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LABORATORY PREDICTION OF SOLUBLE COMPOUNDS BEFORE SOIL RECYCLING OF WASTES

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One of the best alternatives to waste disposal is through the soil-plant system as a fertilizer. Based on contrasting properties, different wastes can be co-recycled in order to take simultaneously the best profit and minimize environmental pollution.

To predict the release of available nutrients and potentially toxic elements, before land application of two wastes (sewage sludge and fly ash), a laboratory experiment had been conducted using leaching and incubation of soil-waste mixtures.

Loading rates of 500 t ha^{-1} of fly ash increased soluble compounds in soil solution, and the sewage loading N-mineralization was inhibited.

The proposed method could give an estimation for soluble elements and mineralizable-N compounds, avoiding undesirable land consequences of waste recycling.

KEY WORDS: Sewage sludge, fly ash, waste reuse, water soluble compounds, laboratory incubations.

INTRODUCTION

Application of wastes on agricultural land has received considerable attention recently because of the cost and environmental problems associated with disposal methods. Since wastes contain nutrients, one of the best alternatives to disposal is to use them as fertilizers in order to recycle nutrients through the soil-plant system. Potentially toxic elements can also be present. Based on contrasting waste properties, it is possible to co-recycle different wastes through the soil.

The application of municipal sewage sludge to agricultural soils is a practice that has been widely recognized, because the organic matter, nitrogen and other nutrients can enhance soil fertility. The fly ash chemical composition is well known.¹⁻³ However, the potential problems associated with land application are related to soluble salts and toxic elements content,⁴⁻⁷ especially B, Mo and Se. Only few studies have demonstrated the beneficial effect of simultaneous soil application of fly ash and sewage sludge.⁸

Taking into account that wastes are usually heterogeneous materials, it is necessary to know their behaviour before land application, in order to avoid undesirable consequences.

Potentially toxic elements in soil can be estimated by soil extraction or plant uptake. To detect the potentially available N from organic wastes, the mineraliza-

Table 1 Chemical composition and physico-chemical properties

	Sewage sludge		Fly ashes		Soil
	SS	MA	TE	S	
Organic matter % ^a	50.5				2.52
Total N % ^b	3.5				0.15
NH ₄ -N mg kg ^{-1c}	4 × 10 ³				8
NO ₃ -N mg kg ^{-1c}	71				32
pH H ₂ O 1/5	7.1	11.1	10.8		7.3
Elec. cond. S m ⁻¹ 1/5	0.44	0.95	0.06		0.02
CEC meq/100 g ^d	61.7	29.0	2.4		37.7
Carbonate %		9.86	3.58		2.92
Ca % ^e	12.08	25.31	3.24		1.21
Mg mg kg ^{-1e}	7140	4320	2000		2340
Na mg kg ^{-1e}	6820	1010	148		536
K mg kg ^{-1e}	3300	7800	1500		557
B mg kg ^{-1f}	19.9	70.08	128.3		5.95
Cd mg kg ^{-1f}	1.96	0.61	0.65		0.35
Mo mg kg ^{-1f}	3.16	21.55	10.48		3.44
Se mg kg ^{-1f}	0.95	16.9	0.35		0.06
Zn mg kg ^{-1e}	1290	91	87		38

^aSludge: ignition at 560 °C; soil: Walkley-Black method.

^bKjeldahl method.

^cExtraction and specific ion electrode.

^dPotentiometric method (1 N BaCl₂, pH = 8.1).

^e2 M HNO₃ digestion at 100 °C and AAS.

^f2 M HNO₃ digestion at 100 °C and ICP.

tion process is commonly accelerated by incubation and mineralizable-N is measured.

In this study, leaching and laboratory incubation procedures⁹⁻¹¹ have been employed before waste disposal to land, to predict water soluble nutrients and toxic elements.

MATERIAL AND METHODS

One sewage sludge, two kinds of fly ash and one soil were used in this experiment.

The sewage sludge (SS) from a domestic source (L'Estartit) on Catalonia, was digested and sand bed dried before being used. Two types of nonweathered fly ash from the combustion of coal in electric power plants were employed, one from Alcudia (Mallorca-MA) and the other from Andorra (Teruel-TE). The soil (S) was sampled from a silty loam Ap-horizon of an Haploxeroll. Sewage sludge and soil were air dried and sieved (≤ 2 mm) before analysis. The chemical composition, the physicochemical properties and the analytical methods employed¹² are shown in Table 1.

The soil was amended with both wastes. Each fly ash was applied at two rates: 50 and 500 t ha⁻¹, also 20 t ha⁻¹ of sewage sludge were incorporated. Two controls were established (soil-S and soil plus sewage sludge-SS) and ten treatments were carried out. For each treatment three replications were done.

The samples (30 g for each treatment) were placed in leaching tubes (18 cm height and 3 cm of diameter) and incubated at 30 °C with a moisture content corresponding to a suction of 34 KPa. Initially and weekly during five weeks the mixtures were leached with 150 mL of water and the moisture content was adjusted by suction.

To quantify the mineralizable N along the incubation period, the soluble N compounds ($\text{NO}_3\text{-N}$ and $\text{NH}_4\text{-N}$) were determined in the percolate. Several water soluble elements, nutrients or toxic (Ca, Mg, K, Na, B, Cd, Mo, Se, Zn), pH and salts were also determined in the percolate. Soluble salts were measured as electrical conductivity and the water soluble compounds released were quantified according to the analytical methods reported in Table 1. All the results are expressed on an oven-dry basis. Data were analyzed by using an analysis of variance with repeated measures.^{1,3}

RESULTS AND DISCUSSION

The soluble compounds determined after water leaching are indicated in Tables 2 and 3. The first columns show the amount released before incubation, it means the initially soluble compounds for each treatment. The second columns show the cumulative amount released, which includes initial and successive five-weeks incubation period. For pH, only initial and final values are reported.

The values of $\text{NH}_4\text{-N}$, Cd, Se and Zn are not indicated because the quantified amount was lower in most cases than the detection level.

In general it can be said that the sewage sludge applied at 20 t ha⁻¹ did not significantly increase the soluble compounds released at any time, excluding obviously $\text{NO}_3\text{-N}$.

The application of fly ash significantly increased the Ca, B and Mo recovered in the percolate, the differences being greater for the highest rates of fly ash applied. For the other soluble elements (Na, K and Mg), the recovered amount had not an uniform behaviour. The amount released by leaching depended not only on the rate but also on the fly ash composition as it can be seen in Table 1.

Boron is considered to be one of the limiting factors for agronomic success of fly ash recycling.^{1,5,8} The B concentration in the percolate does not exceed the plant toxicity threshold—100 mg kg⁻¹ in plant tissue¹⁴ for most agronomic crops.

The physico-chemical properties of percolate change along the experiment according to the soluble compounds released. The total soluble salts are greatly enhanced by fly ash application,^{1,7} the differences being significant at higher rates. The soil salinity seems to be only a problem when 500 t ha⁻¹ of fly ash are recycled, because the cumulative electrical conductivity could reach toxic values for plants. Concerning pH it can be said that fly ash increases significantly the initial values only for the 500 t ha⁻¹ MA treatments. During the incubation period, pH is slowly recuperated and the differences are only significant at the higher rates.

The soil N-mineralization, evaluated as the leached N compounds ($\text{NO}_3\text{-N}$ and $\text{NH}_4\text{-N}$), is not affected by fly ash treatments. Cervelli *et al.*¹⁵ demonstrated that

Table 2 Chemical composition (soluble cations and salts) of the leachates before and after laboratory incubation.

	<i>Ca</i> (mg kg^{-1})		<i>Mg</i> (mg kg^{-1})		<i>Na</i> (mg kg^{-1})		<i>K</i> (mg kg^{-1})		<i>Salts</i> (mg.l^{-1})	
	<i>Initial</i>	<i>Total released</i>	<i>Initial</i>	<i>Total released</i>	<i>Initial</i>	<i>Total released</i>	<i>Initial</i>	<i>Total released</i>	<i>Initial</i>	<i>Total released</i>
Control S	81 a	254 a	5 b	26 b	155 d	244 c	30 b	81 ab	164 a	409 a
S-50 MA	187 b	694 d	4 b	33 c	127 bc	238 c	18 ab	86 b	206 a	740 c
S-50 TE	87 a	267 a	6 b	26 b	106 b	205 b	19 ab	67 a	159 a	425 a
S-500 MA	446 d	1222 h	0.3 a	2 a	80 a	163 a	16 a	90 bc	1071 c	2222 f
S-500 TE	191 b	1031 f	12 d	59 f	160 d	248 c	25 ab	97 c	298 b	1052 e
Control SS	97 a	346 b	8 cd	42 d	162 d	284 d	26 ab	82 b	202 a	555 b
S-50 MA-SS	184 b	807 e	7 c	49 e	177 d	295 d	23 ab	102 c	271 b	892 cd
S-50 TE-SS	99 a	440 c	7 c	44 d	160 d	278 d	23 ab	78 ab	200 a	584 b
S-500 MA-SS	673 e	1702 i	0.4 a	2 a	143 c	262 c	23 ab	144 d	1391 c	2710 f
S-500 TE-SS	379 c	1080 g	26 e	76 g	78 a	162 a	17 a	80 ab	276 b	988 d

Values followed by the same letter are not different at the 99% level of probability. Comparison of means were made for each column.

Table 3 Chemical composition (pH, Mo, B and NO₃-N) of the leachates before and after laboratory incubation

	pH		Mo ($\mu\text{g kg}^{-1}$)		B ($\mu\text{g kg}^{-1}$)		NO ₃ -N (mg kg^{-1})	
	Initial	Final	Initial	Total released	Initial	Total released	Initial	Total released
	Control S	7.8 ab	6.9 a	nd	nd	157 a	957 a	32 a
S-50 MA	10.4 e	7.3 ab	87 a	319 a	331 ab	1712 b	38 bc	134 b
S-50 TE	8.4 c	7.0 a	nd	nd	481 ab	2215 bc	34 ab	128 b
S-500 MA	11.9 f	10.5 d	318 c	3074 d	592 b	1799 b	38 bc	99 a
S-500 TE	8.8 d	8.0 c	214 b	792 b	2977 c	20704 e	39 bc	127 b
Control SS	8.3 c	7.2 ab	nd	nd	185 ab	1117 a	38 bc	210 e
S-50 MA-SS	7.5 a	7.5 b	125 ab	366 a	379 ab	1875 b	37 bc	196 d
S-50 TE-SS	8.6 cd	7.3 ab	nd	nd	344 ab	2452 c	35 ab	204 de
S-500 MA-SS	11.9 f	10.6 d	421 c	3715 e	631 b	3440 d	36 abc	109 a
S-500 TE-SS	8.1 bc	8.0 c	540 d	920 c	4344 d	20527 e	42 c	170 c

nd = Not detectable.

Values followed by the same letter are not different at the 99% level of probability. Comparison of means were made for each column.

from 60 t ha⁻¹ of fly ash the N-mineralization was inhibited. When sewage sludge is added, the NO₃-N released increases, but with fly ash addition N-mineralization is severely diminished especially by MA. It seems due to the high soluble elements content in fly ash that interacts with sludge. Because soluble NH₄-N was only quantified at higher rates of MA-fly ash, it is possible to presume that in this case nitrification was inhibited but ammonification was not.¹⁶

If the data were analyzed as a released percentage of the total apported, it could be observed that at least 50% of soluble compounds are released at the initial leaching, except NO₃-N which is gradually liberated during incubation period. During successive leaching periods, soluble compounds are also liberated in small proportions. It is possible to interpret that soil adsorbs fly ash ions. Consequently, to reduce environmental associated problems and to assure gradual N-mineralization of sewage sludge, it would be better to eliminate soluble compounds by water leaching before soil fly ash recycling.

CONCLUSIONS

According to these results it can be concluded that fly ash disposal to soil enhances the amount of soluble compounds in soil solution when 500 t ha⁻¹ rates are applied; thus, the soil salinity could be a limiting factor for plant development. The release of soluble compounds depends on the loading rates and fly ash chemical composition.

The fly ash pH is high enough to increase soil pH. It minimizes solubilization of heavy metal compounds. In acid soils, fly ash could be used as a soil amendment.^{1,7}

When fly ash and sewage sludge are co-recycled, the soluble elements released are similar to fly ash treatments, and N-mineralization is inhibited probably due to

the content of soluble salts in soil solution since soluble toxic elements do not increase.

From the agronomic point of view this experiment demonstrates that those fly ash can be recycled through soil at lower rates to avoid soil salinity increases. Environmental and food chain hazards would not be expected.

Water soluble compounds percolated by successive extractions along incubation could be a useful method to estimate both soluble elements and mineralized-N compounds, avoiding undesirable land consequences of waste recycling. This method represents also field conditions because soluble compounds are gradually eliminated from soil. It is an easy and quick procedure and inexpensive to perform.

Obviously, to know the fly ash long term effects on soil, field experiments should be done using washed and nonwashed fly ashes.

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