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Influence of Sewage Sludge Application on Crop Yields and Heavy Metal Availability

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A calcareous soil with a low content of organic matter was treated with aerobic or anaerobic sewage sludge. In the same way, chicken manure was used for comparison. Influences on crop yields and the supply of macronutrients and heavy metals to soil and plant tissues were studied.

In the soil amended with the sludges the total N and extractable N and P contents increased, whereas the extractable K level remained nearly constant. Samples which had undergone a period of humification prior to sowing, mineralized part of their organic N and P during this period of time. Through sludge application, the levels of Cu, Zn, and Pb increased, the Fe content decreased while the extractability of Fe, Cu, Mn, Zn, and Pb increased as compared to the control.

Sludge application enhanced the maize and barley yields as is the case of chicken manure. Sewage sludges supplied a considerable amount of available N to the plants, whereas the supply of available K was low. In the barley plants it was observed that the higher the yield, the higher the N and K contents, while in the maize crop this trend was only observed for K. Plants grown on soil amended with sludges absorbed a larger amount of Fe, Cu, and Zn than those grown on the control soil.

It was demonstrated that heavy metal absorption by the plants depended on the vegetal species.

Key Words: **crop yield, heavy metal availability, macronutrients, sewage sludge.**

There is an increasing interest in the agricultural application of sludges obtained in wastewater treatment plants, due to the possibility of recycling valuable components: organic matter, N, P, and other plant nutrients (Sommers 1977; Chaussod et al. 1978; Suss 1979).

In general it has been shown that the addition of sludge to agricultural land increases crop production. Dowdy et al. (1978) reported that the increase of crop yield by sludge application often exceeded that of well-managed fertilized controls.

On the other hand, since sewage sludge contains high concentrations of potentially toxic elements such as Zn, Ni, Cd, and Cu problems may arise when sludge is applied to an agricultural soil (Sanders et al. 1986; Omran and Waly 1988) and heavy metal accumulation in the plant tissues may also occur. The fertility benefits must be balanced against the potential hazards of metal contamination through unrestricted application of sludge to agricultural productive land.

One of the characteristics of the soils in the Southeastern part of Spain is their low content of organic matter. Due to the current limited amounts of organic resources and the decrease in the supply of farmyard manure, the totality of sewage sludge produced in these regions is used for the improvement of soil fertility and crop production. Therefore, studies on sludges are important due to the economic and environmental implications of widespread application of these materials to agricultural lands.

The aim of this paper is to analyze the influence of sewage sludge application to a Calciorthid soil on crop yields and the supply of macronutrients (N, P, and K) and heavy metals (Fe, Cu, Mn, Zn, Ni, Cr, Cd, and Pb) to soil and plant tissues. In the same way, chicken manure was applied in order to make a comparative study between the effect of this farmyard manure and that of the sludges.

MATERIALS AND METHODS

Samples of a Calciorthid soil (Soil Taxonomy 1975) typical of the Southeastern part of Spain were used. The soil contained a large amount of lime, a small amount of organic matter and displayed a clay-loam texture.

Chicken manure (CM) as well as aerobic (AES) and anaerobic (ANS) digested sewage sludges were used. Table 1 lists the chemical and physical characteristics of the soil and organic materials.

Organic residues were added to the soil at such a rate that they raised the soil content of oxidizable carbon by 1.5%. This level represents the minimum average of agricultural soils. Ten-kilogram samples of treated and control soils were placed in pots, each treatment

Table 1. Characteristics of the soil and organic residues.

	Soil	CM	AES	ANS
Conductivity (μS/cm)	264	7,400	2,060	3,710
pH	7.8	8.0	6.4	6.7
$CaCO_3$ (%)	44.0	23.4	32.6	17.7
Ash (%)	ND	33.6	53.4	49.0
CEC (meq 100 g^{-1})	15.0	65.7	48.3	54.5
Oxidiz. carbon (%)	0.73	29.3	22.4	24.9
Extr. carbon (%)	0.20	6.01	3.14	2.81
Humic acid carbon (%)	0.13	2.27	0.80	0.98
Fulvic acid carbon (%)	0.07	3.74	2.34	1.83
Total N (%)	0.07	2.41	2.38	2.95
Extractable N (%)	0.016	0.14	0.08	0.29
C/N	10.4	12.2	9.4	8.5
Heavy metal (mg kg^{-1})				
Fe	16,900.0	1,247.0	8,083.0	10,034.0
Cu	17.0	48.0	152.0	286.0
Mn	336.0	256.0	142.0	169.0
Zn	38.0	612.0	780.0	1,230.0
Ni	42.0	24.0	44.0	62.0
Cr	25.0	23.0	47.0	113.0
Cd	5.0	6.0	8.0	7.0
Pb	55.0	31.0	109.0	86.0

ND, not determined.

being replicated three times.

The three replicates for one treatment were incubated for 6 months with periodic watering to maintain water tensions at levels between 1/3 and 15 atmospheres. By this treatment the process of humification of the organic materials was initiated in soil. The samples were re-mixed and aired at 2 month interval. A second replicate was prepared after 6 months and then in both sets of pots maize was planted, followed by barley. The non-humified and humified series were designated as A and B, respectively. Organic matter was not added in the control sets.

Inorganic fertilizer consisting of $(NH_4)_2SO_4$, $NH_4H_2PO_4$, and K_2SO_4 was applied before the seeds were sown to give 100 kg ha^{-1} N, 200 kg ha^{-1} P_2O_5, and 225 kg ha^{-1} of K_2O. When the maize plants were 15 and 60 cm high, 105 and 140 kg ha^{-1} respectively of N were added as NH_4NO_3. Maize was sown in the spring and harvested after 2 months. Barley was sown in the following autumn without further addition of fertilizer and was harvested in March. The soils in the pots were sampled three times, once prior to the first sowing, and once after the maize and barley harvest and designated as 0, 1, and 2, respectively.

Conventional methods in soil organic matter analysis were used for the determination of the content of oxidizable carbon, total nitrogen and phosphorus (Jackson 1964). Potassium content was determined by flame emission spectrophotometry. Cation exchange capacity (CEC) was determined according to the method developed by Lax et al. (1986) for organic manures.

Total metal contents were determined by atomic absorption flame spectrophotometry after $HNO_3 : HClO_4$ (1 : 1) digestion.

Extracts for metals were obtained by mechanical shaking of 10 g of dry sample for 2 h with 100 ml of 0.05 M DTPA (diethylenetriaminepentaacetic acid), 0.01 M $CaCl_2$, and 0.1 M TEA (triethanolamine) buffered at pH 7.3 (Lindsay and Norvell 1978) and the metal contents were determined by atomic absorption spectrophotometry.

RESULTS AND DISCUSSION

Soil

Table 2 lists the total N and extractable P and K contents in the A and B series samples. It can be observed that the contents of total and extractable N in the soil increased when the organic materials were added, the largest values of total N being recorded when the sludges were applied.

The samples which had undergone a period of humification prior to sowing (series B) transformed part of their organic N into extractable N during this period of time.

Total N content of the samples of the soil treated with chicken manure and the soil treated with anaerobic sludge in series B was lower than that in series A, probably due to the loss of N by denitrification and NH_4^+ volatilization during the humification period.

The application of chicken manure resulted in the largest increase of extractable P content followed by that of aerobic and anaerobic sludge. The samples of series B showed the highest values of extractable P due to the mineralization which occurred during the humification period in which part of the organic P of the samples was transformed into extractable P. These increases were about 65, 11, and 38% for the samples treated with anaerobic sludge, aerobic sludge and chicken manure, respectively.

In the soil amended with chicken manure the extractable K content increased unlike in the sewage sludges. Tables 3 and 4 list the total heavy metal (Fe, Cu, Mn, Zn, Ni, Cr, Cd,

Table 2. Total N and extractable N, P, and K contents in the series A and B samples.

		N (%)		Ext. P	Ext. K
		Tot.	Ext.	(mg/kg)	(%)
S	Series A	0.07	0.016	26	0.42
	Series B	0.08	0.014	27	0.46
S+CM	Series A	0.19	0.032	260	1.70
	Series B	0.15	0.072	359	1.47
S+AES	Series A	0.22	0.028	208	0.46
	Series B	0.23	0.038	231	0.38
S+ANS	Series A	0.23	0.039	86	0.49
	Series B	0.17	0.063	142	0.40

and Pb) contents and the percentages of extractable metals in relation to the total content, respectively in the series A and B samples at the beginning (sample time 0) and at the end (sample time 2) of the experiment.

The total Fe content in the soil decreased slightly with the addition of organic amendments because these materials showed a lower Fe content than the control.

The Fe fraction extracted by DTPA was very small in all the samples. Sewage sludge increased the extractability of this metal since the quantities of extractable Fe were higher than those expected for the amended soils. In all the samples, Fe became more insoluble with time.

In the soils treated with sludges the Cu content increased 2-fold whereas chicken manure did not modify this value in agreement with the Cu content of the organic amendment. It was observed that the extractability of Cu increased with the addition of sewage sludge.

The 6 month period of humification was associated with an increase of the extractable Cu content (see values of series A and B at sample time 0). In the course of the experiment the percentage of extractable Cu in the series A samples increased while in the series B it decreased. The values of extractable Cu in the series B samples were always higher than those of the series A samples except for the soil and S+ANS (soil+anaerobic sludge) samples at the end of the experiment (sample time 2).

Although there were no remarkable changes in the Mn content in the amended soils, these materials, especially chicken manure, increased the extractable Mn content indicating that they promoted Mn extractability. The values of extractable Mn of the amended soils decreased during the experiment although they were always higher than those of the control.

In the course of the experiment the percentages of extractable Mn in both series A and B decreased, presumably due to the insolubilization of released Mn as oxides or carbonates during the degradation of organic matter.

The total Zn content increased when the sludges were added to the soil specially in the anaerobic sludge samples, because the Zn content was higher than that of the soil.

All the organic amendments, particularly the sludges increased Zn extractability.

At the beginning of the experiment (sample time 0), the samples not humified before sowing (series A) showed higher percentages of extractable Zn than those of series B. This Zn fraction decreased during the experiment in both series presumably due to the insolubilization with time of the Zn released in the degradation of organic matter.

The total Ni, Cr, and Cd contents of the soil were not affected by the application of

Table 3. Total metal content in the series A and B samples (mg kg^{-1} dry wt.)

		S		S+CM		S+AES		S+ANS	
		0	2	0	2	0	2	0	2
Fe	Series A	16,900	16,700	16,600	16,700	16,800	17,200	16,400	18,500
	Series B	15,100	16,600	15,300	14,900	16,500	16,000	16,000	16,000
Cu	Series A	17	16	18	19	32	26	29	26
	Series B	15	16	17	18	24	24	30	28
Mn	Series A	336	316	326	358	316	335	316	335
	Series B	335	325	328	335	317	318	322	320
Zn	Series A	38	39	12	11	85	97	168	145
	Series B	39	39	10	9	84	84	178	150
Ni	Series A	42	39	39	41	41	41	44	41
	Series B	40	39	39	39	40	39	41	40
Cr	Series A	25	22	24	23	26	26	25	26
	Series B	23	24	25	23	23	23	24	24
Cd	Series A	5	5	5	5	5	5	5	5
	Series B	5	5	5	5	5	5	5	5
Pb	Series A	55	51	54	52	71	74	71	61
	Series B	55	53	52	56	69	71	67	68

Sampling time: 0, before sowing; 2, after barley harvest.

Table 4. Extractable metal content in the series A and B samples (% of total metal dry wt.).

		S		S+CM		S+AES		S+ANS	
		0	2	0	2	0	2	0	2
Fe	Series A	0.05	0.03	0.05	0.04	0.10	0.08	0.16	0.08
	Series B	0.07	0.02	0.05	0.04	0.08	0.08	0.10	0.08
Cu	Series A	5.88	12.50	5.56	10.53	9.38	11.54	17.24	23.08
	Series B	6.67	6.25	11.77	11.11	16.67	12.50	20.00	14.29
Mn	Series A	0.30	0.35	10.02	6.15	9.10	4.18	7.28	5.07
	Series B	0.30	0.31	12.50	5.67	6.49	5.03	8.07	5.31
Zn	Series A	5.26	5.56	21.05	16.42	27.06	15.46	33.33	22.76
	Series B	5.56	5.13	16.67	14.75	21.43	16.67	27.53	21.33
Ni	Series A	2.38	0.00	2.56	2.44	2.44	2.44	2.44	2.44
	Series B	2.50	0.00	2.56	2.56	2.50	2.56	2.44	2.50
Cr	Series A	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	Series B	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cd	Series A	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	Series B	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Pb	Series A	5.45	5.58	7.41	5.77	8.45	9.46	9.96	11.47
	Series B	7.27	5.66	7.69	5.36	11.59	11.27	10.45	11.76

Sampling time: 0, before sowing; 2, after barley harvest.

organic residues and no variations in the content of extractable Ni, Cr, and Cd were observed during the humification or during the experiment. Extractable Cr and Cd were not detected.

The increase of the content of both, total and extractable Pb by the application of the sludges was attributed to the quantities incorporated by the sludges since chicken manure did not modify the content of this metal in soil.

Sludge application resulted in higher values of extractable Pb than those of the chicken

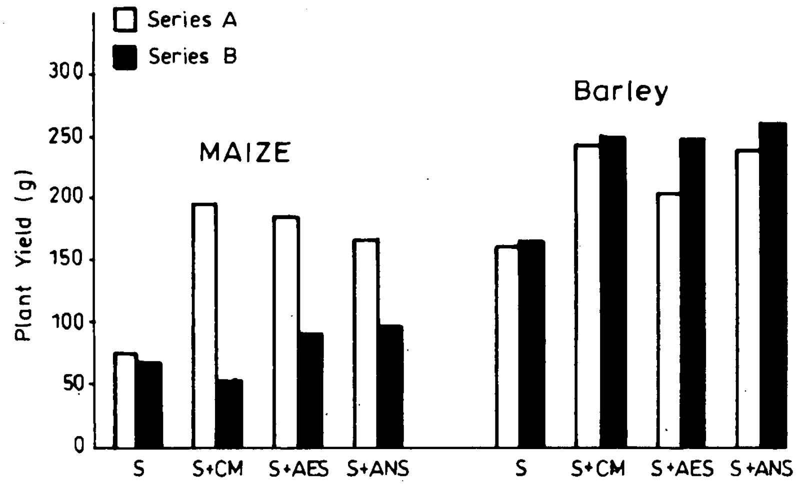

Fig. 1. Dry matter yield in the samples. S, control; S+CM, soil with chicken manure; S+AES, soil with aerobic sludge; S+ANS, soil with anaerobic sludge.

Table 5. Macronutrient contents in maize and barley crops (% dry wt.).

			N	P	K	Ca	Mg
S	Series A	Maize	2.21	0.12	3.61	1.10	0.19
		Barley	2.58	0.21	4.21	0.58	0.29
	Series B	Maize	2.29	0.08	3.27	1.17	0.18
		Barley	3.03	0.17	4.63	1.06	0.43
S+CM	Series A	Maize	2.64	0.42	4.38	0.87	0.34
		Barley	2.65	0.59	4.31	0.71	0.42
	Series B	Maize	3.23	0.42	5.09	1.45	0.22
		Barley	4.25	0.47	4.96	1.40	0.48
S+AES	Series A	Maize	2.77	0.23	3.86	1.02	0.41
		Barley	3.13	0.50	2.58	0.99	0.41
	Series B	Maize	2.93	0.24	3.47	1.53	0.20
		Barley	3.52	0.52	2.87	2.20	0.49
S+ANS	Series A	Maize	2.78	0.27	4.15	1.12	0.34
		Barley	3.58	0.34	2.74	1.66	0.44
	Series B	Maize	2.91	0.17	3.11	2.57	0.26
		Barley	4.11	0.38	3.41	1.55	0.40

manure and the control.

On the other hand, humification did not affect appreciably the extractable and total Pb fraction.

Plant

Figure 1 indicates the yield of the crops. It was observed that the application of organic materials enhanced soil fertility and that crop yield in the amended soils was higher than in the control.

The plant yields obtained with both kinds of organic amendments, chicken manure, and sludges, were similar.

Table 5 shows the macronutrient contents in maize and barley crops. It can be seen that K was the macroelement extracted at the highest rate by maize, followed by N. Both, K and N, accounted for 80–85% of the absorbed macronutrients.

Table 6. Correlation coefficients between plant yield of the crops and contents in different fractions of carbon and nitrogen in the samples.

	r					
	Maize crop			Barley crop		
	Series A	Series B	Series A + Series B	Series A	Series B	Series A + Series B
Ox. C	0.95**	0.93*	0.84**	0.66^{NS}	0.91*	0.42*
Ext. C	0.96**	0.87**	0.88**	0.61^{NS}	0.98**	0.86**
HA	0.87*	0.94*	0.67*	0.79^{NS}	0.60^{NS}	0.62^{NS}
FA	0.97**	0.75^{NS}	0.93***	0.84^{NS}	0.91*	0.88**
Total N	0.91*	0.19^{NS}	0.69*	0.73^{NS}	0.95**	0.73*
Ext. N	0.79^{NS}	0.83^{NS}	0.01^{NS}	0.94*	0.84^{NS}	0.82**

*$p=0.05$; **$p=0.01$; ***$p=0.001$; ^{NS} not significant. Ox. C, oxidizable carbon; Ext. C, extractable carbon; HA, humic acid carbon; total N, total nitrogen; Ext. N, extractable nitrogen; FA, fulvic acid carbon.

Table 7. Carbon and nitrogen contents in the series A and B samples (% dry wt.).

	Sample time	S		S+CM		S+AES		S+ANS	
		Series A	Series B	Series A	Series B	Series A	Series B	Series A	Series B
Total N	0	0.07	0.08	0.19	0.15	0.22	0.23	0.23	0.17
	1	0.10	0.09	0.18	0.16	0.23	0.20	0.24	0.20
Ext. N	0	0.016	0.019	0.032	0.037	0.028	0.032	0.039	0.044
	1	0.019	0.015	0.037	0.079	0.032	0.043	0.044	0.070
Ox. C	0	0.73	0.68	1.82	1.23	2.03	1.45	1.50	1.50
	1	0.65	0.66	1.14	1.12	1.56	1.55	1.46	1.42
Ext. C	0	0.20	0.21	0.42	0.27	0.38	0.27	0.31	0.31
	1	0.19	0.23	1.29	0.29	0.28	0.30	0.25	0.29
HA	0	0.13	0.16	0.23	0.18	0.23	0.19	0.16	0.19
	1	0.11	0.16	0.17	0.16	0.16	0.17	0.14	0.18
FA	0	0.07	0.06	0.19	0.09	0.15	0.08	0.15	0.12
	1	0.08	0.07	0.12	0.13	0.12	0.13	0.11	0.11

Sampling time: 0, before maize sowing; 1, before barley sowing. Total N, total nitrogen; Ext. N, extractable nitrogen; Ox. C, oxidizable carbon; Ext. C, extractable carbon; HA, humic acid carbon; FA, fulvic acid carbon.

P and Mg were the macronutrients less absorbed by the maize crop and they never accounted for more than 5% of the total absorbed.

Compared to the maize crop, the barley crop showed some differences. In both A and B series, K was the macronutrient extracted at the highest rate by the plants grown in the control and in the chicken manure-amended soil. However when sludges were applied to soil, N was the macronutrient most absorbed. This fact is in agreement with the lower extractable K content of the sludges as compared to chicken manure (Table 2) unlike in the maize crop due to the application of K fertilizer.

It was observed that, for each treatment, barley plants grown on previously incubated amended soils (series B) gave a higher yield and contained a higher percentage of N and K than those grown on the series A samples. In the maize crop, the influence of the humification period on the absorption of macroelements by plant was only observed for N.

Maize plants grown on amended soils contained a larger amount of N P K (except for

Table 8. Heavy metal contents in maize and barley crops (mg kg^{-1} dry wt.).

			Fe	Cu	Mn	Zn	Ni	Cr	Cd	Pb
S	Series A	Maize	118	5	121	22	5	9	1	15
		Barley	135	11	73	35	1	1	0	9
	Series B	Maize	212	7	133	28	5	9	1	16
		Barley	198	8	105	35	2	2	1	2
S+CM	Series A	Maize	122	7	147	124	6	10	1	13
		Barley	166	17	45	72	0	4	1	9
	Series B	Maize	275	7	66	85	6	9	1	12
		Barley	165	13	43	70	3	2	0	12
S+AES	Series A	Maize	140	8	91	120	6	8	1	4
		Barley	204	14	45	81	2	1	1	10
	Series B	Maize	216	8	58	119	6	7	2	10
		Barley	209	11	48	85	2	2	1	12
S+ANS	Series A	Maize	133	7	136	171	6	5	1	14
		Barley	277	33	84	159	3	1	1	14
	Series B	Maize	185	9	106	259	6	5	2	9
		Barley	205	13	81	170	3	1	1	13

S+ANS in series B) and Mg than those grown on the control soil. This phenomenon was also observed in the barley crop, but the K content of the plants which had grown on soil amended with sludges was lower than that of the plants grown on the control soil. This fact indicated that although the K content of the sludges was high (13%), K was not available to plants, suggesting that the clay which was carried away by sewage fixed K.

Table 6 shows the relationship between the yield and the contents of the different fractions of C and N in the amended and control soils, which are shown in Table 7. A significant correlation between yield and extractable C, oxidizable C, and fulvic acid contents in both maize and barley crops was observed. This correlation was less conspicuous with humic acid C.

Yield of barley crop was correlated with the total N content of the samples, mainly with the extractable N fraction.

No correlation could be detected between maize crop yield and the content of total and extractable N due to the application of inorganic fertilizer.

Maize yields in series A were always higher than in series B presumably due to the higher fulvic acid content of the series A samples since, as shown in Table 6, maize yields were significantly correlated with the fulvic acid content of the samples. The content of oxidizable and extractable C which was higher in most of the series A samples than in the series B, also plays an important role.

Table 8 shows the heavy metal contents in the maize and barley plants, indicating that the average values of heavy metals in plant tissues were below the levels causing any toxic effect in plants (Chaney 1977; Kabata-Pendias and Pendias 1984).

Plants that had been grown on soil amended with sludges absorbed larger amounts of Fe, Cu, and Zn than those grown on the control soils. This phenomenon may be due to higher extractable metal contents of the amended soil compared with the control.

This trend was similar to that observed in the plants grown on the soil amended with chicken manure, although, in this case, the percentages of extractable Fe and Cu of the amended soil were similar to those of the control.

In general, there were no differences between the Ni, Cr, Cd, and Pb contents of the plants grown on the control and the amended soil. The Mn content of the control plants was, in general, higher than that of the amended soil plants, although the extractable Mn content was lower in the control soil.

In the barley plants, heavy metals mainly absorbed were Fe, Zn, and Mn in that order while in the maize plants no preferential order was detected.

It is known that heavy metal absorption by plants depends on the vegetal species as reported by other workers (Sequi and Petruzzelli 1978; Delcarte et al. 1979; Hani and Gupta 1983; Hovmand 1983). Maize plants accumulated larger amounts of Cr, Ni, Mn, and, except for the control plants, Zn. In contrast, a lower quantity of Cu was accumulated in maize plants than in barley plants.

It should be noted that although only traces of Ni, Cr, and Cd were extracted with DTPA, these metals accumulated in the plants as suggested in the literature (Davis and Stark 1980).

There was no correlation between total and extractable heavy metal contents in the soil samples and metal concentration in plants, except for total and extractable Zn with a significance level of 99.9%. This fact indicates that the extraction power of DTPA is higher than that of plant roots and reflect the heavy metal supply capacity of soil to plants (quantity factor). Presumably, mild extracting solutions could predict more accurately the immediate supply (intensity) of heavy metals to plants, as indicated by Gupta and Hani (1981).

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