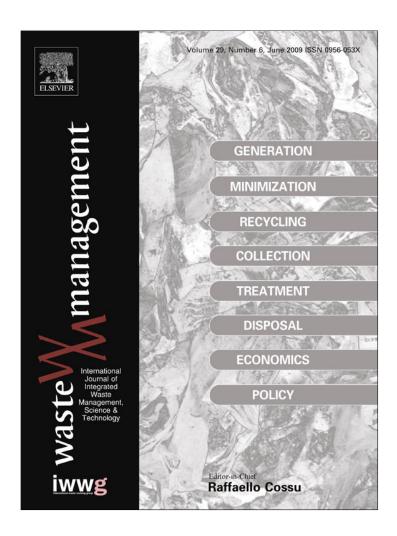
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# Nitrate and phosphate leaching in a Phaeozem soil treated with biosolids, composted biosolids and inorganic fertilizers

M.V. Esteller a,\*, H. Martínez-Valdés a, S. Garrido b, Q. Uribe a

- <sup>a</sup> Centro Interamericano de Recursos del Agua, Facultad de Ingeniería, Universidad Autónoma del Estado de México, Cerro Coatepec S/N Toluca, Estado de México, 50130 México, Mexico
- b Instituto Mexicano de Tecnología del Agua, Paseo Cuauhnáhuac 8532 Progreso, liutepec, Morelos, 62550 Mexico, Mexico

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#### ABSTRACT

The use of organic wastes in agriculture may increase the production of crops by incorporating organic matter and nutrients into the soil, and by improving its physical characteristics; however, this use may cause environmental problems such as the leaching of certain ions. The objective of this study was to establish possible nitrogen and phosphorus leaching under real field conditions in Phaeozem soils. The experimental work was performed in a corn (*Zea mays* L.) field where three plots were conditioned with inorganic fertilizer, three plots with 4.5 Mg ha<sup>-1</sup> of biosolids on dry basis, and three plots with the same amount of composted biosolids. The quality of biosolids and composted biosolids complied with the Mexican Official Standards. Soil water samples were collected with suction cups during two agricultural cycles and were analysed. Soil samples were also taken and analysed.

The N-NO $_3$  concentrations in soil water fluctuated between 0.9 and 98 mg L $^{-1}$  in the composted biosolid treatment, between 0.7 and 64 mg L $^{-1}$  in the biosolid treatment, and between 1 and 61 mg L $^{-1}$  in the inorganic fertilizer treatment. The maximum concentration of N-NO $_2$  and N-NH $_3$  in soil water was 1.02 and 2.65 mg L $^{-1}$ , respectively. The greatest percentage of nitrogen leached is produced when inorganic fertilizer is used (37.4% and 24.0% N leached in the first and second years, respectively), followed by composted biosolids (17.1% and 13.5% N leached in the first and second years, respectively) and last by biosolids (11% for both years). This difference could be related to the form in which nitrogen is present in the fertilizers, while commercial fertilizer is as inorganic nitrogen, organic wastes are basically presented as organic nitrogen. The maximum PO $_4^{3-}$  concentration in soil water was 1.9 mg L $^{-1}$  in the composted biosolid treatment, 1.7 mg L $^{-1}$  in the biosolid treatment and 0.9 mg L $^{-1}$  in the inorganic fertilizer treatment. The estimated percentage of leached phosphorus was less than 1% for all treatments. The minimum leaching that occurred seemed to be due to a sorption–precipitation process.

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#### 1. Introduction

Biosolids and composts provide plants with nutrients such as N, P and K and trace nutrients; and they can also improve the physical, chemical and microbiological characteristics of the soil (Korentajer, 1991; Epstein, 2003; Garrido et al., 2005). However, it may cause environmental problems (Epstein, 2003; Korboulewsky et al., 2002). Studies have been conducted on soils amended with biosolids and/or compost to study the behaviour of contaminants such as nitrogen, phosphorus and heavy metals (Elliot et al., 2002a; Vogeler et al., 2006; Vaca et al., 2006; Brenton et al., 2007). These contaminants can leach toward groundwater causing its contamination. Also, the contaminants that are fixed in the soil

particles may cause contamination of surface waters through runoff (Tian et al., 2006).

Nitrogen leaching is influenced by several factors such as rainfall pattern, irrigation, soil texture and type and rates of fertilizer (Carter, 1997). In a study conducted by Maeda et al. (2003), where the nitrate leaching was studied in an Andisol treated with four N fertilizers (swine compost, coated urea, ammonium or no fertilizers) for seven years, it was observed that excessive N from chemical fertilizers applied to soil can cause substantial nitrate leaching, while compost application is promising to establish high yield and low nitrate leaching for a few years, but would cause the same level of nitrate leaching as in chemical fertilizer plots over longer periods.

On the other hand, the application of biosolids at rates based on crop nitrogen requirements usually has phosphorous in excess for crop needs (Elliot et al., 2002a). This excess of phosphorus in the soil is not harmful to the plants; however, its transference to surface water systems is a serious problem. Unlike nitrogen,

<sup>\*</sup> Corresponding author. Tel.: +53 722 2965550; fax: +52 722 2965551.

E-mail addresses: mvestellera@uaemex.mx, vicenta13@prodigy.net.mx (M.V. Fsteller)

phosphorus leaching has not been traditionally considered a problem for groundwater (Siddique et al., 2000). The leaching of phosphorus will depend mainly on the fertilizer and soil characteristics (mainly its texture and sorption capacity), and so phosphorus fixation can be very high, or it can descend through the soil; as Siddique et al. (2000) found in a study about the leaching potential of P (97% of total P leached was dissolved reactive P) between a range of loam textured topsoils with different native soil P statuses, following treatment with dewatered, biological sewage sludge or inorganic P fertilizer.

Lu and O'Connor (2001) studied the effect of applying biosolids to agricultural soils, showing that the content of phosphorus in the leachate was practically zero, whereas other studies (Eghball et al., 1996) have established that the descending movement of phosphorus through the soil is significant, especially in soils with thick textures and low phosphorus sorption capacity. Elliot et al. (2002a) conducted laboratory and greenhouse column studies to characterize the phosphorus leaching and the forms of leached phosphorus, following the application of biosolids, compost and inorganic fertilizers on sandy soil. Bahiagrass was cultivated in soils with low phosphorus content and low phosphorus sorption capacity in the columns. The leached phosphorus was predominantly inorganic, and was in lower concentrations for biosolids than for inorganic fertilizers. The percentage of applied P leached varied from 1.7% to 21.7% in the inorganic fertilizer treatment. The presence of phosphorus in the leachate from biosolids- and compost-amended soils was lower than 1% of the applied phosphorus.

This diversity in results raises the need to continue carrying out research that will study the environmental impacts of the use of biosolids and compost in agricultural soils, considering that there is little information available with field study data. The objective of this study was to establish possible nitrogen and phosphorus leaching under real field conditions in a Phaeozem soil when biosolids, composted biosolids and inorganic fertilizers are applied to a corn crop.

# 2. Materials and methods

# 2.1. Experimental site

The study site is located in an agricultural area in the Municipality of Toluca, in the State of Mexico, nearby Mexico City, at tropical latitudes (latitude 19°23′57″ and longitude 99°42′47″). The average monthly temperature in the area fluctuates around 20 °C. The rainy season begins in the second half of May, and lasts until the second half of November. Mean annual rainfall is 700 mm.

The soil was characterized as clay loam Haplic Phaeozem soil (CETENAL, 1976). It has high porosity, which facilitates the penetration of plant roots, and has favourable conditions for water filtration. This soil type is the fourth most abundant in Mexico, and it is where a great part of the corn crops of the country are located.

The study area had corn ( $\it Zea\ mays\ L.$ ) in the field. In this area, nine plots of  $6\times 8$  m (separated by 1 m) were established, distributed under a Latin square design. Biosolids were applied to three of the plots, composted biosolids were applied to other three plots, and inorganic fertilizer was used in the remaining three plots. The period of the study covered two agricultural cycles (first cycle in 2002 and second cycle in 2003), each agricultural cycle coinciding with the rainy season (May–November).

The biosolids, composted biosolids and inorganic fertilizers were manually incorporated into the soil (15 cm depth). In this study, the traditional agricultural practices of the region for this type of crop were conducted. The application rate of biosolids was 4.5 Mg ha $^{-1}$  (dry basis), the same as that of composted biosolids, according to the recommendations by Gomez (1998), who

established the optimum rate in relation to content of heavy metals in organic waste in a way that there is no toxic accumulation of these metals in the soil. Only one application was made, and it took place a month before crop cultivation. The biosolids application added 192.0 and 222.8 kg N ha<sup>-1</sup> year<sup>-1</sup> and 23 and 38.4 kg P ha<sup>-1</sup> year<sup>-1</sup> for the first and second agricultural cycles, respectively. The composted biosolids amendment added 229.0 and 307.3 kg N ha<sup>-1</sup> year<sup>-1</sup> and 22.0 and 22.4 kg P ha<sup>-1</sup> year<sup>-1</sup> for the first and second agricultural cycles, respectively. The amount of N and P added changed for biosolids and composted biosolids between the years because the organic waste varied with time.

Local inorganic fertilizer was used in three applications according to the rate that is usually used in this agricultural area: the first application was in April, the second was in May and the third was in June, with a content of urea, granulate triple superphosphate and potassium chloride in a 60-90-60 formulation. The fertilizer was applied at rates of  $120 \text{ kg N ha}^{-1}$  year<sup>-1</sup> and  $60 \text{ kg P ha}^{-1}$  year<sup>-1</sup> for both the agricultural cycles.

Porous ceramic suction cup samplers (Model 1900 Soil Water Samples Soilmoisture Equipment Corp., CA, USA) were used for the collection of soil water samples at different depths. These samplers were formed by a PVC tube with one of its ends sealed by a porous ceramic cup, made with pure silicate. The other end was closed with a plug that had a hole through which Teflon tubes were inserted in order to create a vacuum and take a sample of soil water. This equipment was used because of its easy and comfortable handling and because of its non-destructive nature (Wilson et al., 1995; Romero et al., 2002; Maeda et al., 2003). The samplers were installed before application of soil amendments by manual soil drilling at depths of 15, 30, 60, 90, 120 and 150 cm. The total number of installed samplers was 36, placing in each treatment two samplers at each depth (six samplers were installed in two plots of each treatment).

The use of these samplers had the inconvenience of causing changes in the water chemical composition; to avoid this, the collectors were submitted to previous treatment with 1 N hydrochloric acid and distilled water, allowing to remove certain impurities and release some ions from the cup's walls. Also, a study was conducted on phosphorus retention by the cups, because authors (Wilson et al., 1995) have previously reported the fixation of this element through sorption processes in the material from which these cups are made. In this case, this phenomenon did not take place (Romero et al., 2002).

Tensiometers (Model 2725 Jet Fill Soilmoisture Equipment Corp., CA, USA) were installed by manual drilling in the remaining plots. These tensiometers were calibrated and, started at installation, were checked daily. The total number of tensiometers installed was 18, distributed as follows: six in a plot amended with biosolids, six in a plot where composted biosolids were applied, and the remaining six in a plot conditioned with inorganic fertilizer, with installation at depths of 15, 30, 60, 90, 120 and 150 cm.

# 2.2. Sampling

# 2.2.1. Biosolids and compost

Biosolids were obtained from the municipal wastewater treatment plant "Toluca Oriente", which is a conventional activated sludge plant that produces between 60 and 90 Mg of dry weight biosolids daily. The treatment of sludge obtained from the clarification (sedimentation) process is based on the addition of ferric chloride; then it is carried to a belt thickener, and from there to an aerobic digester, and a partially stabilized sludge is obtained (the organic matter is stabilized at 60–70% as volatile organic matter). The stabilized sludge is supplemented with aluminium sulphate, and is finally pressed in a filter-press, obtaining biosolids as the end product. The sampling of biosolids was performed according

to the EPA methodology (1988), taking individual samples of biosolids of approximately 3 kg during seven days of wastewater plant operation. The individual samples were mixed in order to form a composite sample.

The compost process was carried out in cone-shaped aerated static piles, with 1.5 m of diameter. The conditioning materials were corn straw as a carbon source and tire chips as a bulking agent (Garrido et al., 2005). In these piles, a C/N 30–40 ratio was established. Aeration was conducted through natural ventilation and by turning the piles over every day. The piles were protected with a thin layer of sawdust and corn straw in order to avoid bad smells, the influence of wind, etc. Composting period was 8 weeks. The sampling of composted biosolids was performed according to the EPA methodology (1988), in order to form a composite sample.

The biosolids and composted biosolids samples were placed in polythene bags and were air dried in the shade. Later, they were quartered, finely ground, passed through a 2 mm stainless steel sieve and stored in a refrigerator (4  $^{\circ}\text{C})$  until the analysis was complete.

#### 2.2.2. Soil

Soil samples were obtained using a helicoidal drill. Samples were taken at different depths (15, 30, 60, 90, 120 and 150 cm) in each of the plots, obtaining a composite sample for each depth from five samples. Sampling was conducted before the soil amendment at the first agricultural cycle in order to evaluate the initial conditions of the soil. These samples were kept in polythene bags and stored in a refrigerator (4 °C) until they were analysed.

#### 2.2.3. Soil water

Soil water sampling was conducted after applying vacuum, with a pressure vacuum hand pump, to the interior of the cup samples within three days of each rainfall event. The amount of time between sampling was determined according to rainfall pattern, although it was possible to obtain samples during nearly all the months of the rainy season (six samplings in 2002 and five samplings in 2003).

# 2.3. Chemical, physical and microbiological analyses

#### 2.3.1. Biosolids, composted biosolids and soil

Analyses were conducted in triplicate by determining the following parameters: moisture, pH at 1:2.5 soil:distilled water ratio (McLean, 1982), electrical conductivity (Jackson, 1982), organic matter was measured by the modified method of Walkley and Black's (Jackson, 1982), cation exchange capacity (Chapman, 1991), total nitrogen was measured with the digestion – distillation method of Kjeldahl (Bremner, 1996), available phosphorus with the method of Olsen (Jackson, 1982), exchangeable cations (Ca, Mg, Na and K) with a standard method (Jackson, 1982), and total heavy metals (Cu, Cd, Ni, Pb and Zn) by the EPA 3050 method (1988). For the soil samples, texture was also determined by the Bouyoucos method.

Microorganisms (total and faecal coliforms) were determined in biosolids and composted biosolids by the method of fermentation in multiple tubes (MPN method) (APHA et al., 1995).

## 2.3.2. Soil water

The water samples that were extracted from the sample collectors were emptied into plastic flasks, identified, and covered adequately for later storage in an icebox for transportation to the laboratory, where they were kept until conducting the appropriate analyses.

The description of the analytical methods that were used to determine nitrates ( $4500\text{-NO}_3$  – E Cadmium reduction method), nitrites ( $4500\text{-NO}_3$  – B Colorimetry method), ammoniacal nitrogen

(4500-NH<sub>3</sub> – C Titrimetric method), and phosphates (4500-P Stannous chloride method) by UV–vis spectrophotometry, can be referred in APHA et al. (1995).

It must be mentioned that in some samples the types of nitrogen and phosphorus were not determined because the amount of water sample obtained was insufficient to conduct all the required analytical determinations.

#### 2.4. Estimation of N and P in leachate

In order to estimate the amount of leached nitrogen and phosphorus, a water balance was conducted to estimate the volume of water that infiltrated every month. The water balance was conducted applying the following equations (Quiñónes, 1997):

$$V_{\rm i} = I_{\rm p} * A$$

with

$$\begin{split} I_{p} &= P_{p} - E_{tr} \\ E_{tr} &= K_{c} * K_{s} * E_{To} \\ E_{To} &= K_{t} * E \nu_{t} \\ K_{s} &= \frac{1}{1 + \frac{\left[K_{c} E_{To}}{\left(K_{b}(0)\right)}\right] \left[\frac{1 - H_{r}}{H_{r}}\right]} \end{split}$$

where  $P_{\rm p}$  is the total monthly rainfall in the study area (mm month<sup>-1</sup>),  $E_{\rm vt}$  is the total monthly evaporation in the study site (mm month<sup>-1</sup>),  $H_{\rm r}$  is the average monthly soil residual moisture,  $K_{\rm b}(\theta)$  is the soil hydraulic conductivity in relation to moisture (cm s<sup>-1</sup>),  $K_{\rm c}$  is the corn crop coefficient of growth,  $K_{\rm t}$  is the coefficient of tank, where evaporation recorded in the study site is measured,  $E_{\rm To}$  is the reference evapotranspiration (mm),  $K_{\rm s}$  is the soil coefficient (month<sup>-1</sup>),  $E_{\rm tr}$  is the real evapotranspiration (mm month<sup>-1</sup>),  $I_{\rm p}$  is the infiltrated water (mm month<sup>-1</sup>), A is the cultivated area (1 hectare) and  $V_{\rm i}$  is the volume of infiltrated water (L month<sup>-1</sup>).

The values of precipitation ( $P_p$ ) and evaporation ( $E_{vt}$ ) were obtained from a weather station located 200 m from the study area. The value of average monthly residual moisture ( $H_r$ ) was obtained from the tensiometers installed and from the characteristic curve of the soil (Martinez-Valdes, 2005). Readings were taken from the tensiometers installed at 60 cm depth, a depth chosen based on the root length. The growth coefficient of the corn crop ( $K_c$ ) and the coefficient of the evaporation tank ( $K_t$  = 0.8) were estimated from the graphs and tables exhibited by Quiñónes (1997). It should be noted that the use of the  $K_c$  parameter (Allen et al., 1998) can be questionable for non-irrigated crops because  $K_c$  parameter techniques tend to overestimate evapotranspiration (Mata-Gonzalez et al., 2005).

Reference (or saturated) hydraulic conductivity shows an average value of  $9 \times 10^{-4} \, \mathrm{cm \, s^{-1}}$ , which was determined using a Guelph permeameter. The value of the reference hydraulic conductivity was corrected according to the monthly average moisture content ( $H_{\rm r}$ ) of the soil at 60 cm depth. The values of hydraulic conductivity according to the degree of moisture ( $K_{\rm b}(\theta)$ ) were estimated with the equation of Clapp and Hornberger (1978) (in Lawrence (1994)):

$$K_{\rm b}(\theta) = K_{\rm hsat}(\phi^{-c})(\theta^{c})$$

where

$$c = 2b + 3$$

where  $K_{\rm b}(\theta)$  is the corrected hydraulic conductivity;  $K_{\rm hsat}$  is the reference (or saturated) hydraulic conductivity; b is the empirical parameter;  $\theta$  is the moisture (60 cm depth); and  $\phi$  is the soil porosity.It is worth mentioning that runoff was not considered, since it

**Table 1** Physicochemical characteristics of biosolids and composted biosolids (n = 3, all  $kg^{-1}$  data are expressed on a dry basis).

	Biosolids	Composted biosolids	Biosolids	Composted biosolids	Mexican official standards
	Year 1	Year 1	Year 2	Year 2	
Moisture content, %	82.9	7.0	84.5	11.4	
EC, mS cm <sup>-1</sup>	5.2	8.3	5.4	8.9	
pH (H <sub>2</sub> O)	6.40	7.00	6.55	6.96	
pH (KCl)	6.10	6.90	6.38	6.62	
OM, %	61.64	59.66	67.61	64.90	
C/N	8.4	6.9	5.4	3.8	
Kjeldahl – N, mg kg <sup>-1</sup>	42,624	50,788	49,490	68,300	
Olsen – P, mg kg <sup>-1</sup>	5192	4945	8662	5053	
CEC, cmol kg <sup>-1</sup>	39	41	40	40	
Na <sup>+</sup> , mg kg <sup>-1</sup>	460.0	713.0	1702.0	2162.0	
$K^+$ , mg kg <sup>-1</sup>	5183.1	4984.2	5928.0	9243.0	
$Ca^{2+}$ , mg kg <sup>-1</sup>	4268.0	3412.0	4736.0	4256.0	
$\mathrm{Mg}^{2+}$ , $\mathrm{mg}\ \mathrm{kg}^{-1}$	3948.8	1091.1	1482.0	2330.0	
Cu, mg kg <sup>-1</sup>	352.0	382.0	266.0	249.0	1500
Cd, mg kg <sup>-1</sup>	32.0	nd	3.6	2.8	39
Ni, mg kg <sup>-1</sup>	13.3	13.5	38.1	33.7	420
Pb, mg kg <sup>-1</sup>	78.7	112.4	61.5	56.3	300
Zn, mg kg <sup>-1</sup>	809.0	761.0	758.0	782.0	2800

nd: not detected, EC: electrical conductivity, OM: organic matter, and CEC: cation exchange capacity.

was a levelled agricultural plot where this phenomenon did not take place.

Once the monthly volume of the infiltrated water and the monthly averages of nitrogen concentrations in the form of nitrates and nitrites in the soil water were estimated, the leached nitrogen was quantified by assessing the product of the volume of water infiltrated by the average nitrogen concentration. The means of nitrogen concentrations in each monthly sampling correspond to nitrates and nitrites concentrations in the samples collected at 90, 120 and 150 cm depths, since it was considered that plant roots are not present at depths greater than 90 cm, and therefore they do not absorb the nitrogen, and so it tends to leach. The same assessments were conducted to estimate the amount of leached phosphorus.

# 2.5. Data analysis

An analysis of means using the Student *t*-test at 0.05 significance level and an analysis of variance (ANOVA) using the *F*-test at 0.05 significance level were carried out (Kanjin, 1999) to establish the possible significant differences between the mean values of leached N and P among the different treatments and agricultural cycles.

#### 3. Results and discussion

## 3.1. Biosolids and composted biosolids characteristics

Table 1 lists the characteristics of biosolids and composted biosolids for the two study agricultural cycles. When comparing physical and chemical properties, it is possible to observe that the biosolids tend to be slightly acid, whereas after they are submitted to a composting process, they tend to be neutral. Also, it is observed that electrical conductivity increases after the composting process; this is because, during the mineralization process, the organic complexes break down forming simpler inorganic compounds (Carmona et al., 1994).

The content of total heavy metals in both biosolids and composted biosolids complies with current Mexican Official Standards (NOM-004-SEMARNAT, 2002) for the application of organic wastes (sewage sludge and biosolids) in agricultural soils.

Total and faecal coliforms were  $2.4\times10^8$  and  $9.3\times10^7$  MPN g  $^{-1}$  (dry basis) in biosolids, whereas in the composted biosolids,

total and faecal coliforms were 1500 MPN  $\rm g^{-1}$  and 30 MPN  $\rm g^{-1}$ , which represents a 99.9% decrease regarding the biosolids. This decrease is an effect of the compost process. The values of faecal coliforms comply with the Mexican Official Standards (NOM-004-SEMARNAT, 2002), and these organic wastes were classified as excellent amendments for agricultural use.

#### 3.2. Soil characteristics

Table 2 lists the results of the analysis of soil physicochemical characteristics. From this table it is possible to observe that the soil texture in the shallowest levels (15 and 30 cm) is sandy clay, whereas at greater depths it is sandy loam. In the shallowest levels (15–30 cm), acidic pH is observed, which could facilitate nutrient and heavy metal mobility (Bourg, 1995). The percentage of organic matter and the nitrogen and phosphorus content exhibit maximum values in the shallowest level of the soil, which decrease gradually as depth increase.

### 3.3. Soil water characteristics

N–NH $_3$  concentrations determined in the soil water samples that were collected in the different treatments are listed in Table 3. The values correspond to the mean of the two water samples obtained at each depth in every treatment. The concentrations of ammonia forms were nearly always less than 3 mg N–NH $_3$  L $^{-1}$ , with no significant variations between the different treatments.

 $N-NO_2$  concentrations in the soil water samples in any of the treatments were almost always below 1 mg  $N-NO_2$   $L^{-1}$  throughout the entire soil profile, obtaining minimum values (less than 0.1 mg  $N-NO_2$   $L^{-1}$ ) especially over 120 cm of depth.

The low concentrations of N–NH<sub>3</sub> and N–NO<sub>2</sub> in the shallowest levels of soil can be explained due to the oxidation of these nitrogenous species (Carter, 1997). Also, the exchange process that ammonium can undergo (Wong et al., 2000) can be quite important due to the high CEC (12–17 cmol kg<sup>-1</sup>) that this soil has in the shallowest levels.

The behaviour of N–NO<sub>3</sub> during the study period in the soil water samples is shown in Fig. 1. At the beginning of the first agricultural cycle in June, low nitrate concentrations, with contents lower than 10 mg N–NO<sub>3</sub> L<sup>-1</sup>, were seen in all treatments at 15–30 cm depth, whereas in the month of July, N–NO<sub>3</sub> concentrations tended to increase in the different soil profiles of each treatment,

**Table 2** Physicochemical characteristics of soil (n = 3, SD = standard deviation).

	Depth (cm)	Depth (cm)						SD
	15	30	60	90	120	150		
Sand, %	58.4	54.4	46.4	66.4	66.4	66.4	59.7	7.5
Silt, %	16.0	18.0	46.0	14.0	16.0	19.8	21.6	11.0
Clay, %	25.6	27.6	7.6	19.6	17.6	13.8	18.6	6.8
Texture	SCL	SCL	SL	SL	SL	SL		
EC, μS cm <sup>-1</sup>	35.8	39.8	43.2	58.5	67.1	32.8	46.2	12.4
pH (H <sub>2</sub> O)	5.8	5.80	6.40	7.40	7.20	7.30	6.65	0.68
pH (KCl)	5.2	5.20	5.70	6.80	6.70	6.70	6.05	0.70
OM, %	2.0	1.6	0.2	0.1	0.4	0.1	0.7	0.7
Kjeldahl – N, mg kg <sup>-1</sup>	1100	800	600	550	700	400	692	221
Olsen – P, mg kg <sup>-1</sup>	29.0	44.9	48.2	11.1	37.6	9.7	30.1	15.2
CEC, cmol kg <sup>-1</sup>	13	12	17	15	11	9	12	3
Na <sup>+</sup> , mg kg <sup>-1</sup>	58	37	124	166	145	103	106	46
$K^+$ , mg kg <sup>-1</sup>	783	702	643	433	741	659	659	113
Ca <sup>2+</sup> , mg kg <sup>-1</sup>	2344	2548	3548	3652	2248	1508	2648	760
$\mathrm{Mg}^{2+}$ , $\mathrm{mg}~\mathrm{kg}^{-1}$	807	1200	1543	1708	863	872	1166	352

SCL: sandy clay loam; SL: sandy loam; EC: electrical conductivity; OM: organic matter; and CEC: cation exchange capacity.

**Table 3**Mean N-NH<sub>3</sub> concentrations (mg L<sup>-1</sup>) in soil water samples in the three fertilizer treatments during two agricultural cycles (year 1, n = 12; year 2, n = 10; ±SD standard deviation).

Treatment	Year	15 cm	30 cm	60 cm	120 cm	150 cm	180 cm
IF	1 2	1.08 ± 0.55 1.06 ± 1.17	1.24 ± 0.93 1.59 ± 1.70	1.44 ± 1.29 1.34 ± 0.51	0.93 ± 0.89 1.23 ± 0.90	0.12 ± 0.25 2.70 ± 0.65	0.88 ± 0.76 1.17 ± 1.13
В	1 2	1.29 ± 1.05 2.07 ± 1.42	1.05 ± 0.57 1.25 ± 1.74	1.70 ± 1.47 2.71 ± 1.64	1.25 ± 0.87 2.48 ± 1.66	0.94 ± 0.93 2.65 ± 1.30	1.91 ± 2.68 1.10 ± 0.62
СВ	1 2	0.56 ± 0.20 0.003 ± 0.001	0.02 ± 0.01 1.46 ± 1.50	0.11 ± 0.13 2.31 ± 2.07	2.10 ± 2.04 1.35 ± 1.05	0.93 ± 0.87 1.17 ± 1.30	1.96 ± 1.94 1.26 ± 0.71

IF: inorganic fertilizer, B: biosolids, and CB: composted biosolids.

with concentrations that reach 50 mg N–NO<sub>3</sub> L<sup>-1</sup> in the inorganic fertilizer treatment at 120 cm depth (Fig. 1a), and 100 mg N–NO<sub>3</sub> L<sup>-1</sup> in biosolids and composted biosolids treatments at 60–90 cm depth (Fig. 1b and c). The highest values that were detected in organic wastes treatments could be a consequence of the greater addition of N that the use of these organic wastes involved (from 192 to 307 kg N ha<sup>-1</sup> year<sup>-1</sup>), and to a lesser extent could be due to the nitrification of ammonium and nitrite (Maeda et al., 2003).

Subsequently (August–September), the concentration of nitrates decreased in all the treatments and at all depths, reaching another maximum in the month of October, with a concentration of 60 mg N–NO $_3$  L $^{-1}$  in inorganic fertilizer treatment at 30 cm depth (Fig. 1a) and 20 mg N–NO $_3$  L $^{-1}$  in biosolids and composted biosolids treatments, at 90–120 cm depth (Fig. 1b and c). At the end of the first agricultural cycle (November), the N–NO $_3$  concentration reached minimum values (0.5 mg N–NO $_3$  L $^{-1}$ ) at the different soil profiles of each treatment.

The second agricultural cycle exhibited a different behaviour, because in the first sampling, after the first rain, elevated concentrations of nitrate were obtained, with values higher than 35 mg N-NO<sub>3</sub> L<sup>-1</sup> in the inorganic fertilizer treatment (Fig. 1a), and 60 mg N-NO<sub>3</sub> L<sup>-1</sup> in biosolids and composted biosolids treatment at 15-30 cm depth. These concentrations were detected at up to 150 cm depths (Fig. 1b and c). This elevated concentration of N-NO<sub>3</sub> could be a consequence of the large portion of N from organics wastes which first accumulated in soil in the organic form, which could have been mineralized and thereafter leached (Wong et al., 2000; Maeda et al., 2003; Vieira et al., 2005). It is also important to consider that the organic waste treatments exhibited the highest concentrations of N-NO<sub>3</sub> in the shallowest level of the soil (15-30 cm depth) during the first agricultural cycle (Fig. 1b and c). Likewise, nitrogen added during the second application of fertilizers must be taken into account.

After reaching this maximum, the concentrations decreased, and increased again at the end of the second agricultural cycle (November), though with values lower than  $40 \text{ mg N-NO}_3 \text{ L}^{-1}$ , with the lowest concentrations in the composted biosolids treatment (Fig. 1b).

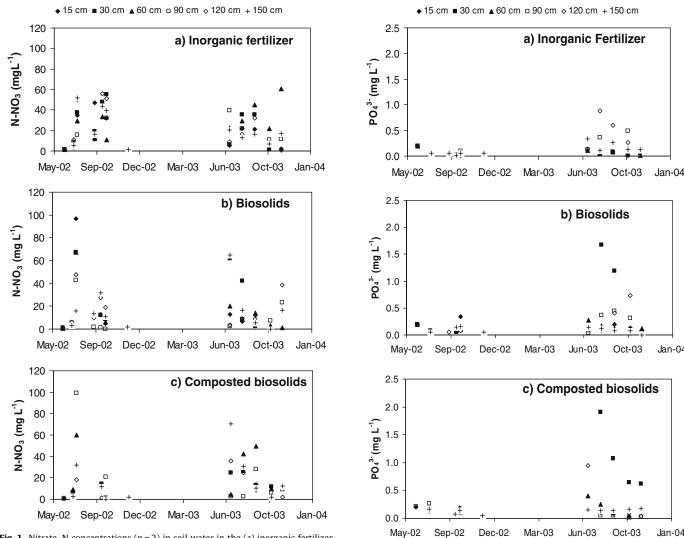
The evolution of the content of phosphorus in the soil water in the different treatments is shown in Fig. 2. In this figure, it is possible to observe the different behaviour between the first and second agricultural cycles. In the first agricultural cycle, the presence of phosphates in the soil water is minimum, with concentrations that rarely exceed 0.4 mg  $PO_4^{3-}$  L<sup>-1</sup> in all the treatments, with no difference between the treatments despite the fact that the amount of added phosphorus is different. In the second agricultural cycle, similar amounts of phosphorus were added, except for biosolids where a greater amount of phosphorus was applied (38 kg P ha<sup>-1</sup>). In this second cycle, it is possible to observe a minimum leaching at the beginning of the cycle (August) in the shallowest levels of the soil, with a maximum concentration of  $1\;mg\;PO_4^{3-}\;L^{-1}$  in the inorganic fertilizer treatment (Fig. 2a) and  $1.6\mbox{ mg}\mbox{ PO}_4^{3-}\mbox{ }L^{-1}$  in the organic wastes treatment (Fig. 2b and c). Subsequently, a decrease in the concentrations in all the treatments was produced at the end of the cycle; with phosphorus further decreasing at greater depths. This decrease in the concentration of phosphorus could be the result of sorption and calcium phosphate precipitation at these pH conditions, and slow dissolution of the phosphate fertilizer after their application (Romero et al., 2006).

# 3.4. N and P concentrations in leachate

The impact of varying nutrient applications on leaching rates can be difficult to be discerned when plants show varying demands for nutrients. Previously published work on these same experi-

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**Fig. 1.** Nitrate–N concentrations (n = 2) in soil water in the (a) inorganic fertilizer, (b) biosolids and (c) composted biosolids treatments.

**Fig. 2.**  $PO_4^{3-}$  concentrations (n=2) in soil water in the (a) inorganic fertilizer, (b) biosolids and (c) composted biosolids treatments.

ments (Martinez-Gutierrez, 2004) concluded that there were no statistically significant differences ( $\alpha > 0.05$ ) in P and N plant uptake rates in relation to the type of treatment at this study site, thus the differences in the leached N and P would not be greatly affected by variations in plant uptake. Table 4 shows that the nitrogen uptake fluctuated between 1.40% in the inorganic fertilizer treatment and between 1.73% in the biosolid treatment, and the phosphorus uptake fluctuated between 0.33% in the inorganic fertilizer treatment and between 0.37% in the composted biosolid treatment.

Water inflow takes place only through rainfall, while outflow occurs through evapotranspiration, which includes direct evaporation and root absorption. The difference between water inflows and outflows constitutes the volume of infiltrated water (Table 5). Annual inflows were 563 and 475 mm for the first and second agricultural cycles, respectively; these values are below average and so these can be considered dry years. The estimated amount of water infiltrated during the first agricultural cycle (282 mm) was twice that during the second agricultural cycle (141 mm), even though the difference in rainfall was not that high. These data could be explained by the higher estimated evapotranspiration that took place in the second agricultural cycle (333 mm). Once the volume of monthly infiltrated water was assessed, it was multiplied by the average concentration of N and P, corresponding to each month for each agricultural cycles (Table 6), and this allowed to estimate the leached N and P (Tables 7 and 8).

The greatest estimated percentage of N leached was produced when inorganic fertilizer was used, followed by composted biosolids and, last by biosolids (Table 7). This difference could be related to the form in which nitrogen is present in the fertilizers, while commercial fertilizer is in the form of inorganic nitrogen, in organic wastes it is basically presented as organic nitrogen, and so firstly its mineralization occurs, and later it can be transformed into its nitrogenous forms (Vieira et al., 2005; Maeda et al., 2003).

The estimated percentage of leached phosphorus was less than 1% for all the treatments (Table 8), values similar to those obtained by Elliot et al. (2002a) in a study with laboratory and greenhouse columns. There was a slightly greater leaching when organic wastes were used, despite the fact that the amount of phosphorus added to these organic waste was fairly minor (22–38 kg P ha<sup>-1</sup>) to that applied with inorganic fertilizer ( $60 \text{ kg P ha}^{-1}$ ). These data could contradict the point made by Elliot et al. (2002b). That study concluded that the treatment process with ferric chloride and aluminium sulphate, to which sewage sludge was submitted, involves a decrease in phosphorus solubility and, therefore, less leaching.

The estimates of leached nitrogen, when applying F-test, indicated that there were no significant statistical differences in

**Table 4** Nitrogen and phosphorus uptake and yield (mean  $\pm$  standard deviation of n = 9).

	Inorganic fertilizer	Biosolids	Composted biosolids
Nitrogen uptake (%)	1.40 ± 0.40a	1.73 ± 0.07a	1.55 ± 0.20a
Phosphorus uptake (%)	0.33 ± 0.02a	0.35 ± 0.02a	0.37 ± 0.01a
Yield (ton ha <sup>-1</sup> )	4.09 ± 0.60a	5.22 ± 0.80 <sup>a</sup>	5.77 ± 0.06a

Same letters indicate no significant differences between treatments at  $\alpha$  > 0.05. Adapted from Martinez-Gutierrez (2004).

relation to the type of treatment ( $\alpha$  = 0.05). For the *F*-test, the six and five monthly estimates of leached nitrogen for first and second cycles, respectively, were used for each treatment. When the same

estimates were analysed by comparing the two agricultural cycles for the same treatment, applying Student t-test using six and five monthly estimates for first and second cycles, respectively, it was found that in the case of inorganic fertilizer treatment, the amount of leached N was statistically different between the two agricultural cycles with a 95% confidence interval ( $\alpha$  = 0.05). This difference may be caused by the lower volume of water infiltrated in the second year, which could lead to less leaching since the concentration of N (or any solute) in the leachate would be a function of the leaching fraction and water volume (Carter, 1997; Brenton et al., 2007). In the case of biosolids and composted biosolid treatments, it was possible to establish that there were no statistically significant differences ( $\alpha$  = 0.05).

**Table 5**Monthly water inputs and estimated leachate volumes during two agricultural cycles.

	June	July	August	September	October	November	Total
P <sub>p</sub> (mm) Year 1 Year 2	140.2 0.0	62.7 184.1	134.4 157.2	141.4 58.0	59.8 53.8	24.9 22.0	563.4 475.1
E <sub>vt</sub> (mm) Year 1 Year 2	104.44	58.64 133.49	52.99 113.94	140.10 54.88	75.05 88.68	72.64 64.49	503.86 455.48
H <sub>r</sub> (%) Year 1 Year 2	38.00	43.48 61.61	36.19 58.31	46.98 49.41	49.48 55.53	51.36 52.89	
$K_{\rm b}(\theta)~({ m cm~s^{-1}})$ Year 1 Year 2	$1.59 \times 10^{-4}$	$7.73 \times 10^{-4} \\ 8.36 \times 10^{-2}$	$7.95 \times 10^{-5} \\ 3.56 \times 10^{-2}$	$\begin{array}{c} 2.41 \times 10^{-3} \\ 4.43 \times 10^{-2} \end{array}$	$4.11 \times 10^{-3} \\ 1.80 \times 10^{-2}$	$6.87 \times 10^{-3}$ $1.12 \times 10^{-2}$	
Kc	0.5	0.8	1.1	0.9	0.55	0.42	
E <sub>To</sub> (mm) Year 1 Year 2	83.55	46.91 106.79	42.39 125.76	112.08 43.90	60.07 70.94	58.11 51.59	403.11 398.98
$K_{\rm s}$ (month <sup>-1</sup> )	0.98	0.99	0.96	0.99	0.99	0.99	
E <sub>tr</sub> (mm) Year 1 Year 2	41.10	37.42 85.35	44.76 138.2	100.67 49.34	33.00 38.98	24.38 21.64	281.33 333.51
V <sub>i</sub> (L) Year 1 Year 2	991 100	252,800 987,510	896,300 190,020	407,100 86,580	268,000 148,190	5000 3560	2,820,300 1,415,860

 $P_p$  is the total monthly rainfall (measured);  $E_{vt}$  is the total monthly evaporation (measured);  $H_r$  is the average monthly soil residual moisture (estimated);  $K_b(\theta)$  is the soil hydraulic conductivity in relation to moisture content (estimated);  $K_c$  is the corn crop coefficient (estimated);  $E_{tr}$  is the reference evapotranspiration (estimated);  $E_{tr}$  is the real evapotranspiration (estimated); and  $V_i$  is the volume of infiltrated water (estimated).

 $\textbf{Table 6} \\ \text{Mean nitrogen and phosphorous concentrations } (\text{mg L}^{-1}) \text{ in soil water at bottom soil during two agricultural cycles.}$ 

Treatment		June	July	August	September	October	November
IF	P						
	Year 1	0.022	0.006	0.016	0.014	0.019	0.016
	Year 2		0.060	0.147	0.102	0.093	0.033
	N						
	Year 1	0.6	47.5	14.6	48.5	40.9	1.6
	Year 2		23.0	17.3	21.1	7.3	9.7
В	P						
	Year 1	0.022	0.011	0.005	0.038	0.028	0.016
	Year 2		0.030	0.063	0.101	0.123	0.005
	Ν						
	Year 1	0.5	40.6	8.4	20.1	10.0	1.5
	Year 2		23.7	11.9	5.8	2.9	26.2
СВ	P						
	Year 1	0.023	0.059	nd	0.032	0.030	0.016
	Year 2		0.134	0.030	0.029	0.032	0.027
	Ν						
	Year 1	0.4	54.9	nd	9.3	7.4	1.6
	Year 2		36.3	19.5	15.6	3.8	7.8

IF: inorganic fertilizer; B: biosolids; CB: composted biosolids; and nd: not detected.

**Table 7**Estimated amount of N leached from soil receiving different types of fertilizer treatments.

Treatment	Year	N added (kg ha <sup>-1</sup> )	Estimated N leached (kg ha <sup>-1</sup> )	Estimated percent of N leached
IF	1	120.0	44.86Aa	37.38
	2	120.0	28.76Bb	24.00
В	1	192.0	21.94Aa	11.43
	2	222.8	26.65Ba	11.94
СВ	1	229.0	39.17Aa	17.10
	2	307.3	41.48Ba	13.50

IF: inorganic fertilizer, B: biosolids, and CB: composted biosolids.

Same capital letters within an agricultural cycle (year) indicate that there is no significant difference between the treatments at  $\alpha$  = 0.05. Same lowercase letters within the same treatment indicate that there were no significant differences between the agricultural cycles (year) at  $\alpha$  = 0.05.

**Table 8**Estimated amount of P leached from soil receiving different types of fertilizer treatments.

Treatment	Year	P added (kg ha <sup>-1</sup> )	Estimated P leached (kg ha <sup>-1</sup> )	Estimated percent of P leached
IF	1 2	60.0 60.0	0.05Aa 0.11Bb	0.08 0.18
В	1 2	23.0 38.4	0.05Aa 0.07Ba	0.23 0.18
CB	1 2	22.0 22.4	0.06Aa 0.14Bb	0.27 0.63

IF: inorganic fertilizer, B: biosolids, and CB: composted biosolids.

Same capital letters within an agricultural cycle (year) indicate that there was no significant difference between the treatments at  $\alpha$  = 0.05. Same lowercase letters within the same treatment indicate that there were no significant differences between the agricultural cycles (year) at  $\alpha$  = 0.05.

The same statistical study was applied to phosphorus. The results of the analysis helped to establish that there are no statistically significant differences between the different treatments for the same agricultural cycle. When the averages of leached P were compared between the agricultural cycles for the same treatment, it was possible to establish that there are no significant differences in the case of composted biosolids treatment, but there was a statistical difference for biosolids and inorganic fertilizer treatments, although with a 99% confidence interval ( $\alpha$  = 0.01), the amount of leached P was statistically the same.

# 3.5. Further work

It would be interesting to continue this study to observe the long-term effects and test, for example, soil retention capacity with regard to phosphorus, as well as the evolution of nitrate leaching at different agricultural cycles when organic wastes are added.

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