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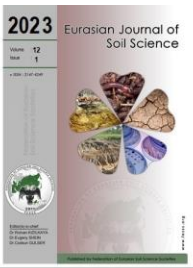

Reducing soluble lead and cadmium in contaminated soils using dairy cattle waste based vermicompost

Zainal Muktamar [a,*], Bandi Hermawan [a], Wulandari [a], Priyono Prawito [a], Fahrurrozi Fahrurrozi [b], Nanik Setyowati [b], Sigit Sudjatmiko [b], Mohammad Chozin [b]

[a] Department of Soil Science, University of Bengkulu, Indonesia
[b] Department of Crop Production, University of Bengkulu, Indonesia

Abstract

Continuous use of synthetic fertilizer can lead to the accumulation of heavy metals in the soil. The use of organic amendment can reduce the solubility of heavy metals such as Pb and Cd in soil. The experiment was undertaken to determine the decline of soluble Pb and Cd in polluted soils treated with dairy cattle waste-based vermicompost. The study used two soil samples; Inceptisols collected from Air Duku Village and Entisol from Beringin Raya Village, Bengkulu, Indonesia. Entisols and Inceptisols contained 2.0 and 0.4 mg kg^{-1} soluble Pb and 0.7 and 0.8 mg kg^{-1}soluble Cd, respectively. The samples were pretreated with either 100 ppm Pb or Cd. Vermicompost was applied at the rate of 0, 10, 20, and 30 Mg ha^{-1} on samples of Inceptisols and Entisol, arranged in Completely Randomized Design (CRD). The mixture was incubated for eight weeks. After the incubation ended, the soil sample was analyzed for soluble Pb and Cd using DTPA extraction before detection using Atomic Absorption Spectroscopy. The study resulted that the soluble Pb and Cd significantly reduced with vermicompost treatment, being the lowest was at the rate of 30 Mg ha^{-1}. Furthermore, the decreased soluble Pb and Cd was more substantial in Inceptisols than Entisols. Soluble Pb in both soils was lower than Cd, suggesting a higher retention affinity of the former. This study summarizes that vermicompost at the rate of 30 Mg ha^{-1} effectively immobilizes Pb and Cd in contaminated soils.

Keywords: Vermicompost, cadmium, lead, Inceptisols, Entisol.

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Author(s)

Z.Muktamar *
B.Hermawan
Wulandari
P.Prawito
F.Fahrurrozi
N.Setyowati
S.Sudjatmiko
M.Chozin

* Corresponding author

Introduction

As an integral part of farming practices, fertilizers and pesticides have a high contribution to the productivity of agricultural crops. FAO (2021) reported that the use of pesticides increased by 39% for a decade (2000-2019), while in the same period, the consumption of N, P, K fertilizer rose from 134.7 million tones in 2000 to 188.5 million tones in 2019 (40%). On average, N, P, K fertilizer per cropland was 122 kg/ha in 2019, rising from 91.3 kg/ha in 2000. As a result, the production of agricultural crops, including cereals, sugar crops, vegetables, oil crops, fruits, roots and tubers, and others, reached 9,356 million tonnes in 2019. This uplifted consumption of these agrochemicals resulted in soil degradation and water pollution. Moreover, the long-term application of inorganic fertilizers and pesticides in agriculture has led to the contamination of soils by heavy metals (AlKhader, 2015; Gebeyehu and Bayissa, 2020; Kinuthia et al., 2020, Wei et al., 2020; Mng'ong'o et al., 2021a).

Phosphate fertilizers, among inorganic fertilizers, are a primary source of heavy metal contaminants in agricultural soils (Kelepertzis, 2014). During the production of certain inorganic phosphate fertilizers from phosphate rocks, inadvertently has added heavy metals such as cadmium (Cd), lead (Pb), arsenic (As), zinc (Zn), strontium (Sr), etc. to the soil (Khan et al., 2018). Phosphate fertilizer contains 11 ppm Cd and 12.5 ppm Pb (Gimeno-García et al., 1996; Setyorini et al., 2003). According to Atafar et al. (2010), the application of

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fertilizer and pesticides significantly increased the concentration of Pb, As, and Cd in soils compared to non-wheat fertilized fields. Mng'ong'o et al. (2021b) detected Cu, Zn, Cd, Pb, and nickel (Ni) in plant samples leading to the possible risk to human and animal health.

In the last few decades, several techniques have been developed to immobilize heavy metals in contaminated soils, such as the use of organic materials. A review by Lwin et al. (2018) indicated that the application of organic amendment could reduce the solubility of heavy metals in soils. A previous study by Wang et al. (2020) revealed that soil treated with 1% organic matter had higher soil pH and lowered soluble Zn, Pb, and Cd than control (without treatment), leading to lower concentration in corn tissues. We have produced vermicompost from dairy cattle wastes for the organic farming system for the last ten years. Our previous study detected that vermicompost improved soil chemical properties. However, the effectiveness of vermicompost on the reduction of soluble heavy metals has not been evaluated intensively. Therefore, our current study aimed to determine the decline of soluble Pb and Cd in polluted soils treated with dairy cattle waste-based vermicompost.

Material and Methods

Soil Sample Collection and Experimental Design

The study used two soil samples collected to represent those from high and low altitudes in the humid tropical environment. The first soil sample was collected from Closed Agricultural Production System (CAPS) Research Station, Air duku Village, Selupu Rejang SubDistrict, Rejang Lebong Regency, Province of Bengkulu at 102°36' 54.96" E and 3°27' 34.26" S, the elevation of 1054 m above sea level. According to USDA Classification, the soil at the location was Andept, the order of Inceptisols. The second soil was sampled from Beringin Raya Village, Muara Bangkahulu SubDistrict, City of Bengkulu, Province of Bengkulu at 102°15'42.0" E, 3°45'20.9" S, elevation of 15 m above sea level. The soil was Psamment, the order of Entisols. The initial sample of Entisols had 0.7 mg kg^{-1} Cd, while Inceptisols 0.8 mg kg^{-1} (Table 1). Sukarjo et al. (2019) reported that soil contained Cd concentration above 9.3 mg kg^{-1}was considered a Cd contaminated soil. Likewise, the initial concentration of Pb in Entisols and Inceptisols was 2.0 and 0.4 mg kg^{-1}, respectively. According to Ericson et al. (2019) critical level of Pb in soil is 140 ppm. Composite soil samples were collected from 0-20 cm depths using a soil probe. The sample was air-dried and sieved using 2 mm screen. An undisturbed soil sample was also collected using a ring sample to determine field capacity (FC) moisture content. The initial characteristics of both soils are presented in Table 1.

Table 1. Initial characteristics of soil samples of Inceptisols and Entisols (Muktamar et al., 2021)

Soil Characteristics	Inceptisols	Entisols
Great Group	Andept	Psamment
Clay (%)	11.10	8.48
Silt (%)	34.94	10.54
Sand (%)	53.96	80.98
Organic-C (%)	2.86	2.34
Total-N (%)	0.19	0.32
Available P (ppm)	4.97	5.27
Exchangeable K (mmol kg^{-1})	1.90	2.41
Exchangeable Ca (mmol kg^{-1})	0.55	0.45
Exchangeable Mg (mmol kg^{-1})	1.17	0.17
Soluble Cd (mg kg^{-1})	0.80	0.70
Soluble Pb (mg kg^{-1})	0.40	2.00
CEC (cmol kg^{-1})	10.33	8.54
pH H_2O (1:1)	5.12	4.47

The study consisted of two set of experiments conducted at the Laboratory of Soil Science, Faculty of Agriculture, University of Bengkulu. Soil samples were pretreated with lead (Pb) or cadmium (Cd) using $Pb(NO_3)_2$ or $Cd(NO_3)_2$ solution, respectively. The study used a Completely Randomized Design (CRD) with two factors and three replications. The first factor was the rate of vermicompost, i.e., 0, 10, 20, and 30 Mg ha^{-1}, equivalent to 0, 5, 10, 15 mg g^{-1}, while the second factor was soil sample, consisting of Inceptisols and Entisols. Vermicompost was collected from CAPS Research Station and prepared using a method developed by Muktamar et al. (2017). The chemical properties of vermicompost were 7.98% C, 3.06% N, 5.96% P_2O_5, 1.27% K_2O, 1.79% Ca, 0.97% Mg, and pH of 5.47 (Muktamar et al., 2021).

Experimental Procedure

Vermicompost was homogeneously mixed with 0.3 kg of soil and placed in a 0.5 l transparent plastic bottle. The mixture was pretreated with 100 ppm Pb or Cd using $Pb(NO_3)_2$ or $Cd(NO_3)_2$ solution. The sample was watered to field capacity (FC) and weighed as a basal weight for field capacity moisture content. Each experiment set was randomly positioned on a 150 cm wooden table and incubated for eight weeks. Distilled water was added to the mixture every day to reach a similar basal weight. Soil pH and temperature were measured for a week, four weeks of incubation, and at the end of the experiment. Soil pH measurement used pH meter at a 1:1 soil and distilled water ratio. Soil temperature measurement used a soil thermometer at 5 cm below the surface. At the end of the incubation, the soil was homogeneously incorporated and air-dried, ground, and passed through a 0.5 mm screen. The soil sample was analyzed for Pb or Cd using AAS after extraction with DTPA to determine their soluble form.

Data Analysis

Data were assessed using ANOVA using SAS University Edition at 5% level. Means among treatments were compared using DMRT at a 5% level.

Results

Treatment of Pb on continuously lowered soil pH during the incubation, as shown in Figure 1. Soil pH of Inceptisols at 4 and 8 weeks of the incubation was 9% and 14% lower than the first week, respectively, when the soil was treated with Pb (Figure 1a), while those of Entisols decreased by 2% and 9%. A similar fashion was observed at Cd treatment (Figure 1b). The soil pH of Entisols decreased by 2% and 12% at the 4 and 8 weeks of incubation, respectively, compared to the first week, while those of Inceptisols were 14% and 24%, respectively. Soil pH decline is more substantial in weeks 4 to 8 than previous one. The decrease in soil pH is more significant when soil is treated with Pb than with Cd.

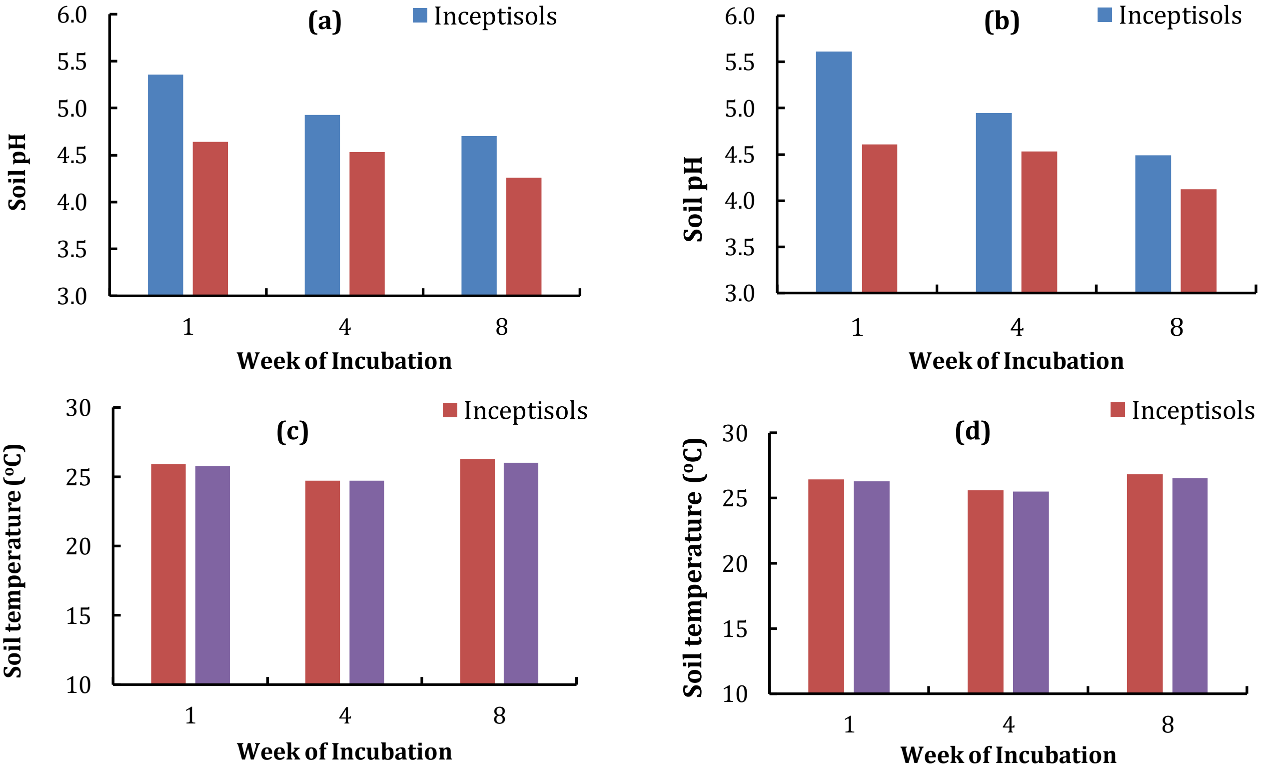

Figure 1. (a) pH of Pb treated soil at 1, 4, and 8 weeks of the incubation; (b) pH of Cd treated soil at 1, 4, and 8 weeks of the incubation; (c) Temperature of Pb treated soil at 1, 4, and 8 weeks of the incubation; (d) Temperature of Cd treated soil at 1, 4, and 8 weeks of the incubation.

Treatment of Pb or Cd did not significantly affect the temperature for both soil samples (Figures 1c and 1d). On average, the temperature of Inceptisols ranged from 24.7 ^{o}C to 26.3 ^{o}C for Pb treated soil, and 25.6 ^{o}C to 26.8 ^{o}C for Cd treated soils. Likewise, the temperature of Pb treated Entisols was 24.7 ^{o}C to 26.0 ^{o}C, while that of treated Cd was 25.5 ^{o}C to 26.5 ^{o}C. These results indicate that temperature has a similar effect on the vermicompost mineralization for soils treated with Pb or Cd.

Even though the soil pH continuously declined for the period of the incubation, vermicompost considerably increased the pH of Pb or Cd treated soils (Table 2). Treatment of vermicompost at a dose of 30 Mg ha^{-1} raised Pb treated soil by approximately 7% at week 1 and 8% at week 4 of the incubation compared to control, but the effect was insignificant at week 8. Table 2 also shows that the pH of Cd-treated soil increased significantly at the first, fourth, and eighth weeks of the incubation as the vermicompost rate increased. Soil pH increased by 8% in the first week and by 7% in the fourth week when the vermicompost rate increased by 30 Mg ha^{-1} compared to control. However, the pH of Cd treated soil at week eight was continuously expanded by applying 30 Mg ha^{-1}vermicompost as compared to control. This result indicates that neutrality of the acidity is faster for Pb than in Cd-treated soils.

Table 2. pH of Pb and Cd treated soil as affected by vermicompost.

Vermicompost (ton/ha)	Pb treated soil			Cd treated soil		
	Week 1	Week 4	Week 8	Week 1	Week 4	Week 8
0	4.81 a	4.51 a	4.48 b	4.84 a	4.56 a	4.11 a
10	4.97 ab	4.69 b	4.43 b	5.11 b	4.69 b	4.29 b
20	5.09 b	4.85 c	4.54 b	5.24 bc	4.82 c	4.33 b
30	5.14 b	4.87 c	4.57 b	5.25 c	4.88 c	4.50 c

Note: Numbers followed by the same letter in the same column are not significantly different using DMRT at 5%

Temperatures for both Pb and Cd treated soils were not affected by the application of vermicompost, as shown in Table 3. On average, soil temperatures of Pb treated soil during the incubation ranged from 24.3 ^{o}C to 26.7 ^{o}C. Meanwhile, the average soil temperatures of Cd-treated soil were 25.5 ^{o}C to 26.8 ^{o}C. These results indicate that vermicompost decomposition does not significantly release the heat during the incubation.

Table 3. The temperature of Pb and Cd treated soil as affected by vermicompost

Vermicompost (ton/ha)	Soil Temperature (^{o}C)					
	Pb treated soil			Cd treated soil		
	Week 1	Week 4	Week 8	Week 1	Week 4	Week 8
0	26.0	24.7	26.0	26.3	25.5	26.5
10	25.8	24.8	26.0	26.5	25.3	26.7
20	25.5	24.8	26.0	26.5	25.7	26.8
30	26.0	24.3	26.7	26.0	25.7	26.7

Note: Numbers followed by the same letter in the same column are not significantly different using DMRT at 5%

The application of vermicompost substantially lowered soluble Pb of the soils treated with lead (Figure 2). The reduction of soluble Pb in Inceptisols was approximately 72% when treated with 30 Mg ha^{-1} vermicompost compared to the control; however, a rate of 10 or 20 had not been able to lower the soluble metal in the soil. A similar trend was observed in Entisols, where the application of 30 Mg ha^{-1} vermicompost reduced soluble Pb by as much as 26% compared to the control. There was no significant reduction of soluble Pb at the rate of 10 or 20 Mg ha^{-1}. Figure 2 also indicates that Inceptisols had lower soluble Pb than Entisols, suggesting that Entisols are more vulnerable to Pb contamination.

Similar to Pb treated soil, vermicompost treatment also lowered soluble Cd, as indicated in Figure 3. It was found that soluble Cd was significantly lower by treating 30 Mg ha^{-1} vermicompost than other rates. Soluble Cd in Inceptisols treated with 30 Mg ha^{-1} is 25.7% lower than the control, but both rates of 10 and 20 Mg ha^{-1} have not affected the reduction of the soluble Cd. Figure 3 also shows that Entisols treated with 30 Mg ha^{-1} had 24.7% lower soluble Cd than the control but were not different from that of 20 Mg ha^{-1}.

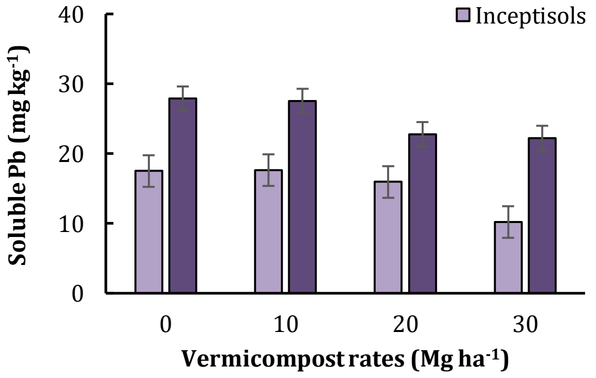

Figure 2. Soluble Pb as affected by vermicompost in Inceptisols and Entisols

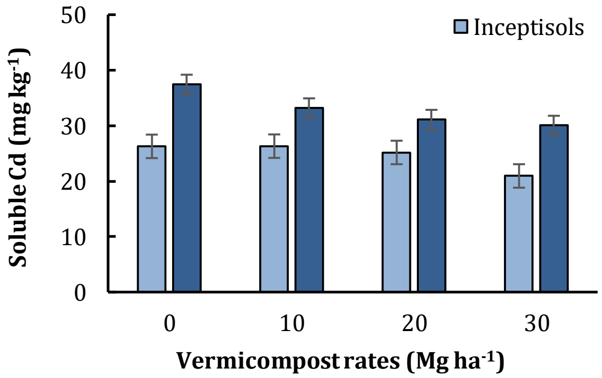

Figure 3. Soluble Cd as affected by vermicompost in Inceptisols and Entisols

A comparison between soluble Pb and Cd is presented in Figure 4. The figure shows that soluble Cd is always higher than soluble Pb at each rate of vermicompost. As the vermicompost rate is higher, the difference between both metals increases. At the control, soluble Pb is 40.4% higher than soluble Cd, while at the highest rate is more than 57%. This result indicates the different characteristics of both heavy metals.

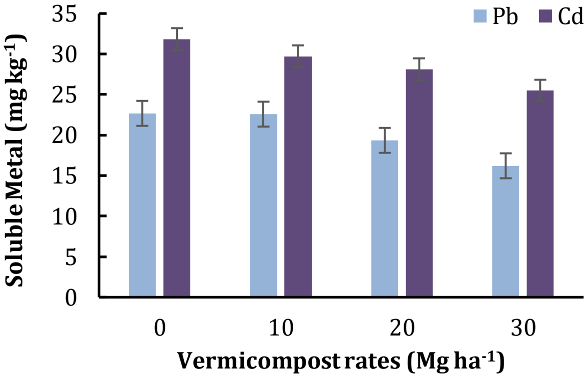

Figure 4. Soluble Pb and Cd as influenced by vermicompost

Correlation analysis shows that soil pH significantly correlates to soluble Pb with an r of 0.75 (Figure 5a). As soil pH is higher, soluble Pb declines. Likewise, soluble Cd decreases with increasing the soil pH, as shown in Figure 5b. Soluble Pb lowers by 59.2% as pH increases from 4.0- to 4.5, while soluble Cd decreases by 22.2% at the same pH range. These results suggest that the solubility of both Pb and Cd in the soil is dependent on the pH of the system.

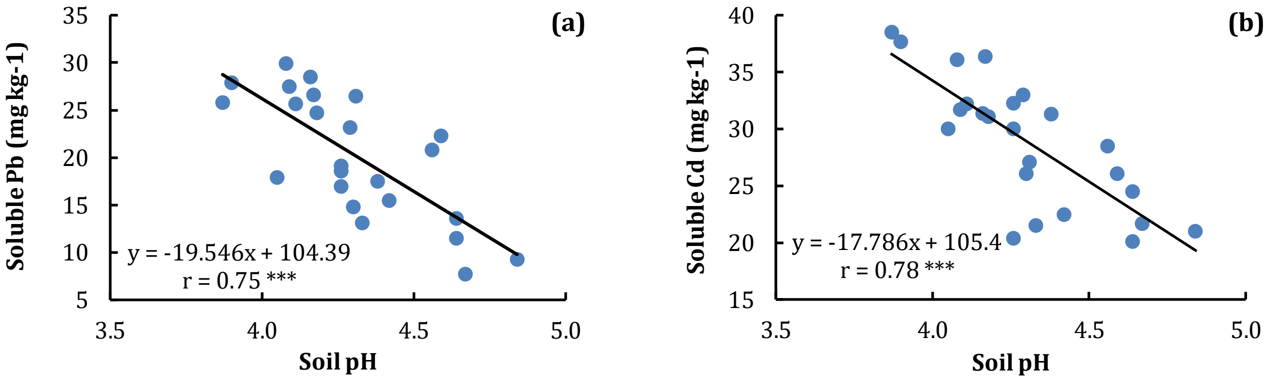

Figure 5. Correlation between soil pH and soluble Pb (a) and soluble Cd (b)

Discussion

In general, soil pH continuously decreases during eight weeks of the incubation, and the application of vermicompost significantly increases soil pH each week for both samples of Inceptisols and Entisols (Figures 1, Table 2). The decline in the pH of Pb or Cd treated soils is associated with the hydrolysis of the heavy metal as follows $M^{2+} + H_2O \rightarrow M(OH)_2 + 2\ H^+$ (Cruywagen and Van der Water, 1993; Barysz et al. 2004); M denotes Pb or Cd. As hydrolysis proceeds, higher proton release will lower soil pH. Meanwhile, the increase in soil pH by adding vermicompost is associated with the formation of organo-metal complexes, reducing their hydrolysis. Consequently, lower Pb and Cd hydrolysis will raise the soil pH.

Application of vermicompost significantly decreased the soluble Pb and Cd in both Inceptisols and Entisols (Figures 2 and 3). Organic matter decomposition releases organic acids, mainly humic and fulvic acids, rich in carboxyl and phenolic functional groups. These groups bond metals to form complexes (Spark, 2003; Barančíková and Makovníková, 2003; Boguta and Sokolowska, 2013), reducing their solubility. Carboxyl is a major functional group from humic acid that interacts with heavy metals (Spark et al., 1997). A previous study by Klučáková and Pavlíková (2017) found that lignitic humic acid strongly bound Pb and Cu. Another possible mechanism suggested by Qu et al. (2019) is the formation of hydroxide-humic acid metal complexes. Their results also showed that Pb and Cd immobilized in co-precipitation complexes were similar to their adsorption. The result of this study corresponds to that suggested by Boechat et al. (2016), where humic and fulvic acids substantially declined in selected heavy metals, including Pb and Cd.

The soil sample used in this study exhibits different retention of Pb and Cd where Inceptisols retains Pb and Cd higher than Entisols, as indicated by the amount of soluble form of both metals (Figures 2 and 3). This result is associated with the initial characteristics of both soils. Inceptisols have higher clay content, soil organic matter, and CEC than the other soil. Higher CEC will adsorb more metals due to higher negative charges in the soil. Humic substances at a pH of more than 3.0 also contribute to a negative charge and the formation of organo-metal complexes (Spark, 2003).

Our study also shows that soluble Pb in both soils was significantly lower than soluble Cd (Figure 4). The affinity of Pb to soil adsorption sites is higher than Cd; consequently, higher retention of the former element. The adsorption affinity (Kd) of Pb was greater than Cd, as reported by Usman (2008) and Covelo et al. (2007). Another study confirmed that the amount of Pb adsorbed by humic substances is higher, and the stability constant of the complex is greater than Cd. On the other hand, Cd bonded weaker to the substance than Pb (Klučáková and Pavlíková, 2017). Higher retention of Pb by the soil will lower its soluble form.

The correlation of pH and soluble Pb or Cd shows the dependency of the solubility of the metal on soil pH (Figure 5). As soil pH rises, soluble Pb and Cd are lower. Some studies show that the concentration of soluble Pb and Cd continuously declines as soil pH increases (Ok et al., 2010; Zhang et al., 2018; Soltan et al., 2018).

Conclusion

Addition of vermicompost significantly reduced the soluble Pb and Cd of Inceptisols and Entisols, being the lowest was achieved at 30 Mg ha^{-1}. The reduction of both heavy metals was more prominent in Inceptisols than Entisols. Soluble Pb in both soil samples was lower than Cd, indicating the higher retention affinity of the former. The decrease in the soluble heavy metals using vermicompost was followed by the increase in soil pH. This finding suggests that vermicompost at the rate of 30 Mg ha^{-1} is effective for the remediation of Pb and Cd contaminated soils.

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