Composting of domestic refuse and sewage sludge. I. Evolution of temperature, pH, C/N ratio and cation-exchange capacity

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ABSTRACT

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A piling test with urban refuse (R-pile) and urban refuse+sewage sludge (R+S-pile) was carried out to study the evolution of organic matter during composting, and compost maturity. The R+S-pile maintains during around 20 days the limit of thermal inactivation of the micro-organisms. Therefore, the exclusive determination of temperature in piling composting plants cannot be considered in all cases as the conclusive criterion to estimate the grade of biological stability of compost. The bio-oxidative phase was 75 days and the optimization of C/N ratio in the water-soluble phase (C/N_w) and cation-exchange capacity (CEC) occurs in the maturation phase. Likewise, the C/N ratio in the solid phase (C/N_s) cannot be considered as a valid parameter to evaluate the maturity degree of compost: the R-pile reached the optimal value of 15 in full thermophilic phase, with an optimal value of the initial material of 28.

The period in which practical temperature stabilization is achieved with a C/N_w ratio lower than 6 and a CEC value higher than 60 meq 100 g^{-1}, on an ash-free material basis, may constitute the most valid criterion for establishing the optimum degree of maturity.

INTRODUCTION

On the island of Tenerife the application of organic matter to agricultural land has decreased considerably in recent years as a result of the almost total disappearance of traditional sources of organic dressings, animal manure and forest residue. One of the most effective means of offsetting this organic matter deficit is the recycling, for agricultural use, of the residues of urban origin, urban refuse and sewage sludge, by means of composting process.

The composting practise is an aerobic one which combines mesophilic and thermophilic phases, brought about by the combined metabolic activities of

a wide range of dynamic mixed populations of aerobic micro-organisms in rapid succession [1,2]. The heterogeneous organic matter in the starting material is transformed, after a suitable composting period which includes bio-oxidative and maturation phases, into a stabilized end-product throu gh partial mineralization and humification [3].

The physicochemical and microbiological characteristics of city refuse composting and the effect of compost on the soil–plant system have been widely studied [3,4,5]. Most of the studies show that the compost exercises a positive influence on crops due to the ability to supply nutrients to the plants [6,7,8]. Moreover, it has been demonstrated that the application of compost to the soil improves some physical properties such as porosity, aggregate stability, water-holding capacity, and bulk density [9,10,11]. It also improves soil buffering capacity and increases the percentage of organic matter and cation-exchange capacity [12]. Nevertheless, negative effects, normally associated with a decrease in yield, have also been reported due to the application of large amounts of compost with high heavy metal levels [13], high soluble salts [14], and most negative of all, the application of insufficiently mature compost, i.e. not sufficiently stabilized as regards its mineralization and humification [15].

The present paper constitutes the first part of a series where the composting process of the urban refuse and sewage sludge of Tenerife Island is studied in a controlled piling test. In this paper some physicochemical parameters which are closely related to biological stabilization and maturity of compost, are evaluated. The fundamental object is to obtain a simple relevant criterion for rapid assessment of maturity degree in commercial composting plants.

MATERIALS AND METHODS

Composting system
The evolution of two windrows has been studied, one exclusively formed by the bio-oxidative fraction of the urban solid waste (R-pile) and the other formed by a mixture of city refuse and sewage sludge (R+S-pile). The trial was carried out between February and August 1986 with 75 days duration of the bio-oxidative phase and 90 days of complementary maturation.

Urban solid waste was collected from different parts of the city of Santa Cruz de Tenerife. Since one of the most notable characteristics of municipal waste is its heterogeneity, a great quantity of refuse material, 3400 kg, was collected. It is approximately the daily waste production of 3600 persons. Representative garbage samples were obtained after a thorough mixing of all samples following hand removal of contaminant materials such as glass, gravel, plastics, and metal, leaving paper only. The R-pile was constructed of a selected sample of this prepared domestic refuse (810 kg and 59.6% moisture) and reached the following dimensions: 3 m long×2 m wide×0.75 m high.

The R+S-pile was prepared from 850 kg of city refuse mixed with 370 kg of dehydrated sewage sludge (17.8% moisture). It reached the following dimensions: 3 m long×2 m wide×1.10 m high. The proportion of the mixture of the R+S-pile (1:2.3, on a fresh weight material basis) was selected to typify the mixture envisaged for the future composting plant to be constructed on Tenerife Island.

The sewage sludge used in this study was aerobic sludge from the Tenerife wastewater treatment plant (activated sludge plant), dewatered on drying beds. When dehydrated, five samples of the sludge were taken for analysis from the uppermost layer (5 cm) in different areas of the drying beds. Table 1 shows the analytical composition of the sludge used in R+S-pile and Table 2 the initial physicochemical analysis of the initial material utilized in both composting piles.

The temperature variation was followed at 10 cm (surface) and 50 cm (centre of pile) depth. Each reading was the average of four measurements taken at symmetrical locations at each level with a digital thermometer probe ($\pm 0.1\,^{\circ}$C), the readings being taken every three days until the temperature stabilized at around $30\,^{\circ}$C.

TABLE 1

Physico-chemical analysis of the sludge utilized in composting of domestic refuse-sewage sludge (R+S-pile). Average of five repetitions

Moisture (%)	17.8	VS (g $100\,g^{-1}$)	51.1
pH (extract 1:5)	6.9	Ash (g $100\,g^{-1}$)	48.9
EC (mS cm^{-1}, $25\,^{\circ}$C)	2.5		
C_t (g $100\,g^{-1}$)	29.4	Total N (g $100\,g^{-1}$)	4.4
C_o (g $100\,g^{-1}$)	25.0	Total P (g $100\,g^{-1}$)	0.9
C_{ex} (gC $100\,g^{-1}\,C_o$)	9.7	Total K (g $100\,g^{-1}$)	0.5
c_{ha} (gC $100\,g^{-1}\,C_o$)	3.9	Total Ca (g $100\,g^{-1}$)	2.4
C_{fa} (gC $100\,g^{-1}\,C_o$)	5.8	Total Mg (g $100\,g^{-1}$)	1.1
C_{ha}/C_{fa} ratio	0.7	Available P (mg kg^{-1})	4850
C/N_s ratio	6.7	Available K (mg kg^{-1})	4450
$C.N_w$ratio	16.2	Available Ca (mg k^{-1})	5590
CEC (meq $100\,g^{-1}$)	38.7	Available Mg (mg kg^{-1})	2075
Fe (mg kg^{-1})	30720	Pb (mg kg^{-1})	217
Cu (mg kg^{-1})	134	Co (mg kg^{-1})	21
Mn (mg kg^{-1})	599	Ni (mg kg^{-1})	298
Zn (mg kg^{-1})	537	Cd (mg·kg^{-1})	5
Cr (mg kg^{-1})	216	Hg (mg kg^{-1})	4

EC: Electrical conductivity. VS: Volatile solids. C_t: Total carbon. C_{ex}: Alkaline-extractable carbon. C_{ha}: Carbon in humic acid-like compounds. C_{fa}: Carbon in fulvic acid-like compounds. C/N_s: C/N ratio in the solid phase. C/N_w: C/N ratio in the water-soluble phase. CEC: Cation-exchange capacity (on an ash-free material basis). Available K, Ca, and Mg were determined on ammonium acetate (1 M, pH 7) extracts and available P by the Olsen method [16].

TABLE 2

Initial chemical analysis of the material utilized in both composting piles. Average of four repetitions

Variable	R-pile	R+S-pile
Moisture (%)	59.6	49.7
pH (H_2O, extract 1:5)	6.1	6.1
EC (H_2O, 1:5) cm^{-1} mS 25°C	9.9	7.2
VS (g $100 g^{-1}$)	86.24	74.60
Ash (g $100 g^{-1}$)	13.76	25.40
C_t (g $100 g^{-1}$)	48.30	42.84
C_o (g $100 g^{-1}$)	45.21	38.56
C_{ex} (gC $100 g^{-1} C_o$)	19.85	14.96
C_{ha} (gC $100 g^{-1} C_o$)	9.72	6.13
C_{fa}/C_{fa} ratio	10.13	8.83
C_{ha}/C_{fa} ratio	0.96	0.69
C/N_s ratio	27.8	15.7
C/N_w ratio	31.1	27.3
CEC (meq $100 g^{-1}$)	39.65	44.60
Total N (g $100 g^{-1}$)	1.74	2.73
Total P (g $100 g^{-1}$)	0.42	0.56
Total K (g $100 g^{-1}$)	1.29	0.77
Total Ca (g $100 g^{-1}$)	2.71	2.67
Total Mg (g $100 g^{-1}$)	0.20	0.53
Total Na (g $100 g^{-1}$)	0.61	0.52
Available P (mg kg^{-1})	806	921
Available K (mg kg^{-1})	9350	6650
Available Ca (mg kg^{-1})	7840	5240
Available Mg (mg kg^{-1})	990	1520
Available Na (mg kg^{-1})	5320	4750
Fe (mg kg^{-1})	6487	14275
Cu (mg kg^{-1})	148	90
Mn (mg kg^{-1})	112	279
Zn (mg kg^{-1})	184	303
Pb (mg kg^{-1})	94	196
Ni (mg kg^{-1})	25	82
Cr (mg kg^{-1})	23	59
Co (mg kg^{-1})	6	12
Cd (mg kg^{-1})	1	3
Hg (mg kg^{-1})	1	2

Moisture level was maintained, during the whole bio-oxidative phase, between 40 and 60% which was considered the optimum range [17]. To measure these levels, tensiometers, placed at depths of 10 and 50 cm, were used. Treated wastewater was used to dampen the pile so that any dissolved nutrients would enrich the final product and offset, in part, N-losses through volatilization of ammonia during composting.

Aeration was achieved by turning over the compost mass. The number of turnings-over required was established from Kochtitzky et al. [18] who

showed that 6 to 8 separate turnings-over are enough to ensure an adequate oxygen supply for a composting period of 8–10 weeks (bio-oxidative phase). The turning-over regimen is shown in Fig. 1.

Analytical determinations

During the composting process samples were collected at regular intervals, coinciding with the turning-over of the mass, with the result that samples were from the centre of the pile, exposed on the surface by the turning. Samples were obtained on the following days: R-pile, 9, 16, 23, 35, 43, 51, 60, 68, 75 and 165; R+S-pile, 10, 18, 25, 34, 46, 62, 75 and 165.

Each triplicated sample (3 kg) was composed of eight subsamples (24 subsamples). From a fraction of approximately 1 kg the moisture was determined (80°C, 5 hours) and the remainder was air-dried and crushed in a hammer mill (mesh size 1 mm) followed by further pulverization using a vibrating cutter, and analysed.

The pH and electrical conductivity (EC) were determined in a 1:5 compost–water suspension. Ash and volatile solids (VS) were determined by weight-loss on ignition in a muffle furnace at 600°C for two hours.

Total organic carbon (C_t) was determined by combustion in a 12-H Omega Carmhograph carbon oven, and nitrogen content by Kjeldahl microanalysis. Inorganic carbon was found to be negligible. Oxidizable carbon (C_o) was determined by dichromatometric oxidation at 150°C ($K_2Cr_2O_7 + H_2SO_4$ mixture). Extractable carbon (C_{ex}) was determined by 0.1 M $Na_4P_2O_7 + 0.1$ N NaOH solution (1:1) and humic acid-like carbon (C_{ha}) after precipitation at pH 1 with sulphuric acid. Fulvic acid-like carbon (C_{fa}) was deduced from the difference between C_{ex} y C_{ha}.

The carbon/organic nitrogen ratio in water extract (C/N_w) was determined following the method proposed by Chanyasak and Kubota [19] using a 1:5 compost–water suspension after filtration through a 0.45 μm membrane filter.

The cation-exchange capacity was determined by the acid-washing method of Harada and Inoko [20].

RESULTS AND DISCUSSION

The evolution of pH, EC, Ash, the forms of carbon (C_t, C_o, C_{ex}, C_{ha} and C_{fa}), maturity parameters (C/N_s ratio, C/N_w ratio and CEC), and "humification" indexes (humification ratio, humification index, percent humic acid, C_{ha}/C_{fa} ratio) during composting in the R-pile and the R+S-pile are shown in Tables 3 and 4 respectively.

TABLE 3

Changes in the forms of carbon, maturity parameters, and "humification" indices during composting of domestic refuse (R-pile)

	Days										
	1	9	16	23	35	43	51	60	68	75	165
Moisture	59.6	58.5	56.3	57.8	52.3	52.0	55.2	47.7	46.8	42.0	35.20
pH (1:5)	6.1	5.6	5.7	6.4	6.9	7.9	8.6	8.8	8.7	8.4	8.4
EC (1:5)	9.9	12.3	13.8	15.5	15.2	15.0	15.0	16.5	15.0	15.0	16.0
Ash %	13.76	17.47	19.39	22.71	28.19	30.38	30.42	31.54	31.05	31.15	38.75
VS %	86.24	85.35	80.61	77.29	71.81	69.62	69.58	68.46	68.95	68.85	61.25
C_t	48.35	45.50	45.30	44.50	41.29	39.96	37.16	36.78	36.63	35.55	32.55
C_o	45.21	43.40	42.15	42.51	40.28	37.12	36.31	32.99	32.72	31.86	30.96
C_{ex}	10.41	11.20	10.68	10.54	10.47	10.88	12.09	10.20	10.12	9.55	11.15
C_{ha}	5.10	5.24	5.32	6.12	6.32	6.53	7.28	6.41	6.38	5.92	7.38
C_{fa}	5.31	5.96	5.35	4.42	4.15	4.35	4.81	3.78	3.73	3.63	3.77
C_{ha}/C_{fa}	0.96	0.88	0.99	1.39	1.52	1.50	1.51	1.70	1.71	1.63	1.96
HR	19.85	21.30	20.42	19.16	18.66	20.41	23.17	21.16	21.32	20.64	22.05
HI	9.72	9.96	10.18	11.13	11.26	12.25	13.96	13.31	13.45	12.79	14.60
P_{ha}	48.97	46.76	49.85	58.09	60.34	60.02	60.25	62.90	63.09	61.97	66.21
C/N_s	27.79	19.78	16.47	15.72	15.35	13.28	11.69	12.18	12.05	10.74	9.83
C/N_w	31.10	26.28	19.25	11.06	8.12	7.41	6.28	5.95	5.48	5.69	5.37
CEC*	34.19	38.53	41.39	42.09	36.91	41.23	49.52	47.74	47.47	50.38	49.08
CEC**	39.65	46.69	51.35	54.46	51.40	59.22	71.17	69.73	68.85	73.17	80.14

EC: Electrical Conductivity. mS cm^{-1} 25°C, extract compost–water 1:5. VS: Volatile solids. dry matter (d.m.). C_t: Total carbon. gC 100 g^{-1} d.m. C_o: Oxidizable carbon. gC 100 g^{-1} d.m. C_{ex}: Alkaline-extractable carbon. gC 100 g^{-1} d.m., on ash-free material basis. C_{ha}: Humic acid-like carbon. gC 100 g^{-1} d.m., on ash-free material basis. HR: Humification ratio ($C_{ex}/C_o \times 100$). HI: Humification index ($C_{ha}/C_o \times 100$). P_{ha}: Humic acid percent ($C_{ha}/C_{ex} \times 100$). C/N_s: C_t/N_t ratio in solid phase. C/N_w: C_o/N_t ratio in water-soluble phase. CEC*: Cation-exchange capacity. meq 100 g^{-1} d.m. CEC**: Cation-exchange capacity. meq 100 g^{-1} on ash-free material basis.

Temperature evolution

Temperature evolution is an indicator of microbial activity during composting and consequently this parameter may be considered a good indicator of the end of the bio-oxidative phase. The temperature curves for the R-pile and R+S-pile are shown in Fig. 1, where it is possible to distinguish three different stages: a 10-day logarithmic phase, clearer in the R-pile; a 30-day stationary phase, which is observed more clearly in the R+S-pile; and a decline phase, or cooling, down, which continues later as the phase of maturation.

As observed in Fig. 1, the initial mesophilic phase (up to 45°C) has a duration of 6–8 hours in both piles. Nevertheless, the R-pile shows a slower temperature evolution rate than in the R+S-pile, since the maximum temperature is reached on the surface after 26 days of composting (70°C), and in the centre at 33 days (67°C). However, temperature evolution in R+S-pile is more exceptional because, after 24 hours, temperatures of 61°C have been

TABLE 4

Changes in the forms of carbon, maturity parameters, and "humification" indices during composting of domestic refuse-sewage sludge (R + S-pile)

	Days								
	1	10	18	25	34	46	62	75	165
Moisture	49.7	52.4	45.5	47.5	45.7	53.7	50.3	45.5	27.7
pH (1:5)	6.1	6.7	7.3	7.4	7.4	7.4	7.4	7.5	7.1
EC (1:5)	7.2	7.5	8.3	9.4	9.2	9.0	10.1	9.5	9.5
Ash %	25.40	33.84	37.13	37.27	41.45	42.47	48.12	49.88	56.38
VS %	74.60	66.16	62.87	62.73	58.55	57.53	51.88	50.12	43.62
C_t	42.84	37.83	34.68	33.31	32.27	31.98	29.39	28.87	25.07
C_o	38.56	35.76	33.38	33.06	29.17	28.34	21.84	20.90	21.66
C_{ex}	7.73	8.19	7.77	7.31	7.04	7.02	5.79	5.85	9.54
C_{ha}	3.17	3.37	3.38	3.78	4.11	4.33	4.26	4.22	6.74
C_{fa}	4.56	4.82	4.39	3.53	2.93	2.68	1.54	1.63	2.80
C_{ha}/C_{fa}	0.69	0.70	0.77	1.07	1.40	1.61	2.77	2.59	4.17
HR	14.96	15.16	14.63	13.87	14.13	14.25	13.76	14.02	16.85
HI	6.13	6.24	6.37	7.18	8.25	8.80	10.11	10.12	13.59
P_{ha}	40.98	41.16	43.54	51.77	58.39	61.75	73.47	72.18	80.65
C/N_s	15.69	12.09	9.71	9.30	8.99	8.71	8.75	8.86	8.89
C/N_w	27.25	26.43	22.14	16.18	11.26	7.44	6.86	6.25	5.19
CEC*	33.27	34.63	35.71	35.91	35.24	33.75	30.63	28.83	27.15
CEC**	44.60	52.34	56.78	57.24	60.19	58.67	59.04	57.52	62.25

EC: Electrical conductivity. $mS\,cm^{-1}$ 25°C, extract compost–water 1:5. VS: Volatile solids. dry matter (d.m.). C_t: Total carbon. $gC\,100\,g^{-1}$ d.m. C_o: Oxidizable carbon. $gC\,100\,g^{-1}$ d.m. C_{ex}: Alkaline-extractable carbon. $gC\,100\,g^{-1}$ d.m., on ash-free material basis. C_{ha}: Humic acid-like carbon. $gC\,100$ g^{-1} d.m., on ash-free material basis. C_{fa}: Fulvic acid-like carbon. $gC\,100\,g^{-1}$ d.m., on ash-free material basis.
HR: humification ratio $(C_{ex}/C_o \times 100)$. HI: Humification index $(C_{ha}/C_o \times 100)$. P_{ha}: Humic acid percent $(C_{ha}/C_{ex} \times 100)$. C/N_s: C_t/N_t ratio in solid phase. C/N_w: C_o/N_t ratio in water-soluble phase. CEC*: Cation-exchange capacity. $meq\,100\,g^{-1}$ d.m. CEC**: Cation-exchange capacity. $meq\,100\,g^{-1}$ on ash-free material basis.

measured on the surface and 54°C in the centre. At 48 hours, the R + S-pile had already reached 70°C in the centre as well as on the surface. Among several possible explanations, this elucidated on the basis of the great richness of indigenous microorganisms present in the sewage sludge which provoke a rapid attack on the easily decomposable organic matter, a fact which is favourable because of the high content of available nutrients in the sludge and the relatively small size of the particles of the organic fraction [21].

This rapid temperature increase in the R + S-pile does not seem to exert any influence on the duration of the bio-oxidative phase. Notwithstanding, a slow evolution of temperature ensures a smaller loss of nitrogen per unit time, considering that the composting process is fundamentally an alkaline reaction [22].
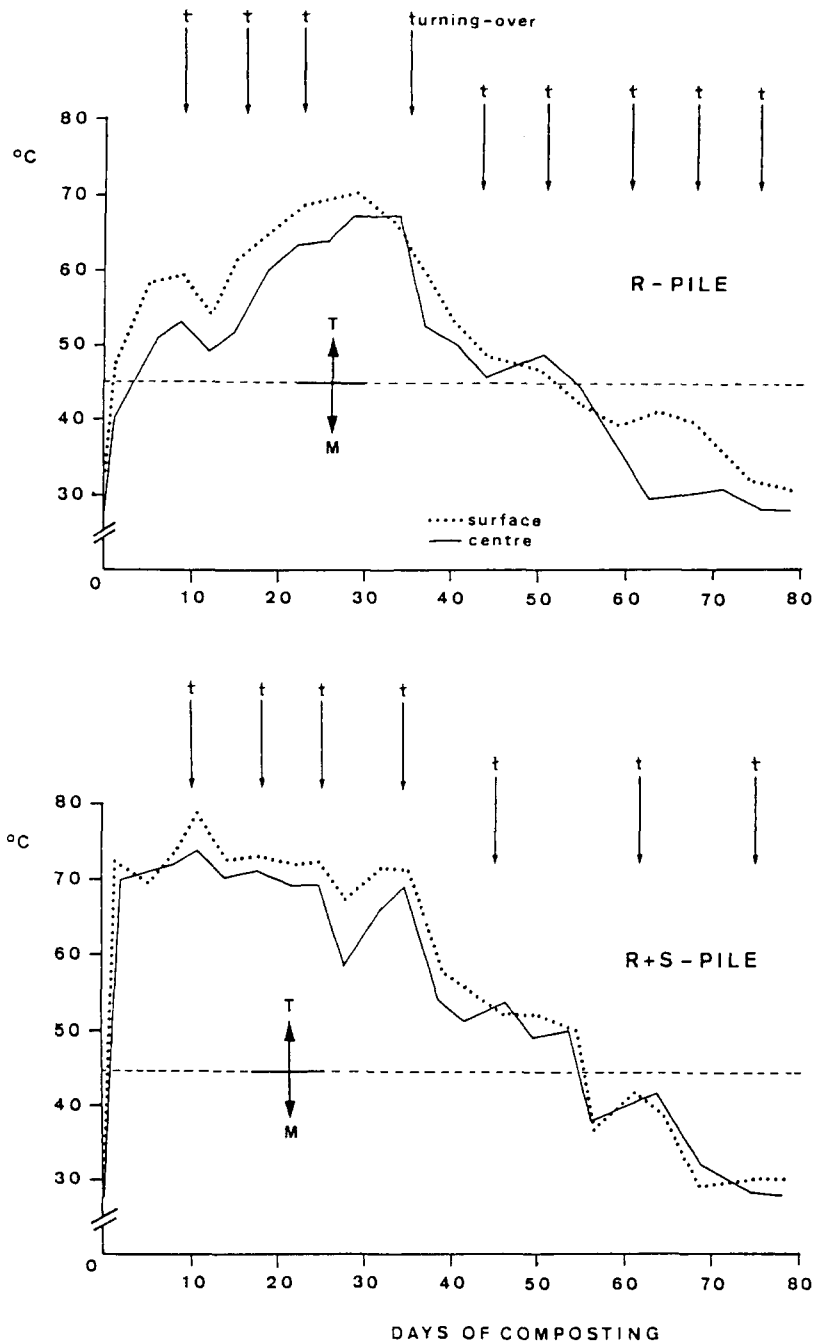
Fig. 1. Temperature evolution during composting of the organic fraction of urban refuse (R-pile) and urban refuse + sewage sludge (R + S-pile). T: Thermophilic phase. M: Mesophilic phase.

All authors concur that the thermophilic phase has to be maintained for at least 10 days and the optimum level should be between 60 and 70°C to destroy thermosensible pathogens [1,3,23] and encourage the development of thermophilic microorganisms, mainly eumycetes and thermophilic actinomycetes [24] The thermophilic phase is maintained in both piles during 50 days, a period which constitutes approximately 70% of the total duration of this biooxidative phase. This long thermophilic period, as well as the number of turnings-over, seems to be sufficient for the thermal destruction of pathogens and seems to favour the germination of spores and seeds and to permit the hatching of parasites eggs, whose larvae are destroyed by the thermal exposure [25].

When the metabolic rate decreases, because of exhaustion of available substrate, the temperature starts to fall and the thermophilic microflora is replaced by mesophilic one, which continues the partial degradation of some bio-resistant compounds, fundamentally cellulose and, to a lesser degree, lignin [3]. As observed in Fig.1, the passage of the thermophilic phase to the mesophilic takes place in both piles at between 50 and 55 days of composting. From this time on, a slow temperature decrease is noted until stabilization at 30°C from the 70th to the 75th day of composting.

Numerous authors have indicated the importance of this second mesophilic phase (and a later maturation phase) because of the spectacular development of mesophilic eumycetes and actinomycetes, which are very active during the degradation of cellulose and lignin [3]. Furthermore, during this mesophilic phase a strong nitrogenase activity has been detected [2]. Many species of N_2-fixing bacteria have been isolated during city refuse composting, mostly in association with the mesophilic phases, fundamentally *Azomonas, Klebsiella,* and *Enterobacter* [2,3].

Rise and fall of temperature are characteristic features of the piling process of compost, and temperature measurements have been proposed as a means of establishing the compost maturity [26]. Notwithstanding, the practical stabilization of the temperature curve in industrial composting plants cannot be considered as a convincing criterion of stabilization since a thermal inactivation of microorganisms can be produced if temperatures exceed 70°C for a long period. Only a few species of thermophilic sporigenous bacteria show metabolic activity above 70°C: *Bacillus subtilis, B. stearothermophilus* and the non-spore forming, Gram-negative, aerobic genus *Thermus* [3]. By observation of Fig. 1, it was deduced that the R+S-pile practically maintains, during 20 days, the limit of this thermal inactivation, exceeding 70°C between the 2nd and 17th days (centre), despite the number of turnings-over and the optimum degree of moisture during this period. The R-pile does not at any moment exceed the limit of 70°C. In spite of this thermal inhibition, when the surface temperature falls below 70°C, a reinvasion of microorganisms takes place from the centre to the surface and also from the exterior.

This mechanism or cycle of destruction–reinvasion really constitutes a feed-back process and explains why the temperature curve in the R+S-pile effectively maintains a stable 70°C during these 20 days. The feedback process, therefore, involves a thermal inhibition of the microflora in the R+S-pile during this time and, for this reason, a partial degradation of the organic matter. Thus, the optimization of the parameters that indicate the maturity degree (C/N_w ratio and CEC) occur in the later phase of maturation. Therefore, the determination solely of the temperature in commercial composting plants (piling composting) cannot be considered in all cases as a conclusive criterion of the degree of biological stability of the compost.

Evolution of pH

Like temperature, pH may be a good indicator of bio-oxidative phase evolution and microbial development. The evolution of pH in the R-pile follows the typical model described in the literature for city refuse composting [1]: acidic during the first days, as a consequence (among other possible causes) of the degradation of easily hydrolyzed polysaccharides, and new synthesis of simple organic acids. Later, as the temperature rises, the pH increases gradually to 8–9, mainly because of the metabolic degradation of these organic acids or loss by volatilization and furthermore, because of intensive proteolysis liberating ammonia compounds. Finally, the pH falls slightly during the cooling phase to values of between 7 and 8.

In the R+S-pile however, the pH maintains the same values during the whole bio-oxidative phase, understood to be between 6.7 and 7.5, very near to neutrality. As observed in Table 3, the pH in the R-pile remains slightly below 7 during the first 35 days of composting. This fact is of great importance with respect to the conservation of N of the original material, since conditions of high temperature, rapid thermal increase and alkaline pH, as well as a low moisture degree and an initial C/N ratio lower than 20, lead to rapid N-volatilization in the form of ammonia [1]. These conditions correspond with the observed ones in the R+S-pile, where the pH remains slightly acidic only until the tenth day. Furthermore, a very rapid thermal increase is produced during the first hours and the initial C/N ratio is very low, i.e. 15.7. Therefore a bigger loss of N is foreseen, in absolute terms, in the R+S-pile relative to the R-pile. The concentration of N in the compost of the R-pile changes from 1.74% to 3.31% after 75 days of the bio-oxidative phase; meanwhile, the change in the R+S-pile is of 2.73% to 3.26%.

Evolution of C/N ratio in the solid phase (C/N_s)

In Fig. 2, the evolution of the C/N_s ratio (and C/N_w ratio) during the bio-oxidative phase in both piles is shown.

One of the major problems in the composting process on an industrial scale is establishing the initial C/N ratio to ensure a good composting rate and
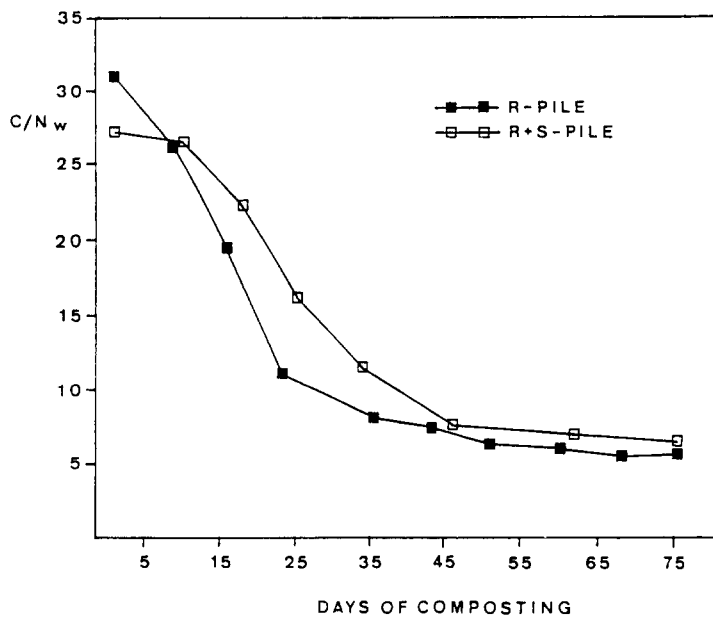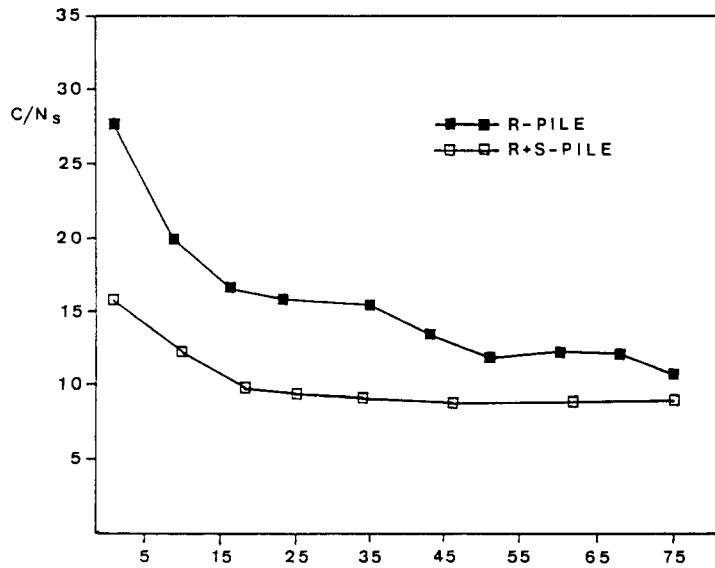
Fig. 2. Evolution of C/N ratio in the solid phase (C/N$_s$) and C/N ratio in the water-soluble phase (C/N$_w$) during composting or urban refuse (R-pile) and urban refuse + sewage sludge (R + S-pile).

effective yield. Accurate calculation of this parameter is made difficult due to the very heterogeneous composition of domestic refuse. Nevertheless, the wide experience gained from studies of composting with different values of C/N, reviewed by Mote and Griffis [27], seems to demonstrate that suitable values for composting should be 26–35. The C/N_s ratio of the raw material in the R-pile is within this optimal range, which proves that the organic fraction of municipal waste does not need any correction of this parameter, as occurs with other types of residue. However, the initial C/N_s ratio of the R+S-windrow is very low which implies an important N-loss during the bio-oxidative phase, as we have just noted.

Furthermore, the C/N ratio in the solid phase is the index traditionally used to establish the maturity degree of compost. A C/N_s ratio below 20 is indicative of an acceptable maturity degree [23,28], a ratio of 15 or less is preferable [25]. Nevertheless, the C/N_s ratio of compost cannot be used as an absolute index of compost maturity since this parameter varies greatly in well-composted materials [29]. For this reason, it is possible to find immature composts with a C/N_s ratio lower than 20 when the relative N-richness in raw materials is high [30]. This is frequently the case when the organic fraction of municipal waste is composted with sewage sludge, as is the case of the R+S-pile where the C/N_s ratio of the initial material is only 15.7, which would classify this material, according to the former criterion, as mature. Because of this fact, Morel et al. [30] note that it is necessary to carry out a periodic monitoring of the C/N_s ratio during composting until stability is reached and propose as the surest criterion the ratio: (Final C/N/(initial C/N). As observed in Fig. 2, during the bio-oxidative phase in both piles, a gradual decrease is produced, stabilising, without appreciable variation, towards the 50th day in the R-pile and 30th day in the R+S-pile. For this reason, the stabilization of the C/N_s ratio cannot be considered in all cases as a valid index of compost maturity. According to our experimental conditions, this stability is reached during the full thermophilic phase, when the material is not degraded.

Evolution of C/N ratio in the water-soluble phase (C/N_w)

As an alternative to the solid-phase C/N_s ratio, Chanyasak and Kubota [19] propose the determination of this parameter in water extracts of compost as a more reliable index of maturity. These authors explain that the composting reaction is a biochemical decomposition of organic matter in raw materials by microorganisms whose metabolism occurs in the water-soluble phase. Chanyasak et al. [31] found that the C/N_w ratio of mature organic materials settles down between 5 and 6. This occurs in all cases studied, irrespective of the origin of the material and its initial and final C/N_s ratios. The results found in our piling test completely confirm the proposition of these authors. In the R-pile the value within the optimum range (5.9) is obtained towards

the 60th day of composting. In the R + S-pile, at the end of the bio-oxidative phase (75 days) this parameter has a value of 6.3, very close to the optimum value, which is obtained during the phase of maturation. Furthermore, the curves corresponding to the R and R + S-piles (Fig. 2) are, except for slight fluctuations, nearly coincident, which obviously does not occur in the case of the C/N_s ratio. Therefore, it seems clear that some common rules exist during the biochemical processes in the water-soluble phase of composting, independently of the type of initial material and of the initial C/N ratio in the solid phase. In short, this parameter can be considered as an excellent index of the evaluation of compost maturity.

Cation-exchange capacity evolution (CEC)

In Fig. 3 the evolution of CEC in both piles is shown. During composting, normally a gradual increase of CEC is produced. Harada et al. [32] found, in a controlled piling test, that the CEC of city refuse compost increases progressively from 40 meq 100 g^{-1} to about 80 meq. 100 g^{-1} after 12 weeks of composting. They also found a negative correlation between CEC and C/N_s ratio. They concluded that a CEC value > 60 meq 100 g^{-1} on an ash-free material basis is the minimum value needed to ensure an acceptable maturity degree. The results of our piling test confirm the results of these authors: a parallel increase in both piles is observed. On the 75th day the compost in the R + S-
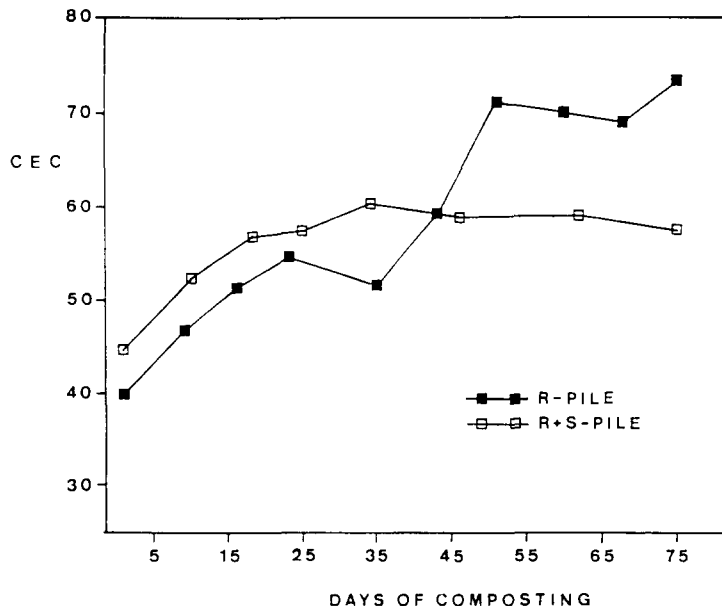

Fig. 3. Evolution of cation-exchange capacity (meq 100 g^{-1}) on an ash-free material basis during composting of urban refuse (R-pile) and urban refuse + sewage sludge (R + S-pile).

pile has a value of 57.5 meq 100 g^{-1}, lower still than the proposed minimum value of 60 meq 100 g^{-1}, a value which is reached during the later phase of maturation. At the end of the composting process (165 days), the R+S-compost has a CEC value of 62.3 meq 100 g^{-1} (ash-free material), and the R-compost 80.1 meq 100 g^{-1}.

The increase in CEC during composting might be explained not only by the accumulation of materials bearing a negative charge, such as lignin-derived products, but also by an increase of carboxyl and/or phenolic hydroxyl groups in the material [33]. Therefore the CEC constitutes an index of the degree of humification of compost and newly-formed organic materials [20,34,35].

CONCLUSIONS

The determination of the cation-exchange capacity by the simple method proposed by Harada and Inoko [20] and/or the analysis of the C/N ratio in water extracts, may constitute a good test for rapid assessment of compost maturity. The use of these parameters in a complementary manner with temperature measurements, may be an excellent criterion to establish the optimum degree of evolution of compost and, therefore, the minimum time of composting needed in commercial composting plants.

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