



Waste paper and clinoptilolite as a bulking material with dewatered anaerobically stabilized primary sewage sludge (DASPSS) for compost production

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Abstract

Environmental problems associated with sewage sludge disposal have prompted strict legislative actions over the past few years. At the same time, the upgrading and expansion of wastewater treatment plants have greatly increased the volume of sludge generated. The major limitation of land application of sewage sludge compost is the potential for high heavy metal content in relation to the metal content of the original sludge. Composting of sewage sludge with natural zeolite (clinoptilolite) can enhance its quality and suitability for agricultural use. However, the dewatered anaerobically stabilized primary sewage sludge (DASPSS) contained a low concentration of humic substances (almost 2%), and the addition of the waste paper was necessary in order to produce a good soil conditioner with high concentrations of humics. The final results showed that the compost produced from DASPSS and 40–50% w/w of waste paper was a good soil fertilizer. Finally, in order to estimate the metal leachability of the final compost product, the generalized acid neutralization Capacity (GANC) procedure was used, and it was found that by increasing the leachate pH, the heavy metal concentration decreased. The application of the sequential chemical extraction indicated that metals were bound to the residual fraction characterized as a stabilize fractions.

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1. Introduction

In the greater region of Athens, with almost 4,500,000 citizens, the main wastewater treatment plant is on the rock-island of Psittalia. At Psittalia, approximately 750,000 m³ day⁻¹ of mainly municipal wastewater along with industrial wastes are subjected to primary treatment, producing approximately 250 t day⁻¹ of dewatered anaerobically stabilized primary sewage sludge (DASPSS; Zorpas et al., 1998). Also in the same region 4000 t day⁻¹ (0.9 kg/person day) of municipal solid waste is produced approximately. Among them, organic fraction is 50%, waste paper is 23%, plastics is 10%, metals is 5% and finally 13% includes wood, glasses, leather and textiles (Zorpas et al., 1997, 1998).

Until now, landfilling is the main disposal method for sewage sludge in Athens, generating potential environmental hazards, including the production of odour and methane gas, as well as the contamination of groundwater by the leachate produced (Zorpas et al., 1997, 1998).

A number of various sludge treatment methods have been recently employed, and these can be divided into physico-chemical, biological and physical methods (Vlyssides et al., 1996). The use of sewage sludge in agriculture is not new, since it is an inexpensive source of organic matter and its use contributes to solving a serious environmental problem (Morenom et al., 1996). However, there are certain risks involved in its use, some of which, such as those derived from the instability of the organic matter or from the mineralizable organic nitrogen content, are only of transitory nature. Such problems are easily eliminated by composting, a process which stabilizes the organic matter content. One of the most serious problems in the application of sewage

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sludge in agriculture is the presence of the metals. Some of these are extremely toxic and when added to the soil along with the organic amendment, negatively affect plant development and even enter the food chain. Composting of sewage sludge with natural zeolite (clinoptilolite) can enhance its quality and suitability for agricultural use. Zeolites have been used world-wide for the last few decades, either for their cation exchange or molecular sieving properties. Natural zeolites nowadays are mostly used in catalysis, in air enrichment, as fillers in paper and rubber industry, in soil beneficiation, as animal feed supplements, and in water and wastewater treatment for the ammonia and heavy metal removal (Zorpas et al., 1997, 1999; Constantopoulou et al., 1994; Zorpas, 1999).

The objective of this study was to present the physico-chemical characteristics of the compost produced from DASPSS, waste paper (WP) and clinoptilolite as well as the leachability and the sequential chemical extraction of the final product.

2. Materials and methods

The DASPSS samples had been collected from the Psittalia wastewater treatment plant. The samples were dried, homogenized and stored, while the waste paper (WP; newspaper, cardboard, magazines paper, plastic paper, etc.) had been collected from the land filled area in Ano Liosia.

The samples were dried at room temperature and used in order to determine the following parameters: pH, conductivity, total organic carbon (TOC), organic matter (OM), total phosphorous (TP), total kjendahl nitrogen (TKN), ammonia, nitrite, nitrate, chlorite, boron, humic substances, lignin, cellulose, C/N ratio, C/P ratio, E4/E6 (absorbance in 415 and 615 nm) ratio and heavy metals (Cd, Cr, Cu, Fe, Mn, Ni, Zn and Pb).

The composting process was carried out in the laboratory using an in-vessel reactor of 1m³ active volume. There was an aeration system which was consisted with an air fan for air supply, an air flow meter, an air humidity meter. A temperature indicator controller was controlling the operation of the fan in order to maintain the temperature about 60 °C, according to the following principle: minimum air flow (2.3 m³ per m³ active volume) was provided at low temperature (<30 °C) and maximum air flow (28 m³ per m³ active volume) was provided at high temperature (>60 °C). The minimum air flow corresponds to the minimum oxygen demand for the micro-organisms and the maximum to the necessary air for cooling. The moisture was in the range of 40–50%. The thermophilic phase lasted for 15 days in the reactor. After the thermophilic period in which the organic material was biodegraded, the compost was piled in an enclosed package where it

Table 1
Samples prepared for composting (dry mass)

Sample	Waste Paper (WP.%)	DASPSS (%)
So	0	100
S1	5	95
S2	10	90
S3	20	80
S4	30	70
S5	40	60
S6	50	50

remained for about 4 months to mature (Zorpas et al., 1997; Vlyssides et al., 1996; Zorpas, 1999). Table 1 presents the samples prepared for composting with 40–50% moisture.

For the determination of conductivity, TOC, TKN and ammonia content, nitrite and nitrate, standard methods of analysis were used (APHA, AWWA-WPCF, 1985; Adams, 1990). pH values were determined according to methods of sampling and analysis of solid wastes using 10 g of each sample with 50 ml of distilled water for 2 h (Methods of Sampling and Analysis of Solid Wastes, 1970). TP was determined by stannous chloride method (APHA, AWWA-WRCF, 1985) while the OM by loss of ignitions in 450 °C according to Handbook of Reference Methods for Soil Analysis (1982). For the total metal concentration, a known quantity (1 g) of sample was digested with 10 ml of conc. HNO₃ as described by Zorpas et al. (1997). After the completion of the digestion, the samples were vacuum filtered and the filtrate was used for the determination of heavy metal concentration by atomic absorption spectroscopy, using a Perkin Elmer 2380 spectrophotometer. Humic substances were extracted using NaOH 0.1 N for 24 h according to Schintzer (1982), while lignin and cellulose were determined by a digestion technique with 72% of sulfuric acid as described by Zorpas et al. (1998). The optimal density (E4/E6) was carried out using NaHCO₃ in 415 and 615 nm.

Finally, in order to estimate the metal leachability of the final product of compost, the generalized acid neutralization capacity (GANC) procedure was used as described by Zorpas et al. (1998). The GANC test procedure was used in the estimation of the leachability 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.

The chemical forms of heavy metals were determined by the use of the sequential extraction procedure of Tessier et al. (1979), with some modifications according to Zorpas et al. (1997). According to the procedure, heavy metals are associated with five fractions: the

Table 2
Physicochemical characteristic of sewage sludge and organic fraction of municipal solid waste

Parameters	Mean value of 20 samples	
	DASPSS	WP
Moisture %	70.45±2.50	12.08±2.00
pH	7.05±0.05	8.00±0.05
Conductivity mS/cm	1.005±0.005	1.101±0.010
Organic matter% *	45.00±1.00	90.00±2.00
Ash % *	27.00±1.00	5.00±2.00
TOC % *	26.10±0.50	55.00±2.00
TKN % *	1.90±0.20	1.80±0.20
Cl ⁻ mg/g *	–	4.75±0.25
B mg/g *	–	–±
N–NH ₄ 10 ⁻¹ mg/g *	9.57±0.50	19.73±0.70
N–NO ₃ 10 ⁻¹ mg/g *	2.53±0.20	0.45±0.05
N–NO ₂ 10 ⁻⁶ mg/g *	258±25	2160±100
P–PO ₄ % *	2.45±0.05	2.60±0.05
Humic substances % *	1.80±0.05	9.50±0.50
E4/E6	1.25±0.01	1.38±0.01
Lignin % *	4.50±0.50	30.50±0.50
Cellulose % *	2.00±0.05	52.10±1.00
C/N	13.37±0.50	30.55±1.00
C/P	10.65±0.50	21.15±1.00

*, Dry basis; –, not detected.

exchangeable fraction, the carbonate fraction, the reducible fraction, the organic fraction and the residual fraction.

In order to take up the heavy metals from the compost, natural zeolite (clinoptilolite) was added in 20 and 25% w/w (Zorpas et al., 1999).

Scli5: 20% clinoptilolite + 80%
(50% DASPSS + 50% WP)
Scli6: 25% clinoptilolite + 75%
(50% DASPSS + 50% WP)

Taylor's statistical techniques were used for analysis of data, specifically the confidence interval 95% (CI) for a mean (Kapetanios et al., 1993).

3. Results and discussion

The physico-chemical characteristic of DASPSS samples and waste paper are presented in Table 2.

Sewage sludge contained lower concentration of organic matter and organic carbon (45 and 26.10%, respectively) than the WP (90% OM and 55% TOC). The pH values were about seven for DASPSS samples and about eight for the WP samples. The total phosphorous content was found in high levels due to the fact that the main load of the treated wastes were municipal. The total humics were found in lower levels (1.80%) in DASPSS than in the WP (11.50%). The E4/E6 ratio shows the characterization of humic materials. As the E4/E6 ratio was below 5 in DASPSS, the samples are characterized as humic acid (whereas if the ratio is above 5 the sample is characterized as fulvic acid; Zorpas, 1999; Schnitzer, 1982). The DASPSS contained high amounts of organic constituents. However, if the sludge was to be used for the production of compost, the organic amount was considered to be low. The DASPSS solid wastes were amenable for composting treatment due to the low C/N ratio. It was necessary to proceed with co-composting with the waste paper which contained a higher amount of organic matter, humic substances, C/N ratio, lignin and cellulose than the DASPSS. The fact that the nitrogen content of WP is low is probably due to the fact that the raw material represented from newspaper, plastic paper, magazines etc.

Table 3 shows the characterisation of the final product after 150 days of maturity. It was obvious that when the amount of the waste paper was increased the concentration of the organic matter in the final product increased.

Table 3
Compost characteristics

	So	S1	S2	S3	S4	S5	S6
Moisture %	27.20	30.00	32.00	29.50	28.75	29.65	31.50
pH	7.01	7.16	7.48	7.16	7.56	7.09	7.25
Conductivity mS/cm	1.001	1.250	1.128	1.002	1.258	1.345	1.450
OM % *	32.81	32.05	36.23	35.34	38.14	38.37	39.03
TOC % *	19.03	23.05	21.01	20.49	20.96	20 + .46	22.17
TKN % *	1.65	1.75	1.61	1.85	1.66	1.62	1.89
P–PO ₄ % *	2.45	2.50	2.48	2.54	2.35	2.50	2.35
N–NH ₄ mg/g *	0.735	0.563	0.011	0.107	0.180	0.090	0.080
N–NO ₃ 10 ⁻¹ mg/g *	2.05	2.10	1.92	2.36	1.87	1.91	2.37
N–NO ₂ 10 ⁻⁶ mg/g *	258	263	248	281	286	271	260
C/N	11.53	13.17	13.04	11.07	12.93	12.62	11.73

*, Dry basis, significantly different at $P < 0.05$.

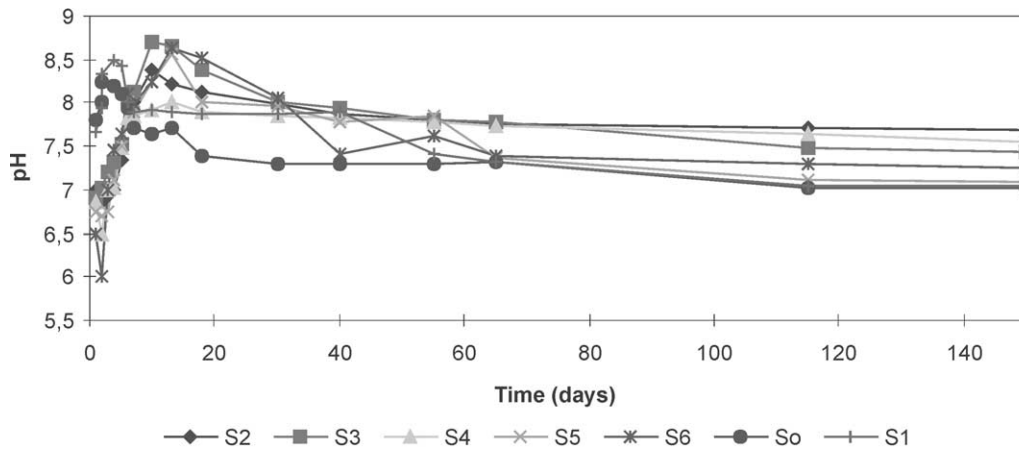


Fig. 1. pH during the composting.

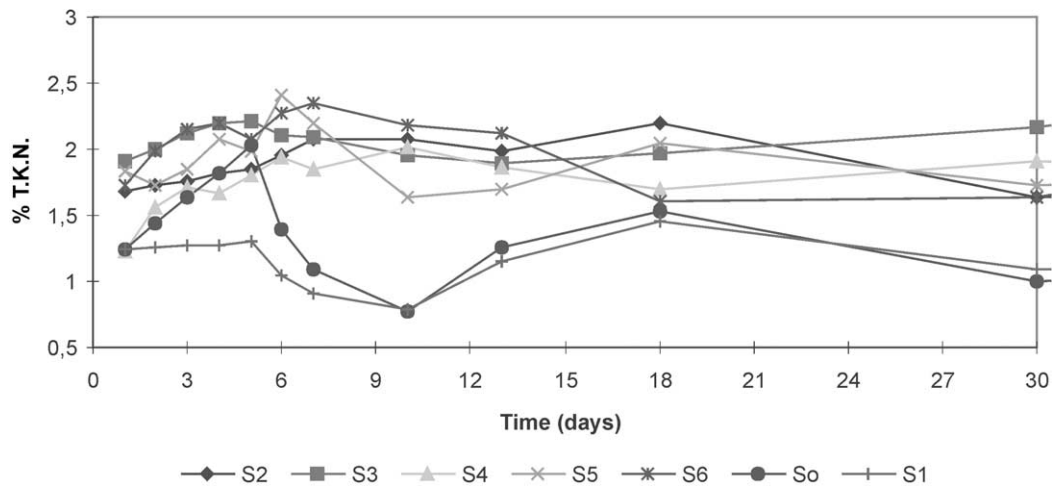


Fig. 2. TKN during composting.

Fig. 1 shows the pH during the composting process. pH is a parameter which affects greatly the composting process. The optimum pH values are 6–7.5 for bacterial development, while fungi prefers an environment in the range of 5.5–8.0 (Kapetanios et al., 1993). The pH was initially low due to the acid formation, then the pH increased and at the latter stage pH was constant.

Figs. 2 and 3 show the TKN content and ammonia content, respectively, during the composting process. The total nitrogen is usually affected by the action of the proteolytic bacteria and by the temperature. At high temperatures the nitrogen is released to the atmosphere (Wagner et al., 1990). It is obvious, from Fig. 2, that the total nitrogen seemed to increase during the first 10 days. This was caused by a decrease in the substrate carbon resulting from CO_2 loss, plus the action of the azotobacterial which fix nitrogen from the atmosphere. It was also observed that increasing the bulking agent ratio (WP ratio) to 40%, the total nitrogen seemed to decrease. A higher bulking agent ratio results in better

aerobic conditions due to greater porosity, which enhances mineralization and volatilization. At the final stage when the compost was matured the TKN was 1.65 for So, 1.75 for S1, 1.61 for S2, 1.85 for S3, 1.66 for S4, 1.62 for S5 and 1.89 for S6.

The compost quality with respect to agricultural use depends on its inorganic nitrogen content. It should not exceed 10% of the total nitrogen and the ammonia content should be less than the 0.04% of the dry matter (Zorpas, 1999). The final total concentration did not exceed the above limit, rendering the compost to be a good soil conditioner.

Fig. 4 shows the changes during the composting process in the organic matter for all the compost samples. At the final stage the organic matter was greater in sample S1 and in the co-composting product S6 followed by S5, S4, S3 and S2. It was obvious that while the amount of the waste paper increased the organic matter in the co-composting products increased. The final C/N ratios after 150 days were 11.53, 13.17, 13.04, 11.07, 12.93, 12.62, 11.73 for So–S6, respectively. The

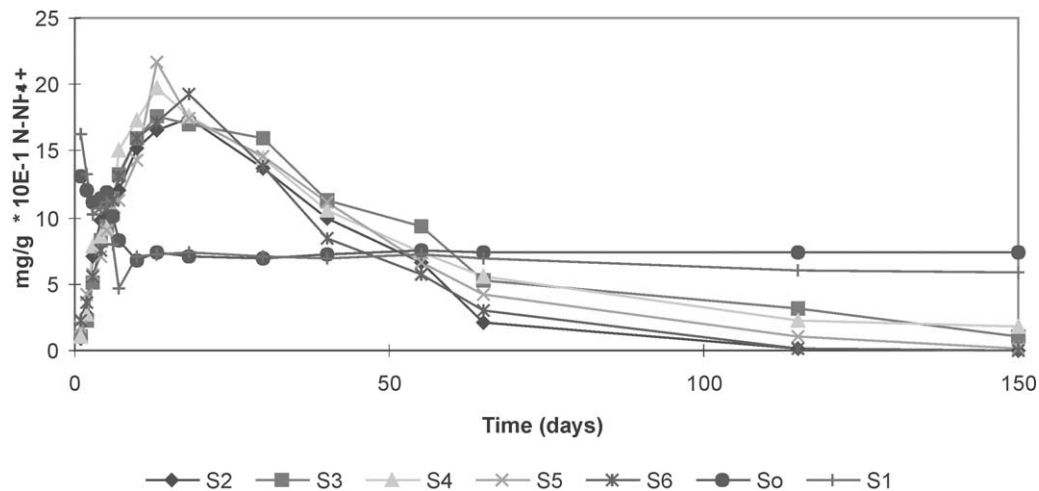


Fig. 3. Ammonia content during composting.

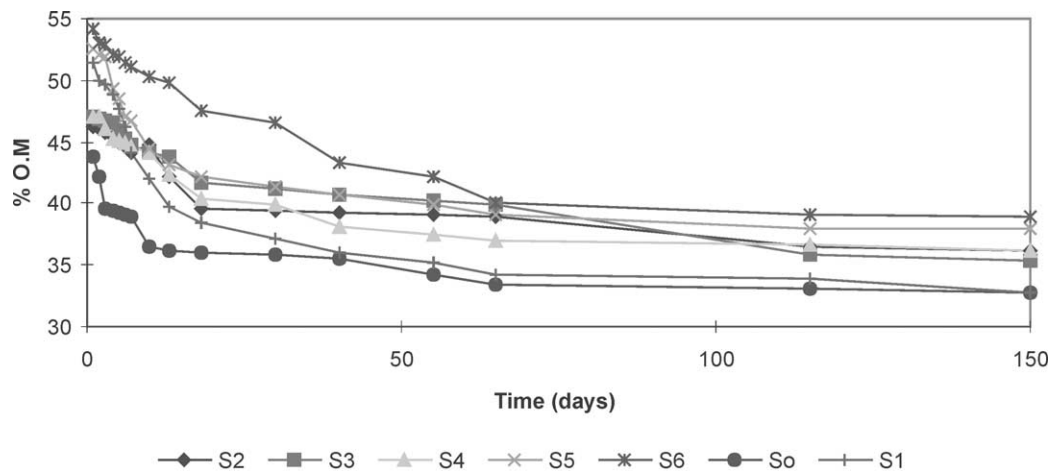


Fig. 4. Organic matter during composting.

Table 4
Humic substances in the final product^a

	So	S1	S2	S3	S4	S5	S6
Total humics	3.50	3.50	6.25	7.82	9.02	10.49	11.95

^a Significantly different at $P < 0.05$.

C/N ratio for this stabilized compost was considered very satisfactory for agricultural use.

Table 4 shows the concentration of the humic substances after 150 days of maturity. The sewage sludge contained a low concentration of humic substances which required the addition of ligno-cellulose product. The WP (Table 2) contained a higher concentration of humics, lignin and cellulose than the sewage sludge. The co-composting of the DASPSS and WP increased the

total amount of the humics substances in the final product and made it a good soil conditioner.

Table 5 presents the metal content in DASPSS, WP, and the final products. When comparing the metal content of the So sample (sewage sludge compost) and DASPSS sample, it can be observed that the concentration of chromium, nickel, manganese, lead and zinc appeared to increase while that of copper and iron to decrease. Composting can concentrate or dilute heavy metals present in sewage sludge. This change in metal concentration depends on the metal loss through leaching and on the overall concentration of metals due to organic matter destruction (Wagner et al., 1990).

Comparing the results in Table 5, with other studies carried out before (Krabe, 1988; Lineres and Petruzzelli, 1988; Nappi et al., 1992), it was observed that all the metals seemed to be in lower concentrations.

Table 5

Concentration of metals in the raw materials and in the final products

	DASPSS	CI	WP	CI	So	CI	S1	CI	S2	CI	S3	CI	S4	CI	S5	CI	S6	CI
Cd	0.002	a	–		0.002	a	0.002	a	0.002	a	0.001	a	0.001	a	–		–	
Cu	0.258	0.025	0.002	a	0.205	0.005	0.238	0.025	0.238	0.025	0.225	0.025	0.212	0.025	0.201	0.025	0.199	0.025
Cr	0.552	0.005	–		0.578	0.023	0.560	0.022	0.561	0.025	0.530	0.024	0.527	0.022	0.508	0.022	0.499	0.022
Fe	5.098	0.310	1.000	0.150	4.118	0.350	5.001	0.450	5.741	0.500	5.547	0.500	5.201	0.460	5.001	0.470	4.099	0.450
Mn	0.150	0.003	0.006	0.001	0.168	0.010	0.148	0.003	0.148	0.003	0.148	0.003	0.140	0.004	0.132	0.004	0.125	0.004
Ni	0.041	0.003	–		0.045	0.002	0.039	0.003	0.040	0.003	0.037	0.003	0.035	0.003	0.033	0.003	0.030	0.003
Pb	0.326	0.004	0.002	a	0.335	0.030	0.322	0.005	0.298	0.005	0.286	0.005	0.263	0.005	0.235	0.005	0.220	0.005
Zn	1.739	0.050	0.003	a	1.801	0.150	1.664	0.050	1.405	0.050	1.129	0.050	0.952	0.045	0.926	0.045	0.884	0.045

Al metals in mg/g dry basis; –, not detected; a: 0.0005.

Table 6

Metals concentration in cured compost

Metasl in mg/g dry sludge	DASPSS	Scli5	CI	Scli6	CI	Directive 278/86/EEC
Cd	0.002	–		–		0.02–0.04
Cu	0.258	0.160	0.004	0.134	0.004	1.00–1.75
Cr	0.552	0.452	0.010	0.405	0.010	a
Fe	5.098	4.105	0.125	3.274	0.125	b
Mn	0.150	0.124	0.002	0.105	0.002	b
Ni	0.041	0.027	0.001	0.021	0.001	0.30–0.340
Pb	0.326	0.201	0.015	0.181	0.015	0.75–1.20
Zn	1.739	0.806	0.050	0.754	0.050	0.016–0.025

^a There is no concentration limit.^b Are not considered to be toxic.

Table 6 presents the heavy metals concentration in samples Scli5 and Scli6 where natural zeolite was added in 20 and 25% w/w, respectively.

It was observed that the zeolite can take up a significant amount of heavy metas by ion exchange. Zeolite was found to take up 100% of Cd, 32% of Cu, 18% of Cr and Mn, 20% of Fe, 15% of Zn, 40% of Ni and 17% of Pb. The concentration of heavy metals (Table 6) was high but not above the EU limits (directive 86/278/EEC) set for the land disposal of sludge (Directive 278/86/EEC, 1986).

The GANC test and the sequential chemical extraction was applied in the raw sludge (DASPSS), in S6 compost with no clinoptilolite and in Scli6 cured compost.

Fig. 5 shows the metal leachability in the DASPSS, S6 and Scli6 cured compost. It was observed that by increasing the leachate pH, the heavy metal concentration decreased.

The metal partitioning in the DASPSS sample as determined by sequential chemical extraction is shown in Fig. 6a. A significant amount of Cr and Cu was bound to the organic and residual fractions with less than 2% in the exchangeable and the carbonate fractions. Almost 60% of Cd and Mn was bound to the reducible fraction. Iron and Fe 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 before (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. 6a 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 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 metal partitioning of the sewage sludge compost were the result of the following:

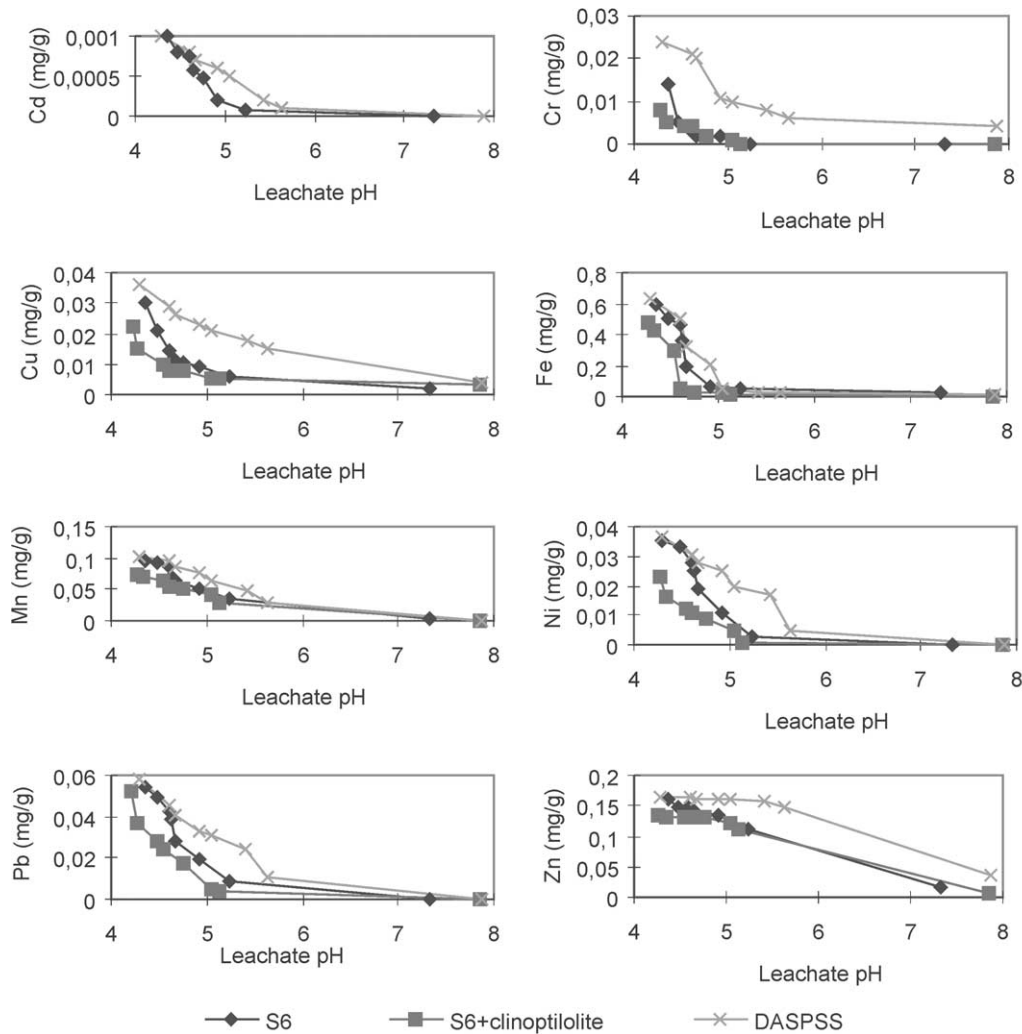
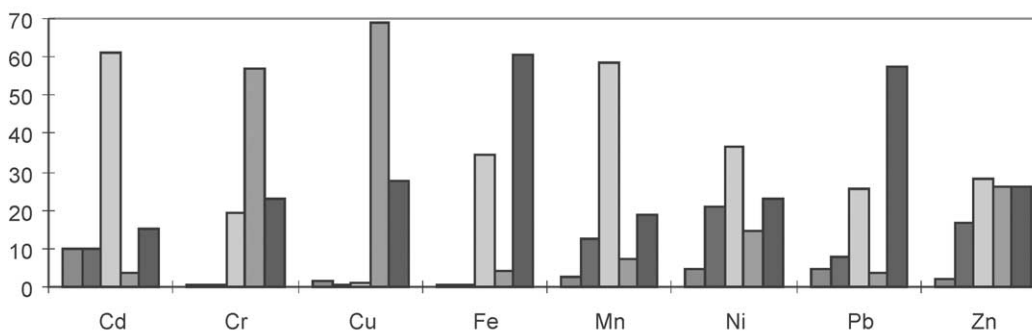


Fig. 5. GANC metals releases to leachates from DASPSS, S6 and S6 with clinoptilolite compost as a function of pH of leachate.

1. 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 (Zorpas, 1999; Wagner et al., 1990);
2. The pH changes, which occurred in the beginning of the composting process, affected the exchangeable and carbonate fraction. The pH changes may be due to acid formation during the decomposition of organic matter contained in the sludge (Zorpas et al., 1999; Zorpas, 1999; Kapetanios et al., 1993); and
3. 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 Scli6 cured compost was found to be bound in the residual fraction at 47–67% (Fig. 7). 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 less than 8%, most of which was bound in the organic fraction. Seventy five per cent 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 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 had taken up all metals bound in exchangeable and carbonate fractions according to the following selectivity series: $\text{Cu} > \text{Cr} > \text{Fe} > \text{Ni} > \text{Mn} > \text{Pb} > \text{Zn}$.

(a) DASPSS



(b) S6 cured compost

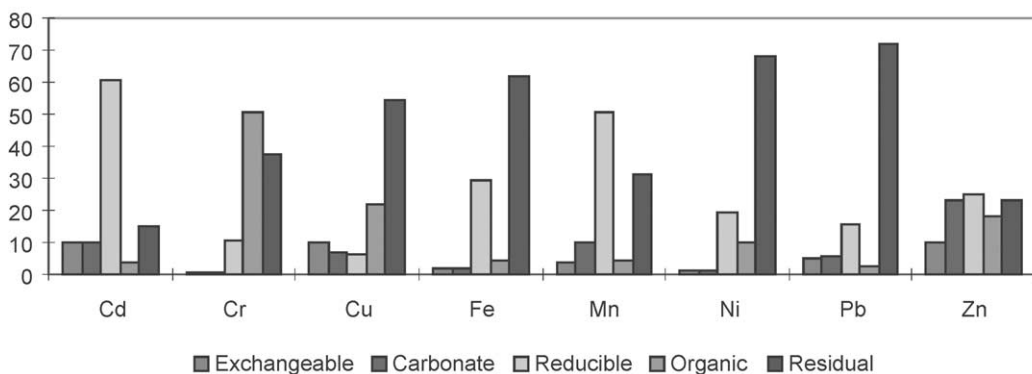


Fig. 6. % Metals partitioning in DASPSS and in sewage sludge cured compost.

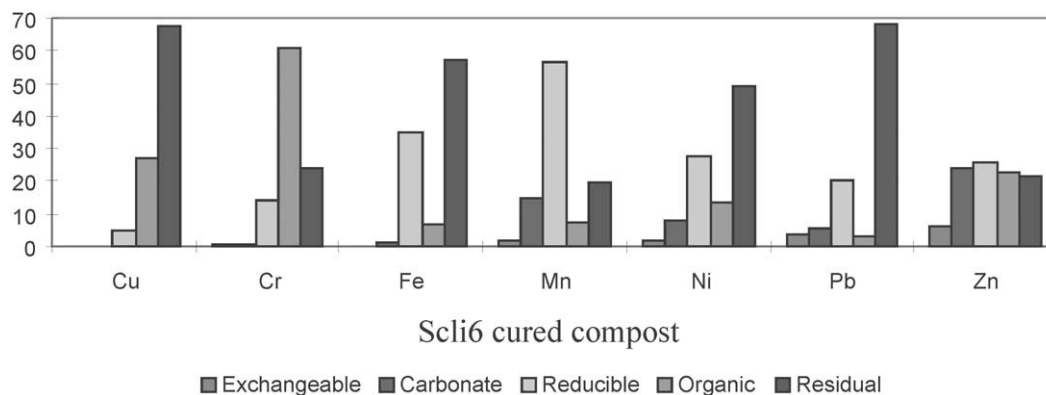


Fig. 7. % Metals partitioning in cured compost.

4. Conclusions

The sewage sludge contained low concentrations of humic materials. The waste paper contained a higher concentration of humics, lignin and cellulose than the sewage sludge and the co-composting of the DASPSS and WP increased the total amount of the humic substances in the final product. The compost quality with respect to agricultural use depends on its inorganic nitrogen content. Inorganic nitrogen should not exceed 10% of the total nitrogen and the ammonia content should be less than the 0.04% of the dry matter. The final total concentration did not exceed the above limit,

rendering the compost to be a good soil conditioner. The C/N ratio for this stabilized compost was considered satisfactory for agricultural use.

All parameters reached a relatively constant level after 80 days (approximately). This indicates the maturity of the final products. S5 and S6 sample which contained 40 and 50% w/w of WP matured faster than the others compared to the So sample due to the fact that the WP had the ability, as a bulking agent, to increase the porosity. This led to faster decomposition.

The final product contained a low concentration of heavy metals and that is not a problem for the use of compost for agricultural proposes. This increase the

quality of the compost. Zeolite was helpful in the uptake of heavy metals. Using the GANC procedure it was found that by increasing the leachate pH, the heavy metal concentration decreased. The GANC test procedure showed that in the case of oxic rain the zeolite had the ability to retain the heavy metals (significant different at $P < 0.05$) and not let them pass to the groundwater.

Using a sequential extraction procedure in the raw sludge (DASPS) and final products (compost after 150 days of maturity) it was found that a significant ($P < 0.05$) amount of sludge metals was bound in the residual fraction, in an inert form. Clinoptilolite had most readily taken up the metal content bound in the exchangeable and carbonate fractions.

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