THE DTPA-EXTRACTABLE HEAVY METALS IN TROPICAL SOILS TREATED WITH LIME MATERIALS

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ABSTRAK

Soils are prone to pollution by heavy metals from various sources, but their capacities to immobilize heavy metals are limited. This research was intended to study the influence of some lime materials on the, extractability of some heavy metals in soils. Tropical soils (Oxisols, Ultisols and Alfisols), collected from Lampung, Indonesia, were used. The soil samples (air-dry, 2-mm-sieved) were spiked with standard solution containing Cu, Cd, Zn, and Mn and treated independently with lime materials (CaCO3, Ca(OH)2, or CaMg(CO3)2) to bring the soils' pH about 2 units higher. The soluble heavy metals were extracted with DTPA and determined with flame AAS after an 8-week incubation at the soils' field moisture capacities. The results showed that in the soils not spiked with heavy metals, at all time, this significantly decreased the extractable Cu, Cd, Zn, and Mn, in a negative relation with the changes in soil pHs, increased with lime addition. The extractable heavy metals in the soils spiked with heavy metals were significantly higher than those in the soils not spiked with heavy metals, but the effect of lime in lowering the extractable heavy metals was also clearly observed. The extractable heavy metals were generally higher in soils treated with CaMg(CO3)2 than in soils treated with CaCO3 or Ca(OH)2, probably because of the effect of Mg ions in displacing the metals cations from soil adsorption surfaces. The degree of recovery of heavy metals suggested that parts of the heavy metals added were strongly adsorbed by the soils. Based on the percent recovery values, the order of the soil adsorption capacities with respect to Cu, Cd, and Zn followed the order of : Alfisols > Oxisols > Ultisols. The soil preference toward the heavy in:tals generally followed the order of : Cu > Cd > Zn.

Keywords: heavy metals, manganese, copper, zinc, cadmium, lime, tropical soils.

ven though some are essential for the living things, all heavy metals have been suggested to be detrimental to human health at elevated concentrations, partly due to their negative effects on human physiological functions. For example, soil and water pollution by cadmium (Cd) has been reported to be the reason for the emergence of "Itai-itai" disease that caused the deaths of the Japanese women in 1940s (Alloway, 1990). Mercury (Hg), which is also highly toxic may cause health problems, exemplified by the "minamata" disease occurred in Japan around 1950s (Steinnes, 1990). While it is essential for plants, at elevated concentrations, copper (Cu) may also cause toxicosis in humans known as Wilson's disease (Baker, 1990). Djuangsih (1992) has documented some of the negative effects of heavy metals on human physiological functions.

mediated by certain mechanisms, of which the heavy-

The intake of heavy metals by human beings may be

metals solubilization in soils is most important, because soils are open systems on which heavy metals from various sources may accumulate easily. Heavy metals in soils are actually distributed in two major phases : solid and liquid. Most parts of heavy metals adsorbed onto the solids phase are considered not being available to plants and may resist leaching processes. Unlike the soluble heavy metals in soil water, the solid phase may restrict plant absorption and the probable ground-water contamination and, thereby, it may restrict heavy metal intake by humans through food chains. Unfortunately, the soil-solid absorption capacity with respect to heavy metals is limited and cannot accommodate excessive amounts of heavy-metal inputs. Therefore, accumulation of heavy metals in soils may finally cause heavy metal influx into human bodies through food chains unless the soil adsorption capacity is enlarged in one way or another.

Some data on heavy metal accumulation are available in current literature (Ma and Lindsay, 1995; 1993; Ville et al., 1995; Alloway, 1990; Davies, 1990, Leung, 1988), but no accessible data on this phenomenon is available for tropical soils of Indonesia. The only data which has been recorded is the heavy metal accumulation in the Bay of Jakarta fed in by the river systems in Ciliwung watershed, which locates hundreds of industrial activities (DPMA, 1983). This data shows high rates of heavy metal transported to the bay. It is estimated that heavy metals were transported into the bay through the river systems at the rates (in kg day⁻¹) of : 325 (Cr), 191 (Cu), 362 (Hg), 154 (Ni), 757 (Pb), and 43.000 (Zn) (DPMA, 1983). By inference, it is probable that heavy metals are also deposited on agricultural farms in the vicinity of Jakarta through fly-ash from the center of industries, which can also occur in any agricultural farms close to any center of industrial activities (Sweet et al., 1993). In addition, agrochemical such as fertilizers and pesticides, which are used intensively, are also important heavy metals sources that may contaminate agricultural lands (McLaughlin, et al., 1996; Nicholson et al., 1994; Herrero and Martin, 1993; Alloway, 1990; Baker, 1990). He and Singh (1994) recently reported that high-Cd NPK-fertilizers significantly increased the soil extractable Cd, which correspond to the total Cd uptake by plants. Nicholson et al. (1994) also showed Cd accumulations in the phosphate-fertilizer-treated soils. These phenomena are of great concern in the long-run and efforts should be done to lower their mobility's and ameliorate their detrimental effects on the living systems.

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There are numerous suggestions that can be done, aside from lowering the disposal of heavy-metalcontaining wastes to agricultural lands, for example, treatment to immobilize heavy metals to restrict their movements in reaching the ground-water and to reduce reasonably their availability's to plants (Salam, 1996; Ruby et al., 1994; Ma et al., 1993). Of these methods, addition of lime materials to soils is most reasonable for tropical soils, which, in general, possess low cation exchange capacities (CEC) or adsorption capacities but high degree of pH-dependent charges (Summer et al., 1991). Addition of lime materials have been shown to increase the soil pHs through neutralization processes of acid components such as soluble and exchangeable H and Al ions. The increase in soil pHs may increase the ionization of H-ions bound on the soil exchange-sites or the soil's organic functional groups such as carboxyl and, thereby, may increase the negative charges on the soil solid surfaces which function to hold cations including the heavy metal cations (Rodella et al., 1995; Salam, 1995; Helling et al., 1964). Researchers also suggested that the specific adsorption of heavy metals in soil increases with increasing pHs (Bohn et al., 1985). All these processes may eventually immobilize heavy metals from the soil solution and decrease their availability to plant and movements in soils. Sing et al., (1995) recently showed that increasing soil pHs decreased the plant uptake of Ni and Zn. Berthelson et al., (1994) also showed that liming decreased the leachability of Zn in soils.

There is evidence on the inverse relationship between soluble heavy metals and soil pH, that can be manipulated with lime addition (Salam, 1996; 1995; Helmke et al., 1995; Tack et al., 1995; El-Falaky et al., 1991; Ma and Lindsay, 1995; 1993; 1990; Workman and Lindsay, 1990). However, such data for tropical soil accessible to the authors is scanty. This research was intended to study the influence of lime materials on the extractability of heavy metals in tropical soil spiked with heavy metals.

MATERIALS AND METHODS

Soil samples were collected from 3 different locations in Lampung, Indonesia, i.e., Tanjungan (south Lampung), Gedongmeneng (Bandarlampung), and Banjaragung (Central Lampung). The soil samples represented three soil orders: Ultisols, Oxisols, and Alfisols, respectively. After collection, the soil samples were air-dried and sieved to 2 mm. Before being used in the experiments, each soil sample was mixed thoroughly. Selected properties of the soil samples are listed in Table

A plastic container containing 300 g of air-dry soil sample was used as an experimental unit. The soil sample was treated with lime solution to raise the soil pH

about 2 units. The amount of lime added (5 Mg Ca (OH)₂ ha⁻¹) was determined based on a soil neutralization curve of Ultisols with Ca (OH)₂. The level of lime materials other than Ca(OH)₂ were calculated based on their respective neutralization powers, which were 136 for Ca(OH)₂, 100 for CaCO₃, and 109 for CaMg(CO₃)₂. Each lime material was given at the same level for every soil sample. Experimental units with soil samples spiked with 40 ml of a standard solution containing 75 mg 1⁻¹ of each heavy metal studied (Cu, Cd, Mn, and Zn) were also prepared. This metal addition was intended to load the soil with 10 mg kg⁻¹ of each heavy metal. For each treatment, a control experimental unit was also prepared. Each treatment was replicated 3 times.

Table 1. Properties of Ultisols, Oxisols, and Alfisols*

| Soil properties | Ultisols | Oxisols | Alfisols |
|-------------------------------------------|----------|---------|----------|
| pH (112), 1:2) | 4.65 | 5.62 | 5.19 |
| CEC (emol _c kg ⁻¹) | 4.35 | 3.99 | 5.47 |
| Exch Cations (cmol,kg ⁻¹) | | | |
| Ca | 5.35 | 4.79 | 2.17 |
| Mg | 9.35 | 1.40 | 0.937 |
| K | 0.530 | 0.260 | 0.641 |
| Na | 0.240 | 0.0865 | 0.130 |
| Al | 0.519 | 0.0250 | 0.100 |
| Soluble heavy metals (mg kg-1) | | | |
| Zn | 18.2 | 10.7 | 10.8 |
| Cu | 2.15 | 0.868 | 2.40 |
| Cd | 0.270 | 0.138 | 0.208 |
| Mn | 90.4 | 535 | 518 |
| Extractable P (mg kg-1) | 11.3 | 3.12 | 10.3 |
| Texture (%) | | | |
| Sand | 23.6 | 45.6 | 22.0 |
| Silt | 29.6 | 19.6 | 21.2 |
| Clay | 46.8 | 34.8 | 56.8 |

^{*} average of 3 replicates

After a thorough mixing, the mixtures were moistened with distilled water up to the soils field moisture capacities and incubated for 8 weeks at room temperature. The soils field moisture capacities were determined by a gravimetric method. During the incubation time, the containers were tightly covered to minimize water evaporation. Soil analysis included: soil pH (1:2 soil: water), M NH4OAc-exchangeable majorcations (exchangeable Ca, Mg, K, and Na), acidity (KCl-Al), Bray-1P, and DTPA-extractable heavy metals, which included Cu, Cd, Mn, and Zn. Cadmium was to represent the non-nutrient heavy-metals, while Cu, Mn, and Zn to represent the nutrient heavy-metals.

RESULTS AND DISCUSSION

Changes in the Soil pHs

As expected, all lime materials increased the pHs of each soil (Figure 1). These changes had indirectly affected the solubility's of heavy metals. The increase in soil pHs may have increased the soil adsorption capacities and, consequently, decreased the solubility's of heavy metals. This important relationship will be discussed later in the following sections.

Difference among the effect of different lime materials on the extent of the pH increase was not observed (Figure 1). Each lime material increased the pH of Ultisols higher than the pH of Oxisols or Alfisols. In soils not spiked with heavy metals, the addition of lime materials on the average increase of the pH of Ultisols was up to 2.57 units, while the pH of Oxisols and Alfisols were increased by 1.47 and 1.60 units, respectively. In soil spiked with heavy metals, the increase was 2.24 for Ultisols and 1.98 and 1.66 for Oxisols and Alfisols, respectively. These observations show that Ultisols possess a lower pH buffering capacity than the Oxisols and Alfisols.

The decrease in soil pHs with the addition of heavymetals standard solution (Figure 1) was due to the acidity of the solution, which was commercially prepared with dilute nitric acid. In average, the decrease in soil pH due to the addition of heavy-metal standard solution were 0.74, 0.65, and 0.47 units for Ultisols, Oxisols, and Alfisols, respectively, in limed soils and 2.57, 1.13, and 0.53, respectively, in unlimed soils. The observation also shows that Ultisols demonstrated a lower pH buffering capacity than the other two soils. The pH buffering capacity of the soil follows the sequence: Alfisols > Since pH buffering capacity is Oxisols > Ultisols. associated with the amount of H-ions bound on functional groups in soil, this sequence clearly shows the sequence of the size of the soil negative charges that can bind with heavy metals/cations. This may explain the fact that the

sequence, as will be shown in the following section, corresponds to the sequence of soil adsorption capacity with respect to heavy metals.

Changes in the Solubility of Heavy metals

The changes in the solubility of heavy metals (Figures 2 to 5) were related to changes in soil pH caused by lime addition (Figure 1). In general, lime addition decreased the solubility's of heavy metals. For example, in Ultisols not spiked with heavy metals, lime addition decreased the soluble Zn as much as 43%, Cu 46%, Cd 31%, and Mn 32%. A similar trend was observed in Oxisol and Alfisols (Table 2). This observation shows that the increase in soil pH might have increased the soil adsorption capacity with respect to heavy-metals cations. Some sources of negative charges which probably cause this phenomenon were functional groups of clay minerals and sesquioxides and high molecular-weight organiccompounds, which might have probably dissociated their H' ions by increase in the soil pHs and, thereby, increased the number of the negative charges. A similar phenomena has been previously observed in a laboratory experiment using CaO (Salam, 1996).

The above pattern was much more clearly observed in soils spiked with heavy metals (Figures 2 to 5). In average, lime decreased the soluble-heavy metals in Ultisols as much as 34% for Zn, 41% for Cu, 12% for Cd. and 25% for Mn. These values were lower than their respective values in Ultisols not spiked with heavy metals. This phenomenon was related to the difference in changes in the soil pHs (ApH) caused by lime addition. The average value of ▲pH for Ultisols spiked with heavy metals was 2.24, lower than that for soils not spiked with heavy metals (▲pH) 2.57). The value of ▲pH has been reported to be inversely related to changes in the solubility of heavy metals (AM) (El-Falaky et al., 1991; Ma and Lindsay, 1990) and, thus, was directly related to the % decrease in the soluble heavy metals caused by lime addition.

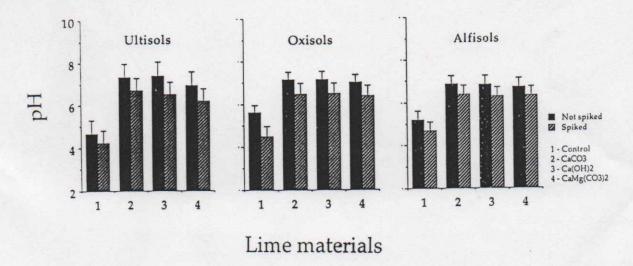


Figure 1. The influence of lime materials (A) and organic composts (B) on soil pH (bar indicates standard error).

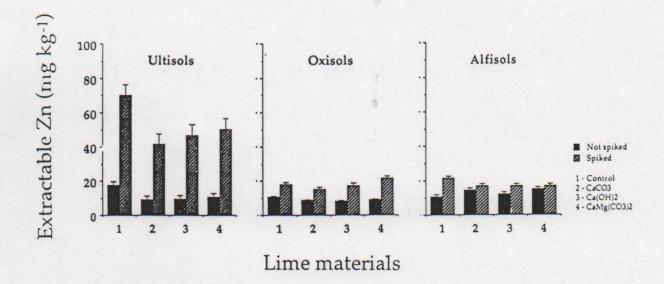


Figure 2. The influence of lime materials on the extractable Zn (bar indicates standard error)

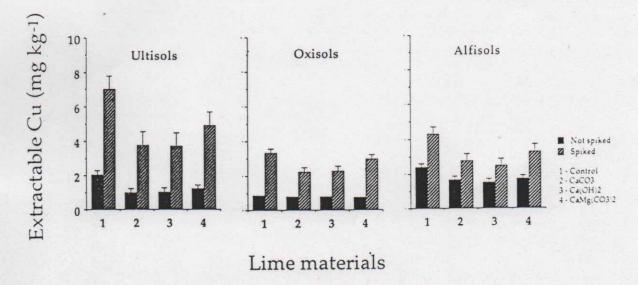


Figure 3. The influence of lime materials on the extractable Cu (bar indicates standard error)

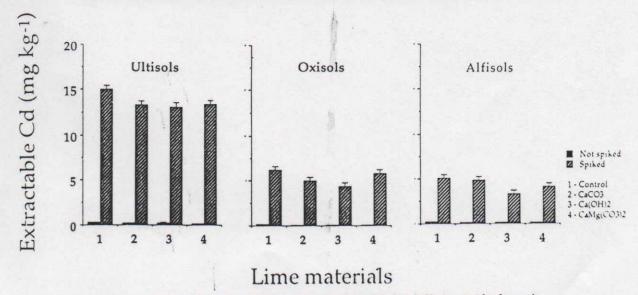


Figure 4. The influence of lime materials on the extractable Cd (bar indicates standard error)

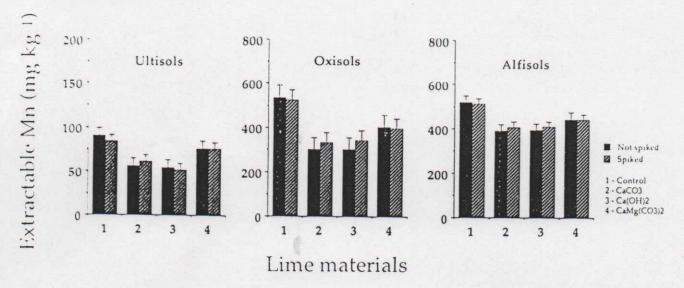


Figure 5. The influence of lime materials on the extractable Mn (bar indicates standard error)

The above patterns were also observed in Oxisols and Alfisols, except for Cu. In contrast to the above patterns, the immobilizing power of lime for Cu in Oxisols and Alfisols (shown by the % decrease in Table were increased with addition of the heavy-metal standard solution. It is probable that decrease in the soil pH brought about by the standard solution in these two soils had shifted Cu ions to a stronger adsorption by oxide minerals in the soils. This possibility is reasonable because Oxisols, which might have had high content of oxide minerals, showed a greater increase in the immobilizing power compared with the Alfisols, which might have had oxide content. Ultisols, which might have been dominated by clay minerals, did not show this phenomenon (Table 2). A careful observation of Figure 2 to 5 also reveals that the absolute adsorption capacities of Oxisols and Alfisols were much higher than that of Ultisols, shown by the disappearance of larger parts of the heavy metals added in Oxisols and Alfisols than in Ultisols (Figure 2 to 5). This property is associated with the higher oxide contents of Oxisols and Alfisols than that of Ultisols.

The addition of heavy-metals standard solution, which based on a simple calculation would have increased each heavy metals solubility in soil as high as 10 mg kg⁻¹, and this indeed drastically increased the solubility of heavy metals, except for Mn. Other than for the heavy metals added into soils through the standard solution, the increase was attributed to decrease in the soil pH caused by the addition of standard solution (Figure 1). However, a calculation indicates that parts or all of the added heavy metals were strongly adsorbed by the soil. For example, in average only 50% out of the 10 mg kg⁻¹ Cu added was recovered from Ultisols not treated with lime and 30% from Ultisols treated with lime. All Mn added into Ultisols was not recovered.

Table 2. Decrease in the average percentage of DTPA-extractable heavy-metal caused by lime addition.

| Heavy metal addition | Soil order | Zn | Cu | Cd | Mn | |
|----------------------|------------|------|------|------|------|--|
| mg kg-1 | | %* | | | | |
| 0 | Ultisols | 43.4 | 46.1 | 31.1 | 31.7 | |
| | Oxisols | 17.4 | 10.7 | 46.3 | 37.4 | |
| | Alfisols | 33.0 | 32.1 | 31.0 | 21.0 | |
| 10 | Ultisols | 34.0 | 41.0 | 11.6 | 24.8 | |
| | Oxisols | 1.11 | 24.7 | 18.5 | 32.1 | |
| | Alfisols | 17.1 | 34.3 | 19.7 | 18.1 | |

* Calculated with an equation of [{M₍₀₎ - (\(\sum_{M(x)}\)3)}*100/M₍₀₎], where M_(x) is the soluble heavy metal in soil treated with lime x; x is either CaCO₃, Ca(OH)₂, or CaMg(CO₃)₂; and M₍₀₎ is the soluble heavy metal in soil not treated with lime.

Decrease in the soil pHs caused by addition of standard solution in fact greatly affected heavy metals solubility, except for Mn. The addition of standard solution increased the solubility of Zn, Cd, and Cu in unlimed Ultisols as high as 5.2, 1.5, and 0.5 times, respectively, and in limed Ultisols as high as 3.6, 1.3, and 0.3 times, respectively, relative to the amount added to the Although the magnitudes were significantly soil. different, Oxisols and Alfisols in general showed the same pattern (Figure 2 to 5). This observation shows that parts of heavy metals previously adsorbed or precipitated was drastically dissolved as a consequence of the decrease in soil pH. This data suggests that disposal of heavy-metal containing wastes into the soil systems may increase heavy-metal solubilization due to its: (1) heavy-metal contents and (2) acidity, which may indirectly dissolve heavy metals immobilized in soils. For such wastes, neutralization processes should be conducted before disposal.

Recovery of the Heavy Metals Added into Soils

The recovery values of heavy metals from all are given in Table 3. The recovery values indicate the percentage of the amount of each heavy metal added into the soil that can be re-extracted by DTPA after soil incuoation. Low values indicate huge immobilization and high values indicate the opposite situation. The values greater than 100 indicate that dissolution or desorption of the heavy metals had occurred in soil while the negative values indicate that some heavy metals previously soluble had precipitated or had been adsorbed along with those added though the standard solution. Dissolution and precipitation processes might have probably occurred due to the changes in soil pH brought about by the addition of standard solution or lime materials.

Table 3. The percent recovery of heavy metals from soils treated with lime

| Soil order | Lime | Zn | Cu | Cd | Mn |
|---------------|------------|------|------|------|---------|
| Ultisols | Control | 523 | 49.8 | 147 | 65.0 |
| | CaCO3 | 321 | 27.5 | 131 | 62.0 |
| | Ca(OH)2 | 371 | 26.7 | 128 | (-)26.0 |
| | CaMg(CO3)2 | 396 | 37.0 | 133 | 3.00 |
| Oxisols | Control | 73.0 | 24.4 | 60.3 | (-)90.0 |
| | CaCO3 | 67.0 | 14.5 | 49.0 | 310 |
| | Ca(OH)2 | 90.2 | 15.0 | 42.8 | 410 |
| | CaMg(CO3)2 | 124 | 21.9 | 56.8 | (-)40.0 |
| Alfisols | Control | 110 | 19.1 | 48.8 | (-)70.0 |
| | CaCO3 | 24.0 | 10.8 | 46.9 | 150 |
| | Ca(OH)2 | 46.0 | 9.50 | 31.7 | 150 |
| | CaMg(CO3)2 | 44.0 | 15.8 | 39.6 | (-)200 |

Calculated with an equation of [(M_{(x)1} - M_(x)))*100/M], where M_{(x)1} is the soluble heavy metal in soil treated with lime x and spiked with heavy metal; x is either CaCO₃, Ca(OH)₂, or CaMg(CO₃)₂; M_{(x)0} is the soluble heavy metal in soil treated with lime x but not spiked with heavy metal; and M is the amount of heavy metal added into soil (in this experiment it is 10 mg kg⁻¹)

Except that for Mn, the recovery values for each heavy metal in Oxisols and Alfisols are lower than that in Ultisols. This observation supports the suggestion that Oxisols and Alfisols possessed higher adsorption capacities than did Ultisols. This was probably related to sesquioxides in Oxisols and Alfisols which had larger specific adsorption surfaces. Based on the recovery values, the adsorption capacities of all soils generally follow the sequence of: Alfisols > Oxisols > Ultisols for Cu, Cd, and Zn. While the preference of all soils towards these heavy metals in general follows the order of Cu > Cd > Zn. The amount of Mn added through the standard solution was relatively low compared to its indigenous

concentration in soils, making the adsorption capacity of soils with respect to Mn is difficult to assess.

The addition of lime significantly reduced the recovery values for every heavy metal, meaning that lime increased the immobilization of heavy metals in soils. In general, CaCO₃ was as effective as Ca(OH)₂ in immobilizing heavy metals in all soils, but was more effective than CaMg(CO₃)₂, even though CaMg(CO₃)₂ possessed a higher neutralization power than the other two lime materials. This is probably attributed to the presence of Mg ions that might have prevented heavy metals from entering the adsorption sites in soils treated with CaMg(CO₃)₂.

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