite), zeolite 2 (control + 10% zeolite) and zeolite 3 (control + 15% zeolite).

Initial characterisations of zeolite were found as follows: pH  $8.85 \pm 0.05$ , electrical conductivity of  $1.58 \pm 0.03 \text{ dS m}^{-1}$ , moisture content of  $3.75 \pm 0.15 \%$ , volatile solids of  $11.73 \pm 0.03 \%$ , nutrients ( $\text{mg kg}^{-1}$ ) of Na– $7100 \pm 100$ , K– $1850 \pm 100$ , Ca– $6575 \pm 0.08$  and Mg– $1175 \pm 125$ , and concentration of total heavy metals ( $\text{mg kg}^{-1}$ ) of Zn– $6.61 \pm 0.54$ , Cu– $1.15 \pm 0.05$ , Mn– $4.60 \pm 0.10$ , Fe– $8.10 \pm 0.26$ , Pb– $0.72 \pm 0.065$  and Cr– $0.08 \pm 0.003$ .

### 2.2 Rotary drum composter design

A pilot-scale rotary drum composter of 550 L capacity was operated by batch-mode operation. The main unit of the composter *i.e.* the drum was 1.22 m in length and 0.76 m in diameter, fabricated by a 4-mm thick metal sheet (Kalamdhad *et al.*, 2009). The capacity of the pilot scale rotary drum composter was determined by considering that a single person can handle around 150 kg of wastes by manual rotation. The composting period of 20 days was decided to ensure degradation and stabilisation based on the performance of earlier studies regarding in-vessel composting reactors (Kalamdhad *et al.*, 2009; Singh and Kalamdhad, 2013a). Turning was done manually after every 24 h through one complete rotation of the rotary drum to ensure that the material on the top portion moved to the central portion, where it was subjected to higher temperature. After that, aerobic conditions were maintained by opening the top half side doors of the two circular faces.

The samples were collected through grab sampling from different locations, mainly from the mid-span and end terminals of the pilot-scale rotary drum composter with a compost sampler to minimise disturbance of the adjacent materials. All the grab samples were thoroughly mixed together to make a homogenised sample. Triplicates homogenised samples were collected on days 0, 4, 8, 12, 16 and 20 after drum turning and the samples were air dried immediately, ground to pass through a 0.2 mm sieve and stored for physicochemical analysis.

### 2.3 Experimental analysis

A digital thermometer was used for temperature monitoring during the composting process. Each sample was analysed for the following parameters: pH (1:10 w/v waste: water extract), moisture content and total organic carbon (TOC) (Kalamdhad *et al.*, 2009). Atomic absorption spectrometer (AAS) (Varian Spectra 55B) was used for analysis of Zn, Cu, Mn, Fe, Ni, Pb, Cd and Cr concentrations after digestion of 0.2 g samples with 10 mL of  $\text{H}_2\text{SO}_4$  and  $\text{HClO}_4$  (5:1) mixture in a block digestion system (Pelican Equipments, Chennai, India) for 2h at 300 °C. The detection limit of the AAS determination for heavy metals was  $0.001 \text{ mg L}^{-1}$ .

The sequential extraction method developed by Tessier *et al.* (1979) for heavy metal speciation was followed. According to this method, metals are bound with five fractions: exchangeable (F1), carbonate (F2), reducible (F3), oxidisable (F4) and residual (F5) fraction. The extraction was carried out with an initial mass of 1.0 g oven dried sample in polypropylene centrifuge tubes of 50 mL capacity. After each successive extraction, the supernatant liquid was removed with a pipette after centrifugation at 10,000 rpm for 5 min and made up to the required volume for analyses of the heavy metals. The residue was washed with 20 mL of Milli Q water by shaking for 15 minutes followed by centrifugation without loss of solids. The extracts were stored in polythene bottles. All extractions were performed in triplicate and the mean values were presented with standard deviation.

The following steps were adopted:

- (1) Exchangeable: about 1 g sample was extracted at room temperature with 8 mL of 1 M  $\text{MgCl}_2$  (pH 7) with continuous agitation for 1 h.
- (2) Carbonate: residue from step (1) was leached at room temperature with 8 mL of 1 M of NaOAc (pH 5 adjusted with conc. HOAc) with continuous agitation for 5 h.
- (3) Reducible: residue from step (2) was extracted with 20 mL of 0.04 M  $\text{NH}_2\text{OH}\cdot\text{HCl}$  in 25% (v/v) HOAc agitated for 6 h at 96°C.
- (4) Oxidisable: to the residue from step (3) was added 3 mL of 0.02 M  $\text{HNO}_3$  and 5 mL of 30%  $\text{H}_2\text{O}_2$  (pH 2, adjusted with conc.  $\text{HNO}_3$ ); heated at 85°C for 2 h with occasional agitation. A second 3 mL aliquot of 30%  $\text{H}_2\text{O}_2$  was added and heated at 85°C for 3 h with occasional agitation. After cooling, 5 mL of 3.2 M  $\text{NH}_4\text{OAc}$  in 20% (v/v)  $\text{HNO}_3$  was added; diluted to 20 mL; agitated for 30 min and centrifuged.
- (5) Residual: residue from step (4) was digested with 10 mL of  $\text{H}_2\text{SO}_4$  and  $\text{HClO}_4$  (5:1) mixture in a block digestion system. The bioavailability factor (BF) was calculated according to Singh and Kalamdhad (2012).

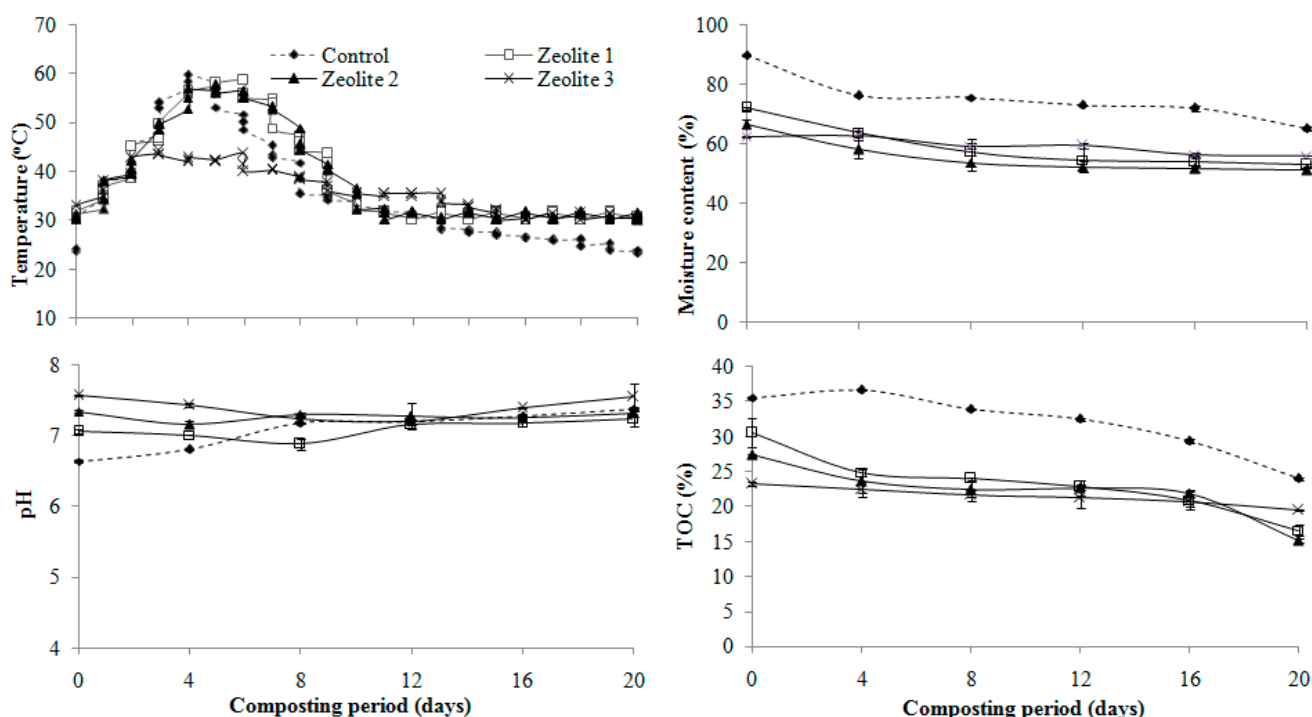
$$\text{BF} = \frac{\text{F1} + \text{F2} + \text{F3} + \text{F4}}{\text{F1} + \text{F2} + \text{F3} + \text{F4} + \text{F5}}$$

All the results reported are the means of three replicates. Repeated measurements treated with analysis of variance (ANOVA) were made using Statistica software. The objective of the statistical analysis was to determine any significant differences among the parameters analysed for different proportions.

### 3 RESULTS AND DISCUSSION

#### 3.1 Physico-chemical analysis

As shown in Figure 1a, the composting drum temperature went through three typical phases (mesophilic, thermophilic and cooling phase) in the control and all zeolite treatments during the composting process. The maximum thermophilic temperatures were observed in the range of 44.4–59.7°C in the control and zeolite treatments due to the degradation of organic matter by microorganisms (Chiang *et al.*, 2007). The lower thermophilic temperature observed in the zeolite 3 treatment in comparison to the control, zeolite 1 and 2 treatments, might be due to the poor degradation of organic matter. Higher additions of zeolite may absorb water and produce anaerobic conditions for the composting microorganisms (Villasenor *et al.*, 2011), consequently reducing the degradation of organic



**Figure 1** Variation of temperature (a), moisture content (b), pH (c), and total organic carbon (d) during composting process (bars denote  $\pm$  SD).

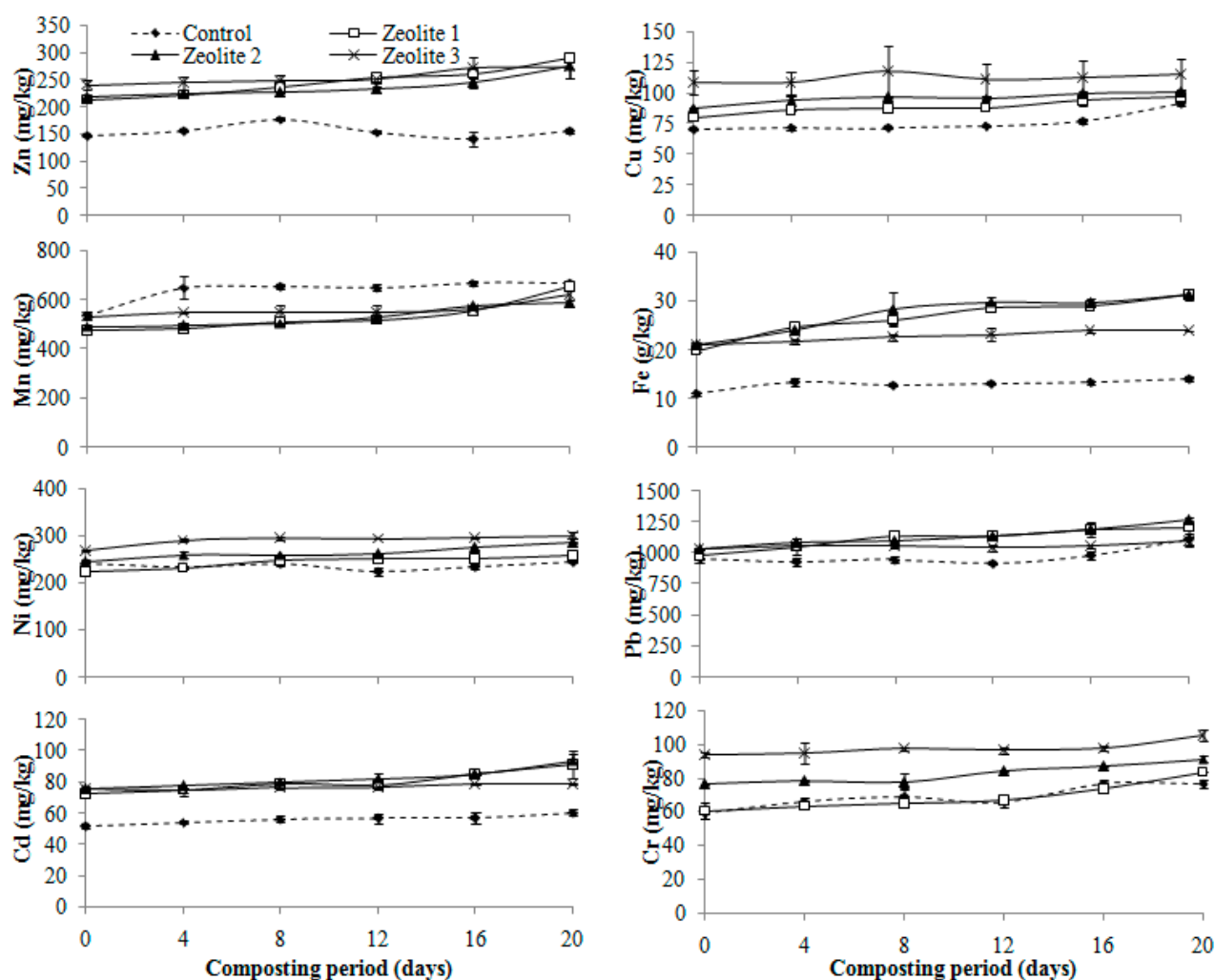
matter during the composting process. Figure 1b shows the significant reduction in moisture content in the control and all zeolite treatments during the process ( $F = 39.9$ ,  $P < 0.001$ ). Moisture loss during the composting process is an indicator of the decomposition rate of the organic matter, because the composting material requires an optimum moisture content for survival of the microorganisms (Singh and Kalamdhad, 2012). The moisture loss was observed in the range of 10.2–27.4% in the control and all zeolite treatments. The higher reduction of moisture content was observed in the control followed by zeolite 1, zeolite 2 and zeolite 3 treatments. A lower reduction of moisture content in all the zeolite treatments might be due to moisture retained by the added zeolite (Villasenor *et al.*, 2011).

Figure 1c shows the significant variation in pH values from 6.7 to 7.7 during the composting process ( $F = 38.5$ ,  $P < 0.001$ ). The pH is an important parameter which greatly affects the composting process and speciation of heavy metals. The pH values were within the optimal range for the development of bacteria 6.0–7.5 and fungi 5.5–8.0 (Amir *et al.*, 2005).

The pH values were within the optimal range for the development of bacteria (6.0–7.5) and fungi (5.5–8.0) (Amir

*et al.*, 2005). The pH of the initial compost mixture increased slightly in all zeolite treatments in comparison to the control. The pH decrease in the initial phase of composting might have been due to acid production during the decomposition of organic matter, a similar result was observed by Zorpas *et al.* (2000). The pH in the initial feed mixtures of zeolite was higher; however, at the end of composting process it reduced and became neutral or slightly alkaline. Figure 1d shows the significant reduction in TOC in the control and all zeolite treatments during the process ( $F = 48.2$ ,  $P < 0.001$ ). The higher reduction of TOC was observed in the zeolite 1 treatment followed by the zeolite 2 treatment, control and then the zeolite 3 treatment. The reduction of the TOC during the composting process is associated with its transformation into  $\text{CO}_2$  and  $\text{H}_2\text{O}$  by micro-organisms (Castaldi *et al.*, 2006). The decomposition of organic matter was enhanced by the addition of natural zeolite which has the capability of increasing the porosity of the substrate, resulting in enhanced microbial action on the organic matter due to the presence of sufficient oxygen (Villasenor *et al.*, 2011).

Figure 2 illustrates the variation in the total concentration of metals (Zn, Cu, Mn, Fe, Ni, Pb, Cd and Cr) in the control and all zeolite treatments during the 20 days of the composting



**Figure 2** Variation of total heavy metal concentration in control and zeolite treatments during the composting process (bars denote  $\pm$  SD).

period. In all the treatments, the concentration of the total heavy metals was increased with respect to the total mass of organic matter due to the reduction of organic matter and release of  $\text{CO}_2$  (Zorpas *et al.*, 2000, Singh and Kalamdhad, 2012, 2013d). The variations in the total concentration of Zn, Cu, Mn, Fe, Ni, Pb, Cd and Cr in the control and zeolite amended compost were significant ( $F = 14.3$ ,  $P < 0.001$  for Zn,  $F = 15.3$ ,  $P < 0.001$  for Cu,  $F = 9.2$ ,  $P < 0.001$  for Mn,  $F = 6.1$ ,  $P < 0.001$  for Fe,  $F = 8.6$ ,  $P < 0.001$  for Ni,  $F = 10.3$ ,  $P < 0.001$  for Pb,  $F = 7.01$ ,  $P < 0.001$  for Cd,  $F = 10.74$ ,  $P < 0.001$  for Cr).

### 3.2 Speciation of heavy metals

#### 3.2.1 Speciation of Zn, Cu, Mn and Fe

Figure 3 shows the speciation of Zn, Cu, Mn and Fe in the control, while Tables 1, 2 and 3 illustrate the speciation of these metals in zeolite 1, 2 and 3 treatments, respectively, during the composting process. For Zn, the F1, F2, F3 and F4 fractions exhibited reduced concentration at the end of the composting process for the control and all zeolite treatments. Higher reductions in the F1 and F3 fractions were observed of about 57.8 and 56.9% of the total fraction, respectively, in the control but a higher reduction in F2 (67.7%) and F4 (44.6%) fractions was observed in zeolite 1 and 2 treatments, respectively.

The F5 fraction of Zn increased in all treatments. The F1 and F2 fractions contributed about 5% of total fraction in the final compost of the control; however, in the zeolite samples, these fractions were reduced to approximately 4.1, 3.2 and 4.9% of the total, respectively. The F5 fraction contributed about 77% in the mature compost of control; however, this fraction was higher (in the range of 82–86%) for the zeolite treatments. The F1 and F2 fractions are the most mobile and bioavailable fractions and were reduced in zeolite treatments in comparison to the control. These fractions were bound with zeolite through ion exchange and converted into the F5 fraction which is the most inert fraction (Zorpas *et al.*, 2000; Venkateswaran *et al.*, 2007). Zn was mainly bound in the F5 fraction throughout the composting process. On the other hand, Zorpas *et al.* (2008) reported that Zn was mainly found in F3 and F4 fractions in sewage sludge compost with natural zeolite. It has been observed that a significant percentage of the inert fraction (F5 fraction) was not adsorbed by the zeolite. Similar results have also been reported by Villasenor *et al.* (2011) during sewage sludge composting with natural zeolite. The variation in F1, F2, F3, F4 and F5 fractions for Zn concentrations in the control and zeolite treatments were significant ( $F = 9.57$ ,  $P < 0.001$  for F1,  $F = 12.54$ ,  $P < 0.001$  for F2,  $F = 34.79$ ,  $P < 0.001$  for F3,  $F = 22.84$ ,  $P < 0.001$  for F4,  $F = 16.94$ ,  $P < 0.001$  for F5).

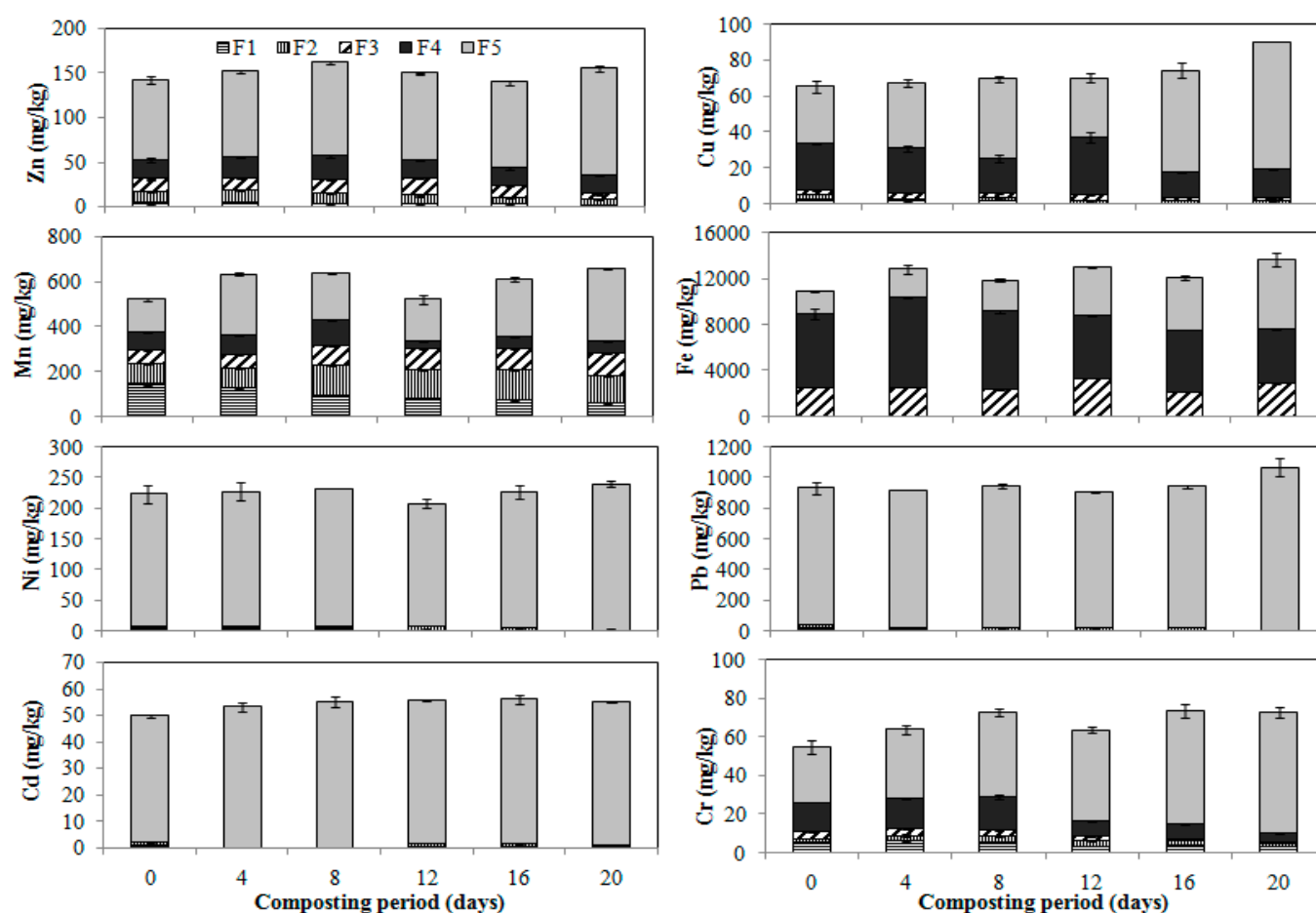


Figure 3 Speciation of heavy metals in zeolite 1 treatment during the composting process (bars denote  $\pm$  SD).

For the zeolite 1 treatment, high reductions in the Cu concentrations of the F1, F2 and F3 fractions were observed of about 81.3, 80.6 and 74.1% of the total fraction, respectively, compared with the control; however, the highest reduction of the F4 fraction was observed in the control. The F4 fraction was increased in the zeolite 3 treatment, while it was reduced in the control, zeolite 1 and 2 treatments. The F5 fraction of Cu was increased in all treatments during the composting process. The F1 and F2 fractions contributed about 2.2% of the total in the final compost of the control; however, these fractions were reduced to about 1.5, 2.1 and 2.0% of the total in zeolite 1, 2 and 3 treatments, respectively. Zorpas *et al.* (2008) have also reported that the amount of Cu bound to the F1 and F2 fractions was less than 2%. The F5 fraction was found to be in the range of about 86–90.6% of the total in all zeolite treatments but in the control it was observed to be about 78.4% of the total Cu. Zorpas *et al.* (2000) reported that Cu was mainly bound to the F4 and F5 fractions during sewage sludge composting with natural zeolite; however, in the present study, Cu was mainly bound in F5 fraction in the control and all zeolite treatments; Furthermore, during the composting process, the breakdown of the organic matter

increased the formation of Cu in the exchangeable fraction (F1 fraction) and helped the ion exchange phenomenon for natural zeolite, resulting in reduced bioavailability of Cu (Villasenor *et al.*, 2011). The maximum reduction of movable fractions (from F1 to F4) of Cu, observed in the zeolite 1 treatment, could be explained as a higher degradation of organic matter followed by conversion of organic matter into humic substances which contain more carboxyl groups (Liu *et al.*, 2008), resulting in a Cu formed complex compound with humic substances. The variations in F1, F2, F3, F4 and F5 fractions of Cu in the control and zeolite treatments were significant ( $F = 14.09$ ,  $P < 0.001$  for F1,  $F = 14.10$ ,  $P < 0.001$  for F2,  $F = 28.63$ ,  $P < 0.001$  for F3,  $F = 29.03$ ,  $P < 0.001$  for F4,  $F = 12.81$ ,  $P < 0.001$  for F5).

The F1 and F4 fractions of Mn were reduced in the control and all zeolite treatments. The F2 fraction was increased in the control; however, it was reduced in all the zeolite treatments. The F3 fraction was increased in the control and zeolite 3 treatment but it was reduced in the zeolite 1 and 2 treatments. The higher reduction in F1 fractions was observed to be about 66% in the control; however, a higher reduction in the F2, F3 and F4 fractions were observed of about 49.8, 63.6 and 46.8% of the total, respectively, in the zeolite 1 treatment. The F1 and

**Table 1** Speciation of heavy metals in zeolite 1 treatment during 20 days of composting period

Days	Heavy metals									
	Zn (mg kg <sup>-1</sup> dry matter)					Cu (mg kg <sup>-1</sup> dry matter)				
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
0	7.78±0.08	13.47±1.30	24.63±0.97	14.38±0.64	136.5±1.50	2.70±0.55	2.88±0.08	1.70±0.10	11.87±0.49	53.80±0.80
4	6.82±0.20	13.22±0.13	24.84±1.16	15.38±1.80	152.2±3.80	1.73±0.63	2.04±0.24	1.28±0.33	11.59±0.86	61.35±3.15
8	6.88±0.01	11.80±0.31	21.98±3.78	17.05±0.78	174.4±11.6	1.63±0.23	1.33±0.18	1.20±0.30	10.63±0.62	64.55±1.75
12	6.19±0.14	11.76±0.11	19.50±0.30	20.11±1.58	178.2±3.20	1.51±0.35	1.43±0.18	1.10±0.20	10.87±0.60	68.30±0.30
16	5.71±0.40	7.62±1.51	21.13±1.43	22.30±0.41	187.9±1.95	1.33±0.03	1.65±0.05	1.25±0.05	9.68±0.56	74.35±4.75
20	5.18±0.18	6.11±0.05	16.10±0.80	20.14±0.52	228.5±1.50	0.67±0.07	0.74±0.02	0.58±0.02	7.09±0.05	87.05±1.15
Days	Mn (mg kg <sup>-1</sup> dry matter)					Fe (mg kg <sup>-1</sup> dry matter)				
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
0	96.5±3.2	76.4±2.0	115.0±2.5	70.9±2.0	106.5±1.7	19.43±0.48	34.25±0.45	2353±128	2901±12	13282±1757
4	90.6±5.4	75.4±1.9	108.0±3.2	62.6±2.7	129.4±4.0	23.90±0.10	24.33±0.93	2296±96	2544±17	17984±946
8	91.0±0.9	71.7±3.7	97.5±6.5	62.5±9.5	158.1±3.3	17.43±4.23	23.43±1.93	2071±81	2452±85	19821±154
12	88.8±6.8	71.7±1.1	89.7±0.4	58.1±3.9	172.7±1.3	16.75±1.90	20.94±0.68	1963±63	2424±139	23850±380
16	73.6±4.0	66.4±1.6	85.6±4.2	56.6±1.0	264.8±2.8	11.93±0.33	17.90±0.75	1931±181	2237±66	24168±1732
20	52.8±5.2	50.5±4.3	55.5±3.5	49.9±2.3	407.0±3.4	11.28±0.33	17.88±0.03	1720±130	2207±54	25289±8635
Days	Ni (mg kg <sup>-1</sup> dry matter)					Pb (mg kg <sup>-1</sup> dry matter)				
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
0	3.58±0.18	2.83±0.13	ND	ND	214.5±4.0	12.75±0.25	10.94±0.29	ND	ND	879.5±20.5
4	2.45±0.05	2.60±0.05	ND	ND	216.7±2.2	12.50±0.50	10.84±0.16	ND	ND	953.0±33.0
8	1.83±0.58	2.23±0.38	ND	ND	234.5±11	10.25±0.25	9.47±0.53	ND	ND	1053.5±21.5
12	1.60±0.10	2.20±0.40	ND	ND	242.8±35	9.70±0.80	8.41±0.10	ND	ND	1094.1±5.90
16	1.25±0.15	2.03±0.08	ND	ND	240.0±0.1	9.75±0.75	8.18±0.18	ND	ND	1127.5±12.5
20	1.20±0.10	0.89±0.09	ND	ND	242.3±0.8	3.55±0.05	4.79±0.21	ND	ND	1157.5±42.5
Days	Cd (mg kg <sup>-1</sup> dry matter)					Cr (mg kg <sup>-1</sup> dry matter)				
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
0	1.33±0.03	1.28±0.07	ND	ND	65.50±2.50	2.65±0.30	0.92±0.035	2.42±0.20	12.74±0.39	37.05±3.55
4	1.27±0.02	0.91±0.05	ND	ND	71.65±2.65	2.25±0.11	0.82±0.04	2.10±0.30	12.11±0.57	43.85±0.65
8	1.20±0.05	0.63±0.23	ND	ND	74.85±2.15	2.02±0.04	0.63±0.02	1.69±0.10	11.01±0.53	46.55±5.05
12	1.23±0.08	0.59±0.19	ND	ND	71.30±2.80	1.89±0.15	0.32±0.08	1.30±0.10	9.84±0.16	52.90±4.10
16	1.18±0.13	0.55±0.20	ND	ND	79.70±3.80	1.55±0.13	0.23±0.03	1.09±0.09	8.06±0.54	60.65±5.35
20	0.62±0.07	0.59±0.09	ND	ND	89.90±3.10	1.14±0.14	0.21±0.01	0.80±0.089	5.00±0.36	72.60±2.60

Mean values in columns are statistically different (ANOVA;  $P < 0.05$ ). ND, not detected

F2 fractions contributed about 28.1% of the total in the final compost of the control; however, these fractions were reduced to about 16.8, 23.9 and 20.0% of the total in the zeolite 1, 2 and 3 treatments, respectively. The F5 fraction contributed about 48.9% of the total in the compost of control, while this fraction was observed to be about 66.1, 49.2 and 36.7% of total fraction in the zeolite 1, 2 and 3 treatments, respectively. The F5 fraction of Mn was the dominant fraction and it was found to be about 36.7–66% of the total in the control and zeolite treatments, while Zorpas *et al.* (2000) reported that approximately 60% of Mn was bound to the F3 fraction during sewage sludge composting with natural zeolite. Wong and Selvam (2006) reported that before the composting of the sewage sludge, Mn was mainly bound with F5 fraction but after composting, it mainly bound with the F3 fraction. However, in the present study, before composting in the control, the dominant fraction of Mn was the F1 fraction but after composting, this fraction was converted into the F5 fraction. However, in all zeolite treatments before composting, Mn bound in the F3 fraction but after composting, the F5 fraction became the dominant fraction. The variation in F1, F2, F3, F4 and F5 fractions of Mn in the control and zeolite treatments were significant ( $F$

= 30.30,  $P < 0.001$  for F1,  $F = 22.91$ ,  $P < 0.001$  for F2,  $F = 28.17$ ,  $P < 0.001$  for F3,  $F = 12.77$ ,  $P < 0.001$  for F4,  $F = 151.74$ ,  $P < 0.001$  for F5).

A higher reduction in the F1, F2, F3 and F4 fractions of the Fe fractions was observed of about 63.1, 66.8, 53.5 and 51.6% of the total, respectively, in the zeolite 1 treatment. Similar to Zn, Cu and Mn, the F5 fraction of Fe was enhanced in the control and all zeolite treatments. The F1 and F2 fractions contributed about 0.54% of the total fraction in the final compost of the control; however, these fractions were reduced in the range of about 0.10–0.19% of the total in all zeolite treatments. The F5 fraction contributed about 44.3% of the total in the compost of the control while this fraction was observed to be about 86.5, 87.2 and 85.5% of the total in the zeolite 1, 2 and 3 treatments, respectively. Zorpas *et al.* (2000) has reported that Fe was mainly bound in the F3 and F5 fractions (about 95% for the total Fe); however, in the present study, Fe was mainly bound in the F5 fraction. The variation in the F1, F2, F3, F4 and F5 fractions of Fe in the control zeolite treatments were significant ( $F = 70.82$ ,  $P < 0.05$  for F1,  $F = 33.29$ ,  $P < 0.001$  for F2,  $F = 42.4$ ,  $P < 0.05$  for F3,  $F = 22.86$ ,  $P < 0.001$  for F4,  $F = 9.56$ ,  $P < 0.001$  for F5).

**Table 2** Speciation of heavy metals in zeolite 2 treatment during 20 days of composting period

Days	Heavy metals									
	Zn (mg kg <sup>-1</sup> dry matter)					Cu (mg kg <sup>-1</sup> dry matter)				
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
0	8.22±0.15	7.58±0.22	26.09±0.52	15.71±2.65	147.2±6.8	2.28±0.53	2.10±0.30	2.45±0.35	10.87±0.21	65.05±0.45
4	7.22±0.33	7.40±0.06	24.16±1.14	13.28±2.08	157.2±23	1.70±0.10	1.33±0.13	1.45±0.35	10.04±0.68	71.35±1.85
8	7.16±0.06	6.81±1.56	22.80±1.35	13.29±0.96	160.0±4.7	1.68±0.18	1.16±0.20	2.05±0.55	10.47±0.33	74.45±1.55
12	6.58±0.01	7.34±0.02	21.93±1.53	12.38±1.10	182.3±2.3	1.70±0.10	1.17±0.24	1.60±0.0	9.33±0.94	77.25±3.75
16	6.20±0.05	6.34±0.51	19.53±1.17	11.54±0.92	197.3±8.3	1.48±0.23	1.18±0.03	1.55±0.15	10.24±0.81	80.30±1.80
20	5.23±0.09	3.24±0.12	18.00±2.10	11.24±0.02	226.8±12.0	1.13±0.03	0.93±0.13	1.29±0.31	9.24±0.06	86.20±2.30
Days	Mn (mg kg <sup>-1</sup> dry matter)					Fe (mg kg <sup>-1</sup> dry matter)				
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
0	121.7±1.65	83.2±5.13	129.4±3.2	83.1±4.8	71.3±6.3	19.23±0.28	31.45±1.25	1953±28	2686±216	14563±963
4	94.0±4.00	81.0±2.73	126.3±1.5	73.0±7.5	87.8±6.8	22.85±0.85	28.10±3.10	1835±65	2503±58	18265±665
8	86.8±3.28	83.8±4.20	121.7±0.7	71.2±1.97	101.8±6.8	22.43±0.28	24.03±2.53	1706±16	2439±99	21738±763
12	94.3±1.30	74.4±6.22	127.4±1.9	66.1±11.0	124.9±4.2	16.75±1.90	25.70±1.90	1720±20	2449±114	22875±1595
16	72.4±2.83	75.2±0.40	120.3±1.1	66.3±10.7	222.5±1.1	11.93±0.33	18.63±0.23	1750±0	2301±101	24958±3942
20	64.6±3.40	71.4±3.48	83.5±8.6	69.2±2.1	280.0±2.0	13.28±1.68	17.50±1.35	1440±50	2361±200	26048±123
Days	Ni (mg kg <sup>-1</sup> dry matter)					Pb (mg kg <sup>-1</sup> dry matter)				
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
0	6.68±0.28	3.50±0.10	ND	ND	210.3±1.8	12.0±0.50	11.93±0.58	ND	ND	940±35
4	5.28±0.23	2.93±0.03	ND	ND	230.8±7.8	12.9±0.15	12.18±0.18	ND	ND	993±73
8	5.37±0.03	2.83±0.43	ND	ND	232.5±3.0	12.0±2.00	11.49±0.02	ND	ND	1068±7.5
12	4.15±0.35	2.70±0.20	ND	ND	228.4±9.8	10.7±0.02	10.13±0.13	ND	ND	1100±100
16	3.38±0.28	2.43±0.27	ND	ND	244.0±4.0	8.0±0.50	8.69±0.83	ND	ND	1115±25
20	2.50±0.40	2.18±0.08	ND	ND	254.5±3.0	5.0±0.38	4.88±0.38	ND	ND	1193±13
Days	Cd (mg kg <sup>-1</sup> dry matter)					Cr (mg kg <sup>-1</sup> dry matter)				
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
0	1.31±0.06	0.94±0.05	ND	ND	71.25±5.25	2.43±0.53	0±0	2.43±0.17	11.31±1.05	59.5±4.0
4	1.13±0.13	0.88±0.08	ND	ND	73.50±0.50	1.83±0.52	0.43±0.03	2.10±0.30	10.99±0.54	62.3±2.25
8	0.98±0.23	0.64±0.07	ND	ND	73.15±0.35	1.88±0.18	0.70±0.05	2.00±0.80	10.80±0.74	62.0±0.50
12	0.90±0.05	0.50±0.05	ND	ND	75.25±3.25	1.60±0.10	0.30±0.05	1.84±0.56	9.70±0.31	66.0±1.0
16	0.86±0.01	0.40±0.05	ND	ND	79.00±4.50	1.49±0.01	0.23±0.02	1.09±0.11	8.48±0.12	72.3±6.25
20	0.73±0.03	0.23±0.03	ND	ND	85.50±2.50	1.33±0.03	0±0	0.95±0.05	3.80±0.45	86.0±1.0

Mean values in columns are statistically different (ANOVA;  $P < 0.05$ ). ND, not detected



The BF of Zn, Cu, Mn and Fe decreased in the control and zeolite 1, 2 and 3 treatments, respectively (Figure 4). A higher reduction in the BF of Cu, Mn and Fe was observed of about 64.2, 56.0 and 52.6%, respectively, in the zeolite 1 treatment; however, higher reduction in the BF of Zn was observed of about 49.4% in the zeolite 2 treatment during the process. A higher reduction in the BF of Zn, Cu, Mn and Fe was observed in the zeolite 1 and 2 treatments. This could be explained as the higher degradation of organic matter followed by conversion of organic matter into humic substances which have a high carboxyl group content (Liu *et al.*, 2008; Singh and Kalamdhad, 2012a). This results in all the metals forming a complex compound with the humic substances. It has been observed that the BF of metals reduced in the zeolite 1 and 2 treatments in comparison to the control and zeolite 3 treatment. This indicated that the mobile fractions (F1 to F4) were bound to zeolite. The present study, recommends that the addition of natural zeolite could prevent the mobility and bioavailability of metals during the composting process.

### 3.2.2 Speciation of Ni, Pb, Cd and Cr

Figure 3 shows the speciation of Ni, Pb, Cd and Cr in the control, whereas Tables 2 and 3 illustrate the speciation of these metals in the zeolite 1, 2 and 3 treatments during the

process. A higher reduction in the F1 and F2 fractions of Ni was observed pertaining to 69.7 and 71.5% of the total in the zeolite 1 treatments. This can be attributed to zeolite taking up the Ni bound in the F1 and F2 fractions (Zorpas *et al.*, 2008). The F1 and F2 fractions contributed in the range of about 1.44–2.36% of the total in the final compost of the control and all zeolite treatments. Zorpas *et al.* (2008) reported that Ni was found to be associated with the F3 (36%) and the F5 (23%) fractions during the sewage sludge compost with natural zeolites. In the present study, Ni mostly bound in F5 fraction contributed about 97.6–99.2% of the total; however, the F3 and F4 fractions were not detected over all the composting process. Similar results have also been reported by Fuentes *et al.* (2004) in different sewage sludges. The significant increase in the F5 fraction of Ni may be due to an alkaline stabilisation process (Gupta and Sinha, 2007). The variation in the F1, F2 and F5 fractions of Ni in the control and zeolite treatments were significant ( $F = 49.61$ ,  $P < 0.05$  for F1,  $F = 18.88$ ,  $P < 0.001$  for F2,  $F = 4.11$ ,  $P < 0.001$  for F5).

The F1 fraction of Pb was reduced in the range of 20.7–79% and the F2 fraction was reduced in the range of about 35–67% of the total fraction in the control and all zeolite treatments, but a higher reduction in F1 and F2 fractions was observed in the zeolite treatments as compared to the control. There was no significant reduction in F1 and F2 fractions

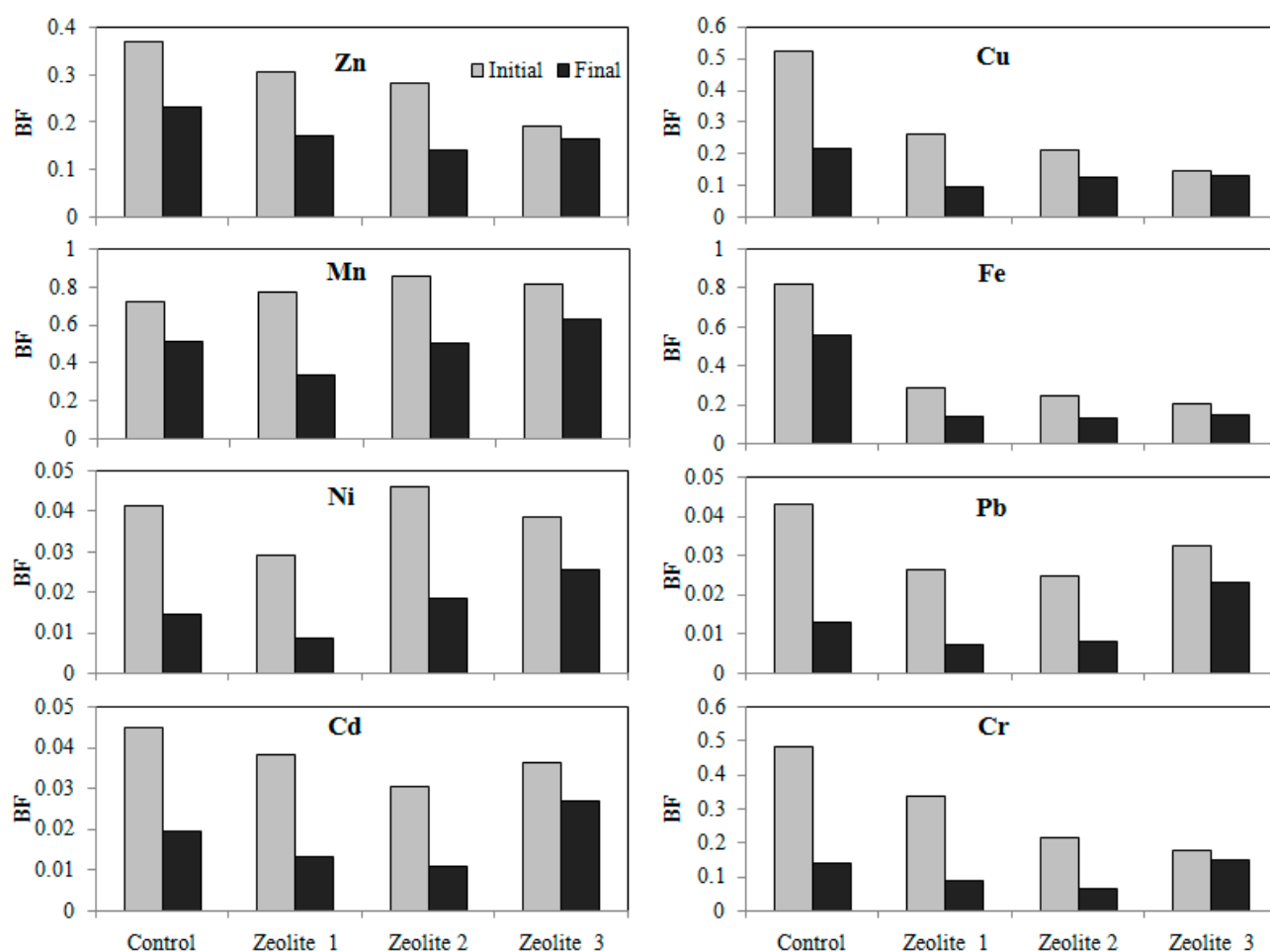


Figure 4 Changes in bioavailability factor (BF) of heavy metals in control and zeolite treatments during the composting process.

of Pb observed between the control and zeolite treatments. Sprynskyy *et al.* (2007) has also stated that zeolite addition had no influence on Pb speciation in the sewage sludge composting. The F5 fraction of Pb was increased in the control and all zeolite treatments. Zorpas *et al.* (2000) have reported that F3 and F5 fractions of Pb contributed about 83% of the total Pb. However, in the present study, Pb was mainly bound in F5 fraction, found to be about 97–98% of the total. The F3 and F4 fractions of Pb were not detected during the composting process. The variation in F1, F2 and F5 fractions of Pb in the control and zeolite treatments were significant ( $F = 41.54$ ,  $P < 0.05$  for F1,  $F = 25.29$ ,  $P < 0.001$  for F2,  $F = 6.19$ ,  $P < 0.001$  for F5).

About 1.95% of Cd was bound to the F1 and F2 fractions in the final compost of the control; however, these fractions were measured to be about 1.32, 1.11 and 2.69% of the total in the zeolite 1, 2 and 3 treatments, respectively, during the process. A higher reduction in the F1 and F2 fractions was observed of about 68.4 and 79.1% of the total in the control and zeolite 2 treatment, respectively. The reduction in the F1 and F2 fractions of Cd was attributed to the fact that

oxygen containing functional groups with a higher affinity to Cd, such as phenolic and benzene–carboxylic groups, were formed during the composting process (Xiong *et al.*, 2010). Cd was mainly bound to the F5 fraction in the range of about 97–99% of the total in the compost of control and all zeolite treatments. Sprynskyy *et al.* (2007) has also reported that the F5 fraction of Cd was the dominant fraction during sewage sludge composting. Similar to Ni and Pb, the F3 and F4 fractions of Cd was not found throughout the composting process. The variations in the F1, F2 and F5 fractions of Cd in the control and zeolite treatments were significant ( $F = 16.60$ ,  $P < 0.001$  for F1,  $F = 21.44$ ,  $P < 0.001$  for F2,  $F = 9.9$ ,  $P < 0.001$  for F5).

The F1, F2 and F3 fractions of Cr were reduced in the control and all the zeolite treatments. The higher reduction in F1 and F2 fractions was observed to be about 70.1 and 84.0% of the total in the zeolite 1; conversely, higher reduction of F3 and F4 fractions were observed in the control. The F4 fraction was increased in the zeolite 3 treatment. The F2 fraction was not observed initially but during the thermophilic stage it appeared and again it was not detected in the mature compost.

**Table 3** Speciation of heavy metals in zeolite 3 treatment during 20 days of composting period

Days	Heavy metals									
	Zn (mg kg <sup>-1</sup> dry matter)					Cu (mg kg <sup>-1</sup> dry matter)				
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
0	6.55±0.20	6.61±0.03	18.27±0.10	11.39±0.26	179.8±8.6	1.43±0.03	3.05±0.25	8.20±0.12	86.0±2.0	65.05±0.45
4	6.86±0.28	7.13±0.26	16.99±0.69	11.60±0.14	185.1±9.1	1.55±0.10	3.55±0.25	8.47±1.23	84.75±6.25	71.35±1.85
8	6.77±0.45	7.86±0.61	17.85±0.45	11.48±0.30	188.7±6.0	1.40±0.05	3.50±0.0	9.03±0.77	92.15±5.85	74.45±1.55
12	6.41±0.72	7.36±0.10	19.40±0.86	11.43±0.26	183.8±2.3	1.35±0.05	4.10±0.50	9.49±0.58	91.50±5.50	77.25±3.75
16	7.20±0.95	6.80±0.74	20.88±0.51	11.77±0.27	210.2±15.2	1.38±0.23	4.00±0.30	9.67±0.38	85.04±1.54	80.30±1.80
20	6.42±0.02	7.0±0.62	18.78±0.17	12.59±0.03	227.3±0.8	1.03±0.03	2.65±0.05	9.58±0.22	93.35±12.15	86.20±2.30
Days	Mn (mg kg <sup>-1</sup> dry matter)					Fe (mg kg <sup>-1</sup> dry matter)				
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
0	95.4±2.6	86.6±1.2	125.0±1.2	85.4±2.9	88.0±3.0	37.33±0.98	1850±50	2175±95	15813±288	14563±963
4	87.6±2.4	78.2±0.1	140.7±12.9	89.3±8.5	97.0±4.0	32.40±4.10	1879±108	2098±148	17715±1215	18265±665
8	72.9±9.7	72.8±4.8	154.9±2.6	91.5±2.2	123.1±5.6	27.63±4.18	1812±90	2071±69	18258±717	21738±763
12	72.3±9.8	68.8±8.2	168.8±6.7	91.1±6.2	129.9±0.9	27.15±0.30	1670±42	1993±42	17735±735	22875±1595
16	66.5±1.1	68.5±3.8	171.3±2.1	93.9±3.1	156.5±5.0	26.65±0.60	1665±292	2308±293	19700±800	24958±3942
20	57.4±0.2	63.2±1.3	168.6±0.5	92.2±0.9	220.8±7.3	24.30±0.60	1213±23	2083±23	19713±1213	26048±123
Days	Ni (mg kg <sup>-1</sup> dry matter)					Pb (mg kg <sup>-1</sup> dry matter)				
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
0	5.70±0.10	3.78±0.18	ND	ND	236.25±5.8	17.43±0.07	ND	ND	988±13	940±35
4	5.45±0.05	3.13±0.17	ND	ND	255.75±3.8	15.43±3.07	ND	ND	993±28	993±73
8	4.68±0.28	2.73±0.48	ND	ND	263.75±5.7	13.99±2.52	ND	ND	1020±60	1068±7.5
12	4.48±0.03	3.18±0.28	ND	ND	257.50±2.0	13.38±3.13	ND	ND	1003±3	1100±100
16	4.30±0.10	3.33±0.38	ND	ND	262.50±1.5	13.29±1.29	ND	ND	1023±3	1115±25
20	3.45±0.35	3.00±0.75	ND	ND	267.25±1.8	11.88±1.38	ND	ND	1053±3	1193±13
Days	Cd (mg kg <sup>-1</sup> dry matter)					Cr (mg kg <sup>-1</sup> dry matter)				
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
0	1.48±0.12	1.20±0.01	ND	ND	71.0±1.0	0.60±0.05	2.36±0.05	10.39±0.26	70.0±4.5	59.5±4.0
4	1.24±0.04	1.21±0.50	ND	ND	68.2±4.65	0.46±0.015	2.39±0.09	11.10±0.16	78.3±6.3	62.3±2.25
8	1.28±0.18	1.22±0.10	ND	ND	69.3±2.25	0.38±0.020	2.04±0.17	10.97±0.72	80.8±0.8	62.0±0.50
12	1.06±0.30	1.40±0.50	ND	ND	72.0±3.50	0.34±0.010	2.07±0.07	10.72±0.85	81.4±0.1	66.0±1.0
16	0.95±0.00	1.10±0.00	ND	ND	69.5±4.0	0.36±0.005	1.26±0.28	11.69±1.44	82.5±1.5	72.3±6.25
20	0.89±0.01	1.098±0.15	ND	ND	71.8±1.25	0.27±0.03	1.07±0.14	13.26±0.90	89.0±1.0	86.0±1.0

Mean values in columns are statistically different (ANOVA;  $P < 0.05$ ). ND, not detected

During the thermophilic stage, it was released due to higher degradation of organic matter and, at the final stages, this fraction was adsorbed by the zeolite. Cr was bound to the F1 and F2 fractions (about 7.2% of total) in the final compost of the control; however, these fractions observed in the range of about 1.42–1.68% of the total fraction in all zeolite treatments, these results were consistent with other findings (Zorpas *et al.*, 2008). Zorpas *et al.* (2008) reported that Cr was mainly bound to the F4 and F5 fractions during sewage sludge composting. However, in the present study, the F5 fraction of Cr contributed about 84–94% of total in the compost of the control and zeolite treatments. Zorpas *et al.* (2000) reported that Cr was mainly associated with the F4 and F5 fractions in the zeolite amended sewage sludge compost. However, in the present study, the F5 fraction of Cr was the dominant fraction. The variations in F1, F2, F3, F4 and F5 fractions of Cr in the control and zeolite treatments were significant ( $F = 36.0$ ,  $P < 0.001$  for F1,  $F = 55.20$ ,  $P < 0.001$  for F2,  $F = 15.44$ ,  $P < 0.001$  for F3,  $F = 26.39$ ,  $P < 0.001$  for F4,  $F = 14.45$ ,  $P < 0.001$  for F5).

The BF of Ni, Pb, Cd and Cr was reduced in the control and zeolite 1, 2 and 3 treatments, respectively (Figure 4). The highest reduction in the BF of Ni, Pb, Cd and Cr was observed of about 70.5, 72.7, 65.4 and 73.3%, respectively, in the zeolite 1 treatment during the composting process. A higher reduction in the BF of Ni might be due to the effect of zeolite, which has the ability to absorb and exchange easily available fractions of the Ni (Zorpas *et al.*, 2008). The maximum reduction in the BF of Pb and Cd which occurred in the zeolite 1 treatment, can be attributed to the higher degradation of organic matter, resulting in the conversion into a humus-like substance which formed a metal–humus complex during composting (Tiquia *et al.*, 1997; Zorpas *et al.*, 2008). The reduction in the readily available fractions of Cr could be explained by the zeolite having the ability to absorb and exchange metals (Zorpas *et al.*, 2008).

#### 4 CONCLUSION

The addition of natural zeolite during the composting process increased the pH of the initial feed mixture and organic matter degradation, consequently the speciation of the heavy metals was influenced. The higher reduction of the most bioavailable fractions (exchangeable and carbonate) and bioavailability factors of all the selected heavy metals was observed in the zeolite 1 and 2 treatments in comparison to the control and zeolite 3 treatment. The order of the BF of different metals in the compost of control was: Fe (0.56) > Mn (0.51) > Zn (0.23) > Cu (0.22) > Cr (0.14) > Cd (0.019) > Ni (0.014) > Pb (0.013); however, the order of these metals in the zeolite treatments was: Mn (0.23) > Zn (0.14) > Fe (0.13) > Cu (0.09) > Cr (0.066) > Cd (0.011) > Ni (0.009) > Pb (0.007). The total concentration of Zn, Cu, Mn, Ni, Cd and Cr was lower than Pb while the BF of these metals was higher. The orders of BF in the control and zeolite treatments revealed that the addition of natural zeolite was successful for reducing the BF of heavy metals and confirmed that the toxicity of metals was associated with its bioavailable fractions rather than the total

metal concentration. Therefore, the utilisation of natural zeolite was successful for reducing the bioavailability of heavy metals during rotary drum composting of the water hyacinth mixed with cattle manure and sawdust. On the basis of the overall results, the present study concludes that the use of the natural zeolite (optimum dose) leads to a better composting product suitable for agricultural or other purposes.

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