Nickel in soil and maize plants grown on an oxisol treated over a long time with sewage sludge

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ABSTRACT

The major limitation for the use of sewage sludge in agriculture is the risk of soil contamination with heavy metals, and their possible transference to man via the food chain. The objective of this study was to evaluate the content of nickel (Ni) in soil by the two methods of digestion ($HNO_3 + H_2O_2 + HCl$ and $HClO_4 + HF$), and in different parts of maize plants grown on a tropical soil classified as Typic Eutrorthox, that had been treated with sewage sludge for nine consecutive years, and the effects on dry matter and grain production. The experiment was carried out under field conditions in Jaboticabal-SP, using a randomized block design with four treatments and five replicates. Treatments consisted of: 0.0 (control, mineral fertilization), 45.0, 90.0 and 127.5 t ha^{-1} sewage sludge (dry basis), accumulated during nine years. Sewage sludge was manually applied to the soil and incorporated at 0.1 m depth before sowing the maize. Soil Ni evaluated by Jackson's method was 76.8% higher than evaluated by the United States Environmental Protection Agency method that digests the samples by heating with concentrated HNO_3, H_2O_2 and HCl. Sewage sludge rates did not affect Ni content in the soil. Ni was accumulated in leaf and stem but was not detected in grain. Sewage sludge and mineral fertilization applied to soil for a long time caused similar effects on dry matter and grain production.

Keywords: *Zea mays* L., biosolids, heavy metal, soil pollution, urban residues, mineral nutrition

INTRODUCTION

Population growth in urban centres is one of the main causes of waste production, which can be hazardously accumulated in the environment if not adequately treated, disposed of and recycled. Sewage sludge is one of the most important types of waste. Melo *et al*. (2002) reported that chemical and biological compositions of sewage sludges depend on the wastewater composition.

Because sewage sludge contains considerable amounts of organic matter and essential elements to plants, it can partially replace mineral fertilizers playing an important role in crop production and in the maintenance of soil fertility (Nascimento *et al*., 2004). It has been used in Brazil for the remediation of degraded areas (Teixeira *et al*., 2005; Bezerra *et al*., 2006), as fertilizer for some crops such as sorghum, maize, common bean and sugarcane (Marques *et al*., 1999; Revoredo and Melo, 2006; Gomes *et al*., 2006; Nogueira *et al*., 2006; Camilotti *et al*., 2007).

The continuous use of sewage sludge in agriculture has caused increases in heavy metal soil concentration, among them Ni (Melo *et al*., 2007; Krebs *et al*., 1998), which represents an important environmental problem due to the possibility of their transference

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Table 1 Chemical characteristics[a] of a Typic Eutrorthox in the 8th year of experimentation and before sewage sludge applications

Treatment ($t\ ha^{-1}$)	pH $CaCl_2$	OM ($g\ dm^{-3}$)	P ($mg\ dm^{-3}$)	K	Ca	Mg	H + Al	SB	CEC	V (%)
				(mmol dm^{-3})						
0.0[b]	5.1	25	58	4.8	29	12	42	45.8	87.8	52
40.0	5.3	27	65	3.9	41	13	38	57.9	95.9	60
80.0	5.6	28	132	4.1	58	13	31	75.1	106.1	71
107.5	5.0	28	88	4.1	32	10	52	46.1	98.1	47

[a]Raij *et al*. (2001). [b]Only mineral fertilization.
OM, organic matter; SB, sum of bases; CEC, cation exchange capacity; V, base saturation.

into the food chain, with sometimes unknown risks (Nriagu and Pacyna, 1988). Concern over the availability of soil Ni for plant uptake has increased among Brazilian researchers in the last years (Martins *et al*., 2003; Rangel *et al*., 2004; Nogueira *et al*., 2007a).

The determination of total heavy metals in soils has been used to estimate their accumulation caused by the continuous application of wastes. In most of the studies this determination has been made by digestion of soil samples with strong acids as nitric acid (HNO_3), hydrochloric acid (HCl), perchloric acid ($HClO_4$) complemented or not by the attack with fluoridric acid (HF) which is able to dissolve soil silicates (Abreu *et al*., 1996).

The United States Environmental Protection Agency (USEPA, 1986) treats soil samples with the sequence hydrogen peroxide (H_2O_2), HNO_3 and HCl to evaluate heavy metal contents. The obtained value is named as total heavy metal content and is used as a reference for soil quality monitoring when treated with wastes such as sewage sludge (Abreu *et al*., 2001). This method has been adopted in Brazil by the São Paulo State Environmental Agency (Cetesb, 2001).

Melo *et al*. (2007) comparing the USEPA method with the method Jackson (1958), which complements the soil digestion with HF, verified that the extracted Ni content determined by Jackson's method was higher than that obtained by the USEPA method.

Nickel is a heavy metal, but is actually also considered a plant nutrient by some authors (Malavolta and Moares, 2007) since it takes part in the constitution of the urease molecule (Jasmim *et al*., 2002; Malavolta, 2006). Considering that sewage sludge may contain high concentrations of Ni, the objective of this study was to evaluate the effects of nine annual application of increasing rates of that residue to a tropical soil classified as Typic Eutrorthox on its Ni concentration and on Ni concentration in different parts of maize plants grown on that soil.

MATERIALS AND METHODS

The experiments were carried out under field conditions in Jaboticabal, Brazil (21°15′S and 48°15′W, 618 m above sea level) using an experimental design of randomized blocks with four treatments (sewage sludge rates) in 60 m^2 plots (6 × 10 m), with five replicates. The sewage sludge applications started in the season 1997–1998, and the results here presented correspond to the season 2005–2006.

The rates of sewage sludge applied in the first year were 2.5, 5.0 and 10.0 $t\ ha^{-1}$, dry basis, and control. The rate 5.0 $t\ ha^{-1}$ was established in order to supply the nitrogen (N) required by maize plants assuming that one-third of the sewage sludge N would be available to the plants. From the second year on, the control treatment received mineral fertilization based on soil chemical analysis (Raij and Cantarella, 1997). Since the 2000–2001 season (fourth year of experimentation) the 2.5 $t\ ha^{-1}$ rate was raised to 20.0 $t\ ha^{-1}$ in order to induce a possible plant toxicity. In this way, the accumulated rates of sewage sludge up until now are 0.0, 45.0, 90.0 and 127.5 $t\ ha^{-1}$.

Maize was planted in the first six years. In the seventh year, however, it was changed to sunflower (*Helianthus annuus* L.), in the eighth to the legume *Crotalaria juncea* L., and in the ninth it returned to maize.

The soil that has been used in the experiment is a tropical clayey Typic Eutrorthox. It was chemically analysed before the ninth application of sewage sludge according to the methodology described in Raij *et al*. (2001) and the data are shown in Table 1.

Sewage sludge was obtained from the sewage treatment plant operated by ''Companhia de Saneamento Básico do Estado de São Paulo''

Table 2 Chemical composition of the sewage sludge used during the nine years of experimentation

Year	N	P	K	Cu	Mn	Zn	Cr	Cd	Ni	Pb
	($g\ kg^{-1}$)			($mg\ kg^{-1}$)						
1997–98	32.00[a]	16.60	4.85	664	228	1800	290	8	268	152
1998–99	37.31	11.30	1.70	551	294	3810	1190	12	595	371
1999–00	28.72	17.41	1.47	660	257	2328	764	8	360	180
2000–01	28.94	15.58	1.85	719	263	1745	699	10	354	171
2001–02	36.75	15.54	2.74	627	287	2354	778	9	350	155
2002–03	34.08	21.62	1.90	722	222	2159	808	11	231	186
2003–04	40.87	19.49	0.09	690	194	2930	736	10	297	173
2004–05	33.67	18.70	1.30	998	206	2474	798	8	299	169
2005–06	33.67	18.70	1.30	998	206	2474	798	8	299	169

[a]Data expressed on dry basis.
Methodology of analysis: N, microKjeldhal method; P, vanado–molybdate spectrophotometric method; K, flame photometry; heavy metals, atomic absorption spectrometry (AAS). Except for N, the other determinations were carried out in the extracts obtained by heating the samples with concentrated HNO_3, H_2O_2 and HCl.

(Sabesp) in Barueri, Great São Paulo, whose chemical analysis of the residue used during the nine years of experimentation is presented in Table 2. Total N in sewage sludge was determined by the microKjeldhal method (Bremner *et al*., 1996), potassium (K) by flame photometry, phosphorus (P) by the vanado-molybdate spectrophotometric method (Malavolta *et al*., 1997), the heavy metals, including Ni, by atomic absorption spectrometry (AAS). Except for N, all the other methods were applied to the extract obtained by heating the samples with concentrated $HNO_3 + H_2O_2 + HCl$ (USEPA-3050, 1986).

In October 2005, herbicide (glyphosate) was applied on weeds, whose residues were incorporated into the soil by a light harrowing. Dolomitic limestone was applied at the rate of 20.0 t ha^{-1} in order to bring the base saturation to 70% (1.8 and 2.5 t ha^{-1} to treatments and to control, respectively) according to Raij and Cantarella, (1997). After that, the sewage sludge (moisture, 80%) was manually and uniformly distributed on the plots and incorporated to the 0–10 cm depth through a light harrowing. Mineral fertilizers were applied manually into the furrow for the control plots. Fertilization at sowing consisted of 277 kg ha^{-1} of simple superphosphate (18% P_2O_5), 86.2 kg ha^{-1} potassium chloride (58% K_2O) and 150 kg ha^{-1} ammonium sulfate (20% N). The treatment that received 5.0 t ha^{-1} of sewage sludge received potassium chloride to complement that applied to the control. Side dress fertilization was applied 27 and 40 days after sowing, at rates of 156 kg ha^{-1} urea (45% N) and 69 kg ha^{-1} potassium chloride each time. N was applied only in the control and K was applied to all the treatments. Table 3 shows the NPK rates applied in the nine years of experimentation. Maize, hibrid Syngenta NK Traktor SL was sown November 26, in a $0.9\,m \times 0.15\,m$ spacement (7 pl m^{-1}) as soon as mineral fertilizers were applied.

Soil samples were collected 60 days after sowing from the 0–20 cm layer at about 5 cm from the side of the plants (10 simple samples per plot to obtain one composite sample). They were air dried for 48 h, sieved through 2 mm and analysed for total Ni by the methods of USEPA-3050 (1986) ($HNO_3 + H_2O_2 + HCl$) and Jackson (1958) digestion with $HClO_4 + HF$.

Plant sampling was carried out at the end of the cycle (April, 2006). Six plants were collected in the useful area of each plot, cut 5 cm above the soil surface, separated into leaves, stems, husk, cob and grain, washed, dried at 65°C until constant weight, and milled through a 40 mesh stainless steel mill for chemical analysis.

Ni in the plant samples was determined according to the method USEPA-3050 (1986), which digests the material with the sequence nitric acid → hydrogen peroxide → hydrochloride acid. The extracts were analysed by atomic absorption spectrometry using an air–acetylene flame. The detection limit for the method was established according to Giné-Rosias (1998).

For soil data analysis, the values were grouped in a 2 × 4 factorial design and submitted to the variance analysis. In the cases the F test was significant at $P < 0.05$, Tukey's test was used for comparison of means (Pimentel-Gomes, 2000). The results were analysed statistically using the statistical program SAS System for Windows 6.11 (SAS Inst., 2002).

Table 3 Mineral fertilization applied during nine years

Year	Sewage sludge[a]	N		P_2O_5	K_2O	
		Sowing	Dressed	Sowing	Sowing	Dressed
	($t\ ha^{-1}$)	($kg\ ha^{-1}$)				
1997–98	0.0	–	–	–	–	–
	2.5	–	–	50	29	–
	5.0	–	–	44	26	–
	10.0	–	–	33	20	–
1998–99	0.0	15	61	30	30	–
	5.0	–	–	–	25	–
	10.0	–	–	–	20	–
	20.0	–	–	–	10	–
1999–00	0.0	30	110	50	50	40
	7.5	–	–	–	46	40
	15.0	–	–	–	41	40
	30.0	–	–	–	32	40
2000–01	0.0	30	120	50	50	40
	20.0	–	–	16	39	40
	2.5[b]27.5	–	–	–	28	40
	40.0	–	–	–	5	40
2001–02	0.0	30	140	50	50	40
	25.0	–	–	–	34	40
	47.5	–	–	–	17	40
	50.0	–	–	–	–	40
2002–03	0.0	30	140	50	50	40
	30.0	–	–	–	38	40
	60.0	–	–	–	27	40
	67.5	–	–	–	4	40
2003–04	0.0	10	40	20	20	–
	35.0	–	–	–	–	8
	70.0	–	–	–	–	–
	87.5	–	–	–	–	–
2004–05	0.0	–	–	18	18	–
	40.0	–	–	–	–	–
	80.0	–	–	–	–	–
	107.5	–	–	–	–	–
2005–06	0.0	30	140	50	50	40
	45.0	–	–	–	24	40
	90.0	–	–	–	–	40
	127.5	–	–	–	–	40

[a]Sewage sludge rates applied during nine years.
[b]Starting in the fourth year, the 2.5 rate $t\ ha^{-1}$ was replaced by 20.0 $t\ ha^{-1}$.

RESULTS AND DISCUSSION

Soil chemical properties and sewage sludge composition

The chemical properties of the soil of the plots of the different treatments before sewage sludge and limestone application (Table 1) indicate a high fertility soil (Raij *et al*., 1997).

The sewage sludge used in the experiment is rich in OM, plant nutrients (except K) and also in heavy metals such as Ni (Table 2). The high content of heavy metals is due to that fact that the plant treats sludge collected from a highly industrialized region.

The Ni content in the sewage sludge of the second year of experimentation was higher than the 420 mg kg^{-1} (dry weight basis), the maximum established

Table 4 Total Ni in a Typic Eutrorthox treated with sewage sludge for nine years determined by the methods 3050-USEPA (1986) and Jackson (1958)

Sewage sludge	USEPA	Jackson
(t ha^{-1})	(mg kg^{-1}) (dry basis)	
0.0	13.68 a	62.20 a
45.0	14.11 a	59.15 a
90.0	15.23 a	64.27 a
127.5	14.75 a	62.92 a
Mean	14.44 B	62.13 A
CV %	8.0	

Values followed by the same letters (small case compare rates and capital letters compare methods) are not different by the Tukey's test ($P < 0.05$). CV, Coefficient of variation.

by Cetesb (1999) in its legislation to regulate sludge application to agricultural soils. In most of the years, the concentration of Ni was close to that limit, so that the high Ni content could be taken as a limitation for the application of this residue to agricultural soils. Since this limit was not established using tropical soils, it becomes important to understand the Ni behaviour in tropical soils amended with sewage sludge, the forms in which Ni occurs, its availability to plants, and the translocation to other plant parts, in order to set guidelines for applications that have no risk for the environment and the food chain.

Ni in the soil

The interaction between the values obtained for total Ni in the soil by the two tested methods was not significant. Soil total Ni was not affected by the sewage sludge accumulated (Table 4). This finding corroborates results obtained by Martins *et al*. (2003) who also found no effect from the addition of sewage sludge to soil on the content of total Ni. However, other authors (Rangel *et al*., 2004; Oliveira *et al*., 2005) have found increasing values of total Ni concentration in the soil when sewage sludge from Sabesp–Barueri was applied to Brazilian Oxisols.

Jackson's method provided 77% higher values in relation to the USEPA method (Table 4). Melo *et al*. (2007) found, for a Brazilian Oxisol, 47% more Ni by Jackson's method as compared to the USEPA method. One possible explanation for this finding is that heavy metals may be occluded in iron hydroxides and manganese oxides, present at high levels in tropical soils (Malavolta, 1994). Whereas the USEPA method does not dissolve the entire soil sample, Jackson's does. Revoredo and Melo (2006) applied sewage sludge enriched with Ni to a Typic Haplorthox and found that part of the added Ni was not recovered immediately after the application of the residue and considered this fact due to a fraction of soil Ni that was not dissolved by the USEPA method. According to Keller and Védy (1994) the *aqua regia* extracts all the metals present in the residual fraction of soil samples except those that take part in the structure of silicates so that the percentage of extraction varies from 60 to 100%.

Considering that Cetesb establishes the value 50 mg kg^{-1} Ni as a reference value for soils of the São Paulo State (Cetesb, 2001), after nine years of application of sewage sludge the soil total Ni was much below this limit (Table 4).

Both the methods for Ni determination in soil samples, USEPA and Jackson, were not a suitable extractors for estimating Ni availability to maize (r, 0.02 and 0.12, respectively).

Maize dry matter production

Dry matter of the aerial part of maize plants of the last year of the experiment was affected by the application of sewage sludge, the highest value (16.3 t ha^{-1}) occurring for the treatment that received the accumulated amount 90.0 t ha^{-1} and the lowest (12.7 t ha^{-1}) for the treatment that received 45.0 t ha^{-1} as shown in Figure 1f.

The same trend was observed for the different parts of the plant (Figure 1a to e), but only for husk there was a significant difference for the accumulated amounts of 45.0 and 90.0 t ha^{-1}. Therefore, except for the lowest accumulated amount, sewage sludge yielded the, represented by the control. This is in agreement with Melo (2002) after applying sewage sludge for three consecutive years in the same soil. Other reports show increases in dry matter of maize plants when grown on soil treated with sewage sludge (Simonete *et al*., 2003; Galdos *et al*., 2004; Nascimento *et al*., 2004) in a few cases the dry matter

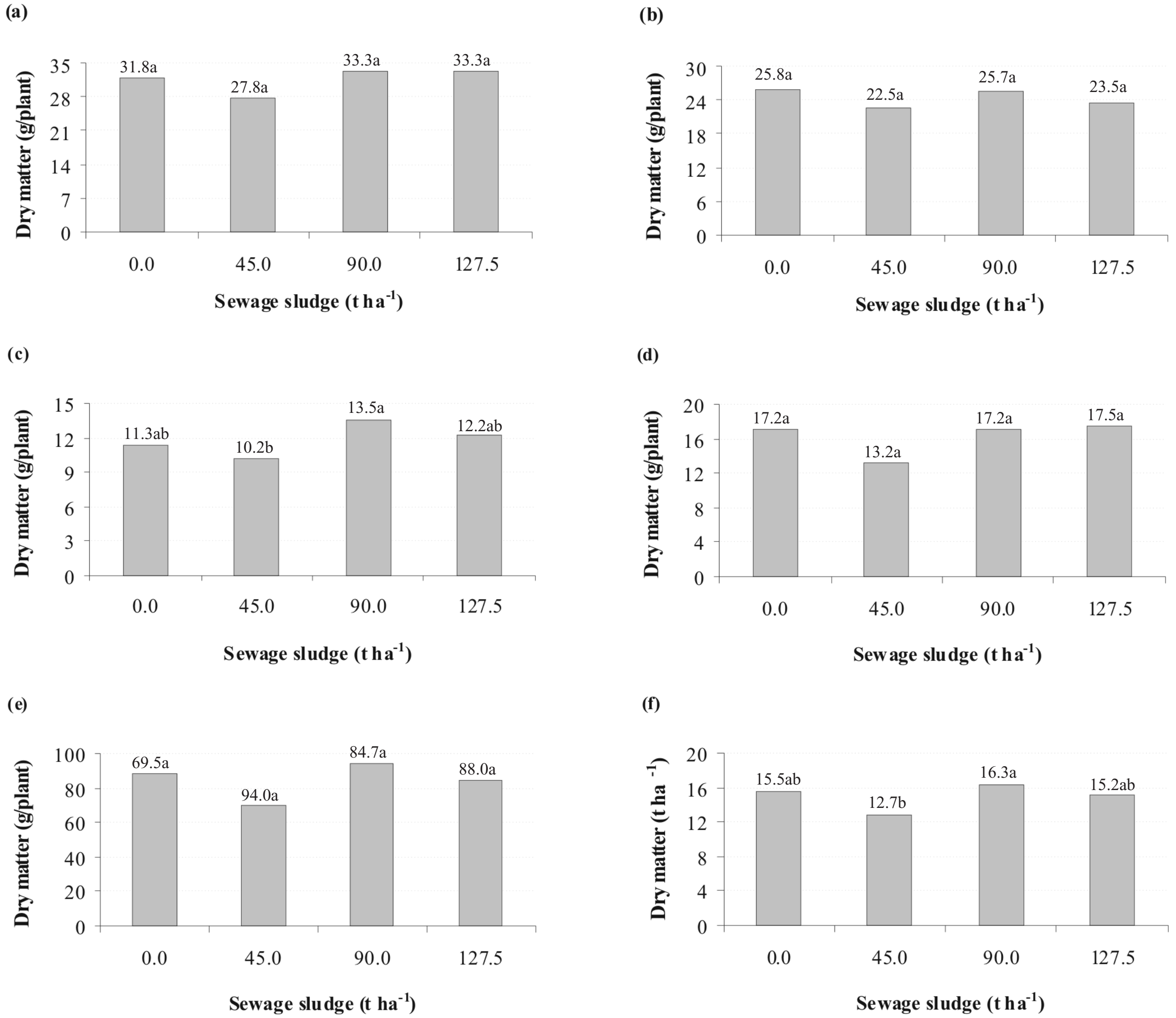


Figure 1 Dry matter production by maize plants grown on a Typic Eutrothox treated with sewage sludge for nine consecutive years. (**a**) Stem, (**b**) leaves, (**c**) husk, (**d**) cob, (**e**) grain and (**f**) total aerial part. Means followed by the same letters are not different by the Tukey's test ($P > 0.05$).

production is higher than that due to the mineral fertilization.

Ni in maize plants

The Ni content in husk, cob and grain was below the detection limit of the analytical method (Table 5).

Other authors (Alves *et al*., 1999; Anjos and Mattiazzo, 2000; Rangel, 2003) were also not able to evaluate Ni in maize due to the method used.

Nickel in the stem was greater in the plants of the control treatment. A possible explanation for this finding should be based on the content of this heavy metal in the simple superphosphate. According to Malavolta (2006) this fertilizer may contain from 10 to 117 mg kg^{-1} Ni. However, considering the Ni concentration in leaves, the highest concentration occurred in the treatment that has received 10 t ha^{-1} yr^{-1} sewage sludge. Therefore the absorption of Ni by the two treatments was similar and the observed difference was due the Ni mobility inside the plant.

Ni concentration in leaves was lower than the 60 mg kg^{-1}, considered toxic for plants (Malavolta, 1994). For this same experiment, but in the fifth year of experimentation, Oliveira *et al*. (2005) did also not find differences in the concentration of Ni in the aerial part of the maize plants.

Considering the Ni accumulated in the different parts of maize, the stem presented the highest values

Table 5 Nickel contents in the different parts of maize plants grown on a Typic Eutrorthox in the ninth consecutive year of sewage sludge application as determined by AAS in the extracts obtained by heating the samples with concentrated HNO_3, H_2O_2 and HCl

Treatments	Stem	Leaves	Husk	Cob	Grain
	(mg kg^{-1})				
Control[a]	0.65 a	1.20 b	<0.5	<0.5	<0.15
45.0 t ha^{-1} SS	0.57 ab	1.53 ab	<0.5	<0.5	<0.15
90.0 t ha^{-1} SS	0.41 c	1.80 a	<0.5	<0.5	<0.15
127.5 t ha^{-1} SS	0.44 bc	1.38 ab	<0.5	<0.5	<0.15
CV %	13.8	18.9	–	–	–

[a]No sludge application and mineral fertilization.
SS, Sewage sludge. Means followed by the same letter in the column are not different by the Tukey's test ($P<0.05$). CV, Coefficient of variation.

Table 6 Nickel accumulation in parts of maize plants under varying sewage sludge rates

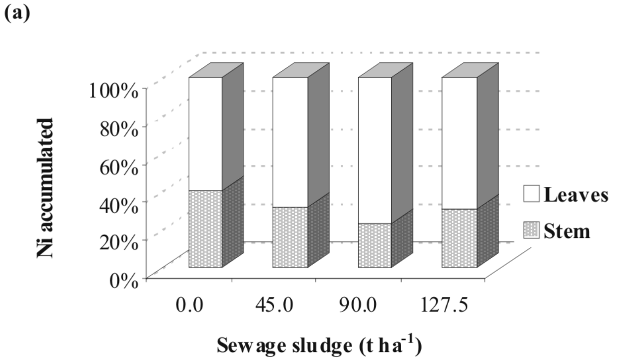
Treatments	Stem	Leaves	Husk	Cob	Grain
	(mg plant^{-1})				
Control[a]	0.021 a	0.031 b	Bdl	Bdl	Bdl
45.0 t ha^{-1} SS	0.016 b	0.034 ab	Bdl	Bdl	Bdl
90.0 t ha^{-1} SS	0.014 b	0.045 a	Bdl	Bdl	Bdl
127.5 t ha^{-1} SS	0.015 b	0.033 b	Bdl	Bdl	Bdl
CV %	15.3	16.9	–	–	–

[a]No sludge application and mineral fertilization.
SS, Sewage sludge. Means followed by the same letter in the column are not different by the Tukey's test ($P<0.05$). CV, Coefficient of variation. Bdl, below detection limit.

for in the control, while for leaves this occurred for the treatment that received 90.0 t ha^{-1} sewage sludge (Table 6). The content of Ni in the aerial part of the plant was 0.052 and 0.059 mg/plant, respectively for the control and treatment 90.0 t ha^{-1}.

Wang *et al*. (1997) reported for wheat plants grown on a soil treated with 60 kg ha^{-1} of Ni from sewage sludge, that the concentration was higher in leaves when compared to grain. According to Kabata-Pendias and Pendias (1992) plants may accumulate heavy metals in some tissues as a way to self-protect them against toxic effects. However, the plateau theory considers that after successive residue applications to the soil, the concentration of the different heavy metals tends to be limited (Corey *et al*., 1987).

The percentage of the total Ni was higher in leaves, agreeing with the results of Malavolta and Moraes (2007) (Figure 2a), however, Pierrisnard (1996) found an accumulation of the metal in the cob, grain and roots. For cotton plants Nogueira *et al*.



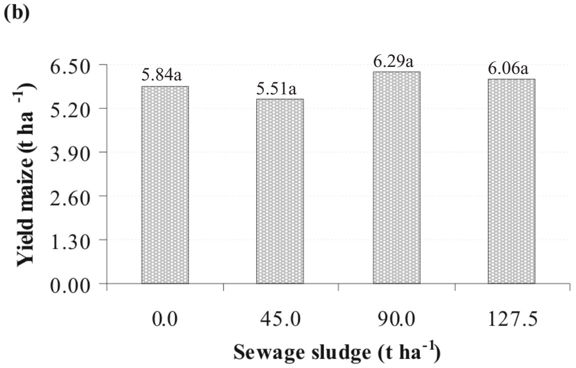


Figure 2 Perceptual distribution (**a**) of nickel accumulated in different parts of the maize plant grown on a soil treated with sewage sludge for nine consecutive years and yield maize (**b**). Means followed by the same letters are not different by the Tukey's test ($P<0.05$).

(2007b) found metal accumulation in the aerial part. For two oxisols, Typic Eutrorthox and Typic Haplorthox treated with sewage sludge Melo (2002) found the following sequence for Ni concentration in maize plants: leaf > stem > husk and cob > grain, for both soils.

It is important to state that Ni in leaves may enter the food chain if the whole plant is used for silage production (Silva *et al*., 2006).

The treatments did not affect grain production (Figure 2b). There are two ways to explain this fact: (1) the different rates of sewage sludge were able to supply the required amounts of nutrients; and (2) this soil was sufficiently fertile so that fertilization was not necessary.

CONCLUSIONS

Sewage sludge evaluated through the accumulated amounts applied for nine consecutive years did not affect soil Ni concentration. Jackson's method detected more Ni in the soil than the USEPA's method, what is explained by the fact that the USEPA's method does not solubilize all the soil mineral fraction. Accumulated Ni was higher in leaves than in stem. Dry matter and grain production were not different for the treatments mineral fertilization and sewage sludge.

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