# HEAVY METALS IN SOIL TREATED WITH SEWAGE SLUDGE COMPOSTING, THEIR EFFECT ON YIELD AND UPTAKE OF BROAD BEAN SEEDS (Vicia faba L.)

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(Received 11 August 2004; accepted 28 April 2005)

Abstract. The final use that may be given to biosolids that result from the treatment of residual municipal waters depends on their physicochemical and microbiological characteristics. Their organic matter content and wealth of essential elements may allow their use for agricultural fertilization purposes. The objective of this research project was to evaluate the physicochemical interactions between soil treated with biosolids and compost from municipal residual waters, and the nutritional parameters of broad bean seeds (Vicia faba L.). The studied area is located in the central region of the Mexican Republic. The biosolids were treated with aerated static pile composting. The experimental work was performed in the area surrounding the East Toluca Macroplant, where nine  $2 \times 3$  m plots were defined and distributed in a Latin square; 3 plots were used as controls (without conditioning), 3 were conditioned with 4.5 Mg ha-1 of biosolids on a dry base, and 3 were conditioned with the same amount of compost. The parameters determined for biosolids, compost, and soil were: pH, electrical conductivity (EC), organic matter (OM), total nitrogen, available phosphorus, cation exchange capacity (CEC), exchangeable cations (Ca, Mg, Na and K), total and available heavy metals (Cd, Cr, Cu, Ni and Zn); for the plant: height reached, green seeds productivity and yield per treatment (ton  $ha^{-1}$ ); for the seeds: humidity, ashes, fiber, fats, protein, starch and total and available heavy metals (Cd, Cr, Cu, Ni and Zn). pH was slightly acid in soil treated with biosolids (6.71). OM and CEC did not represent significant differences. Total concentrations of Cr, Zn, Ni and Cu in soil presented significant differences (p < 0.05) between treated soil and the control, Cd was not detected. Cu was the most available metal in soil treated with compost (15.31%), Cd and Cr were not detected. The plants had higher growth rates with biosolids (112.22 cm) and compost (103.73 cm); higher green broad bean productivity and higher seed yield, especially in plots containing biosolids, which had rates three times higher than the control. In regards to broad bean seeds, content of ashes, fiber, fats, protein, starch and heavy metals (Cu, Ni and Zn), there were no significant differences between the treatments. Cd and Cr were not detected. In conclusion, it has been proven that the use of biosolids and compost studied in this broad bean crop do not involve an environmental risk, and thus give way to a solution to the problem of final disposition of biosolids in the region.

Keywords: Agricultural land application, biosolids, compost, heavy metals, nutritional quality

# Introduction

The use of biosolids and compost in agriculture can improve the soil's productive capacity and physical characteristics, as it allows the incorporation of organic matter

Water, Air, and Soil Pollution (2005) 166: 303-319

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and nutrients. However, an extended use of biosolids may cause environmental problems (Korentajer, 1991; NRC, 1996; Korboulewsky *et al.*, 2002, Binder *et al.*, 2002), such as lixiviation of pollutants, mainly nitrogen and phosphorus, through the soil (Nikolaidis *et al.*, 1999; Siddique, 2000; Lu and O'Connor, 2001; Elliot *et al.*, 2002). The organic matter in biosolids and compost increases the soil's ability to retain water. It improves the structure and increases its organic content, which makes it possible to have agricultural activities in soils with high clay content and decreasing soil erosion (Scott and Ahlstrom, 1985). Thorne *et al.* (1998) applied vegetation over the debris of a mine where biosolids were used as a stratum for restoring nutrients and microorganisms associated with the development of the soil, and thus created substratum that maintained the area's native vegetation. Brofas *et al.* (2000), used biosolids as fertilizer to recover soil in a calcareous bauxite mine, and observed that a 35% increase was produced in water retention capacity, therefore increasing the concentration of organic matter, nitrogen and phosphorus.

On the other hand, biosolids and compost provide plants with nutrients such as: nitrogen, phosphorus, calcium and potassium (Oberhaster, 1991); and trace nutrients – which are considered indispensable for plant growth – , such as: boron, copper, iron, magnesium, nickel and zinc. However, these elements, in addition to cadmium and chromium in high concentrations, can be toxic for the plant, causing a decrease in productivity or yield. Furthermore, they may occasionally produce symptoms that are less marked or are absent, however, at a cellular level, various processes are affected by the local increase of the concentration, thus producing stress in the plant (Vangronsveld and Clijsters, 1994). Such is the case regarding fiber content, fat, protein and starches in the plant, which may be modified by increasing the content of heavy metals.

Broad bean (*Vicia faba L.*) cultivation in Latin American countries is of high importance for local economics and nutrition. Mexico is one of the main broad bean producers in the world. Reports show an average national yield of 4.70 Mg  $ha^{-1}$  of green broad bean and 0.90 Mg  $ha^{-1}$  of broad bean seeds, and a production of 3,983 tons for 1990 (SARH, 1990).

The objective of this study is to investigate the accumulation of heavy metals in the soil and in broad bean crops, and the nutritional quality of broad bean seeds grown on soils treated with biosolids and compost from a residual water treatment plant in the Mexican Plateau.

# **Materials and Methods**

#### EXPERIMENTAL SITE

The study site is located in an agricultural area of the Municipality of Toluca, in Mexico's central region, where 9 land plots of  $2 \times 3$  m each were defined and distributed in a Latin square. Three plots were used as controls, another three were

treated with 4.5 Mg ha<sup>-1</sup> of biosolids on a dry base, as recommended by Gomez (1998), and the remaining three plots were treated with equal doses of compost. The plots were one meter apart from one another and the treatments were repeated three times. Biosolids and compost were applied in May, 2000 and were manually incorporated into the soil at a depth of 15 cm.

The soil is Phaeozem soil (FAO-UNESCO, 1990); it has thick, dark topsoil with clay loam texture, it is rich in organic matter content (>5%), and has a low C/N ratio, with a mean value around 9.

The leguminous broad bean (*Vicia Faba L.*), which has a low absorption of toxic elements, was chosen as temporary crop (Reichman, 2002). It was planted in May 2000 and harvested in September. The crop has a maturity period of 90 to 220 days. In order to obtain high yields in the cultivation of broad beans, the following is required: an annual rainfall of 650 to 1000 mm (in this region, mean yearly precipitation is 700 mm), deep, fertile and well drained soils, with a pH of between 6 and 7, and enough organic matter reserves (Kay, 1979).

### Biosolids and Compost

Biosolids were obtained from the municipal residual water treatment plant "East Toluca Macroplant", which is an activated sludge treatment plant.

The compost process was carried out in cone-shaped, 1.5-m diameter static piles. The piles were installed with the following characteristics: N/C 30–40 ratio. Aerating was performed through natural ventilation and by turning the piles over every day. The conditioning materials were: corn straw as a source of carbon, and shredded tire chips as a bulking agent (Garrido *et al.*, 2002). In general, these materials provide energy, absorb humidity (increasing the content of solids), provide structural integrity, and generate empty spaces; in other words, they introduce the necessary porosity to allow airing and ventilation in the system (Kuhlman, 1990). The piles were protected with a thin layer of sawdust and corn straw in order to avoid bad smells, the influence of the wind, etc., (Photo 1).



Photo 1. Composting piles.

The compost period lasted approximately eight weeks, and included the following stages: material homogenization, pile construction, thermophilic digestion, treatment, and separation of the conditioning material from the material obtained (compost). A temperature variation of 15 to 42 °C was observed, the compost could not stabilize during 5 days at 40 °C as stated by Standard 40 CFR-257 published by the EPA (1985), this was due to rain and low temperatures that were present during the days when the compost was being made.

The quality of biosolids and compost complied with Mexican Official Standards (NOM-004-SEMARNAT-2002), in metals and microorganisms.

# Sampling

Biosolids and compost sampling was performed according to EPA methodology (1988), taking an individual sampling of biosolids of approximately 3 kilos during seven days of wastewater plant operation. These were placed in polythene bags, air dried in the shade, and mixed in order to form a compound sample. Later, they were quartered, finely ground, passed through a 2 mm stainless steel sieve and stored in a refrigerator (4  $^{\circ}$ C) until the analysis was complete.

### Analysis

Biosolids and compost analyses were performed with three replications, and the following parameters were determined: pH 1:2.5 distilled water (Jackson, 1982); OM, by weight differences determined by Walkley and Black method, modified by Jackson (1982), CEC (Jackson, 1982); total nitrogen, Kjeldahl method (Bremmer, 1996); available phosphorus, Olsen method (Jackson, 1982) and electrical conductivity, EC, (Porta *et al.*, 1999). Concentrations of exchangeable cations (Ca, Mg, Na and K) were obtained using the ammonium acetate method (Chapman, 1973), total heavy metals (Cd, Cr, Cu, Ni and Zn) by EPA's 3050 method (1988) and available metals by Lindsay and Norvell (1978). The quantification of exchangeable cations and heavy metals was determined by atomic absorption spectrophotometry using Varian Spectronic 20D equipment.

Faecal and total coliforms were determined by the method of fermentation in multiple tubes (MPN) (APHA, AWWA and WEF, 1995).

Tables I and II show physicochemical characteristics and the concentration of total and available heavy metals, in biosolids and compost, respectively.

# SOIL

## Sampling

Once the terrain area was established, considering  $6 \text{ m}^2$  land plots, and 1-m separation avenues, a random sampling was performed. Due to the homogeneity of the terrain, according to several studies performed in the Science Department of the Mexico State University (UAEMex) (Gomez, 1998; Vaca Paulin, 1999), no significant differences were found in the physical and chemical analyses of the soil.

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Parameter	Biosolids	Compost	Variation, (%)		
Humidity (%)	80.91	56.65	29.98		
pH H <sub>2</sub> O (1:2.5)	6.27	7.37	-14.92		
pH KCl 1N (1:5)	6.2	7.12	-12.92		
EC (S m <sup>-1</sup> )	0.37	0.68	-45.59		
OM (%)	53.65	49.24	8.22		
N (%)	6.36	5.02	21.07		
P available (%)	0.41	0.57	-28.07		
C/N	4.69	5.45	-13.94		
CEC (cmol kg <sup>-1</sup> )	75.6	61.31	18.90		
Ca (cmol kg <sup>-1</sup> )	19.03	10.22	46.30		
Mg (cmol kg <sup>-1</sup> )	29.18	6.94	76.22		
Na (cmol kg <sup>-1</sup> )	4.43	6.09	-27.26		
K (cmol kg <sup>-1</sup> )	10.58	19.18	-44.83		

TABLE I	
Physical and chemical characteristics of biosolids and compost	

# TABLE II

Concentration of total and available heavy metals in biosolids and compost (mg kg $^{-1}$ , d.w.)

	Bi	osolids	Co	mpost	(NOM-004-SEMARNAT
Metal	Total	Available	Total	Available	-2002)Mexico
Cadmium	3.75	0.8	3.5	1.38	39–85
Copper	169.6	40.05	148.05	123.88	1500-4300
Chromium	49.2	ND	31	ND	1200-3000
Nickel	38.1	19.09	33.7	22.19	420
Zinc	583.3	66.22	544.75	66.2	2800-7500

ND: Not detected, detection limit. d.w.: dry weight.

Three random samples were taken in the Ap horizon study area (the plowable strata, which are the first 30 cm of depth) in order to evaluate the soil's initial conditions. After the harvest, 2 samples of the Ap horizon were taken from each plot to form a compound sample, with a total of 9 samples. These were kept in polythene bags and stored in a refrigerator (4  $^{\circ}$ C) until they were analyzed.

#### Analysis

Analyses were performed with three repetitions, determining the following parameters: pH 1:2.5 distilled water (Jackson, 1982); electric conductivity (Porta *et al.*, 1999); texture, Bouyoucos method (1963); organic matter, Walkley-Black method (1947) modified for soil (Jackson, 1982); cation exchange capacity (Jackson, 1982); total nitrogen, Kjeldahl method (Bremmer, 1996) and available phosphorus

Property	$\mathbf{T}^{\dagger}$	$\mathbf{B}^{\ddagger}$	C§
Texture Clayish crumb		-	_
Туре	Feozem	_	_
pН			
H <sub>2</sub> O (1:2.5)	7.18 <sup>a</sup>	6.71 <sup>b</sup>	7.33 <sup>a</sup>
KCl (1:5)	6.55 <sup>a</sup>	6.11 <sup>b</sup>	6.41 <sup>a</sup>
EC (S $m^{-1}$ )	0.38	0.33	0.36
OM (%)	5.22 <sup>a</sup>	5.39 <sup>a</sup>	5.45 <sup>a</sup>
N (%)	0.3	0.28	0.28
P available (mg kg <sup>-1</sup> )	57.14 <sup>a</sup>	106.9 <sup>b</sup>	123.2 <sup>b</sup>
C/N	10.1	11.18	11.28
CEC (cmol kg <sup>-1</sup> )	30.58	30.38	30.48
Ca (cmol kg <sup><math>-1</math></sup> )	18.21 <sup>a</sup>	17.78 <sup>b</sup>	18.10 <sup>a</sup>
Mg (cmol $kg^{-1}$ )	9.91	10.73	9.51
Na (cmol kg <sup>-1</sup> )	0.86	0.79	0.82
K (cmol kg <sup><math>-1</math></sup> )	0.49	0.53	0.51
Sand (%)	42.4	44.7	44.4
Slime (%)	27.2	24.97	26.4
Sand (%)	30.4	30.3	29.2
Total Coliforms (MPN g <sup>-1</sup> )	5480	3074	3566
Faecal Coliforms (MPN g <sup>-1</sup> )	<300	2200	1190

TABLE III Soil physicochemical and microbiological characteristics

<sup>†</sup>Control soil; <sup>‡</sup>Soil treated with biosolids; <sup>§</sup>Soil treated with compost. NOTE: Different letters (a and b) indicate significant differences (p < 0.05). The parameters that have no letters did not present significant differences in the treatments (p > 0.05).

measured by the Bray I method, (1966). Exchangeable cations and total and available metals were also determined using the same methods described for biosolids and compost.

Table III shows the physical and chemical characteristics of the control soil, the soil treated with biosolids and the soil treated with compost.

# BROAD BEAN ANALYSIS

Broad bean seeds, dried at a temperature of  $55 \,^{\circ}$ C for 24 h, were analyzed. They were later ground in a mortar and, finally, in a mill.

#### Analysis

The analyses used to evaluate nutritional quality were: percentage of humidity and ashes, raw fiber determined by the acid detergent method (Bateman, 1970), fat, using

the Goldfish method (NOM-F-90-S-1978), nitrogen and protein, using the Kjeldahl method (Bateman, 1970), starches, using the DNS method (AOAC, 1990) and heavy metals, using Van Loon's method (1998). Quantification was performed with atomic absorption spectrophotometry, using Varian Spectronic 20D equipment.

The plant's height was determined from its base to the highest tip of its main stem. Crop productivity and yield were also determined.

## Data analysis

An Analysis of variance (ANOVA) and Tukey's test were performed in order to compare the means of the different treatments. In addition, simple and multiple correlations were performed in order to determine possible dependencies between the characteristics of the soil treated with biosolids and compost and the broad beans' nutritional quality. The results were analyzed at 95% confidence level.

### **Results and Discussion**

#### **BIOSOLIDS AND COMPOST**

As shown in Table I, humidity decreased during composting due to microbial activity involved in the process, which requires significant amounts of water in its metabolism, and because of the temperature reached. pH increased approximately 1 unit, fitting within the 6-8 range recommended by the Composting Council (TCCA), 1993. Electrical conductivity increased by 45.27% during composting. This result is related to an increase of 27.26% and 44.83% in the concentration of sodium and potassium. It must be taken into account that a conductivity of >4 mmhos cm<sup>-1</sup> restrains plant growth, causing high osmotic pressures in the roots. C/N ratio in the biosolids and compost was of 4.69 and 5.45, respectively, which is relatively low compared with the optimum range, which is between 10 and 16 (Carmona et al., 1994). This is possibly due to the fact that the biosolids and compost had high nitrogen content. The content of organic matter, Ca, Mg (8.22%, 46.29%, 76.22%) in the compost decreased due to the action of degrading microorganisms in the organic matter, which mineralize it and, at the same time, dispose of some ions, including heavy metals. On the other hand, phosphorus levels increased by 28.07% after composting, and potassium increased by 44.83%. Darmody et al. (1983) considered that composting increases the values of these elements, possibly due to the mineralization presented by these nutritional elements during the process.

Total and faecal coliforms were 40,000 and <3,000 MPN g<sup>-1</sup> on a dry base. The compost was classified as "Class C", by the NOM-004-SEMARNAT-2002, which considers this compost fit for agricultural uses.

The values of total heavy metals (Table II) decreased during composting. He *et al.* (1992), mentioned that in studies performed with composted sludge, lower concentrations of heavy metals were found, as compared with biosolids, even if

their availability increased. Such is the case of copper, which increased by 83%. This is attributed to the fact that the availability of metals such as Cd and Cu is increased during composting because microbial activity contributes to the loss of exchange sites, as those found in organic matter during its decomposition. As a matter of fact, Zn as well as Cu may be mobile during composting because of the direct correlation that both have with organic carbon (Darmody *et al.*, 1983).

## SOIL TREATED WITH BIOSOLIDS AND COMPOST

The control soil and soil treated with compost presented a neutral pH of 7.18 and 7.33 (Table III), respectively, while soil treated with biosolids had a pH of 6.71, and was slightly acid, presenting significant differences (p < 0.05). This slight decrease in pH is possibly due to the formation of organic acids, resulting from the addition of organic matter to the soil without going through a process of mineralization, as in the case of biosolids. The percentage of organic matter and the cation exchange capacity in the soil did not present significant differences among the treatments.

Regarding exchangeable cations, it was observed that even when calcium is being added through the biosolids, there is a concentration of 17.78 cmol kg<sup>-1</sup>, which is significantly lower (p < 0.05) compared to the control (18.21 cmol kg<sup>-1</sup>) and to soil treated with compost (18.10 cmol kg<sup>-1</sup>) (Table III). There were no significant differences among the treatments regarding magnesium, sodium and potassium (p > 0.05), and therefore, there was also no significant difference regarding conductivity.

Even though biosolids are considered an excellent medium to replenish the nitrogen that the soil loses in cultivation, there were no significant differences between the treatments (p > 0.05). C/N ratio shows a greater tendency in biosolids and compost treatments, when compared to other treatments. This indicates that the soil has a good fertility level as, according to Tisdale and Nelson (1982), a C/N ratio greater than 15 can produce immobilization of N (Castellanos *et al.*, 2000).

Available phosphorus in soil treated with biosolids and compost increased by 43–53% in relation to the control soil (Table III), presenting significant differences among the treatments. These results are according to the Darmody *et al.* (1983) and James and Aschman (1992) studies, which found that when adding compost to the soil, the available phosphorus increases up to several times in soil treated with biosolids.

The percentage of sand, slime and clay is presented in Table III, which shows that there are no significant differences (p > 0.05) between the treatments. According to the classification of the United States Department of Agriculture, it presented a clayish crumb texture (Porta *et al.*, 1999).

The concentration of total heavy metals in the soil presented the following sequence Zn > Ni > Cu > Cr, Cd was not detected (detection limit). The sequence for available heavy metals was Zn > Cu > Ni > Cr = Cd (Table IV). Figure 1 shows metal availability percentage in the soil.

Concentration of total and available heavy metals in the soil (mg $g^{-1}$ , d.w.)												
		Cr			Cu			Ni		Zn		
	$\mathbf{T}^{\dagger}$	$B^{\ddagger}$	C§	$\mathbf{T}^{\dagger}$	$\mathbf{B}^{\ddagger}$	C§	$\mathrm{T}^{\dagger}$	$\mathbf{B}^{\ddagger}$	C§	$\mathrm{T}^{\dagger}$	$\mathbf{B}^{\ddagger}$	C§
Total	4.43 <sup>a</sup>	8.82 <sup>b</sup>	7.51 <sup>b</sup>	11.52 <sup>a</sup>	14.53 <sup>b</sup>	11.11 <sup>a</sup>	13.43 <sup>a</sup>	23.38 <sup>c</sup>	20.02 <sup>b</sup>	46.09 <sup>a</sup>	58.38 <sup>b</sup>	52.44
Available	ND	ND	ND	1.33 <sup>a</sup>	1.93 <sup>b</sup>	1.71 <sup>a</sup>	1.11 <sup>a</sup>	1.40 <sup>b</sup>	1.27 <sup>b</sup>	7.03 <sup>a</sup>	8.00 <sup>b</sup>	7.81 <sup>b</sup>
SD, total	1.19	1.59	1.18	1.71	1.35	1.29	3.76	2.25	2.43	6.08	5.26	6.82
SD, available	; –	-	-	0.36	0.24	0.23	0.2	0.21	0.15	1.03	0.41	0.07

TABLE IV Concentration of total and available heavy metals in the soil (mg  $g^{-1}$ , d.w.)

<sup>†</sup>Control Soil; <sup>‡</sup>Soil treated with biosolids; <sup>§</sup>Soil treated with compost.

ND: Not detected, detection limit; SD:  $\pm$  Standard deviation; d.w.: dry weight.

NOTE: Different letters (a, b and c) indicate significant differences (p < 0.05).

The parameters that have no letters did not present significant differences in the treatments (p > 0.05).

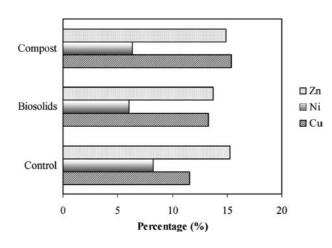


Figure 1. Metals availability percentage in the soil.

The application of biosolids and compost increased the total content of Cr by almost two times as compared with the control soil (Table IV). Therefore, significant differences were obtained (p < 0.05) between the control soil and the two treatments. In spite of this, available Cr was not detected.

The lowest concentrations of Cu occurred in the control soil and in soil treated with compost (Table IV), which was significantly different from the biosolids treatment. This element has low mobility because it forms stable links with organic matter, thus decreasing availability (Zhu and Alva, 1993).

Total Ni concentration was 13.43 mg kg<sup>-1</sup> in the control soil, and 23.38 mg kg<sup>-1</sup> and 20.02 mg kg<sup>-1</sup> in the biosolids and compost treatments, respectively, presenting significant differences (p < 0.05) among them. The same occurred with available Ni, which increased in concentration with the application of biosolids. Nevertheless,

the percentage of availability is very low compared with total Ni. Kabata-Pendias (2001), mentioned that the high content of organic matter in residual sludge may decrease its availability for plants.

The content of Zn presented a higher concentration in soil conditioned with biosolids (58.38 mg kg<sup>-1</sup>) than in the compost treatment (52.44 mg kg<sup>-1</sup>). Both were significantly different (p < 0.05) from the control (Table IV). Regarding concentration of available Zn and total Zn, there were significant differences (p < 0.05) between the control and the treatments. Zn availability corresponds to a seventh part of the total concentration. It is important to consider that high concentrations of Zn in biosolids may increase Zn concentration in the soil, and in high doses, it may compete with other nutrients such as iron and manganese, generating a nutritional unbalance (Otte and White, 1993).

Special attention is given to Zn because the biosolids applied to the soil present high concentrations of this element, which may be available to the plants. However, organic matter helps decrease its effects, because stable complexes are formed, which reduces the metal's availability. (Cripps *et al.*, 1992). Zn presented a negative correlation (r = -0.91, p < 0.05) with Cu, suggesting that both metals are absorbed through the same mechanism, which makes them compete with each other (Kabata-Pendias, 2001). According to Alloway (1990), Bohn (1993) and Kabata-Pendias (1995) none of the heavy metals presented concentrations beyond the criteria limits for agricultural use (Table V).

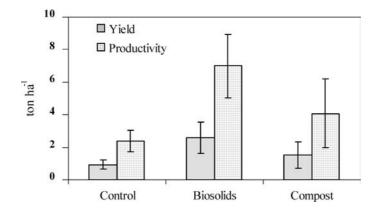
Regarding total and faecal coliforms, the application of biosolids did not alter much the concentration of coliforms (Table III), which are naturally in the soil, due to the competition existing among microorganisms, and to the fact that biosolids, being exposed to environmental conditions before the soil is cultivated, may present a decrease in the population of pathogenic microorganisms (Oberhaster, 1991). Helminth ova were not detected in the biosolids.

Piatkin (1986), mentioned that, due to contamination, fertilization, and farm work, most of the microorganisms that are disseminated throughout the soil do not form spores, so they remain in the soil for a short period of time; the soil being an unfavorable medium for most pathogenic species.

Criteria for concentrations of total heavy metals in biosolids for agricultural use (mg $g^{-1}$ , d.w.)											
Metal	E.U. (Page <i>et al.</i> , 1983)	U.E. (Lester, 1987)	Alloway (1990)	Bohn (1993)	Kabata and Pendias (1995)						
Cadmium	50	20	0.01-2.4	0.01–7	3–5						
Copper	800-1,000	1,000	2-250	2-100	50-120						
Chromium	1,000	750	5-1,500	5-1,000	50-125						
Nickel	100-200	300	2-1,000	10-1,000	20-100						
Zinc	2,000–2,500	2,500	10-300	10-300	70–400						

TABLE V

d.w.: dry weight.



*Figure 2.* Productivity and yield of the broad bean (*Vicia faba L.*) crop. Mean and standard error of n = 3. The results demonstrate statistically significant differences (p < 0.05) and standard deviation.

## THE EFFECT OF BIOSOLIDS AND COMPOST ON THE PLANT

### Productivity and Yield

Regarding productivity and yield, the plants that were conditioned with biosolids and compost were bigger, greener and had good foliage, presenting significant differences (p < 0.05). In fact, the plants that first flowered and bore fruit were in land plots conditioned with biosolids, and their height from the base to the highest point of the tallest stem reached 112.22 cm; in soil conditioned with compost, height was 103.73 cm and in the control soil, 94.62 cm.

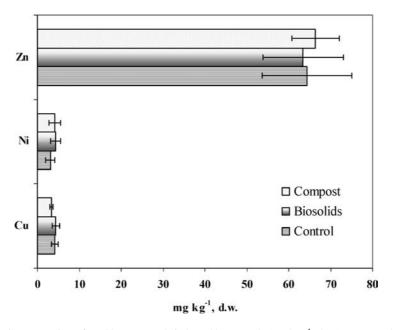
Results in Figure 2 show that soil conditioned with biosolids had a productivity of 7 Mg ha<sup>-1</sup> and a yield of 2.58 Mg ha<sup>-1</sup>, which is almost three times higher than the control soil (productivity 2.39 Mg ha<sup>-1</sup> and yield 0.93 Mg ha<sup>-1</sup>). In addition, soil conditioned with compost presented a productivity of 4.08 Mg ha<sup>-1</sup> and a yield of 2.58 Mg ha<sup>-1</sup>; which is almost twice as large as the control soil. These results demonstrated statistically significant differences (p < 0.05) in productivity and yield among the treatments, which may be attributed to the nutritional benefits provided by the biosolids. However, it is important to mention that due to constant rains, the crop was harvested 20 to 30 days before due and so productivity is not proportional to the yield and, therefore, a greater quantity of seeds might have been obtained at the crop.

Although the yield obtained may be lower than expected due to the above mentioned factors, if we compare it to productivity and yield in the State of Mexico in 1995, which was of 5.45 and 1.58 ton  $ha^{-1}$ , respectively, and in 1995 which was of 3.84 to 5.11 ton  $ha^{-1}$  in productivity and 0.8 ton  $ha^{-1}$  in broad bean yield (SARH, 1990; SAGAR, 1995; INEGI, 1999), the production and yield obtained in this project were favorable.

CHEMICAL ANALYSES

#### Heavy Metals in the Seeds

The presence of total heavy metals in biosolids may have toxic effects on the crops and cause the plants to wither, thus decreasing production (Sterritt and Lester, 1980); for this reason, heavy metals are a limiting factor in the preparation of land for cultivation (Logan et al., 1987). In this study, the concentration of total heavy metals in seeds did not present significant differences (p > 0.05) among the treatments and was not above the intervals recommended by several authors (Bohn et al., 1993, Cd: 01–0.8; Cu: 4–15; Ni: 1; Zn: 8–15 mg kg<sup>-1</sup> p.s. and Kabata-Pendias, 2001, Cd: 0.5; Cu 30; Cr: 2; Ni: 3; Zn: 55 mg kg<sup>-1</sup> p.s.). Figure 3 shows that the concentration of Cu is slightly greater in seeds treated with biosolids  $(4.40 \text{ mg kg}^{-1})$ , while it tends to decrease in the compost treatment,  $(3.42 \text{ mg kg}^{-1})$  with respect to the control (4.13)mg kg $^{-1}$ ). An important factor that might have produced a decrease in Cu is that the biosolids used contained a great amount of Zn, which is an antagonistic element in plants absorption, so Cu is at a disadvantage. Cd and Cr were not detected (detection limit). It is important to mention that heavy metals may be accumulative in plants in various degrees, depending on the type of soil, pH concentration of elements, and plant species rates of application (Alberici et al., 1989). The physiological processes, as well as plant transpiration, breathing and photosynthesis (which easily accumulates Zn, Cu, Cd, Ni, among others), are affected.



*Figure 3*. Concentration of total heavy metals in broad bean seeds (mg kg<sup>-1</sup>, d.w.). Mean and standard error of n = 3. The results demonstrate statistically significant differences (p < 0.05).

As far as concentrations of Ni are concerned, the application of biosolids and compost caused a tendency to increase the content of this metal in broad bean seeds, 4.35 and 4.20 mg kg<sup>-1</sup>, respectively (control 3.12 mg kg<sup>-1</sup>). This may be related to the fact that Ni is a metal that, in its soluble phase, is rapidly absorbed by the plant and stored in its leaves and mainly in the seeds. The ranges of toxicity due to Ni in plants, per Kloke *et al.* (1984); Macnicol *et al.* (1985) are within 10–100 mg kg<sup>-1</sup>; but some vegetables are affected by ranges of 10–30 mg kg<sup>-1</sup>. Plants did not present symptoms of toxicity, such as chlorosis, caused by lack of Fe due to excessive Ni (Kabata-Pendias, 2001).

Zn concentration is slightly higher in seeds treated with compost 66.43 mg kg<sup>-1</sup> than in seeds treated with biosolids 63.43 mg kg<sup>-1</sup> (control 64.3 mg kg<sup>-1</sup>). Zn is a movable element of rapid absorption in the plant because composting permits a greater availability of heavy metals (Castellanos *et al.*, 2000). Therefore, the concentration of this element increased in the seeds and Cu decreased, because these are antagonistic elements.

### SEEDS NUTRITIONAL VALUE

The parameters analyzed for the nutritional quality of the broad bean seeds presented significant differences (p > 0.05) among the treatments. However, there are certain tendencies in some of the analyses, as can be seen in Table VI.

No difference was observed regarding the percentage of humidity and ashes among the treatments. The percentage of humidity in broad bean seeds reported by INEGI (1995) is 78%, but it is variable according to the varieties of broad bean; the same occurs with the percentage of ashes, which is 4.0%. Protein and fat increased by 1.83 and 21% in the broad bean seeds with biosolids application, because the seeds treated with biosolids presented a higher concentration of Cu, which has an important role in protein and lipids synthesis (Baron *et al.*, 1995; Cervantes, 1999; Castellanos *et al.*, 2000). Thus, the mechanism used by plants when there is a high concentration of Cu is to induce the production of protein and lipids in order to stabilize the concentration of this metal in the cell (Tomsett and Thurman, 1988; Zeiger, 1998; Cervantes, 1999).

	Humidity Ash		Protein			Fat			Fiber			Starch						
	$T^{\dagger}$	$\mathbf{B}^{\ddagger}$	C§	$\mathbf{T}^{\dagger}$	$\mathbf{B}^{\ddagger}$	C§	$\mathbf{T}^{\dagger}$	$\mathbf{B}^{\ddagger}$	C§	$\mathbf{T}^{\dagger}$	$\mathbf{B}^{\ddagger}$	C§	$\mathbf{T}^{\dagger}$	$\mathbf{B}^{\ddagger}$	C§	$\mathbf{T}^{\dagger}$	$\mathbf{B}^{\ddagger}$	C§
	Percentage (%)																	
	7.93	72.72	71.2	4.14	4.31	4.61	23.2	25.49	24.27	2.81	3.4	3.33	11.11	9.85	10.34	41.02	39.15	42.92
1*		78			4			21-41			2.0-3.0			8.0–15			30-42.3	
SD	1.75	4.98	1.64	0.2	0.35	0.12	0.97	1.15	2.4	0.53	0.48	0.3	1.34	0.98	1.09	4.24	5.24	4.4

TABLE VI Analysis of nutritional qualities in broad beans (*Vicia faba L.*)

<sup>†</sup>Control Soil; <sup>‡</sup>Soil treated with biosolids; <sup>§</sup>Soil treated with compost; SD:  $\pm$  Standard deviation. 1<sup>\*</sup>: Chaven (1989) and INEGI (1995).

The concentration of fiber decreased by 11.34 and 6.94% in the treatment with biosolids and compost. Lignin is the principal component of fiber and, when in contact with stress agents, such as bacteria, fungi and heavy metals, is rapidly synthesized to form a barrier or wall to block out pollutants (Zeiger, 1998).

Starch concentration in the seeds was lower in the treatment conditioned with biosolids (4.56%), but it presented a tendency to increase in the treatment conditioned with compost (4.63%), which is attributed to the fact that, in this treatment, the seeds presented a greater concentration of available Zn, which is an element that participates actively in the formation of starch. Vaca *et al.* (1999), observed through microscopic analyses of the root of the plant that, when applying different concentrations of biosolids in the cultivation of broad beans, the content of starch in the cells decreased as biosolids concentration increased.

The previous results show that the nutritional quality, the values of humidity, ashes, protein, fats, fiber and starch, are within the ranges reported by Chaven *et al.* (1989), INEGI (1995), (Table VII). No significant correlations were present (p > 0.05) between the physical and chemical parameters of the soil and the nutritional quality of broad bean seeds.

# Conclusions

- It was proved that the use of biosolids and compost in this study does not imply environmental risks, which would permit a solution to the problem of the final disposal of biosolids in this region.
- The concentration of heavy metals in biosolids was found to be within the permitted limits reported by Mexican Official Standards, therefore, there was low availability for the plants, and they were considered stable for their potential use in agriculture.
- The biosolids compost allowed a decrease in total and faecal coliforms, which allows a better handling of the biosolids.
- The total concentrations of Cr and Ni presented a significant increase of more than 40% and more than 20% in Cu and Zn, in the plots that contained biosolids and compost. However, none of the treatments went beyond their permissible limits.
- The availability of Cu, Zn and Ni, was significantly higher in soils conditioned with biosolids and compost.
- The broad bean plants showed greater growth in plots containing biosolids, which allowed a yield three times greater than the control.
- Broad bean seeds did not present significant differences in concentrations of Cu, Ni and Zn.
- The percentage of humidity, ashes, fiber, protein, fats and starches did not present significant differences between the treatments. However, the percentage of starch and fiber was smaller in plants conditioned with biosolids, but the content of

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protein and fat was higher. In plants conditioned with compost, starch content did not tend to increase.

 Greater productivity and yield of broad bean crops was observed in plots treated with biosolids. However, compost gives greater stability, eliminating pathogenic microorganisms and bad smells.

## Acknowledgements

We thank ECOSISTEMAS S.A. de C.V. (Macroplanta Toluca Oriente, Mexico) for their assistance in the sample collection and for their financial support.

#### References

- Alberici, T. M., Sopper, W. E., Storm G. L. and Yahner, R. H.: 1989, 'Trace metals in soil, vegetation and voles from mine land treated with sewage sludge', *J. Envion. Qual.* 18, 115–120.
- Alloway, B. J.: 1990, Heavy Metals in Soils, John Wiley and Sons, New York.
- Association of Official Analytical Chemists (AOAC): 1990, *Official Methods of Analysis*, vol. II, AOAC, Washington, DC.
- APHA, AWWA, WEF: 1995, *Standard Methods for the Examination Water and Wastewater*, 19th edn., APHA, AWWA & WEF. Washington, D.C.
- Baron, M., Arellano, J. B. and Lopez, G. J.: 1995, 'Copper and photosystem II. A controversial relationship', *Physiol. Plant.* 94, 174–180.
- Bateman, J. V.: 1970, Manual de métodos analíticos. Ed. Herrero Hermanos. Mexico.
- Binder, D. L., Dobermann, A., Sander, D. H. and Cassman, K. G.: 2002, 'Biosolids as nitrogen source for irrigated maize and rainfed sorghum', *Soil Sci. Soc. Am. J.* 66, 531–543.
- Bohn, H. L., Mcneal, B. L. and O'Connor, G. A.: 1993, Quimica de suelos, LIMUSA Mexico.
- Bouyoucos, G. J.: 1963, 'Directions for making mechanical analysis of soil by hydrometer method', *Soil Sci.* **42**, 23–30.
- Bray, R. H. and Kurt, L. T.: 1966, 'Determination of total, organic and available forms of phosphates in soils', *Soil Sci.* 59, 39–45.
- Bremner, J. M.: 1996, Nitrogen-Total. p. 1103–1108. *In* Sparks DL. Ed. Methods of soil analysis. Part 3 Chemical methods. SSSA Book Ser. 5. SSSA, Madison, WI.
- Brofas, G., Michopoulos, P. and Alifragis, D.: 2000, 'Sewage sludge as an amendment for calcareous bauxite mine spoils reclamation', J. Environ. Qual. 29, 811–816.
- Carmona, A., Garcia, M. and Azaola, E.: 1994, Determinación de las condiciones óptimas para la obtención de compostas de alta calidad agronómica. Biotecnología. Revista de la Sociedad Mexicana de Biotecnología y Bioingeniería, AC. 34, 12–20.
- Castellanos, J. Z., Uvalle, B. J. and Aguilar, S. A.: 2000, Manual de interpretación de análisis de suelos y aguas. Instituto de Capacitación para la Productividad Agrícola. 2ª edn. Mexico.
- Cervantes, C.: 1999, Contaminación ambiental por metales pesados. Ed. A.G.T. 1ª edn. Mexico.
- Chapman H. D.: 1973, Métodos de análisis para suelos, plantas y aguas Trillas. Mexico.
- Chaven, J. K., Kute, L. S. and Kadam, S. S.: 1989, Hand Book of World Legumes. Ed. Salonkhe and S. S. and Kadman. Florida U.S.A.
- Cripps, W., Winfree, S. K. and Reagan, J. L.: 1992, 'Effects of sewage sludge application method on corn production', *Soil. Sci. Plant Anal.* 23, 1705–1715.
- Darmody, R. G., Foss, J. E., McIntosh, M. and Wolf, D. C.: 1983, 'Municipal sewage sludge compost amended soils: Some spatiotemporal treatment effects', J. Environ. Qual. 12, 231–235.

- Elliot, H. A., O'Connor, G. A. and Brinton, S.: 2002, 'Phosphorus leaching from biosolids-amended sandy soils', *J. Environ. Qual.* **31**, 681–689.
- EPA (Environmental Protection Agency): 1985, Composting of municipal wastewater sludges. EPA/625/4-85/014. Cincinnati Ohio, USA.
- EPA (Environmental Protection Agency): 1988, Sampling procedures and protocols for the national sewage sludge survey. WH-552, EPA, Office of Water Regulations and Standards. Washington D.C., USA.
- FAO-UNESCO: 1990, Soil Map of the World, revised legend. World Soil Resour. Rep. 60 FAO, Rome.
- Garrido, S. E., Vilchis, J. A., Andre, C., García, J. J., Alvarez, A. and Gorostieta, E.: 2002, 'Aerobic thermophilic composting of wastewater sludge', *Resources, Conservation and Recycling* 34, 161– 173.
- Gomez, B. G.: 1998, Variación estacional de metales pesados en lodos residuales de la planta Toluca Oriente México. Tesis de Licenciatura, Facultad de Ciencias, Universidad Autónoma de México. Mexico. 72 pp.
- He, X. T., Traina, S. J. and Logan, T. J.: 1992, 'Chemical properties of municipal solid waste composts', *J Environ. Qual.* 21, 318–329.
- INEGI (Instituto Nacional de Estadística, Geografía e Informática): 1995, El Sector Alimentario en México.
- INEGI (Instituto Nacional de Estadística, Geografía e Informática): 1999, Anuario Estadístico del Estado de México. Mexico.

Jackson, L. M.: 1982, Análisis Químico de Suelos, 4ª edn., Omega, Barcelona, España.

- James, B. R. and Aschmann, S. G.: 1992, 'Soluble phosphorus in a forest soil Ap horizon amended with municipal wastewater sludge or compost', *Soil. Sci. Plant Anal* **23**(7–8), 861–875.
- Kabata-Pendias, A.: 1995, 'Agricultural problems related to excessive trace metal contents of soils', in W. Salomons, U. Förstner and P. Mader (eds.), *Concerning Heavy Metals: Problems and Solutions*, Springer-Verlag, Berlin, Heidelberg. New York. Printed in Germany, pp 19–31.

Kabata-Pendias, A. and Pendias, H.: 2001, *Trace Elements in Soils and Plants*, 3<sup>a</sup> edn., Edition CRC Press, Boca Ratón, FL. 413 pp.

- Kay, D. E.: 1979, Legumbres Alimenticias. Ed. ACRIBIA Zaragoza. España.
- Kloke, A., Sauerbeck, D. R. and Vetter, H.: 1984, The contamination of plants and soils with heavy metals and the transport of metals in terrestrial food chains. Changing metal cycles and human health. Ed. Dahlem. Berlin. 113 pp.
- Korboulewsky, N., Dupouyet, S. and Bonin, G.: 2002, 'Environmental risks of applying sewage sludge compost to vineyards: Carbon, heavy metals, nitrogen and phosphorus accumulation', *J. Environ. Qual.* 31, 1522–1527.
- Korentajer, L.: 1991, 'A review of the agricultural use of sewage sludge: Benefits and potential hazards', *Water, Air and Soil Pollution* **17**(3), 189–196.
- Kuhlman, L. R.: 1990, 'Windrow composting of agricultural and municipal wastes', *Resources*, *Conservation and Recycling* 4, 151–160.
- Lester, J. N.: 1987, Heavy metals in wastewater and sludge treatment processes. Vol. II. Treatment and Disposal. CRC Press, Inc. Florida. USA.
- Lindsay, W. L. and Norvell, W. A.: 1978, 'Development of DTPA soil test for zinc, iron, manganese and cooper', *Soil. Sci. Soc. Am. J.* **42**, 421–428.
- Logan, T. J., Chang, A. C., Page, A. L. and Ganje, T. J.: 1987, 'Accumulation of selenium in crops grown on sludge treated soil', J. Environ. Qual. 16(4), 349–352.
- Lu, P. and O'Connor, G. A.: 2001, 'Biosolids effects on phosphorus retention and release in some sandy Florida Soils', J. Environ. Qual. 30, 1059–1063.
- Macnicol, R. D. and Beckett, P. H. T.: 1985, 'Critical tissue concentrations of potentially toxic elements', *Plant Soil* 85–87.
- Nikolaidis, N. P., Chheda, P., Lackovic, J. A., Guillard, K., Simpson, B. and Pedersen, T.: 1999, 'Nitrogen mobility in biosolid-amended glaciated soil', *Water Environ. Res.* **71**(3), 368–376.

- NOM-004-SEMARNAT-2002, Protección ambiental. Lodos y biosólidos. Especificaciones y límites máximos permisibles de contaminantes para su aprovechamiento y disposición final. Secretaria de Medio Ambiente y Recursos Naturales. México. Diario Oficial de la Federación. Mexico, 15 de agosto de 2003.
- NOM-F-90-S-1978, Determinación de grasa por el método de Goldfish. Diario Oficial de la Federación. Mexico.
- NRC (National Research Council): 1996, Use of Reclaimed Water and Sludge in Food Crop Production, National Academies Press, Washington D. C.
- Oberhaster, G.: 1991, 'South African practice in land disposal of sludge, including legislation and health aspects', *Wat. Sci. Tech* **15**, 151–155.
- Otte, M. L. and Wijte, A. H. B. M.: 1993, 'Environmental variation between habitats and uptake of heavy metals by Urtica dioica', Environ. Monitoring and Assess. 25, 263–275.
- Page, A. L., Gleason, T. L., Iskandar, I. K. and Sommers, L. E.: 1983, Utilization of municipal wastewater and sludge on land. Proc. Workshop USEPA. University of California, USA.
- Piatkin, K: 1986, Microbiología, Editorial MIR Moscu. 2nd edn., USSR.
- Porta, J., Lopez, A. M. and Roquero, C.: 1999, *Edafología para la agricultura y el medio ambiente*, Mundi Prensa, Spain.
- Reichman, S. M.: 2002, The responses of plants to metal toxicity: A review focusing on copper, manganese and zinc. Ed. Australian Minerals & Energy Environment Foundation. Australia.
- SAGAR, Secretaria de Agricultura: 1995, Anuario Estadístico de la Producción Agrícola de los Estados Unidos Mexicanos. Tomo I. Centro de Estadística Agropecuario. Mexico.
- SARH, Secretaria de Agricultura y Recursos Hidráulicos: 1990, Anuario estadístico de la producción agrícola de los Estados Unidos Mexicanos. Tomo I. Datos Básicos Agrícolas de la Subdelegación de Planeación, Estado de México. Mexico.
- Scott, B. and Ahlstrom, P. E.: 1985, 'Irradiation of municipal sludge for agricultural use', *Radiat. Chem.* 28, 1–3.
- Siddique, M. T., Robinson, J. S. and Alloway, B. J.: 2000, 'Phosphorus reactions and leaching potential in soils amended with sewage sludge', J. Environ. Qual. 29, 1931–1938.
- Sterritt, R. M. and Lester, J. N.: 1980, 'Concentrations of heavy metals in forty sewage sludges in England', *Water Air and Soil Pollution* 14, 125–131.
- TCCA. The Composting Council: 1993, Compost Facility Operating Guide. VA, USA: Alexandria.
- Tisdale, S. L. and Nelson, W. L.: 1982, Fertilidad de los suelos y fertilizantes. Mexico City. Mexico.
- Tomsett, A. B. and Thurman, D. A.: 1988, 'Molecular biology of metal tolerances of plants', *Plant Cell Environ.* **11**, 383–394.
- Thorne, M. E., Zamora, B. A. and Kennedy, A. C.: 1998, 'Sewage sludge and mycorrhizal effects on secar bluebunch wheatgrass in mine spoil', *J. Environ. Qual.* 27, 1228–1233.
- Vaca Paulin, R.: 1999, Especiación de metales pesados en suelos y sedimentos de la cuenca alta y media del río Lerma. Tesis de Maestría. Facultad de Ciencias, División de Estudios de Posgrado. Universidad Nacional Autónoma de México (UNAM). México D.F.
- Van Loon, J. C.: 1998, Selected methods of trace metal analysis. Biological and environmental samples. Vol 18 Department of Geology and Chemistry and the Institute of Environmental Studies. University of Toronto. Wiley Science Publication. Toronto, Canada.
- Vangronsveld, J. and Clijsters, H.: 1994, Plants and the chemical elements (Biochemistry, uptake, tolerance and Toxicity) VCH Weinheim.
- Zeiger, E. and Taiz, L.: 1998, *Plant Physiology*, Sinauer Associates, Inc Publisher 2<sup>a</sup> Ed. Massachusetts.
- Zhu, B. and Alva, A. K.: 1993, 'Differential adsorption of trace metals by soils as influenced by exchangeable cations and ionic strength', *Soil Sci* 155, 61–66.