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Dairy industry sewage sludge as a fertilizer for an acid soil: a laboratory experiment with *Lolium multiflorum* L.

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Abstract

The aim of this study was to assess the efficacy of dairy industry sewage sludge as a fertilizer for an acid soil, and to evaluate the leaching of its nutrients and pollutants. The sludge was applied to pots containing an acid pasture soil (pH 4.4, organic matter 9.6%) in which *Lolium multiflorum* was then sown. Two different doses of sewage sludge (80 and 160 m^3 ha^{-1}) and a dose of 15:15:15 NPK (675 kg ha^{-1}) were compared. Changes in soil properties (leachate and soil fraction) and the production and nutritional status of the crop were monitored for nine weeks. The addition of the sludge led to substantial increases in crop production, which was attributed to improvements in crop nutrient concentrations (mainly N and P) and soil properties (pH, total N and available P). The sludge-treated soils lost less NO_3^-, Ca, Mg and K through leaching than those treated with the mineral fertilizer. The amounts of nutrient accumulated in the crop showed, however, that the input of K and to a lesser extent of Mg, were insufficient to cover requirements. Supplements of these elements are therefore necessary.

Key words: balance of nutrients, organic sludge, Italian ryegrass, waste management

Resumen

Evaluación de la eficacia de un lodo de depuradora procedente de industria láctea como fertilizante de un suelo ácido en un ensayo de laboratorio con *Lolium multiflorum* L.

En el presente trabajo se evaluó la eficacia fertilizante, así como la transferencia de nutrientes y contaminantes al medio hídrico de un lodo de depuradora de industria láctea. Se llevó a cabo a partir de un balance entre las cantidades de nutrientes asimiladas por el cultivo y las pérdidas por lavado. El experimento consistió en un ensayo en macetas que contenían un suelo ácido de pradera (pH 4,4 y materia orgánica 9,6%), que recibieron tres tipos de tratamiento: dos dosis de lodo de depuradora de una industria láctea (80 m^3 ha^{-1} y 160 m^3 ha^{-1}), una de fertilizante tipo NPK 15:15:15 (675 kg ha^{-1}) así como un control. Una vez realizados los tratamientos se sembraron con *Lolium multiflorum* L. Durante nueve semanas se analizó la evolución de las propiedades de los suelos (percolados y fracción sólida), así como de la producción y el estado nutricional del cultivo. El aporte del lodo derivó en incrementos substanciales de la producción, lo que se relaciona con el incremento de las concentraciones de nutrientes en el cultivo (N y P, fundamentalmente) y con la mejora en algunas propiedades de los suelos (incremento de pH, N total, P disponible). En comparación a la fertilización mineral, la fertilización con lodo dio lugar a menores pérdidas de NO_3^-, así como de Ca, Mg y K a través de los lixiviados. El análisis de las cantidades asimiladas por el cultivo indica que el aporte de K y Mg a través de la aplicación de lodo no fue suficiente para abastecer las necesidades de cultivo; en consecuencia estos elementos deberían ser suplementados adicionalmente.

Palabras clave: balance de nutrientes, lodos orgánicos, raigrás, gestión de residuos.

Introduction

The disposal of the different types of organic sludge produced from sewage has become one of the major problems of environmental management. The majority of developed countries are now looking into reusing these materials as fertilizers. In Spain, some 861,000 tonnes (dry weight) of sludge were produced from urban sewage in 2000, a figure that may rise to 1.5 million tonnes by 2005 (Rodríguez, 2002): production will no doubt increase in the rest of the EU as well. Such large quantities will be difficult to eliminate or store.

Studies undertaken in different areas show that adding these sludges to agricultural soils can improve

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soil structure, root penetration and nutrient levels (especially N and P) (Sommers, 1977; Khaleel *et al.,* 1981). However, some urban sewage sludges can have negative effects – a consequence of their relatively high content in heavy metals, salts, organic toxins and pathogens (Purves, 1985). Dairy industry sludges, however, have a low heavy metal content, and therefore the risk of large quantities of these pollutants entering the food chain is considerably reduced. In fact, experiments in which this type of sludge has been directly applied to the soil show this practice to be a safe alternative for its disposal (IDF, 2000).

In Galicia (NW Spain), where the dairy sector is particularly important, a programme for dealing with all the sludges produced by this industry is being developed. The aim is to analyse the processes of treatment, elimination and recycling of wastes in order to reach an equilibrium between costs and the need to meet legal requirements. A number of studies have been undertaken to establish the environmentally and agriculturally correct doses of sludge for use in mixed prairies (López Mosquera *et al.*, 2000 and 2002a) and forestry-pasture systems (Omil *et al.*, 2000).

Though the results of such studies have been positive, little is known about the efficacy with which the nutrients supplied by sewage sludge are used. Further, this type of waste contains relatively high amounts of N, and its consequent low C/N ratio could cause strong mobilisation of NO_3^-, a possibility that has not been sufficiently studied. The aim of the present study was to obtain data on the efficacy of dairy industry sewage sludge as a fertilizer, to optimise its use, and to determine the risk of mobilisation of elements towards the groundwater (especially inorganic N). These variables were studied by analysing the soil-plant nutrient balance.

Material and Methods

A nine-week trial, consisting of ryegrass growth assesment in soil pots to which different dairy sludge doses were added, was performed. The sludge used was a product of treating the waste water from a dairy industry in Galicia by the activated sludge method. The waste water from this industry contains discarded milk and cleaning solutions composed of water, sodium hydroxide and nitric acid. The sludge produced is characterised by its near-neutral pH, high N, Ca, P and Na contents, and a C/N ratio of 5.5 (Table 1). Its heavy metal concentration is very low–well below the limits established by the EU for sludges produced by urban sewage farms [EU Directive 86/278 CEE (OJ, 1986), the best reference in the absence of a more specific norm].

Table 1. Sludge composition (totals for dry sludge)

Composition	Total
Dry sludge (g L^{-1})	23.4
pH (H_2O)	6.8
N (%)	7.3
P (%)	2.7
Ca (%)	1.8
Mg (%)	0.3
Na (%)	2.8
K (%)	0.8
Cd (mg kg^{-1})	0.2
Cr (mg kg^{-1})	17.3
Cu (mg kg^{-1})	47.8
Hg (mg kg^{-1})	0.3
Ni (mg kg^{-1})	8.9
Pb (mg kg^{-1})	13.7
Zn (mg kg^{-1})	427.2

The soil used in this trial, which was taken from a meadow at a depth of 20 cm, was derived from schists and classified as a humic cambisol according to FAO-Unesco criteria (FAO-Unesco, 1998). It had a sandy loam texture, was moderately acidic (pH 5.5), had a good organic matter content (9.6%) and a low effective exchange capacity [3.6 cmol (+) kg^{-1}].

The pots used were made of PVC, had a diameter of 15 cm and were 9 cm deep; all were filled with 1.5 kg of soil. The base of the pots had a hole in order to allow leachate to be collected by simple percolation. These pots were then divided into three groups of six and received either 140 ml of sludge (80 m^3 ha^{-1} [D80]), 280 ml (160 m^3 ha^{-1} [D160]) or 1,200 mg (675 kg ha^{-1}) of inorganic 15:15:15 NPK fertilizer. Six control pots received neither sludge nor NPK. The quantities of sludge provided are the same as those used by López Mosquera *et al.* (2002b) in a field trial involving the same sludge. These were reported to be agriculturally efficient doses that involved no risk to soil or vegetation. The dose of NPK used in the present experiment was the same as that normally used on the prairies of Galicia (Piñeiro Andión and Pérez Fernández, 1992).

After treating the soil, the pots were sown with Italian ryegrass (*Lolium multiflorum* L.) at a rate of 30 seeds per pot. This corresponds to the normal sowing density used on Galician prairies. The pots were maintained in environmental chambers under conditions of controlled moisture and temperature for nine weeks. Mean temperature oscillated between 15ºC at night and 25ºC during the day, relative humidity was 65% at night and 50% during the day. Every two days the pots were watered with 200 ml of water. The leachate from each was removed every two days for analysis.

At the end of the nine weeks, the ryegrass and the soil were removed from each pot. The soil was air-dried, passed through a 2 mm sieve, and its pH determined in 0.1 N KCl (1:2.5 soil:solution). Assimilable P, Ca, Mg, K, Mn, Fe, Ni, Cu, Cr, Cd, and Pb were extracted with Mehlich 3 reagent (Mehlich, 1984). This extracts Ca, Mg and K in quantities similar to those achieved with ammonium acetate, slightly more P is extracted than with the Olsen or Bray methods, and the remaining ions are extracted in amounts similar to those provided by the DTPA (trisodium calcium diethylenetriaminepentaacetate) method (Sen Tran and Simard, 1993).

The fresh weight of the ryegrass was recorded. The aerial and root compartments were then separated and both were dried at 65ºC. Following digestion in concentrated H_2SO_4 and H_2O_2 at 390ºC (Jones *et al*., 1991), the concentrations of P, Ca, Mg, K, Fe, Ni, Cu, Cr, Cd, Zn and Al were determined using an atomic absorption/emission spectrophotometer (Perkin Elmer, Wellesley, MA, USA). The pH and the concentrations of inorganic N (NO_3^- and NH_4^+), P, Ca, Mg and K were also determined for the leachates.

The total C, S and N concentrations of the soil and ryegrass were determined by combustion using a CNS-2000 autoanalyser (LECO, St. Joseph, Michigan, USA). Soil, leachate and plant P contents were determined by colorimetry with ascorbic acid and sulphomolybdic reagent. The $N\text{-}NO_3^-$ and $N\text{-}NH_4^+$ contents of the leachate were determined using a FIAstar 5000 flow injection analyser (Foss Tecator, Barcelona, Spain). Ca, Mg, K, Fe, Ni, Cu, Cr, Cd, Zn and Al concentrations were determined as above.

The nutritional status of the crop was determined based on leaf nutrient concentrations; these were compared to the reference levels indicated by Crush *et al*. (1989).

The results were examined by analysis of variance. Tukey's test was used to distinguish groups showing significant differences. The data were also examined using regression analysis and the Pearson correlation test.

Results

Leachate analysis

Figure 1 shows that the provision of NPK provoked a fall in pH of almost one unit compared with that of the control treatment. Consistent with this, the leachates of the NPK treatment pots contained high $N\text{-}NO_3^-$, K, Ca and Mg concentrations. The leachate from the high dose sludge-treated pots, however, showed a gradual increase in pH, coinciding with higher concentrations of Ca. In this treatment, leaching losses of $N\text{-}NO_3^-$ and cations were very reduced over the entire experimental period compared with controls. The concentration of $N\text{-}NH_4^+$ was greater than that of $N\text{-}NO_3^-$ in this high dose sludge treatment. In all treatments, leachate P concentrations were very low (<0.1 mg L^{-1}, data not shown).

Soil analysis

Table 2 shows the results of the chemical analysis of the solid fraction of the different soils after nine weeks. NPK treatment led to a slight decrease in soil pH. The application of sludge, however, led to a slight increase in soil pH, total N and available P (in proportion to the dose applied). The available Ca, Mg and K contents did not vary significantly across the different treatments, although there was a trend towards higher Ca levels after sludge application.

The Mn, Fe, Cr, Cd, Cu, Pb and Ni contents were not greatly affected by any treatment; values remained low. The only effect attributable to the treatments was the significant reduction in available Ni with the higher sludge dose.

Production and nutrition of vegetation

The production of *Lolium multiflorum* L., both in terms of its aerial and root compartments, increased significantly in the pots treated with NPK and sludge (Fig. 2). The greatest effect was seen with the higher sludge dose, in which increments of 70% and 21% were recorded for these two compartments respectively.

The response to the different treatments was assessed in terms of the concentration (Table 3 and Fig. 3) and absolute quantities of elements absorbed by the plants (Table 4). Increases were seen in N concentration in the aerial compartment with both sludge doses, but especially with the higher dose. Fertilization with NPK, however, led to no changes in plant N concentration.

The higher soil P content occasioned by sludge application led to increases in P levels in the aerial compartment of the plants, with values reaching 2.5 mg g^{-1} for both treatments (Fig. 3). Though NPK

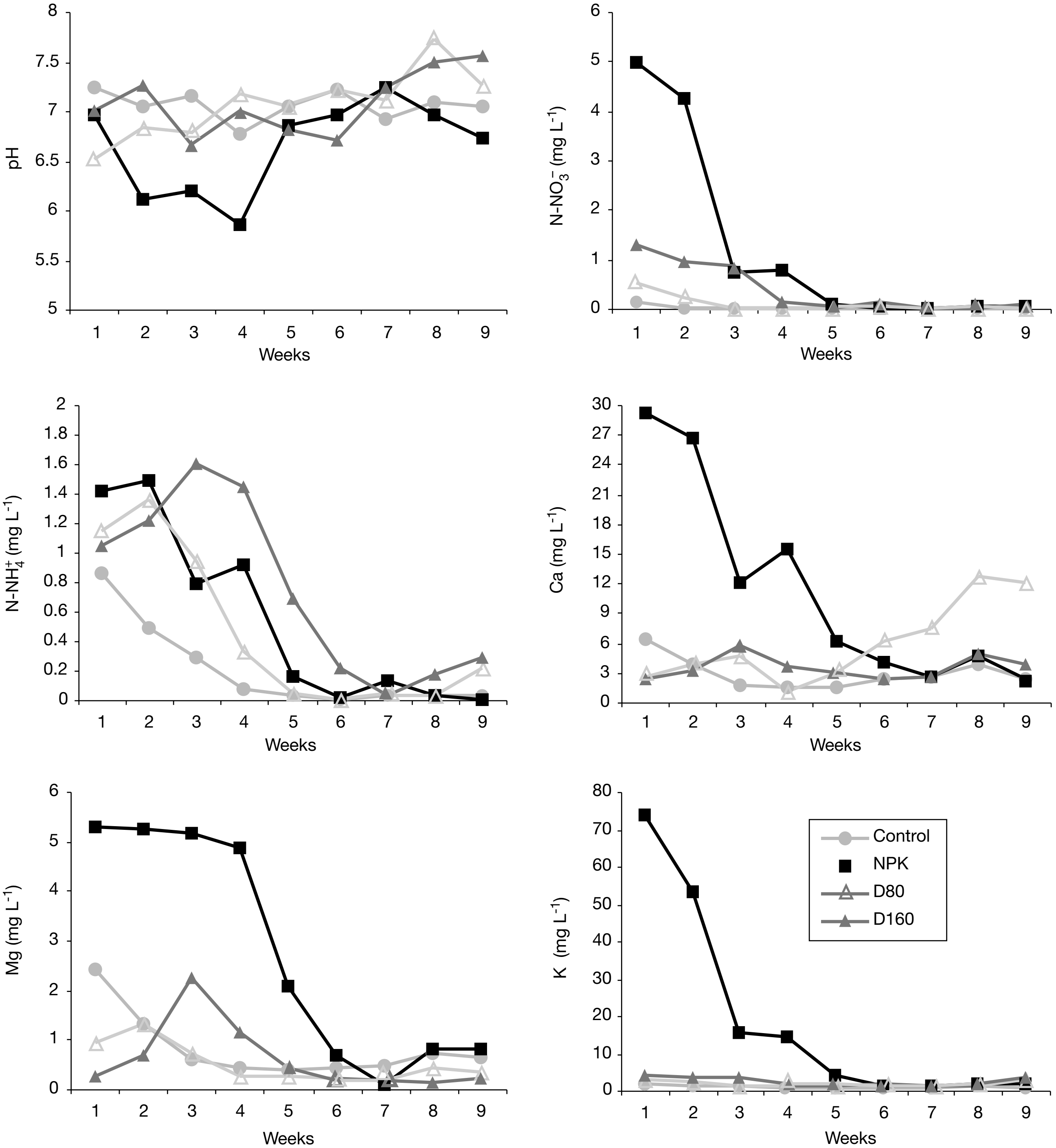

Figure 1. Changes in pH and NO_3^--N, NH_4^+-N, Ca, Mg and K concentrations in leachates. NPK: 675 kg ha^{-1} of NPK 15:15:15. D80: sludge at 80 m^3 ha^{-1}. D160: sludge at 160 m^3 ha^{-1}.

Table 2. Means and SD for pH and chemical composition of the soils after nine weeks. C, N and S are total amounts expressed as percentages. Values for all other elements (in mg kg^{-1}) are for those extracted with Mehlich reagent

Treatment	pH (KCl)	C	N	S	P	Ca	Mg	K	Mn	Fe	Ni	Cu	Cr	Cd	Pb
		(%)			(mg kg^{-1})										
Control	4.34ab (0.07)	5.60a (0.2)	0.42ab (0.02)	0.05a (0.01)	2.6a (1.5)	701.0a (353.6)	71.4a (36.1)	53.2a (1.91)	1.78a (1.05)	267.50a (54.5)	1.10b (0.2)	0.82a (0.62)	1.50a (2.9)	0.08a (0.04)	2.90a (1.53)
NPK	4.27a (0.04)	5.20a (0.3)	0.40a (0.02)	0.04a (0.02)	2.5a (1.3)	909.0a (151.6)	72.3a (4.99)	60.5a (10.75)	1.78a (0.26)	307.70a (28.5)	1.00ab (0.09)	0.59a (0.40)	0.07a (0.2)	0.12a (0.04)	2.20a (1.48)
D80	4.37b (0.04)	4.40a (0.04)	0.43ab (0.01)	0.05a (0.01)	3.8b (1.03)	940.0a (675.2)	69.3a (34.88)	63.8a (7.73)	8.98a (15.66)	266.70a (133.8)	1.07ab (0.17)	0.70a (0.32)	2.00a (2.4)	0.12a (0.04)	5.40a (1.68)
D160	4.49c (0.03)	5.60a (0.2)	0.45b (0.01)	0.05a (0.01)	3.7b (1.6)	1104.0a (97.9)	91.5a (2.62)	60.0a (7.07)	2.25a (0.54)	356.30a (36.2)	0.73b (0.3)	0.33a (0.19)	0.18a (0.4)	0.10a (0.001)	5.40a (2.34)

fertilization led to no increase in soil P concentration, it did lead to greater P assimilation by the plants (Table 4).

Plant Ca, Mg and K concentrations were higher in the aerial than in the root compartment, but were not significantly affected by either NPK or sludge treatment. However, there was a trend towards higher Ca and Mg concentrations with the sludge treatments, and towards a higher K concentration with the NPK treatment (Table 4).

Correlations between the concentrations of the different elements and crop production were only significant with respect to P ($r = 0.62$, $p < 0.05$ and $r = 0.52$, $p < 0.05$ for the aerial and root compartments respectively).

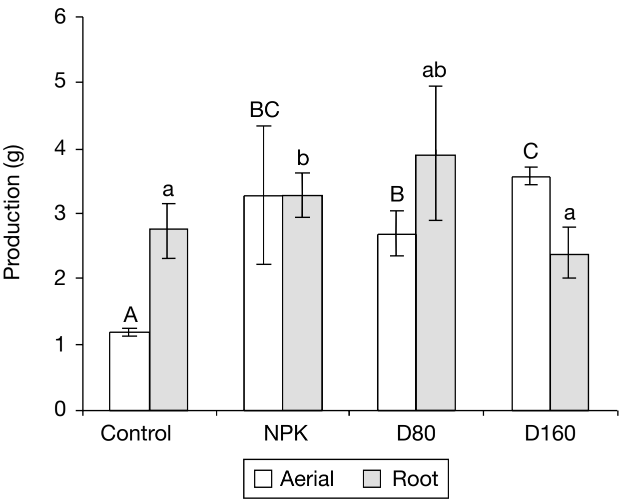

Figure 2. Production of *Lolium multiflorum* L. over the nine weeks of the experiment. NPK: 675 kg ha^{-1} of NPK 15:15:15. D80: sludge at 80 m^3 ha^{-1}. D160: sludge at 160 m^3 ha^{-1}. Different letters indicate significant differences at $p < 0.05$. Capitals and lower case letters refer to the aerial and root compartments respectively.

Nutrient balance

Table 4 shows the quantities of elements provided by the different treatments, those accumulated by the plant, and leachate losses. These data reveal the different efficacies of the treatments in terms of the quantities of elements assimilated and those lost by leaching. Of the total N supplied by the treatments, the plants absorbed between 27% and 45%, the rest was mainly accumulated in the soil. The efficacy of NPK fertilization was slightly greater in terms of providing an appropriate nutrient balance, but a very large amount of N was lost through leaching. A similar effect was seen for P, with NPK being slightly more efficient than the sludge.

The amounts of Ca and Mg accumulated by the plants were similar in all treatments. Both sludge doses covered Ca requirements, but the lower dose did not supply sufficient amounts of Mg. The NPK treatment reduced soil Ca and Mg levels, because of a lack of their provision, and also because of greater leaching losses linked to the mobilization of NO_3^-. Finally, the sludge treatments were unable to provide enough K to meet the needs of the plants. The plants assimilated 49% of the K supplied by the NPK treatment, but the sludge treatments led to a 17% loss through leaching.

Discussion

The results of the present study, wich are consistent with those of López-Mosquera (2000 and 2002a)

Table 3. Concentration of elements in the aerial and root compartments of *Lolium multiflorum* L. after nine weeks of treatment

Treatment	Fraction	Mass (g)	N	S	P	Ca	Mg	K	Mn	Fe	Ni	Cu	Cr	Cd	Zn	Al
			(mg g^{-1})													
Control	Aerial compartment	1.17	12.8	1.1	0.97	4.42	3.07	18.94	0.10	0.29	0.08	0.01	0.03	0.002	0.03	1.51
	Root compartment	2.74	9.5	1.7	2.13	0.62	1.22	3.66	0.16	21.63	0.24	0.01	0.56	0.001	0.03	10.39
NPK	Aerial compartment	3.28	7.6	2.7	1.86	0.90	1.20	5.65	0.12	11.40	0.30	0.01	0.24	0.001	0.04	3.57
	Root compartment	3.28	12.6	0.5	1.26	2.13	1.77	14.50	0.10	14.50	0.08	0.01	0.03	0.002	0.02	1.01
D80	Aerial compartment	2.69	17.5	0.7	2.51	4.54	3.19	8.91	0.10	2.94	0.15	0.02	0.11	0.002	0.06	0.91
	Root compartment	3.89	8.1	2.2	1.56	0.71	1.06	3.94	0.20	12.43	0.45	0.02	0.87	0.001	0.04	4.97
D160	Aerial compartment	3.57	28.1	0.6	2.25	4.80	3.02	24.60	0.10	0.31	0.08	0.01	0.03	0.001	0.04	1.25
	Root compartment	2.40	11.6	2.7	1.78	0.84	1.10	4.18	0.20	11.94	0.16	0.01	0.30	0.001	0.04	4.20

confirm that the application of sewage sludge improves the nutritional status of the crop, leading to substantial increases in production.

Sludge treatment increased the N content of both the aerial and root compartment of the plant in proportion to the dose applied. The use of the higher dose led to aerial compartment N values similar to the reference values for ryegrass (2.8%) indicated by Crush *et al*. (1989). Other studies with sludges rich in N have also reported increases in the concentration of N in the tissues of the same species (Bernal *et al*., 1998). Despite this effect, and unlike that seen with the NPK treatment, sludge application did not lead to an increased N-NO_3^- concentration in the leachate. Some studies performed under similar conditions have shown that this may be due to a process of microbial mobilization as a consequence of providing organic C, which is easily used by soil organisms (Babarika *et al*., 1985; Bernal *et al*., 1998).

The acidification of the soil caused by the NPK treatment coincided with the appearance of NO_3^- in solution. This effect may be linked to the nitrification of NH_4^+. Acidification occurs because of the leaching of NO_3^- in the absence of vegetation that can assimilate it. The acid produced by nitrification, along with NO_3^- mobilization, would promote K, Ca and Mg losses. These are displaced from the exchange complex and are lost through leaching, as the analysis of the leachates shows.

Nitrification was favoured in the experimental soil despite its acidic character – which normally tends to reduce the activity of nitrifying microorganisms (Haynes, 1986). Some studies (e.g., Meiwes *et al*., 1998) show that the repeated application of NH_4^+ stimulates nitrification in acidic soils; this appears to be related to an increase in the microbial population in response to greater amounts of N (Johnson, 1991). As well as this acidification process and the environmental risks it entails, the loss of N by leaching also implies a reduction in the efficacy of the fertilizer.

In addition to leaching losses, gaseous losses must also be taken into account, mainly through denitrification. This is potentiated when NO_3^- levels are high (Kaiser *et al*., 1998). A recent study (Merino *et al*., 2003) indicates that following applications of organic or inorganic fertilizers to similar soils, the loss of N_2O-N in natural conditions is < 5 kg ha^{-1} $year^{-1}$. However, other studies report greater losses for both kinds of fertilizer (Estavillo *et al*., 1994).

The Ca and Mg losses caused by the NPK treatment may have adverse effects and should therefore be compensated for by supplements. These losses could be the reason why plants receiving this treatment had lower Ca and Mg concentrations than the controls.

The sludge applications led to the assimilation of greater quantities of Ca, Mg and K by the crop. However, neither treatment increased final plant

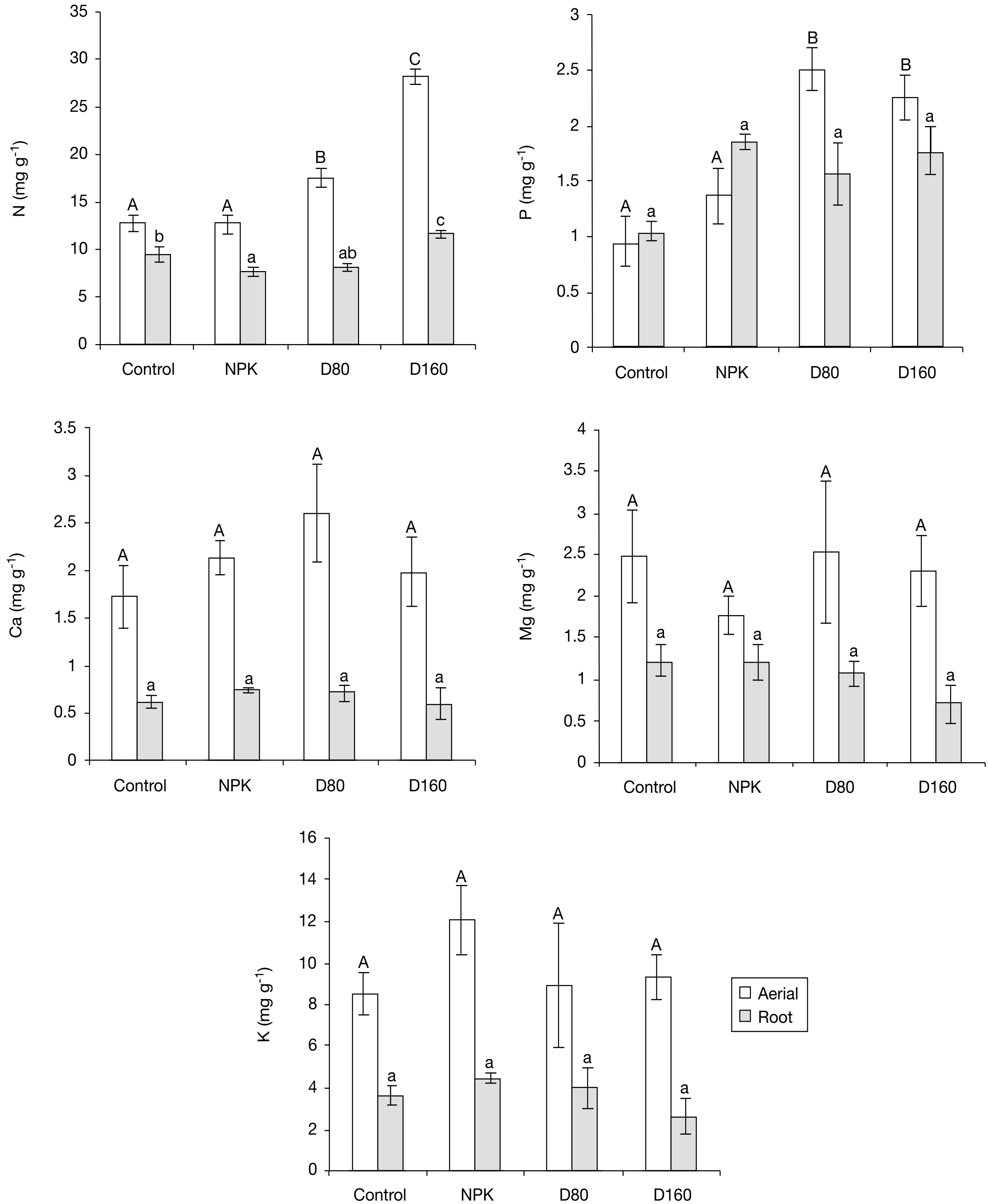

Figure 3. Concentration of elements in the aerial and root compartments of the plant. NPK: 675 kg ha^{-1} of NPK 15:15:15. D80: sludge at 80 m^3 ha^{-1}. D160: sludge at 160 m^3 ha^{-1}. Different letters indicate significant differences at $p < 0.05$. Capitals and lower case letters refer to the aerial and root compartments respectively.

Table 4. Amounts of nutrients supplied, lost through leaching, and accumulated by the plants under the different treatments

	Nitrogen (g m^{-2})				Phosphorus (g m^{-2})			
	Control (mg)	**D80 (mg)**	**D160 (mg)**	**NPK (mg)**	**Control (mg)**	**D80 (mg)**	**D160 (mg)**	**NPK (mg)**
Amount provided	0.00	13.6	27.20	10.2	0.00	4.20	8.40	4.44
Plant	2.24	4.4	7.3	4.6	0.27	0.7	0.70	0.83
Aerial compartment	0.84	2.7	5.7	2.3	0.06	0.39	0.46	0.25
Root compartment	1.4	1.75	1.6	2.3	0.20	0.30	0.24	0.40
Leachate	0.00	0.03	0.1	0.15	0.00	0.00	0.00	0.00
Provided-plant-leachate	**–2.3**	**9.15**	**6.20**	**5.41**	**–0.08**	**3.51**	**7.69**	**3.61**
	Calcium (g m^{-2})				**Magnesium (g m^{-2})**			
	Control (mg)	**D80 (mg)**	**D160 (mg)**	**NPK (mg)**	**Control (mg)**	**D80 (mg)**	**D160 (mg)**	**NPK (mg)**
Amount provided	0.00	3.35	6.70	0.00	0.00	0.51	1.03	0.00
Plant	0.22	0.45	0.52	0.65	0.40	0.63	0.61	0.69
Aerial compartment	0.12	0.33	0.41	0.39	0.16	0.41	0.47	0.33
Root compartment	0.10	0.15	0.11	0.26	0.20	0.22	0.14	0.36
Leachate	0.18	0.22	0.25	0.88	0.05	0.03	0.05	0.23
Provided-plant- leachate	**–0.57**	**2.34**	**5.44**	**–1.53**	**–0.45**	**–0.22**	**0.25**	**–0.92**
	Potassium (g m^{-2})							
	Control (mg)	**D80 (mg)**	**D160 (mg)**	**NPK (mg)**				
Amount provided	0.00	1.58	3.17	8.45				
Plant	1.15	1.75	2.43	4.12				
Aerial compartment	0.57	1.01	1.91	2.51				
Root compartment	0.58	0.74	0.52	1.61				
Leachate	0.05	0.08	0.15	1.47				
Provided-plant-leachate	**–1.89**	**–0.54**	**–2.15**	**2.86**				

concentrations – possibly because levels were sufficient. The concentration of these elements recorded in the aerial part of the plant were similar to the reference values indicated by Crush *et al*. (1989), 4.4 mg g^{-1} for Ca, 2.2 mg g^{-1} for Mg, and 32.5 mg g^{-1} for K. In addition, soil NH_4^+ competes with plant assimilation for ions such as Ca, Mg or K (Fangmeier *et al*., 1994): increases in NH_4^+ were seen with all treatments.

Despite the large quantities of P provided by both the NPK and sludge treatments, no leaching losses were recorded. The soil and/or the plant therefore have a strong P-retaining capacity. In agreement with similar studies (Soler Rovira, 1998), soil analysis showed only slight increases in available P with sludge application. Large increases were seen, however, in plant P in the aerial compartment, reaching the reference value of 2.8 mg g^{-1} indicated by Crush *et al*. (1989). The plant P concentrations reached with the NPK treatment were lower than those achieved with the sludge, possibly because of a reduction in its availability through acidification.

The reductions in Ni availability with the high sludge dose were related to the lower solubility of this element with increasing pH (Kabata-Pendias and Pendias, 1984). This has also been observed when other types of alkaline residues with a low metal content have been used as fertilizers (Krejsl and Scanlon, 1996).

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