



Evaluation of digested sludge as an amendment to chromium and lead contaminated Gangetic alluvial soils of India

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ABSTRACT

Indo-Gangetic belt of India possesses many industrial sites whereby effluents containing very high levels of heavy metals, especially Cr and Pb are directly discharged into the environment without any treatment or cleaning process leading to soil, plant and ecological toxicities. There has been increasing interest amongst scientific community to remediate the contaminated soils with cost-effective and environmentally safe methods. Keeping this in view, pot experiments were conducted to evaluate the effect of different levels of digested sludge addition to Gangetic alluvium, spiked with Cr or Pb up to 500 mg/kg on phytotoxicity in corn (*Zea mays* L.) and radish (*Raphanus sativus* L.) plants. Increasing sludge levels resulted in decreased levels of DTPA-extractable Cr and Pb in the soil. The recovery of these metals by DTPA declined considerably with the duration following treatment application. Lowest biomass yield of test plants was observed in the treatment where no sludge was applied. Increasing levels of digested sludge enhanced the dry matter yields and decreased the tissue concentrations of Cr and Pb in plants. Plant tissue metal concentrations across all treatments decreased from first to second crop which could be ascribed to decrease in their DTPA-extractable levels in soil, a result of reversion of the metals into less available form with time. The findings reveal that digested sludge has a potential to reduce the phytotoxicity of Cr and Pb and, therefore, can be used as an amendment for soils which have been adversely polluted with these metals by anthropogenic and industrial activities.

Key words: Amendment, Cr, Digested sludge, Pb, Phytotoxicity

Several carpet, leather, tannery and battery manufacturing industries operative in Indo-Gangetic belt of India discharge their effluents containing extremely high levels of metals particularly Cr and Pb into the environment posing a potential hazard to soils, ecosystem or food chain. Heavy metals have limited downward movement in soil (McGrath 1987) and therefore, accumulate in surface horizons (Boon and Soltanpour 1992). Unlike organic contaminants, these cannot be degraded by microorganisms. Currently, cleanup methods of soil metal pollution are expensive and environmentally destructive (Moffat 1995, Nanda *et al.* 1995). Recently, scientists and engineers have started to generate cost-effective and community acceptable remediation technologies for the metal contaminated soils.

Sewage sludge is the primary organic solid by-product from sewage water treatment plants treating domestic or urban sewage. It is a major waste product and produced in increasing quantities as additional sewage treatment is required to reduce the polluting impact of discharges from waste water systems. Application of sewage sludge to land has been demonstrated to be a feasible alternative for the disposal of sludge and also to reutilize the residual resource in the sludge. The residual resource in sludge consists of high nutrient and organic matter content, which represent a good fertilizer and/ or conditioner for soil and plants (Casado-Vela *et al.* 2006, Mahdy *et al.* 2007, Bozkurt *et al.* 2010). Organic matter constitutes the solid fraction of the sludge and it improves the physical condition of soil by decreasing bulk density, increasing soil aeration, water holding capacity, aggregate stability and promoting greater water infiltration (Lagae *et al.* 2009, N-Dayegamiye 2009).

The use of sewage sludge as an amendment for contaminated lands can reduce the bioavailability of a wide range of inorganic pollutants including metals while simultaneously enhancing re-vegetation and, thereby,

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Table 1 Initial characteristics of experimental soil and digested sludge

| pH (1:2.5) | EC (dS/m) | Organic carbon (g/kg) | Soil available levels (mg/kg) | | | | DTPA-extractable (mg/kg) | | | |
|------------------------|-----------|-----------------------|-------------------------------|-------|-------|-------|--------------------------|-------|------|------|
| | | | N | P | K | S | Zn | Ni | Cr | Pb |
| 7.5 | 0.24 | 5.5 | 106.9 | 10.7 | 117.3 | 11.6 | 0.7 | 0.4 | ND | 0.1 |
| <i>Digested sludge</i> | | | | | | | | | | |
| 6.2 | 1.97 | 94.0 | 16 800 | 4 000 | 3 400 | 2 800 | 594 | 108.2 | 68.4 | 47.3 |

ND, Non-detectable

protecting against offsite movement of contaminants by wind and water (Chiu *et al.* 2006; Roongtanakiat *et al.*, 2008). As such, it can be used in situations ranging from time critical contaminant removal actions to long-term ecological revitalization projects. In addition, this approach offers the benefit of recycling municipal residuals to reclaim damaged or disturbed land rather than disposing of what is generally considered to be waste in landfills or by incineration. This study was undertaken to find out the effect of digested sludge on phytotoxicity of Cr and Pb spiked in the form of metal salts to Gangetic alluvium, as measured by DTPA-extractable metal levels in the soil, biomass production and plant metal concentrations.

MATERIALS AND METHODS

Surface (0-22.5 cm) alluvial soil (Dystric Eutrudepts, texture sandy clay loam) of Indo-Gangetic plains was amended with air-dried and homogenized digested sewage sludge at 0, 30, 60, 240 and 480 g/kg and filled in five kilogram polythene-lined earthen pots. The initial characteristics of soil and digested sludge are given in Table 1.

The sludge was collected from a sewage water treatment plant located in Varanasi, Uttar Pradesh, India. The sludge amended pot soils were spiked with Cr or Pb so as to raise their levels up to 500 mg/kg via aqueous solutions of analytical grade chromium oxide and lead chloride salts. The high metal concentration was used to ensure complete phytotoxicity for the crops being tested. The treatment details of experiments are given as under:

| Treatment | Treatment symbol | |
|---|-------------------------------------|-------------------------------------|
| Digested sludge at 0 g/kg + Cr or Pb at 500 mg/kg | DS ₀ Cr ₅₀₀ | DS ₀ Pb ₅₀₀ |
| Digested sludge at 30 g/kg + Cr or Pb at 500 mg/kg | DS ₃₀ Cr ₅₀₀ | DS ₃₀ Pb ₅₀₀ |
| Digested sludge at 60 g/kg + Cr or Pb at 500 mg/kg | DS ₆₀ Cr ₅₀₀ | DS ₆₀ Pb ₅₀₀ |
| Digested sludge at 240 g/kg + Cr or Pb at 500 mg/kg | DS ₂₄₀ Cr ₅₀₀ | DS ₂₄₀ Pb ₅₀₀ |
| Digested sludge at 480 g/kg + Cr or Pb at 500 mg/kg | DS ₄₈₀ Cr ₅₀₀ | DS ₄₈₀ Pb ₅₀₀ |

The treatments were arranged in quadruplicate in a completely randomized design in a pot chamber at normal temperatures (25°C ± 5°C) and left to equilibrate at field

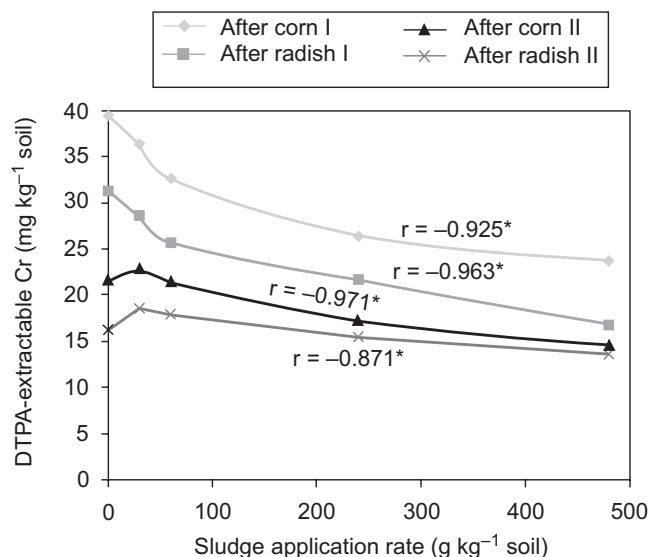
capacity for one year. Border pots were established to completely encircle all the treatments. At the end of equilibration period, the soil was again pulverized and the pots were refilled. Seeds of corn cv Kanchan and radish cv. Early Menu were sown twice in the order, corn - radish - corn - radish, during summer and winter seasons of 2005 and 2006. Upon emergence the plants were thinned to four per pot and grown for 6 weeks. Uniform doses of 60 mg N, 40 mg P₂O₅ and 30 mg K₂O to corn and 50 mg N, 30 mg P₂O₅ and 30 mg K₂O to radish/kg soil were applied as urea, diammonium phosphate and muriate of potash at the time of sowing. Nitrogen supplied through diammonium phosphate was adjusted in the amount of urea. Corn was cut just above the ground level with a stainless steel knife. Individual radish plants were uprooted manually and shoots were separated from roots with the stainless steel knife. The harvested samples were washed with double distilled water, dried at 70°C, weighed, crushed in a stainless steel grinder and stored in glass vials for metal analyses. Soil samples were also taken at each harvest by sampling the pots after remixing, air-dried, ground, passed through a 2 mm stainless steel sieve and stored in polythene bags until analyses. Organic carbon, nitrogen, phosphorus, potassium and sulphur in sludge and soil were determined following standard procedures advocated by Jackson (1973). Sludge, soil and plant samples were digested in 2:1 HNO₃-HClO₄ mixture (Jackson 1973) prior to determination of Cr and Pb by atomic absorption spectrophotometer (UNICAM, Model No. 969). Soil samples were extracted with 0.005 M DTPA (diethylene triamine penta-acetic acid) + 0.1 M TEA (triethanol amine) + 0.01 M CaCl₂ buffered at pH 7.3 according to Lindsay and Norvell (1978) and analyzed for Cr and Pb using atomic absorption spectrophotometer (UNICAM, Model No. 969). Suitable dilutions were made to extraction filtrates prior to analysis in case the metal concentrations were found outside the range of the instrument. Analytical quality assurance was addressed by the systematic use of blanks and duplicate samples.

RESULTS AND DISCUSSION

DTPA-extractable metals

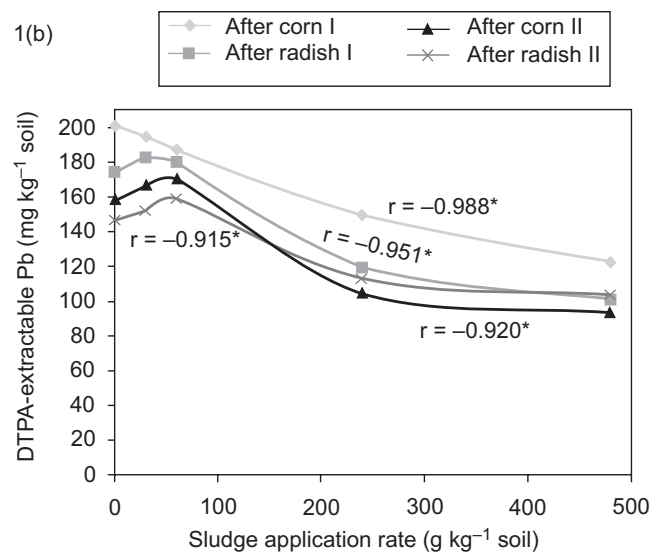
Results indicating the effect of digested sludge on DTPA-extractable Cr and Pb levels in soil are depicted in Fig 1 and 2.

Only a fraction of applied Cr and Pb appeared to be extractable with DTPA. Lead was extracted in larger amounts



*Correlations are significant at the 0.05 level
 ** Correlations are significant at the 0.01 level

Fig 1 Effect of digested sludge on DTPA-extractable or levels in soil



*Correlations are significant at the 0.05 level
 ** Correlations are significant at the 0.01 level

Fig 2 Effect of digested sludge on DTPA-extractable pb levels in soil.

in comparison to Cr by DTPA. Increasing sludge levels decreased the levels of DTPA-extractable Cr and Pb in the soil. The decreased extractability of Cr by DTPA could be explained on the basis of increased reduction of Cr (VI) to Cr (III) with the application of digested sludge followed by its subsequent adsorption on to sludge borne organic matter or on to negatively charged soil particles. The results confirm the findings of Bolan and Thiyagarajan (2001). Stewart *et al.* (2003) concluded that Cr (VI) is readily mobile while Cr

(III) is immobile in the soil environment. The decreased extractability of Pb by DTPA could be due to its complexation with high molecular weight humic substances generated during sludge decomposition (Bell *et al.* 1991). Basta *et al.* (2001) reported that municipal biosolid application to soil enhanced its ability to immobilize Pb, thereby limiting its plant availability. The results reveal that digested sludge possesses the ability to sorb Cr and Pb and remove them from the soil solution.

DTPA-extractable Cr and Pb levels tended to decrease after each crop however, extractability of Pb by DTPA at higher sludge levels (240 and 480 g/kg) was more after the third compared to the last crop. The decline in recovery of applied metals by DTPA with crops could be attributed to increase in the binding of the metals to soil colloids as well as their conversion to insoluble forms. Bruemmer *et al.* (1988) demonstrated that some mineral forms of contaminants may recrystallize over time into more stable minerals or adsorbed species may diffuse into host mineral or may be incorporated during the growth of the mineral surface. Fendorf *et al.* (2004) reported that the solubility and hence the mobility potential of metallic contaminants in soils decreases with time. This phenomenon, termed as ‘aging effect’ has been attributed to the conversion of more soluble solid-phase metal species into less soluble or chemically more stable species. Higher DTPA-extractable Pb levels at higher sludge levels after the third than the last crop might be assigned to production of low molecular weight fulvic acids from sludge that increase metal mobility, as they can complex metals previously bound on to soil solid constituents affecting their speciation and solubility (Harter and Naidu 1995).

Table 2 Effect of digested sludge on dry matter yields of corn and radish

| Treatment | Dry matter yield (g/pot) | | | |
|-------------------------------------|--------------------------|---------------------|---------------------|---------------------|
| | Corn I | Radish I | Corn II | Radish II |
| DS ₀ Cr ₅₀₀ | 6.5 ^a | 2.9 ^a | 18.0 ^a | 7.9 ^a |
| DS ₃₀ Cr ₅₀₀ | 9.4 ^{a,b} | 2.9 ^a | 20.9 ^a | 10.1 ^a |
| DS ₆₀ Cr ₅₀₀ | 13.0 ^{a,b} | 4.3 ^{a,b} | 26.6 ^{a,b} | 13.0 ^{a,b} |
| DS ₂₄₀ Cr ₅₀₀ | 18.7 ^b | 5.8 ^b | 41.0 ^b | 17.3 ^b |
| DS ₄₈₀ Cr ₅₀₀ | 34.6 | 8.6 | 69.1 | 26.6 |
| LSD (P=0.05) | 10.1 | 2.2 | 16.6 | 6.5 |
| SEm ± | 3.6 | 0.7 | 5.8 | 2.2 |
| DS ₀ Cr ₅₀₀ | 15.8 ^a | 13.9 ^a | 23.0 ^a | 30.2 ^a |
| DS ₃₀ Cr ₅₀₀ | 21.6 ^{a,b} | 18.3 ^a | 28.1 ^{a,b} | 32.4 ^{a,b} |
| DS ₆₀ Cr ₅₀₀ | 24.5 ^{a,b} | 22.8 ^{a,b} | 31.6 ^{a,b} | 35.3 ^{a,b} |
| DS ₂₄₀ Cr ₅₀₀ | 28.8 ^{b,c} | 30.1 ^b | 39.8 ^{b,c} | 43.4 ^{b,c} |
| DS ₄₈₀ Cr ₅₀₀ | 37.4 ^c | 40.6 | 51.8 ^c | 49.6 ^c |
| LSD (P=0.05) | 9.7 | 10.1 | 13.7 | 11.5 |
| SEm ± | 3.2 | 3.6 | 4.3 | 3.6 |

a,b,c = Values followed by the same letter are not significantly different at P=0.05

Dry matter production

Results pertaining to the effect of digested sludge on dry matter production of plants in Cr and Pb spiked soil are presented in Table 2.

A perusal of the results reveals that the dry matter yields of corn and radish were lowest in no sludge treatment which increased almost linearly as a function of increasing sludge levels to Cr or Pb spiked soil. The enhancement in dry matter production could be ascribed to improved physical and nutritional environment of soil caused by sludge application as well as decreased uptake of organo-sorbed metals. Rappaport *et al.* (1988) applied an anaerobically dry digested sludge from a waste water treatment plant with major industrial inputs at the rates of 0, 42, 84, 126, 168 and 210 Mg/ha to Bojac loamy sand, Davidson clay loam and Groseclose silt loam and noticed that the grain and straw yield of corn at the three locations increased linearly with increasing rate of sludge application. Chiu *et al.* (2006) noticed that manure compost application increased the yield of vetiver in Pb and Zn mine tailings. Roongtanakiat *et al.* (2008) found that combination of soil amendment materials, especially DTPA and compost, was more effective than sole chelating agents in enhancing growth and nutrient uptake of vetiver in iron ore mine areas. Sludge application rate correlated significantly and positively and DTPA-extractable Cr and Pb levels significantly but negatively with the dry matter yield of plants (Table 4). The results show that plants exhibit greater tolerance to metals applied with digested sludge than when they are added as inorganic salts alone.

Plant metal concentrations

Reductions were noticed in shoot Cr and Pb concentrations of corn and shoot and root Cr and Pb concentrations of radish as a result of increasing rates of digested sludge application to Cr or Pb spiked soil (Table 3).

Plant tissue concentrations of metals in both the crops were lowest in the highest sludge treatment. Sludge application rate correlated negatively with the tissue metal concentrations (Table 4).

Correlations of DTPA-extractable metal levels were positive with tissue metal concentrations. The reduced plant metal concentrations might have been caused by a change in metal speciation in soil solution with the addition of digested sludge. Maclean *et al.* (1969) observed that the Pb content of oat and alfalfa in soil, pretreated with PbCl₂ at rates 0, 20, 100 and 1000 mg/kg in pot tests varied inversely with the organic matter content. Cunningham *et al.* (1975) compared Cr uptake by corn and rye on soil treated with inorganic salt of chromium with that when sewage sludge was amended with this metal before addition to the soil and found that the treatments involving the inorganic salt resulted in general higher metal concentration than the equivalent sludge treatments. Bolan *et al.* (2003) examined the effect of municipal biosolid application (0-1 000 g organic carbon/kg

Table 3 Effect of digested sludge on heavy metal concentrations in corn and radish plants

| Treatment | Concentration in plant tissues (mg/kg dry weight) | | | | | | | |
|-------------------------------------|---|---------------------|---------------------|--------------------|--------------------|---------------------|-----------|--|
| | Corn I | | Radish I | | Corn II | | Radish II | |
| | Shoot | Shoot | Root | Shoot | Shoot | Shoot | Root | |
| Cr | | | | | | | | |
| DS ₀ Cr ₅₀₀ | 13.7 ^a | 12.5 ^a | 16.2 ^a | 8.6 ^a | 8.8 ^a | 10.6 ^a | | |
| DS ₃₀ Cr ₅₀₀ | 11.4 ^a | 9.7 ^{a,b} | 13.7 ^{a,b} | 7.2 ^{a,b} | 7.1 ^{a,b} | 9.4 ^{a,b} | | |
| DS ₆₀ Cr ₅₀₀ | 9.7 ^a | 8.2 ^b | 11.3 ^{b,c} | 5.8 ^{b,c} | 5.5 ^{b,c} | 9.0 ^{a,b} | | |
| DS ₂₄₀ Cr ₅₀₀ | 4.9 ^b | 7.9 ^b | 9.6 ^{b,c} | 3.5 ^{c,d} | 4.8 ^c | 7.5 ^{a,b} | | |
| DS ₄₈₀ Cr ₅₀₀ | 3.8 ^b | 7.2 ^b | 8.4 ^c | 1.6 ^d | 4.4 ^c | 6.9 ^b | | |
| LSD | 4.5 | 3.8 | 4.5 | 2.5 | 3.0 | 3.3 | | |
| <i>P</i> =0.05) | | | | | | | | |
| SEm ± | 1.4 | 1.2 | 1.4 | 0.8 | 1.0 | 1.1 | | |
| Pb | | | | | | | | |
| DS ₀ Cr ₅₀₀ | 11.4 ^a | 13.8 ^a | 42.6 ^a | 10.2 ^a | 10.3 ^a | 37.2 ^a | | |
| DS ₃₀ Cr ₅₀₀ | 9.7 ^a | 12.0 ^{a,b} | 38.1 ^{a,b} | 8.8 ^a | 11.4 ^a | 32.4 ^{a,b} | | |
| DS ₆₀ Cr ₅₀₀ | 8.2 ^{a,b,c} | 11.7 ^{a,b} | 34.7 ^{a,b} | 7.7 ^{a,b} | 9.9 ^a | 28.5 ^{a,b} | | |
| DS ₂₄₀ Cr ₅₀₀ | 5.8 ^{b,c} | 9.1 ^{a,b} | 25.4 ^b | 5.2 ^{b,c} | 7.9 ^a | 21.9 ^b | | |
| DS ₄₈₀ Cr ₅₀₀ | 4.6 ^c | 8.3 ^b | 23.7 ^b | 3.7 ^c | 7.4 ^a | 21.3 ^b | | |
| LSD | 3.7 | 5.4 | 17.0 | 3.0 | 3.5 | 14.8 | | |
| <i>P</i> =0.05) | | | | | | | | |
| SEm ± | 1.2 | 1.7 | 5.6 | 1.0 | 1.1 | 4.9 | | |

a,b,c,d Values followed by the same letter are not significantly different at *P*=0.05

Table 4 Correlations of sludge application rate and DTPA-extractable Cr and Pb levels with dry matter yield and tissue metal concentrations

| | Corn I | Corn II | Radish I | Radish II |
|-----------------------------------|---------------|----------------|-----------------|------------------|
| <i>Dry matter yield</i> | | | | |
| Sludge application rate | | | | |
| Cr | 0.990** | 0.997** | 0.987** | 0.990** |
| Pb | 0.957* | 0.985** | 0.979** | 0.981** |
| DTPA | | | | |
| Cr | -0.917* | -0.959* | -0.975** | -0.813 |
| Pb | -0.959* | -0.882* | -0.917* | -0.910* |
| <i>Plant tissue concentration</i> | | | | |
| | <i>Corn I</i> | <i>Corn II</i> | <i>Radish I</i> | <i>Radish II</i> |
| | <i>Shoot</i> | <i>Shoot</i> | <i>Shoot</i> | <i>Shoot</i> |
| | | | <i>Root</i> | <i>Root</i> |
| Cr | -0.918* | -0.953* | -0.730 | -0.857* |
| Pb | -0.918* | -0.949* | -0.921* | -0.910* |
| DTPA | | | | |
| Cr | 0.997** | 0.935* | 0.882* | 0.964* |
| Pb | 0.956* | 0.891* | 0.907* | 0.914* |

Significant at the 0.01 level, * significant at the 0.05 level

soil) on the uptake of Cr by Indian mustard from a soil treated with Cr (VI) up to 1 200 mg/kg soil and noticed that increasing levels of Cr increased the Cr concentration in plants, whereas increased additions of biosolid decreased the Cr concentration in plants. Zheljzakov and Warman (2004)

applied 0, 20, 40 and 60% municipal compost to soil by volume, but did not found any increase in the tissue concentration of Pb in Swiss chard and basil despite the relatively high metal content in the applied compost. Ryan and Chaney (1994) concluded that potentially toxic metals in sewage sludge treated soils are maintained in chemical forms not readily available for plant uptake.

Plant tissue Cr and Pb concentrations across all treatments were highest in the first harvest and decreased for the next harvest of both the crops. The decrease in tissue concentrations of metals with crops can be as a result of the reversion of the metals into less available forms either by adsorption on soil colloids or stabilization of previously formed organo-metal complexes or to a decrease in available metals as a result of plant uptake by the crops.

The outcome of this investigation reveal that digested sludge can be used as an amendment in sequestering and mitigating phytotoxic effects of heavy metals such as Cr and Pb in contaminated Gangetic alluvial soils of India.

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