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Sawdust and natural zeolite as a bulking agent for improving quality of a composting product from anaerobically stabilized sewage sludge

Antonis A. Zorpas [a,*], Maria Loizidou [b]

[a] *ENVITECH Ltd., Laboratory of Environmental Technology (Research Institute), Griva Digeni 37, P.O. Box 34073, 5309 Paralimni, Cyprus*
[b] *National Technical University of Athens, Department of Chemical Engineering, Laboratory of Environmental Science 9, Heroon Polytechniou Street, Zographou 157 00, Greece*

Abstract

This study has dealt with the production of compost from dewatered anaerobically stabilized primary sewage sludge (DASPSS) and sawdust (SWD). SWD is added in order to increase the humic substances in the final product. The DASPSS is mixed with clinoptilolite (Cli), which is used as a bulking agent at 20% w/w, and the mixture is amended with sawdust at 10%, 30% and 40% (w/w). The final results have indicated that by increasing the sawdust concentration in the initial mixture, the humic substances in the final product increase too. The natural zeolite that was added in the initial mixture takes up a significant amount of heavy metals. In order to observe the maturity of the final product, the germination index is used in oat cultivation. The results indicate that the substrate appears to be non-phytotoxic after 75 d of maturity. Also, in order to estimate the metal leachability of the final compost product, the generalized acid neutralization capacity procedure is applied, and it is found that by increasing the pH values, the heavy metal concentrations decrease.
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Keywords: Composting; Zeolite; Leach ability; Partitioning; Phytotoxic

1. Introduction

The mineralization of biogenic substances is a part of the natural recycling process, which occurs at any place where organic material is synthesized by plants and degraded by animals and by microflora. This mechanism keeps the global balance upright. Environmental problems associated with sewage sludge disposal have prompted strict legislative actions over the last years. At the same time, the upgrading and expansion of wastewater treatment plants have greatly increased the volume of produced sludge. The sludge is classified as solid waste that requires special methods of disposal, because of its noxious properties. However, much of the sludge originating from urban wastewater treatment is contaminated with heavy metals (Wozniak and Huang, 1982; Hasit and Christensen, 1987; Sims and Skline, 1991; Langenbach et al., 1994; Karvelas et al., 2003; Wei and Liu, 2005). These metals may leach from sludge and enter the ecosystem, the food chain and finally the human body. Also, the total concentration of heavy metals cannot provide useful information about the risk of bioavailability, toxicity and capacity for remobilization of heavy metals in environment (Fernandez et al., 2000; Kunito et al., 2001; Liu et al., 2007).

The most common treatment methods of sludge is landfill, composting, incineration and the agricultural use. All these methods present tend to have some potential environmental impacts. Landfill presents to have emissions of CH_4, CO_2, odours and also produces leaching of salts, heavy metals and persistent organics to ground water. Also landfill and the uncontrolled use of sludge in agriculture accumulate the hazardous substances in soil and the toxic substances in food chain. Incineration is a very promising treatment method due to that fact that the volume of the

* Corresponding author. Tel.: +357 23743440; fax: +357 23743441.
  E-mail address: antoniszorpas@yahoo.com (A.A. Zorpas).

raw material is limited, however, it presents with environmental impacts like emission of SO_2, NO_x, CO_x, dioxins, and heavy metals if the incineration is not controlled.

In the greater region of Athens, with almost 4,500,000 citizens, the main wastewater treatment plant is located at the rock-island of Psittalia. At this plant, $\sim$750,000 $m^3\,d^{-1}$ of mainly municipal wastewater along with industrial wastes is subjected to biological treatment, producing approximately 250 ton d^{-1} of dewatered anaerobically stabilized primary sewage sludge (DASPSS) (Zorpas et al., 1998). Landfill is the main practice for the management of the sewage sludge, and this fact generates potential environmental risks such as the production of odors and methane gas, and the contamination of groundwater by the leachate produced in landfill site (Zorpas et al., 1997). Composting provides a simple and cost-effective alternative treatment method for sewage sludge by decomposing organic matter, producing a stabilized residue and disinfecting pathogens (Fang et al., 1999). The composting method is parallel with the landfill directive (1999/31/EC), which requires the minimization of biodegradable waste in landfills. The composted product can also be used as a fertilizer or soil conditioner because of its large content of stabilized organic matter. However, the high content of heavy metals in sewage sludge compost has proven to be a limiting factor in the land application of sewage sludge compost (Wong et al., 1997). The addition of natural zeolite, clinoptilolite, during sewage sludge composting has been proven to be a promising way to reduce the heavy metals content (Zorpas et al., 2000), since zeolite (clinoptilolite) has the ability to take up heavy metals. Zeolite utilization had become popular in the last decade, due to its cation exchange and molecular sieving properties (Ouiki and Kavannagh, 1997; Zorpas, 1999).

According to European Union the use of sewage sludge in agriculture should be in such a way as to prevent harmful effects on soil, vegetation, animals and man, thereby encouraging the correct use of such sewage sludge (directive 86/278). According to the directive 86/278 the member States shall prohibit the use of sludge where the concentration of one or more heavy metals in the soil exceeds the limit values which they lay down in accordance with Annex IA and shall take the necessary steps to ensure that those limit values are not exceeded as a result of the use of sludge.

The aim of this work is to present the effect of natural zeolite and sawdust (SWD) in the maturity of the co-composting product with DASPSS and the phytotoxicity of the final product in oat cultivation.

2. Methods

The DASPSS samples were collected for a period of six months (April–September: 200–250 kg every two days from the Psittalia wastewater treatment plant. Then the samples were dried, homogenized and stored. Natural zeolite, clinoptilolite (Cli), used as a bulking agent, was col-

Table 1
Composition characteristics of DASPSS, Cli and SWD

Parameters	Mean value of 20 samples		
	DASPSS	SWD	Cli
Moisture %	70.10 ± 2.01	12.00 ± 2.15	7.15 ± 0.50
pH	7.05	8.00	7.85
Conductivity mS cm^{-1}	1.005 ± 0.005	1.101 ± 0.010	0.155 ± 0.002
Organic matter %	45.05 ± 1.05	90.15 ± 2.21	Not detected
Ash %	27.23 ± 1.10	5.03 ± 1.25	80.65 ± 1.50
TOC %	26.10 ± 0.50	55.05 ± 2.35	not detected
TKN %	1.90 ± 0.20	1.80 ± 0.20	0.032 ± 0.002
N-NH_4 mg g^{-1} dry samples	9.57 ± 0.50	19.73 ± 0.70	1.40 ± 0.15
P-PO_4 %	2.45 ± 0.25	2.60 ± 0.25	Not detected
Humic substances %	1.80 ± 0.05	9.50 ± 0.50	Not detected
E4/E6	1.25 ± 0.01	1.38 ± 0.01	Not detected
Lignin %	4.50 ± 0.10	30.50 ± 0.50	Not detected
Cellulose %	2.06 ± 0.05	52.10 ± 1.00	Not detected
C/N	13.73 ± 0.50	30.55 ± 1.00	Not detected
C/P	10.65 ± 0.50	21.15 ± 1.00	Not detected
Germination index	17 ± 2	63 ± 2	75 ± 5

Significant difference at $p < 0.05$.

lected from Evros (Region in North Greece). Selected composition characteristics of the materials used (Zorpas, 1999) are given in Table 1.

The composting process was carried out in the laboratory using an in-vessel reactor of 1 m^3 active volume (Finstein et al., 1992). The thermophilic phase in the reactor lasted for 15 d. The temperature in the center of the reactor was about 60–65 °C and the moisture percentage was between 40 and 50%. The samples were aerated using an aerated air force (oxygen concentration range in the reactor was between 5 and 8%). A temperature indicator controller was controlling the operation of the fan in order to maintain the temperature at about 60 °C, according to the following principle: minimum air flow (2.3 m^3 per m^3 active volume) was provided at low temperature (<30 °C) and maximum air flow (28 m^3 per m^3 active volume) was provided at high temperature (>60 °C). The minimum airflow corresponds to the minimum oxygen demand for the microorganisms and the maximum airflow corresponds to the necessary air for cooling. After the thermophilic period, in which the organic material was biodegraded, the compost was piled to an enclosed package where it remained for about four months to mature (periodically this was stirred). Also it is very important to know that there was not any leachate during the composting processes. The reason is that the reactors at the first step of composting were closed and during the thermophilic phase the contents were evaporated.

The ratio of the materials used for the preparation of the samples for co-composting is

SSC1: 80% w/w (70% w/w DASPSS + 10% w/w SWD) plus 20% w/w Cli.
SSC2: 80% w/w (50% w/w DASPSS + 30% w/w SWD) plus 20% w/w Cli.

SSC3: 80% w/w (40% w/w DASPSS + 40% w/w SWD) plus 20% w/w Cli.
So: 100% w/w DASPSS.

For all the raw materials and prepared samples for composting, the following parameters were measured: pH value, electrical conductivity (EC), total organic carbon (TOC), organic matter (OM), total phosphorous (PO_4-P), total kjeldahl nitrogen (TKN) and ammonia content, C/N ratio, and heavy metals content (Cd, Cr, Cu, Fe, Mn, Ni, Zn and Pb) (APHA, 1985; Adams, 1990; Zorpas et al., 1998). Humic substances were extracted according to the following scheme (Schnitzer, 1982): 2.5 g of sample was shaken for 24 h at ambient temperature using 100 mL of NaOH 0.5 N. Suspensions were filtered through Whatman No. 1 filter paper. Then the filtrates were dried at 80 °C.

Seed germination and root length tests were performed on water extracts obtained by mixing 10 g of each sample with 20 mL of distilled water, for 2 h. Suspensions were centrifuged at 5000 rpm for 30 min before being filtered through Whatman No. 1 filter paper. Ten milliliter of each test solution was pipetted into a sterilized plastic petri-dish. Also, one blank with only double-distilled water was carried out. Twenty oat seeds were placed in each petri-dish and incubated at 22 °C, 50% relative humidity, in the dark for 7 d, % grow index G. The germination index (GI) was calculated according to the following formula (Tiquia and Tam, 1998):

$$GI = \frac{(\%G)(\%L)}{100} \quad \text{where } \%G = \text{Grow index} \quad (1)$$

$$\%L = \text{Root Length} = L = \frac{\text{RootLength of Treatment}}{\text{RootLenght of Control}} * 100 \quad (2)$$

In order to estimate the metal leachability of the final product of compost, the generalized acid neutralization capacity procedure was used as described by others (Zorpas et al., 1998; Zorpas et al., 1999). The generalized acid neutralization capacity test procedure was used in the estimation of the leach ability of metals from the sludge samples (Isenburg and Moore, 1992). This test is a single batch procedure that utilizes a series of sludge samples extracted with increasingly acidic leachant.

Finally, the chemical forms of heavy metals (metals speciation) were determined by the use of the sequential extraction procedure of Tessier et al. (1979), with some modifications by others (Zorpas, 1999; Zorpas et al., 2000). According to the procedure, heavy metals are associated with five fractions: Exchangeable, carbonate, reducible, organic fraction and residual fraction.

All the results obtained from the measurements and chemical analyses are shown in Tables 2 and 3, and Figs.

Table 2
Compost characteristics

	SSC1	SD	SSC2	SD	SSC3	SD
Moisture %	27.20	1.15	32.06	1.25	31.50	1.15
pH	7.01	0.05	7.48	0.05	7.25	0.05
Conductivity mS cm^{-1}	1.001	0.005	1.128	0.015	1.450	0.020
OM %	32.81	1.50	36.23	1.50	48.23	1.75
TOC %	19.03	1.50	21.01	1.60	22.17	1.60
TKN %	1.65	0.05	1.61	0.05	1.89	0.05
P-PO$_4$ %	2.45	0.25	2.48	0.25	2.35	0.25
N-NH$_4$ mg g^{-1}	7.35	0.25	7.90	0.25	9.28	0.25
Total humics %	3.95	0.50	7.50	0.50	12.05	0.50
C/N	9.53	0.50	11.04	0.75	11.73	0.62

SD: standard deviation of 20 samples, significant difference at $p < 0.05$.

Table 3
Metals concentration in raw materials and in the final products

Metals (mg/g)	DASPSS	SWD	Cli	SSC1[a]	SSC2[a]	SSC3[a]	86/278/EU directive
Cd	0.002	Not detected	Not detected	Not detected	Not detected	Not detected	0.02–0.04
Cu	0.258	0.002	Not detected	0.242	0.234	0.221	1.00–1.75
Cr	0.552	Not detected	Not detected	0.518	0.502	0.474	
Fe	5.098	1.004	1.901	4.102	3.974	3.574	
Mn	0.150	0.006	0.015	0.139	0.135	0.132	
Ni	0.041	Not detected	Not detected	0.032	0.024	0.023	0.3–0.4
Pb	0.326	0.002	0.110	0.306	0.296	0.281	0.75–1.2
Zn	1.739	0.003	0.005	1.634	1.581	1.394	2.5–4.0
Na	0.724	0.012	1.786	2.012	2.025	2.024	
K	0.723	0.014	5.493	6.102	6.107	6.112	

Significant difference at $p < 0.05$. All metals in mg g^{-1} dry samples.
[a] Zeolite had been removed from the final products (the zeolites were manually removed from the final products using sieving. The diameters of the sieve were ⩽2.3 mm).
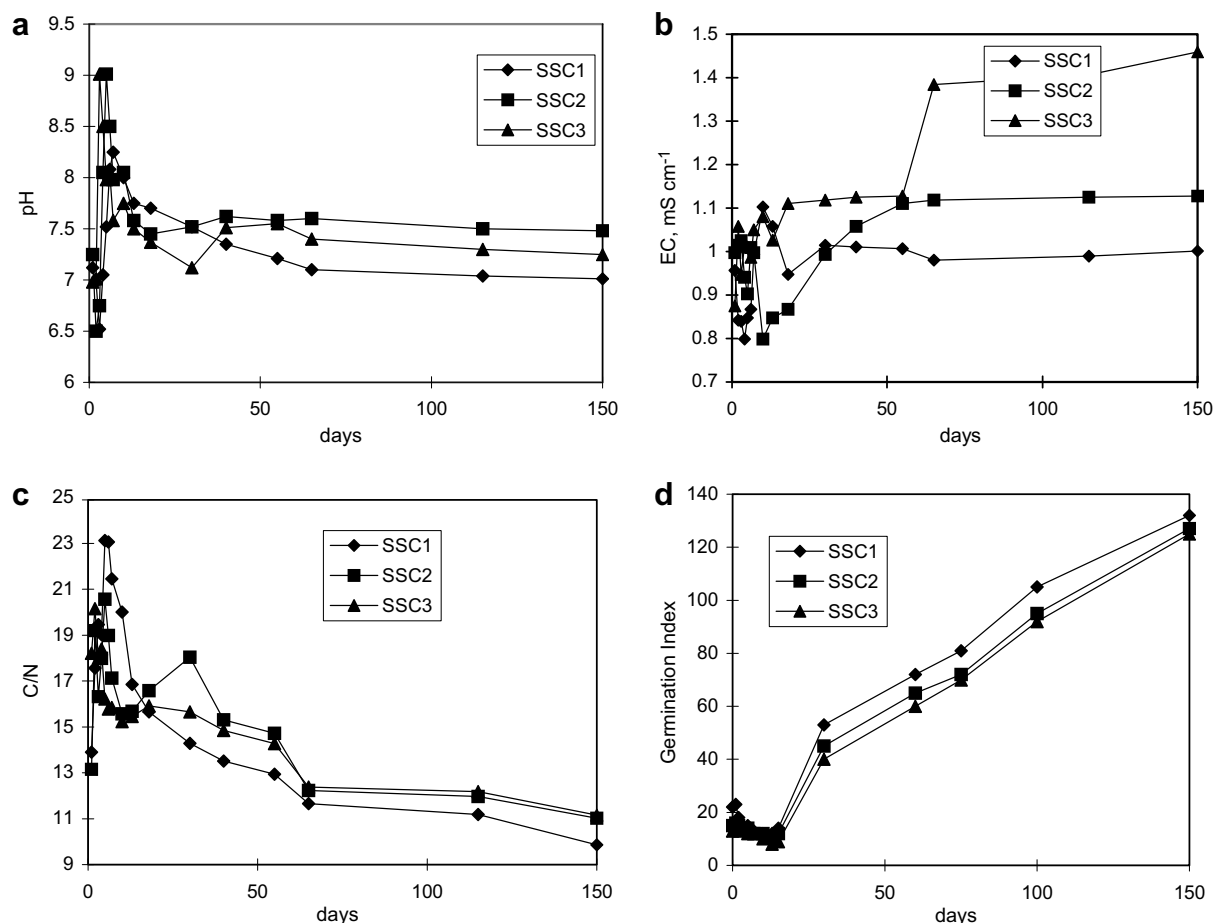
Fig. 1. pH, electronic conductivity and C/N values during the composting process and germination index in oats cultivation.

1–4. Also it is very important to mention that zeolite has been removed from the final products before any chemical analysis (the zeolites were manually removed from the final products using sieving. The diameters of the sieve were ⩽2.3 mm).

For the data statistical analysis, Taylor's techniques have been applied (Taylor, 1990).

3. Results and discussion

As it is observed from Table 1, sewage sludge contains a lower concentration of organic matter and organic carbon (45.05% and 26.10%, respectively) than the SWD (90.15% OM and 55.05% TOC). The pH values of DASPSS and SWD samples were about 7 and 8, respectively. The phosphorous (P-PO$_4$) content was found in high levels due to the fact that sludge was produced mainly from the treatment of municipal wastewater. The total humics in DASPSS were found in lower levels (1.80%) than in the SWD (11.50%). The E4/E6 reflects the presence of humic acid or fulfic acids. As the E4/E6 ratio is below five, the samples are characterized as humic acid, whereas if the ratio is above five the sample is characterized as fulvic acid (Zorpas et al., 1998). The DASPSS contains high amounts of

organic constituents. However, if the sludge is used for the production of compost, the organic amount is considered to be low. (This is because at the final stage it is better to have C/N ratio close to 14:1 and organic matter >35%).

Table 2 shows the results for the characterization of the final product after 150 d of maturity. The use of SDW in the composting process increases the humic substances in the final product. The initial total humic substances in the three samples were 2.21, 3.12 and 4.51, (SSC1, SSC2 and SSC3), respectively, while their concentrations in the final products were 3.95%, 7.50% and 11.73%, (SSC1, SSC2 and SSC3), respectively. Moreover it is observed that by increasing the sawdust amount added in the initial samples mixture the humic substances in the final product increases, too.

Fig. 1a and b, presents the changes in pH, the changes in electrical conductivity (EC) and the changes in C/N (Fig. 1c) during the development of the process. It is obvious that during the first day pH increased to nine (due to the presence of ammonia) (Zorpas, 1999), and then decreased gradually to seven. The decrease in pH value may be due to the formation of organic acids during the decomposition of organic matter (Verdonck, 1988; Zorpas, 1999). EC is usually measured during the composting pro-
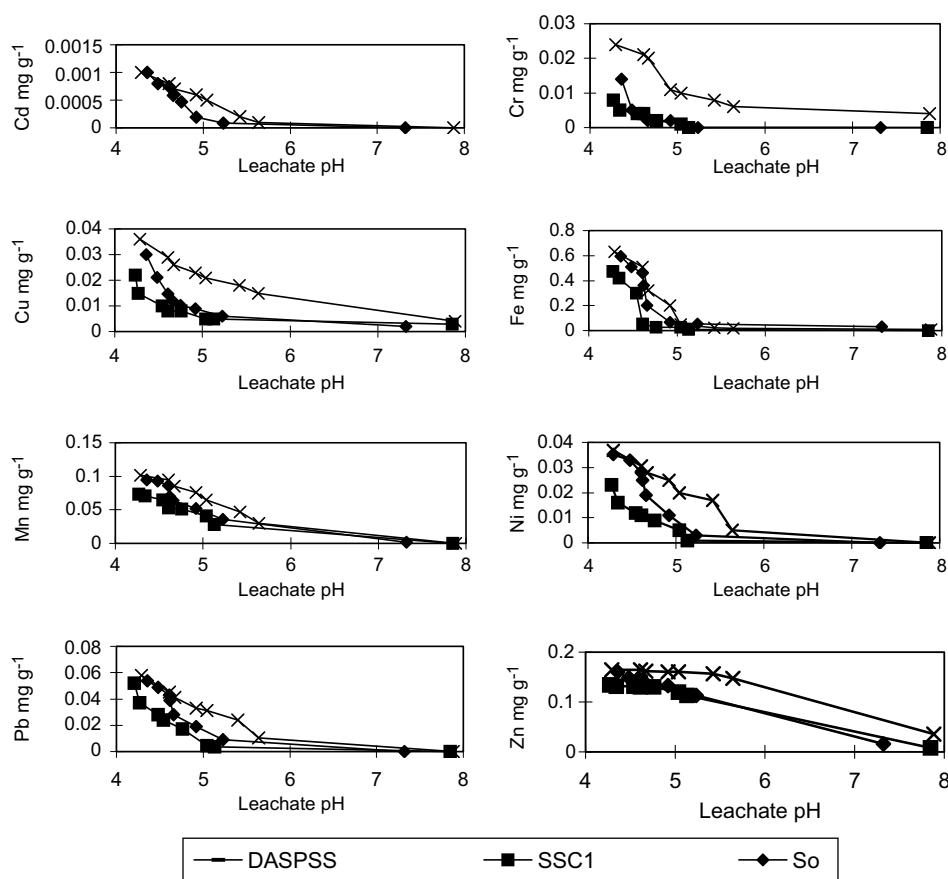
Fig. 2. Metals release to leachates from DASPSS, So and SSC1 with clinoptilolite compost as a function of pH.

cess because it reflects the salinity of the composting products and their suitability for plant growth. Electrical conductivity increased from 0.957 to 1.001 mS cm^{-1} for SSC1, 0.998–1.128 mS cm^{-1} for SSC2 and 0.875–1.459 for SSC3. It is observed that the zeolite does not affect the changes in pH and EC. However, both pH and EC reach relatively constant values after approximately 75 d. This indicates that the compost samples start to become stabilized and matured.

C/N ratio is used for the determination of compost maturity. The C/N ratio (Fig. 1c) decreased from 14 to 10 for SSC1, from 13 to 11 for SSC2 and from 18 to 12 for SSC3. Compost with a C/N between 9 and 10 was considered as mature (Bernal et al., 1998; Zorpas, 1999). The C/N ratio is considered relatively constant after 75 d (approximately). This indicates the maturity of the final products.

Table 3 presents the metals concentration in the raw materials and in the final products. As it is observed Cli takes up a significant amount of heavy metals during the composting process. In particular Cli seems to take up 100% of Cd in all samples, 6–14% of Cu, Cr and Pb, 19–30% of Fe, 7–12% of Mn, 24–43% of Ni, 6–19% of Zn, while at the same time the concentration of sodium and potassium seems to increase.

Fig. 1d also presents the germination index in oat cultivation. According to Zorpas (Zorpas, 1999), if germination

index is between 0 and 26 the substrate is characterized as very phytotoxic, if germination index is between 27 and 65 the substrate is characterized phytotoxic, if germination index is between 66 and 100 the substrate is characterized non-phytotoxic, while if the germination index >101 the substrate is characterized phyto-nourishing. DASPSS has germination index = 17 ± 2 which is characterized as very phytotoxic (Table 1) and is not suitable for agricultural usage. SWD has germination index = 63 ± 2 while clinoptilolite has germination index = 75 ± 5 and as it is observed it does not affect the phytotoxicity. The appearance of the phytotoxicity in the first step of the composting process has to do with the decomposition of the organic matter. The unstable composts seem to have high phytotoxicity due to the fact that phytotoxic compounds are produced by microorganisms (Bernal et al., 1998). Fig. 1d indicates that the substrate is considered to be non-phytotoxic after 75 d of maturity while phyto-nourishing is present after 110 d of maturity. Also, in Fig. 1d it is clarified that the SSC1, SSC2 and SSC3 have similar results, which means that SWD does not affect the germination index.

The generalized acid neutralization capacity test and the sequential chemical extraction were applied in the DASPSS, without clinoptilolite addition (So) and with cured compost (SSC1).

Fig. 2 shows the metal leach ability in the DASPSS, So and SSC1 cured compost. It has been observed that by
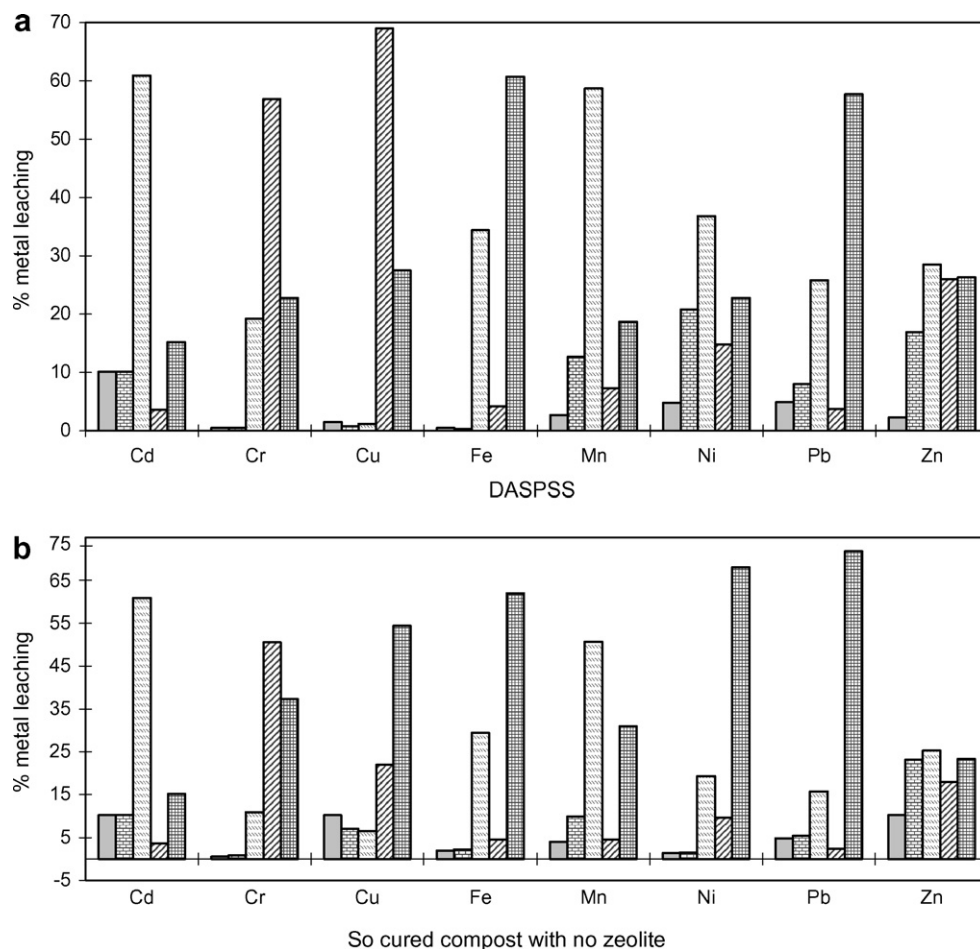
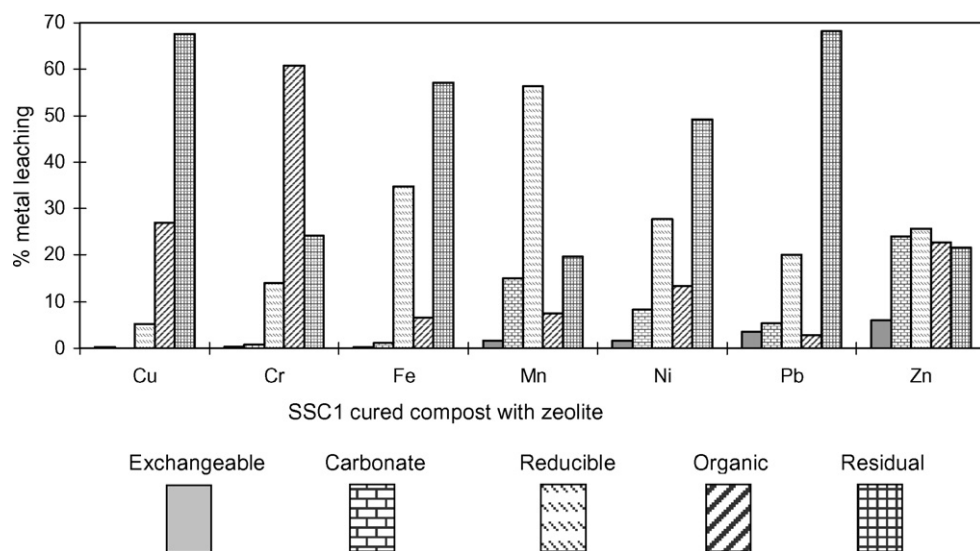Fig. 3. % Metals partitioning in DASPSS and in sewage sludge cured compost.

Fig. 4. % Metals partitioning in cured compost.

increasing the leachate pH, the heavy metal concentration decreased.

The metal partitioning in the DASPSS sample as determined by sequential chemical extraction is presented in Fig. 3a. A significant amount of Cr and Cu was bound to the organic and residual fractions with <2% in the exchangeable and carbonate fractions. Almost 60% of Cd and Mn was bound to the reducible fraction. Iron and lead were

found to be bound in the reducible and residual fractions (about 95% for the Fe and 83% for the Pb). Only Ni and Zn were found in substantial proportions in all phases (very little in exchangeable phase). Comparing the results with studies carried out earlier (Angelidis and Gibbs, 1988; Rudd et al., 1988; Brennan, 1991; Garcia-Delgado et al., 1994), chromium and copper were found in similar proportions in all fractions while iron was mainly associated with the residual and reducible ones. Nickel was found in substantial proportions in all phases, whereas zinc was mostly found in the organic and reducible fractions. Lead in the reducible, organic and residual fractions was over 90%.

Comparing the results in Fig. 3a and b, it was found that Cd, Fe and Zn were not affected during the composting process. Additionally, Cr seemed to be transformed from the reducible and the organic fraction to the residual fraction. The organic fraction of Cu was found to change to other fractions, especially residual and exchangeable. The residual fraction increased from 27.53% to 45.37% and exchangeable from 1.55% to 10.22%. Approximately 72% of Pb was found to be bound in the residual fraction. A substantial percentage of Mn was removed from the reducible, carbonate and organic fractions and transferred to the residual fraction. The changes observed in the metal partitioning of the sewage sludge compost were a result of the following:

- The thermophilic phase of composting, which was the first step in composting and which affected the exchangeable fraction. During the first step in composting changes in pH (acetic acid formation) and in ammonia content (affected by the action of the proteolytic bacteria and by the temperature) occurred (Wagner et al., 1990; Zorpas, 1999).
- The pH changes, which occurred at the beginning of the composting process, affected the exchangeable and carbonate fractions. The pH changes may be due to acid formation during the decomposition of organic matter contained in the sludge (Kapetanios et al., 1993; Zorpas et al., 1999; Zorpas, 1999).
- The oxic and anoxic conditions (produced by acetic acid and ammonia) at the first step of composting (Wagner et al., 1990) affected the reducible and organic fractions.

Copper in the SSC1 cured compost was found to be bound in the residual fraction at 47–67% (Fig. 4). Chromium in the residual fractions was about 32%. A substantial amount (63%) of Cr was found in the organic fraction. Iron was found to be bound in the residual fraction at 67%. The other three fractions contained <8%, most of which was bound in the organic fraction. Seventy five percent of Ni was bound in the residual and in the organic fraction, while Mn was found in the reducible fraction at 48% and in the residual at 35%. In the case of Zn all fractions had substantial proportions of the metal, while lead was found to be bound in the residual and reducible fractions at almost 90%. It was observed that clinoptilolite had the ability to

readily take up almost all metals that were bound to the exchangeable and the carbonate fractions. Clinoptilolite took up all metals bound in exchangeable and carbonate fractions according to the following selectivity series: Cu > Cr > Fe > Ni > Mn > Pb > Zn.

4. Conclusions

The DASPSS contains low concentration of humic substances (1.80%). The final compost product appears to have 3.95%, 7.50% and 11.73% (SSC1, SSC2 and SSC3, respectively) of humic substances. All compost samples appear to have germination index almost 80, after 75 d, a fact that indicates non-phytotoxicity. Therefore, all samples reached maturation after 75 d, which was in accordance with the results obtained for C/N, pH and EC values.

Clinoptilolite takes up a significant amount of heavy metals. Using the generalized acid neutralization capacity procedure, it was found that by increasing the leachate pH, the heavy metal concentration decreased. Finally, using a sequential extraction procedure in DASPSS and final products (compost after 150 d of maturity) it was found that a significant amount of sludge metals was bound in the residual fraction, in an inert form. Clinoptilolite most readily took up the metal content bound in the exchangeable and carbonate fractions.

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