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Transition Metal Rates in Latosol Twice Treated With Sewage Sludge

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ABSTRACT

Agricultural recycling of sewage sludge has been a source of accumulation of heavy metals in the environment which may reach toxic levels and cause serious damage to the biota. Field experiments were undertaken for two agricultural years (2000 and 2002) and effects of two sewage sludge applications were evaluated through the extraction of (essential and non-essential) transition metals by diethylenetriaminepentaacetic acid *(DTPA) extractor in a medium texture dystrophic Dark Red Latosol. Cd, Ni, Co, Pb and Cr were not detected. Application of sewage sludge initially caused a slight pH rise in the soil; later pH lowered and kept itself close to the starting level. It could be concluded that through consecutive sludge application, extractable rates of Fe and Mn in soil samples gradually increased during the two agricultural years in proportion to sewage sludge doses and sampling period. In fact, they were higher than rates of control. Due to low concentrations of soil samples, extractor had a restricted capacity for evaluation of its phytoavailability.*

Key words: Heavy metals, sewage sludge, DTPA, biosolid, nutrient rates

INTRODUCTION

In mankind's efforts to find a suitable place for different types of residues disposal, agricultural recycling has frequently been the best option to produce large quantities of nutrients, improve the chemical, physical and biological qualities of soil and find a solution to dispose of large quantities of wastes. Although sewage sludge is a nutrient source for soil, it is also highly risky. Besides lixiviation of nitrates and the accumulation of heavy metals in the non-homogenized soil, sewage sludge may also be a source of disease that may be transmitted by water. Biosolids may contain harmfull elements, especially pathogens, persistent and toxic organic substances, excessively nitrogenized compounds and heavy metals, which restrict their use in agriculture (Andreoli and Fernandes 1997). Successive applications of sewage sludge may cause an accumulation of toxic agents, especially heavy metals. Since the latter are non-degradable, they are of major concern for environmental safeness necessary for such practice. Depending on to their availability in their solution in soil, these compounds may directly contaminate soil organisms and plants at phytotoxic levels, transfer their pollutants to the food chain through plants and their fruit or through

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the contamination of surface or under-surface waters (Chang, 1987).

Many authors, (Chang et al., 1997; Raij, 1998) have discussed restriction in the agricultural use of biosolids, mainly heavy metals. Page et al. (1987) reviewed studies on the subject, providing important information on the behavior of heavy metals added to the soil-plant system through sludge. Two relevant aspects could be identified: sewage sludge is a source and, at the same time, an immobilizing agent of heavy metals in the soil. Further, absorption of heavy metals by plants in proportion to the residue's application rates has different responses.

Nutrient or non-nutrient metal rates available in the soil's solution are low and dependent on certain variables, such as pH, levels of organic matter and clay, soil mineralogy, competitions for other cations in absorption sites, soil temperature and humidity. Availability decreases at low temperatures and humidity (Petruzzelli, 1989; Hooda and Alloway, 1996). Immobilization of metals in the soil is caused by the formation of slightly soluble compounds with a group of anions in the soil and by the formation of complexes with the active sites of the soil's organic substances, among which may be mentioned humus components. pH plays an important role among the above variables in the formation or suppression of active sites.

pH in soil may be changed by various agents. The type of biosolid incorporated to the soil may cause alterations in pH (Gloria, 1992). As a general rule, its addition increases pH and the soil's negative charges (Dias, 1994; Marques, 1996). Berton et al. (1989) showed the effect on the behavior of pH in various soil types when increasing doses of biosolid are added. Alkalinized sewage sludge has neutral and alkaline pH and is able to increase the residues's pH. Another pH modifier is the environmental microbiological activity with the formation of inorganic and organic acids. Research by Pietz et al. (1989) and Dowdy et al. (1991) stated that certain types of sewage sludge may acidify the soil. Reactions of nitrification of ammoniac nitrogen, the probable oxidation of sulfites, and the production of organic acids during the degradation of residues may account for such acidification. Logan et al. (1997) reported a decrease in pH for the lowest sewage doses immediately after the application of residues in calcareous rock-derived soil during the first year of application of sewage sludge (without any previous treatment with lime).

The pH of the soil is one of the most important factors in the control of the bioavailability of metals. These are more unstable in low pH, due to the displacement of the metallic cations by the protons, to the occurrence of hydrolyzed species of hydroxides and to the solubility of other solid mineral phases, such as carbonates and phosphates (Logan and Chaney, 1983). The availability of metals tends to be smaller in the case of high pH, due to the formation of precipitates, very strong adsorption and the increase of the stability of compounds with humus substances (Petruzzelli, 1989).

Since Brazilian soils are different from those in temperate zones, greater concern is required in the extrapolation of metals that may be incorporated to them, because these soils are generally acid, with low cation-exchange capacity (CEC) and low contents of organic matter. Soils classified as latosols, (Curi et al., 1993) are: normally compact soils, generally formed in humid tropical regions, characterized to present low molecular relation of SiO_2/Al_2O_3 (or silica/sesquioxides) in the clay fraction, low cation-exchange capacity (CEC), low clay activity, low content of primary minerals easily decomposed by the physical, chemical and biological conditions and low content of soluble constituents. The dystrophic dark red latosol is a poor nutrient-soil, which presents low saturation of base (SB).

Since few researches have been undertaken involving the addition of metals through organic residues, no agreement exists with regard to sewage sludge doses and their frequency of application in cultures. Their composition is not constant and there are some difficulties in foreseeing the behavior of certain metals vis-à-vis soil types. Actually these facts restrict the application of higher doses of residues. This work used a medium texture dystrophic Dark Red Latosol, which, in the past, was alternatively utilized to cultivate maize and bean, and evaluated the effects of two times applications of sewage sludge on the availability of transition metals

MATERIALS AND METHODS

General characteristics of site, soil and sewage

Field experimental work was undertaken in the Experimental Farm of the State University of

Maringá, at Iguatemi, municipality of Maringá PR Brazil during the agricultural years 2000-2001 and 2001-2002. Site, with a slightly rolling relief, lies at 23º 25’00”S; 51º 25’00”W; at 555m above sea level. Soil belongs to the large group of dystrophic Dark Red Latosols with medium clay texture. Rates of fine sand, thick sand, silt and clay were 570, 280, 20 and 130 g kg^{-1} respectively. Sewage sludge, resulting from an anaerobic fluidized bed reactor, came from Sewage Treatment Station STS-l, of the Paraná Water Supply Company (SANEPAR), Maringá PR Brazil. Tempered and stored sewage sludge was solid at the moment of collection, with its natural humidity and without any addition of lime. A representative sample was collected to determine its humidity and chemical characteristics.

Three aliquots of known mass were removed to determine their humidity. They were dried in buffers at 65ºC till constant weight. Humidity was determined by difference of mass, whereas the amount of humid sewage sludge needed to be distributed in each experimental unit was calculated to obtain the doses for the different treatments applied to the soil (Andreoli and Bonnet, 1998). Aliquots (10 g) of recently collected *in natura* sewage sludge were separated to determine pH, following Andreoli and Bonnet (1998).

To determine the chemical composition of the sewage sludge, a composite sample was dried, ground, homogenized and sieved. The resulting sample was reduced to constant mass. Nutrient and non-nutrient metals were determined. 0.5000 g-aliquots were humidly decomposed with a nitric-perchloric mixture, in a digester. Metal concentrations were determined by atomic absorption method, flame mode (Welz and Sperling, 1999). Kjeldahl and thermal decomposition gravimeter methods were used to determine nitrogen and organic matter, respectively (Horwitz, 1980). Whereas Table 1 presents results, Table 2 shows heavy metals analyzed.

Experiment

Field experiment was performed in a randomized experimental block design in subdivided lots, with four repetitions. Doses (0, 6, 20, 40, 60, 80 t ha^{-1}) of sewage sludge were distributed in lots and sampling time (0, 30, 60, 90 and 120 days) was fixed for each sub-lot. Each block consisted of six experimental units. Sub-lots consisted of five 23 m-long rows, spaced 1.0 m between the row and 0.20 m between plants. Although totaling 115 sq. m., used area for sampling soil and plants consisted of 3 internal rows of 20 m and totaled 60 sq. m. Sewage sludge was applied on the surface with spade and hoe. Incorporation was done by harrowing at approximately 0-20 cm depth in both applications.

Duration of the experiment

The experiment was carried out along two agricultural years. In the first (2000-2001) the applications of the sewage sludge doses: 0, 6, 20, 40, 60, 80 t ha^{-1} in the following periods: 0, 30, 60, 90 and 120 days were evaluated with four repetitions. In the second agricultural year (2001-2002) the application of the same sewage sludge doses, in the followings periods: 0, 60, 90 and 120 days, were evaluated with four repetitions. During the two agricultural years, the immediate effects of the two sludge application, summer harvest and the residual effect of the two application winter harvest, were evaluated.

Collection, preparation and analysis of soil samples

At 0, 30, 60, 90 and 120 days after incorporation of sewage sludge in the two agricultural years collection of soil samples of each one of the six experimental units was undertaken. The six units comprised 5 treatments and 1 control for each randomized block at a 0-20 cm depth layer. A sample composed of 4 simple samples came from each sub-lot. Samples were identified, air dried, filtered through 2mm-hole polypropylene sieves, homogenized and stored as Air Dried Fine Earth (ADFE).

Analysis of soil characterization

ADFE samples were analyzed routinely for usual parameters following IAPAR (1992): pH was determined in $CaCl_2$ solution; potential acidity or $[H^+ + Al^{3+}]$ was determined with the Shoemaker, Mclean and Pratt or SMP buffer solution; P, K and Na were determined in the soil solution extracted with Mehlich solution, and P determined by UV-Vis method, K and Na determined by atomic absorption method; Ca, Mg were determined by atomic absorption method in the soil solution extracted with KCl 1,0 mol L^{-1} solution; the concentration of C was determined by the Wakley-Black method, N by Kjeldhal method and S determined by sulfate turbidimetric method. The value denominated by Sum of Bases (SB) was calculated with the equation [01]. The result is expressed in cmol$_c$ dm^{-3} units.

$$SB = Ca^{2+} + Mg^{2+} + K^{+} + Na^{+} \quad [01]$$

The CEC values were determined by equation [02] in cmol$_c$ dm^{-3} units.

$$CEC = SB + [H^{+} + Al^{3+}] \quad [02]$$

Table 3 shows results for soil samples, without treatment, at time 0 of incorporation.

Analysis of total rates of metals in the soil

Total rates of metals in the soil were determined by their respective concentrations by atomic absorption in the solution obtained from the decomposition of samples, humidity mode, using a mixture of HNO_3 and $HClO_4$ (Horwitz, 1980).

Analysis of available rates of metals in the soil

Soluble rates of metals, available for plants in soil were determined by their respective concentrations of metals by atomic absorption, in the solution obtained from the extraction of 20.0000 g of ADFE with 40 mL of extracting mixture comprising DTPA (diethylenetriaminepentaacetic acid) 0.005 mol L^{-1} + TEA (triethanolamine) 0.1 mol L^{-1} + $CaCl_2$ 0.01 mol L^{-1}, corrected to pH 7.3 (Lindsay and Norwell, 1978).

Statistical analysis

The conventional statistical method was employed for sub-lots for each period. Surface response methodology was used for regression analysis. It was done from the square polynomial model with two independent variables, according to equation [03] (Regazzi and Campos, 1988).

$$Y_i = B_O + B_1N_i + B_2T_i + B_3N_i^2 + B_4N_iT_i^2 + B_5N_iT_i + E_i \quad [03]$$

where: Y_i = variable responses; N_i = biosolid levels; T_i = time levels; E_i = randomized error; B_i with i = 0, 1, 2, …, 5 = parameters to be estimated.

The complete model above shows that the equation with the best adjustment to data was based on the coefficient of determination (CD), or r^2, in the context of the regression coefficient by Student's *t* test (up to 10% probability) and by analysis of residues. Since only three evaluations were undertaken during the second agricultural year, the mathematical model was formula [04]

$$Y_i = B_O + B_1N_i + B_2T_i + B_3N_i^2 + B_4N_iT_i^2 + E_i \quad [04]$$

RESULTS AND DISCUSSION

The results of the chemical analysis of sewage sludge and soil are presented in Tables 1-3.

Table 1 - Averages of chemical characteristics of sewage sludge used in the experiment, associated to soil fertility (†).

Sewage sludge	N	C	OM(‡)	C:N	Mn	Cu	Zn	Fe	pH (*)
	⟵	g kg^{-1}	⟶		⟵		mg kg^{-1}	⟶	
1st application	37.0	264.0	405.0	7:1	274.1	480.0	1,320.0	42,800.0	4.68
2nd application	32.2	235.8	406.5	8:1	298.0	450.00	1,540.0	48,600.0	4.72

(†) – Analyses were undertaken at the Agrochemical and Environment Lab of the State University of Maringá. (‡) - OM = Organic Matter. (*)- pH rates were calculated from the original material and others from matter dried at 65^{0}C.

Table 2 - Concentration of heavy metals non-essential for plants in the sewage sludge in soil treatments (†).

	Cd	Ni	Cr	Co	Pb
Sewage sludge		⟵		(μg g^{-1}) ⟶	
1st application	2.8	88.0	72.5	72.5	492.0
2nd application	2.1	142.0	125.0	125.0	380.0

(†) – Analyses were undertaken at the Agrochemical and Environment Lab of the State University of Maringá.

Table 3 - Chemical characteristics of soil used in experiments.

Soil (0-20 cm)	**pH $CaCl_2$**	**P**	**S**	**N**	**C**	**Ca**	**Mg**	**K**	**H^++Al^{3+} (†)**	**CEC (‡)**	**SB**	**C:N**	**V%**
		←mg dm^{-3}→		← g kg^{-1}→		←		cmol$_c$ kg^{-1}			→		
dDRL	5.0	1.8	6.6	0.6	7.6	3.58	0.88	0.08	3.42	7.96	4.54	12:1	57.0

dDRL – Dystrophic Dark Red Latosol. (†) - [H^+ + Al^{3+}] = potential acidity. (‡) - CEC = Cation-Exchange Capacity. Results above are mean of analyses done in two repetitions. SB = Saturation of base.

CEC and pH

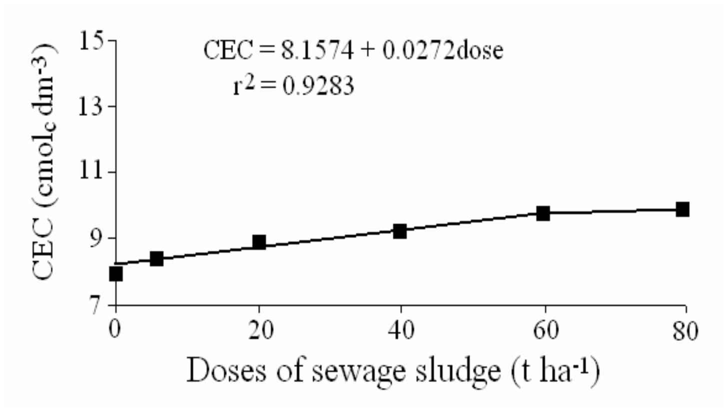
Regression analysis (Fig. 1) showed that sewage sludge dose significantly affected the soil's CEC. Variance analysis of the soil's CEC, (data not presented) also showed that significant effects only occurred in the two agricultural years due to sewage sludge doses. CEC increased because of residual doses in the two agricultural years (Fig. 1).

Variable had a linear trend in Harvests 1 and 2 during the first year (2000-2001), and in Harvest 3, during the second year (2001-2002). However, Harvest 4, during the second year (2002), average 7.16 cmol$_c$ dm^{-3}, failed to show any significant effect from treatments. Due to residual doses, CEC increase had a linear design (Fig. 1). Variable's behavior showed an increasing linear trend when 0.0272 cmol$_c$ dm^{-3} for each dose of 1 t ha^{-1} of residue were added to the soil. An increase in CEC caused by sewage sludge application was also suggested by Melo et al. (1994); Cavallaro et al. (1993). Who reported a CEC increase due to sewage sludge application in doses between 0 and 240 t ha^{-1} (dry base). Increase was attributed to the fact that organic matter has active sites, with a capacity for linking and exchanging ions, according to their pH. Carboxylic, phenol, carbonyl, alcohol, amino and other groups integrated the sites, which was directly proportionate to the quantity of organic matter. Although results from the application of increasing doses of sewage sludge on soil's pH had a significant effect (Fig. 2), adjustment of adequate regression model failed. pH increased up to level 6 t ha^{-1} and stabilized itself for other doses at Harvest 1.

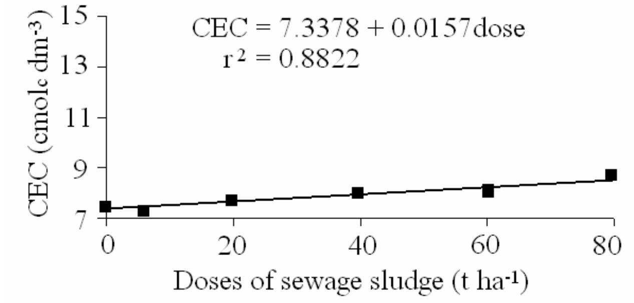
In Harvest 2 of the same year, a pH decrease occurred when residual doses increased. Residue affecting pH of soil decrease stabilized during harvest and maintained itself close to start.

Since residue affected pH of soil and such effect decreased in time, this fact suggested that nitrification, a phenomenon accounting for pH decrease, was restricted by the environment's acidity. Logan et al. (1997) reported a decrease in pH for the lowest doses of sludge, 7.5 and 15 t ha^{-1}, in soil from calcareous rocks during the first year of non-treated sludge with lime. Nitrification reactions of ammoniacal nitrogen for rise in pH in the highest doses and reactions were responsible involved in the degradation of the residue's organic charge for the verified acidification.

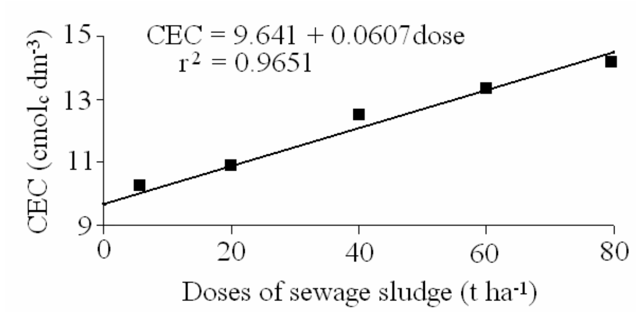
Oliveira (2000) and Bertoncini and Mattiazzo (1999) reported an increase in pH rates when increasing biosolid doses were added. The alkalinity of the material was responsible for pH increase. Difference in results was associated to different characteristics of sewage sludge used in the different experiments. Harvests 3 and 4 (Fig. 2) during the second year showed no significant effect on pH (average pH 4.40 and 4.35, respectively) in proportion to the levels of sewage sludge applied and to the culture's time of development.



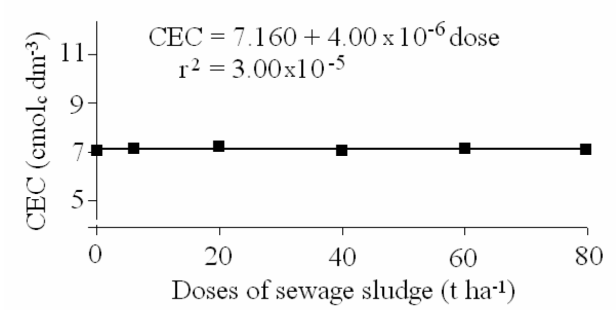
(a) Harvest 1 (conventional or Summer harvest)



(b) Harvest 2 (Winter harvest)

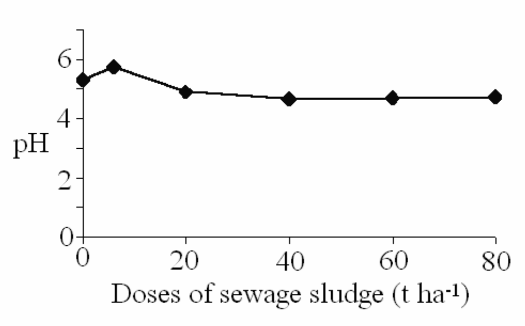


(c) Harvest 3 (conventional or Summer harvest)

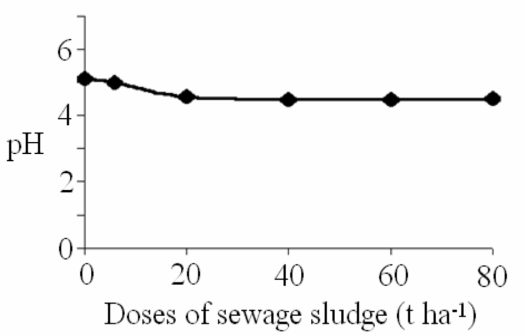


(d) Harvest 4 (Winter harvest)

Figure 1 - Cation Exchange Capacity (CEC) of the soil during agricultural years 2000-2001 and 2001-2002, Harvests 1 (a) - conventional or Summer harvest, 2 (b) - Winter harvest; 3 (c) - conventional or Summer harvest and 4 (d) - Winter harvest, due to increasing doses of sewage sludge.



(a) Harvest 1(conventional or Summer harvest)



(b) Harvest 2 (Winter harvest)

(c) Harvest 3 (conventional or Summer harvest)
Mean: pH = 4.40

(d) Harvest 4 (Winter harvest) - Mean: pH = 4.35

Figure 2 - Mean rates of pH of soil during agricultural years 2000-2001 and 2001-2002, in harvests 1(a), 2 (b), and mean rates of pH in harvests 3 (c) and 4 (d), in proportion to increasing doses of sewage sludge.

Table 4 - Mean rates of Fe and Mn, extracted by DTPA-TEA, obtained from analytic result averages of 5 samples from soil collected during periods 0, 30, 60, 90 and 120 days of biosolid incorporation, by increasing doses of sewage sludge.

Agricultural	**Doses of sewage sludge t ha^{-1}***						**CD**
year	**0**	**6 (12)**	**20 (40)**	**40 (80)**	**60 (120)**	**80 (160)**	**(r^2)**
				Fe (mg kg^{-1})			
2000-IE	27.06	35.91	51.85	71.45	85.62	94.32	0.97
2001-RE	28.19	40.16	57.66	83.87	97.10	105.19	0.95
2001-IE	31.15	47.94	74.55	97.72	108.00	129.36	0.93
2002-RE	34.53	58.15	88.76	114.69	127.75	140.08	0.98
				Mn (mg kg^{-1})			
2000-IE	17.88	18.80	19.82	21.96	25.84	28.28	0.88
2001-RE	20.45	21.90	24.18	26.12	29.84	33.02	0.97
2001-IE	22.70	26.70	33.30	37.86	42.83	46.29	0.95
2002-RE	27.05	34.63	42.47	50.01	54.53	58.81	0.98

* doses between brackets are for 2001/02; IE: Immediate Effect (1st and 3rd - Conventional Harvest); RE: Residual Effect (2nd and 4th - Winter Harvest). CD - Coefficient of Determination or r^2.

Heavy Metals (non-essential elements): Cd, Cr, Ni, Co and Pb

Table 2 showed Cd, Cr, Ni, Co and Pb in sewage sludge when total decomposition of the sample by nitro-perchlorate mixture was performed. However, these heavy metals failed to be detected when soil extraction was undertaken with sewage sludge applied yearly, (12, 40, 80, 120 and 160 t ha^{-1}), corresponding to different treatments during the two agricultural years with extractor DTPA-TEA. Or, rather, their concentrations were below the method's detection level (Long and Winefordner, 1983).

Oliveira (1995) and Bertoncini (1997) also failed to show Cr removal by DTPA solution. When Anjos (1999) worked with Dark Red Latosol and Red Latosol treated with different sludge doses, totaling 388 t ha^{-1} (dry base), it was found that Cd and Pb rates evaluated by DTPA-TEA extractor were below the method's detection limit. In field work, Oliveira (2000) evaluated the phytoavailability of heavy metals in Red Latosol, treated with 33, 66 and 99 t ha^{-1} of sewage sludge (dry base) during the first year and with 37, 74 and 110 t ha^{-1} (dry base) in the second year. Cd, Cr and Pb rates, evaluated by three extractors (one of them was DTPA-TEA) were below the analytic method's detection limit. In a Red Argisol treated with 0, 10, 20, 30, 40 and 50 t ha^{-1} of sewage sludge (dry base) in a green house, Simonete (2001) showed that Cd, Ni, Pb and Cr rates, extracted by DTPA-TEA, were below the method's detection limits for all treatments.

Evaluation of available rates of Fe and Mn in soil

Amounts of Fe and Mn removed from soil samples by extracting solution increased in proportion to the doses of sewage sludge and occurred in the following sequence Fe > Mn. This showed rates in sewage sludge (respectively, 42,800 and 48,500 mg kg^{-1}) and doses incorporated in the soil (Table 4). This could be accounted for the concentrations of metals in the residue. Average increases in rates of elements available were reported when compared to those of control. Table 4 presents the variations from the lowest to the highest dose for the four harvests, or rather, Fe: 33 to 249%; 42 to 273%; 54 to 315%; 68 to 306%, respectively. This has also been reported by Simonete (2001), who applied sewage sludge in soil cultivated with corn in a green house and registered a greater increase in Fe rates. The increase was attributed to the high metal rate in the residue.

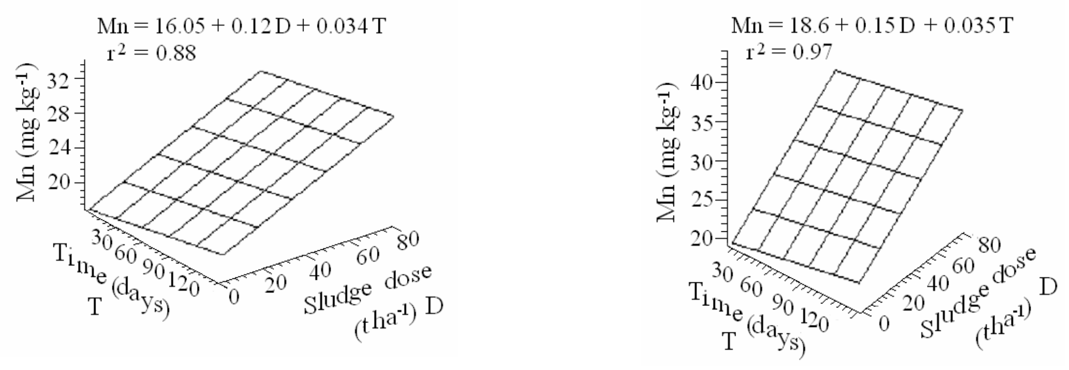
If amounts of the above metals in the soil during the first agricultural year (1st and 2nd harvests) were taken into account, average Fe and Mn extraction in treatments with sewage sludge doses was estimated at 7 and 120%, respectively, for DTPA. During the second agricultural year (3rd and 4th harvest) rates changed to 5 and 218%, respectively (Table 4).

Fe and Mn rates from soils by the extractor were mainly affected by doses and periods with metal increase (Figs. 3 and 4).

In the case of manganese, DTPA solution extracted 120 and 218%, respectively. In the case of Mn^{2+} removal, when rates of the metal in treatments were compared with and without sewage sludge, Anjos (1999) reported a significant difference with extractor DTPA pH 7.3. Lowest rates of Mn^{2+} were extracted from treatments with biosolid. Such behavior was also registered by Silva (1995) and was related to final pH of the treatments. It was actually higher (pH 7.4) than that of controls (pH 4.8).

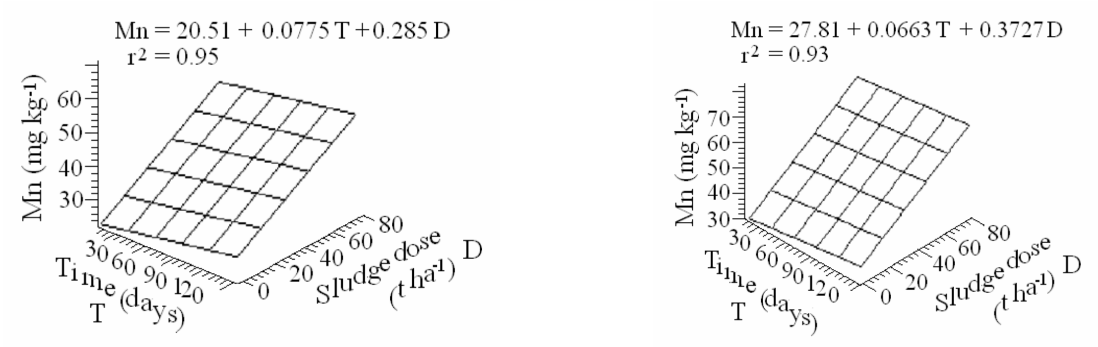
Manganese

Variance analysis showed that availability of manganese in the soil had significant effects on sewage sludge doses and sampling periods, although no interaction between these factors was observed (Fig. 3). In proportion to residue doses and sampling periods, rate increase was linear during the agricultural years 2000-2001 and 2001-2002 for the four harvests (Figs. 3a, b, c, d).



(a) Harvest 1 (conventional or Summer harvest)

(b) Harvest 2 (Winter harvest)



(c) Harvest 3 (conventional or Summer harvest)

(d) Harvest 4 (Winter harvest)

Figure 3 - Variation of average rates of Mn (a) Harvest 1, (b) Harvest 2, (c) Harvest 3 e (d) Harvest 4, removed by DTPA pH 7.3 in proportion to the sewage sludge in the four harvests.

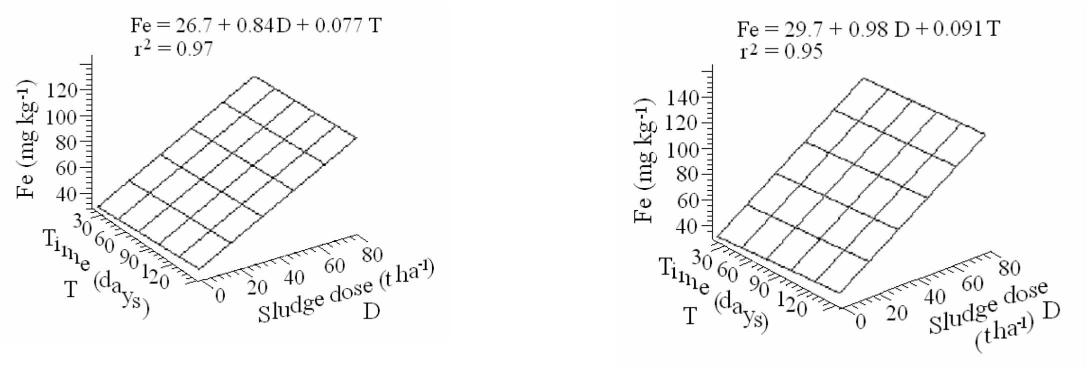
Availability of Mn in the soil mainly depends on pH, oxy-reduction potential, organic matter and balance with other cations. Soil pH is one of the chief factors that controls availability. Liming triggers oxidation of Mn^{2+} to a higher valence and lower solubility. Complexation of organic matter may also account for a decrease in the element's availability through liming (Borkert et al., 2001).

In the case of Quartz Sand and Dark Red Latosol, pH adjusted to 3.9 and 4.9 and then treated with 0; 13.5; 29.7 and 40.5 t ha^{-1}, Oliveira (1995) found that solution of HCl 0.1 mol L^{-1} and DTPA extracted larger amounts of Mn than those added to soil through sewage sludge. High pH rates favored the formation of more stable organic compounds. Activity of microorganisms that oxidizes soluble Mn to unavailable forms reached its peak at pH 7.0 (Tisdale et al., 1985).

Iron

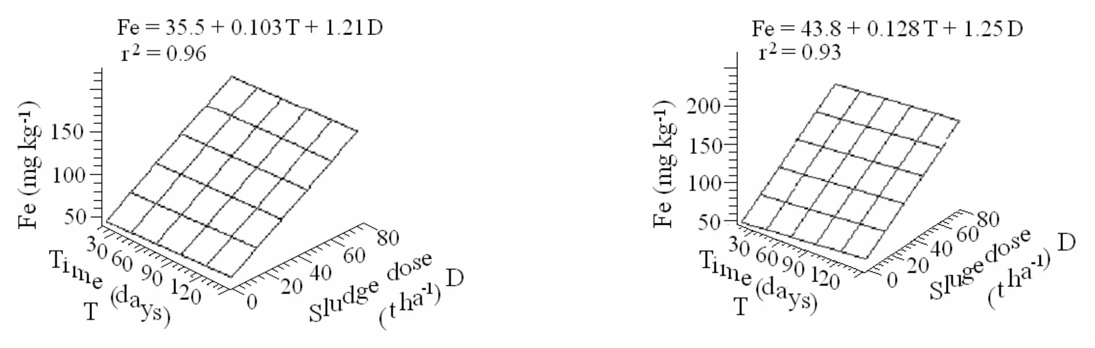
By variance analysis, iron presented an increasingly linear trend between time and sewage sludge doses applied to the soil for the agricultural year 2000-2001 (Figs. 4a and b). Since similar data were found in Harvests 3 and 4 of the second year, Fe showed an increasingly linear trend both in doses and in time (Figs. 4c and d). Availability of element was higher during the second agricultural year in the two harvests. This was due to the cumulative effect of two sewage sludge applications to the soil with high concentrations of the element in the residue and pH increase. Availability of the element decreased with a rise in pH (Matos et al., 1996).

pH influences the solubility of Fe compounds. According to Pigozzo et al. (2000), average rates of Fe in the dry material of the different residues showed a higher potential in proportion to nutrient and pH in the soil. In fact, in acidic soils, the availability of Fe is higher than that in increased pH. Decrease up to 0.5 pH units may be important in metal availability in the soil (Hooda and Alloway, 1996). This fact could explain an increase in availability of Fe with a decrease of pH, as reported in current research.



(a) Harvest 1 (conventional or Summer harvest)

(b) Harvest 2 (Winter harvest)



(c) Harvest 3 (conventional or Summer harvest)

(d) Harvest 4 (Winter harvest)

Figure 4 - Variation in average rates for Fe: (a) Harvest 1, (b) Harvest 2, (c) Harvest 3 and (d) Harvest 4, removed by DTPA pH 7.3 on applying sewage sludge in the four harvests.

CONCLUSIONS

- Successive applications of sewage sludge in soil showed an increase in CEC and, at the same time, a decrease in pH; later, CEC became constant and close to start rate.
- Heavy metals, non-essential to plants, showed concentrations lower than the analytic method's detection limits.
- Fe and Mn rates in soil gradually increased according to rise of sewage sludge doses.
- Effects of sewage sludge application could be taken as a source of essential metals.

RESUMO

A reciclagem agrícola do lodo de esgoto tem provocado o acúmulo de metais pesados no solo e na água, podendo atingir níveis tóxicos e causar danos às plantas cultivadas, aos animais e ao homem, por meio da cadeia trófica. Neste intuito foi desenvolvido o presente experimento, em condições de campo, entre 2000 e 2002, onde foram avaliados os efeitos da aplicação de lodo de esgoto por dois anos, sobre a extração de metais de transição (essenciais e não) pelo extrator DTPA em um Latossolo Vermelho distrófico (LVd) de textura média. As concentrações dos elementos metálicos: Mn, Fe, Cd, Ni, Co, Pb e Cr não foram detectados pelo método da absorção atômica na solução obtida com o extrator DTPA. A aplicação de lodo de esgoto causou inicialmente pequena elevação no pH do solo, posteriormente a diminuição do mesmo, e manteve-se próximo ao original. Foi possível concluir que, com a aplicação consecutiva do lodo, os teores extraíveis de Fe e Mn nas amostras de solos aumentaram gradativamente nos dois anos agrícolas, com as doses do lodo de esgoto aplicado, época de amostragens, e foram superiores ao tratamento testemunha. O extrator apresentou capacidade restrita para avaliação da fitodisponibilidade dos metais pesados decorrentes das baixas concentrações nas amostras de solo.

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