THE WATER-SOLUBLE ORGANIC FRACTION AND ITS RELATIONSHIP TO THE DEGREE OF MATURITY OF ORGANIC MATTER DURING COMPOSTING.

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1. ABSTRACT

The evolution of different components of the water-soluble organic matter, water soluble carbon (C_{OW}), carbohydrates and phenols were studied during the composting of six different mixtures of organic wastes prepared with sweet sorghum bagasse, cotton waste, sewage sludge, municipal solid waste, urea, pine bark and brewery sludge. The C_{OW}, carbohydrate and phenol concentrations decreased in the six composting mixtures as a consequence of the organic matter degradation carried out by the microbial activity. The intensity of this organic matter degradation and the evolution of the water-soluble compounds depended on the kind of material used in the starting mixtures. Therefore, these parameters were not considered suitable to be used as the basis for a general organic matter stabilisation index.

The changes in the water-soluble organic carbon to water-soluble organic nitrogen ratio (C_{OW}/N_{OW}) and the water-soluble organic carbon to total organic nitrogen ratio (C_{OW}/N_{OT}) were considered to be suitable as general stabilisation indices since these ratios did not depend on the material used. The evolution of these two ratios showed a similar pattern during the composting of the six mixtures studied. All mature composts reached values for these ratios which were in agreement with the ranges proposed by other authors with other type of materials. These maturity indices were also compared with Lepidium Sativum L. germination assays and no phytotoxic effects were found in materials with C_{OW}/N_{OW} and C_{OW}/N_{OT} ratios values between the limits established for mature composts (C_{OW}/N_{OW} between the range 5-6, and $C_{OW}/N_{OT} < 0.40$).

2. INTRODUCTION

Composting is a useful way of transforming organic wastes into a valuable organic matter for use as an organic amendment, or a substrate in soil-less culture. However, the maturity or stability degree of the organic matter, which means, when the compost can be used for agriculture proposes without the phytotoxic effects of the fresh organic matter is a key issue in the use of the material.

Compost maturity has been determined by different workers using different indices based on physical, chemical, biological, biochemical and humification aspects of the organic matter (Bernal *et al.*, 1998; Iglesias and Pérez. 1989; Riffaldi *et al.*, 1986; Senessi, 1989). From all of these indices, those related to the water-soluble fraction of the organic matter during composting have a special interest because it is in this fraction that most of the biochemical transformations of the organic matter take place. The enzymes produced by the microbial activity lead to the solubilization of simple soluble organic compounds causing the breakdown of other compounds with more complex structure. This newly soluble organic matter formed is metabolised again by the microbial biomass as a source of carbon, energy and nutrients. Therefore, the organic compounds quantified in the water-soluble fraction are involved in continual synthesis and degradation processes. This fraction includes compounds such as sugars, small carbohydrates, aminoacids, small peptides, phenols, carboxilic acids, etc. The quantification of these parameters during the composting process has led to the conclusion that simultaneously there is a degradation of the organic matter by the microganisms and also a synthesis as a consequence of the transformation of the simple organic molecules.

Chanyasak and Kubota, (1981), García *et al*.,, (1992), Saviozzi *et al.*, (1987), Zucconi *et al.*, (1985) proposed different parameters to asses the degree of maturation of compost based on the study of this water-soluble fraction. These parameters were: the water-soluble organic carbon (C_{OW}), the ratios of water-soluble organic carbon to water-soluble organic nitrogen (C_{OW}/N_{OW}), and water-soluble organic carbon to total organic nitrogen (C_{OW}/N_{OT}), the germination index, the quantification of phytotoxic substances such as ammonia, the low molecular weight organic acids, phenols, etc. However, it is difficult to determine the degree of maturation of composting organic matter by only one indicator. Hence, it is necessary to consider the value of some of them simultaneously. Some of these indices have been validated against plant germination tests and growth assays. The C_{OW}/N_{OW} ratio has been correlated with the results obtained with plant assays with de *Brassica Rapa* var. *pervidis* and it has been proposed that materials with C_{OW}/N_{OW} ratio values in the range 5-6 did not have any phytotoxic effect (Chanyasak *et al.*. 1983).

The aim of this work was to study the evolution of the C_{OW}, carbohydrates, phenols, and the C_{OW}/N_{OW} and C_{OW}/N_{OT} ratios in the water-soluble fraction of the organic matter of six organic waste mixtures during composting. The degree of maturation of the composting mixtures will be established with these parameters and will be compared with the germination index of these materials.

3. MATERIALS AND METHODS

3.1 Materials

Six different organic wastes were used: a primary aerobic sewage sludge, cotton waste, sweet sorghum bagasse, pine bark, brewery sludge from the wastewater treatment of the brewery factories and the organic fraction of selectively collected municipal solid wastes (MSW). Table 1 shows the main physico-chemical properties of the organic wastes. All the organic wastes used had high humidity with the exception of cotton waste and pine bark. The pH values of all the residues were similar and suitable for composting. Sewage sludge, brewery sludge and urea were used as a nitrogen sources, whereas sweet sorghum bagasse, cotton waste and pine bark were used as bulking agents. The mixtures were prepared in order to reach an initial C/N ratio between 25 and 30.

3.2 Composting performance

Static or turned-pile composting systems were used to compost the six mixtures prepared from seven different organic wastes in the following proportions (fresh weight):

- - By the Rutger static-pile system:
  - Mixture 1: 47 % sewage sludge (SS) + 53 % cotton waste (CW)
  - Mixture 2: 88 % sorghum bagasse (SSB1) + 11 % pine bark (PB) + 1 % urea
  - Mixture 3: 86 % sorghum bagasse (SSB1) + 11 % pine bark (PB)+ 3 % brewery sludge (BS)
  - Mixture 4: 90 % municipal solid waste (MSW) + 10 % sorghum bagasse (SSB2)
- - By the turned pile system:
  - Mixture 5: 90 % municipal solid waste (MSW) + 10 % sorghum bagasse (SSB2)
  - Mixture 6: 100 % municipal solid waste (MSW)

	SS	MSW	BS	SSB1	SSB2	CW	PB
Humidity (%)	51.4	59.4	51.4	78.0	68.3	10.5	9.2
pH	6.8	6.6	7.6	6.3	7.8	7.7	5.3
EC (dSm^{-1})	0.42	5.50	1.60	0.39	2.36	0.41	0.20
OM (%)	67.3	65.3	54.1	96.7	88.8	88.0	86.5
C_{OT} (%)	35.4	32.5	26.3	47.3	44.8	42.2	53.9
N_T (%)	5.79	2.17	4.54	0.42	1.49	1.82	0.28
NH_4-N (%)	0.57	0.19	0.70	0.01	0.03	0.06	--
NO_3-N (%)	0.00	0.00	0.00	0.04	0.00	0.01	--
C_{OT}/N_{OT}	5.40	14.95	5.80	113.8	30.01	23.21	191.0
C_W (%)	2.77	3.24	1.64	2.28	2.15	2.49	1.88

EC: electrical conductivity; OM: organic matter; C_{OT}: total organic carbon; N_T: total nitrogen; C_W: water-soluble carbon; --: not calculated

SS: sewage sludge, MSW: municipal solid waste, BS: brewery sludge, SSB1 and SSB2: sweet sorghum bagasse, CW: cotton waste, PB: pine bark

.Table 1. General characteristics of the waste materials used (dry matter).

About 1500-2000 Kg of mixtures 1, 2, 3 and 4 were composted in a pilot plant by the Rutgers static-pile composting system. This system maintains a temperature ceiling in the pile, through the on-demand removal of heat by ventilation. This encourages a high decomposition rate by avoiding high temperatures, which inhibit and slow down decomposition by reducing microbial activity. Air was blown from the base of the pile using three perforated PVC tubes, 3 m in length and 12 cm in diameter. The timer was set for 30 s ventilation every 15 min. The ceiling temperature for continuous air blowing was 55°C. Mixtures 5 and 6 were composted by the turned-pile system. They were turned every two days during the first week, twice during the second week and only once per week thereafter.

The biooxidative phase of composting (active phase) was considered to be finished when the temperature of the pile was near to that of the atmosphere (final stage). This stage was reached after 49, 63 and 42 days in mixtures 1, 2 and 3, respectively, and after 77 days in mixtures 4, 5 and 6. Air blowing (piles 1, 2, 3 and 4) and turning (piles 5 and 6) were stopped and the mixtures were then allowed to mature over a period of two months (maturation phase). The moisture levels of the piles were controlled weekly during the biooxidative phase of composting and adjusted by adding the necessary amount of water to obtain values between 45-65% of total weight. One representative sample was taken weekly by mixing six subsamples from six sites of the pile and from the whole profile (from the top to bottom of the pile), it was air dried and ground to 0.5 mm for analysis.

3.3 Analytical methods

Total nitrogen was determined by automatic microanalysis. All water soluble parameters were determined in the aqueous extract (1:10, w:v): Water-soluble organic carbon and water soluble total nitrogen by automatic microanalysis (Sánchez-Monedero *et al.*., 1996). Water-soluble phenols by a modified version of the Folin method (Kuwatsuka and Shindo, 1973). The water-soluble carbohydrates by the anthrone method (Brink *et al.*., 1959). Water-soluble NH_4-N by a colorimetric method based on Berthelot's reaction (Sommers *et al.*. 1992), adding sodium citrate to complex divalent cations. Water-soluble NO_3-N by ion chromatography. Water-soluble organic nitrogen was calculated by subtracting the NH_4-N and NO_3-N from water-soluble total nitrogen. The germination index (G.I.) was determined using seeds of *Lepidium sativum L.* (Zucconi *et al.*. 1985) but in the aqueous extract 1:10 (w:v).

4. RESULTS AND DISCUSSION

4.1 Water soluble organic matter degradation

Figure 1 shows the evolution of the C_{OW} concentration of the six mixtures during the composting process. The organic matter of mixtures 1, 2 and 3 underwent less intense degradation than for the other three mixtures. This can be seen from the change of the C_{OW} during composting. C_{OW} values of mixture 1 were almost constants during the two first weeks (2.43 and 2.45%) and after that diminished to reach a value of 0.92% at the end of the composting process. In mixture 2, prepared with urea, the C_{OW} concentration varied from an initial value of 2.08 to 2.99% in the third week and then diminished until the end of the composting process. The rise in C_{OW} concentration occurred at the beginning of the thermophilic phase, when the activity of the cellulolotic and proteolitic microorganisms start to develop (Riffaldi *et al.*, 1986), but in this mixture the microbial activity was retarded for two weeks as demonstrated by the temperature evolution of the mixture during composting (data not shown). During these two weeks, the solubilization of simple organic compounds was probably greater than the degradation of these compounds by the microbial biomass and it is reflected in the rise of C_{OW} concentration. In mixture 3 is where C_{OW} was degraded more slowly than in the rest of the mixtures. A fall in C_{OW} concentration was observed during the maturation process which could have been due to a premature cessation of the ventilation during composting.

The three mixtures prepared with MSW (mixtures 4, 5 and 6) showed a similar pattern. Mixture 6 had a higher C_{OW} concentration for the starting material (3.24%) but this parameter decreased in the three mixtures to very similar final values (between 0.31 and 0.48%). These values indicate that the final product reached a good degree of maturity according to Garcia *et al.*., (1992) who proposed a C_{OW} value of 0.5% for mature compost prepared with MSW.

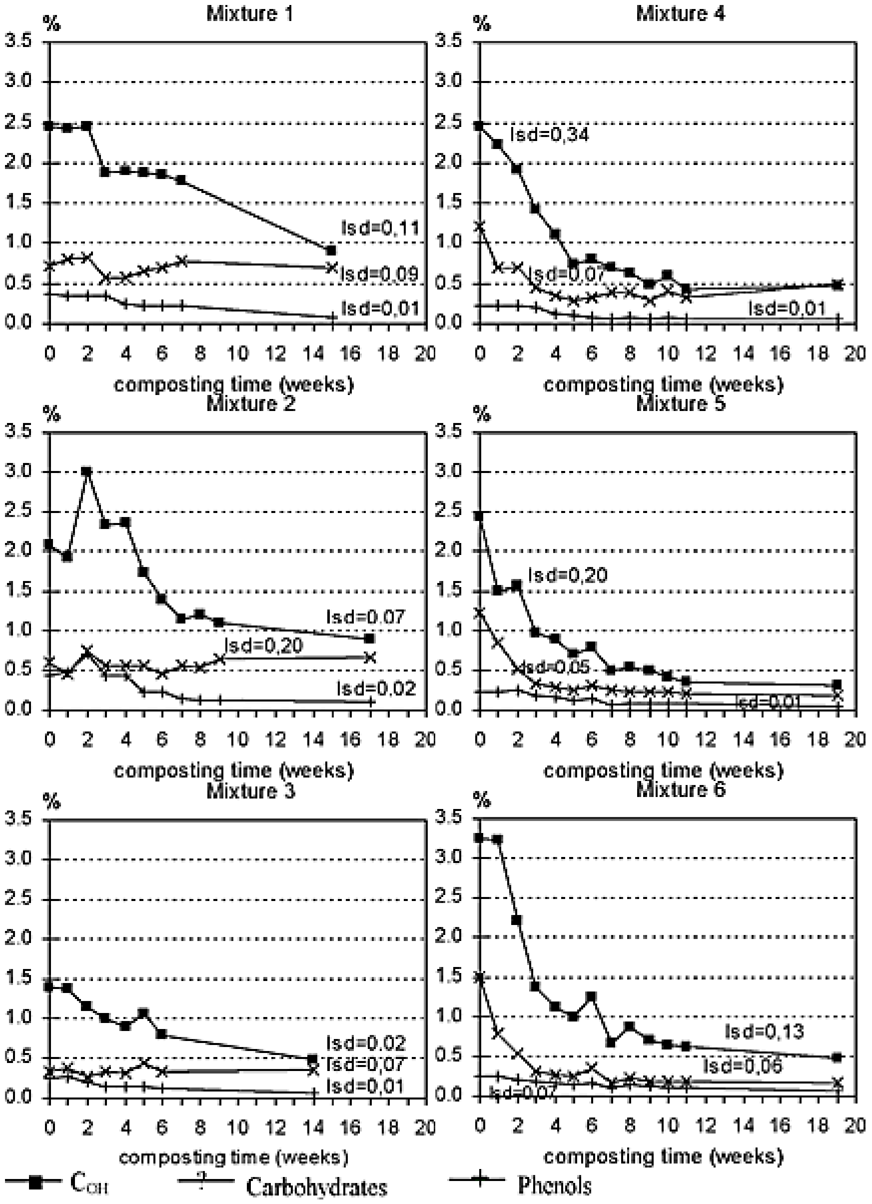

Figure 1. Changes in C_{OW} (% C), carbohydrate (% glucose) and phenol (% p-cumaric acid) concentrations during composting of the different organic waste mixtures. lsd: Least significant difference at the probability level of $P<0.05$

Figure 1 shows the water-soluble carbohydrates and phenols changes in the six mixtures during composting. In the mixtures prepared with MSW, the concentration of carbohydrates fell sharply during the first three weeks by more than 60% of the initial values, although there was very little variation during the rest of the composting process, including the thermophilic phase when degradations were more intense. The carbohydrate concentration of mixtures 1, 2 and 3 showed no clear tendency during composting, ranging from 0.3 to 0.8% during the process. According to these results, the evolution of carbohydrate concentration could be used as an index for following the composting progress of materials with high carbohydrate concentration that undergo an intense degradation as happen with MSW. However, this parameter cannot be generally used for other materials, where organic degradation is less intense, and the effect of carbohydrate degradation could be masked by the simple carbohydrate generation from cellulose and hemicellulose hydrolysis.

The total phenolic fraction is generated during the partial degradation of lignin and is more resistant to degradation that carbohydrates due to its aromaticity. However, the water-soluble phenol fraction, which has a more simple structure and a smaller molecular size, is sensitive to the transformations occurring during composting and has been used as an indicator of the composting process (Saviozzi *et al.*., 1987). This fraction behaved similarly in all six mixtures, although it showed a slight increase in mixture 2 after three weeks, as was the case with the carbohydrate fraction. In all the mixtures there was a gradual fall throughout composting (Figure 1). Initial values of between 0.23 and 0.44% fell to less than 0.1%, which suggests that they were used by the microorganisms either as an energy source or as precursors for the synthesis of new molecules. According to these results, the evolution of water-soluble phenol concentration could be used as parameter for following the composting progress as it decreases in all mixtures. The phenolic fraction degradation was more intense than the formation of new soluble phenols from lignin degradation. However, Saviozzi *et* *al.*, (1987) and Hänninen and Lilja, (1994), using a mixture of wastewater sludge from a paper-processing factory and chopped wheat-straw, and a mixture of slaughter wastes, respectively, observed that this fraction behaved otherwise. It decreased rapidly during the first months of composting and then increased to levels higher than the initial values. Their observations suggested that phenol polymerisation and/or its degradation exceeded the lignin degradation since the phenol fraction did not decrease during the composting process.

Figure 2 shows the changes in the C_{OW}/N_{OW} ratio of the six mixtures during composting. The values of this ratio for the final products of the six composting mixtures, except for mixture 2, were very close to the range 5-6 proposed by Chanyasak and Kubota (1981) as a maturity index for compost prepared with different materials. Mixtures 4, 5 and 6 reached these values in the 5th, 3rd and 7th week, respectively, and could be considered as a mature compost according to this C_{OW}/N_{OW} ratio. However, at these stages of the composting process the humification and nitrification processes would not be completed as they take place during maturation. Therefore it is very difficult to conclude that these materials have really reached a stability degree appropriate to mature compost.

In mixtures 1 and 3, the composting mixtures reached values of the C_{OW}/N_{OW} ratio in the range 5-6 at the end of the active phase or after the maturation process. These results suggest that the water-soluble fraction transformations had been occurring during the whole composting process and the composting mixtures only reached a good degree of stability at the end of the composting process, which is different to the MSW mixtures. Mixture 1, prepared with sewage sludge, had a initial C_{OW}/N_{OW} value around 7, close to the range 5-6, but began to rise from the second week of composting, after which it began to fall following the composting process despite the initial value. This behaviour was described by Chanyasak and Kubota, (1981) who found initial C_{OW}/N_{OW} values close to the range for mature materials. This could also mask the "true" value of maturity index.

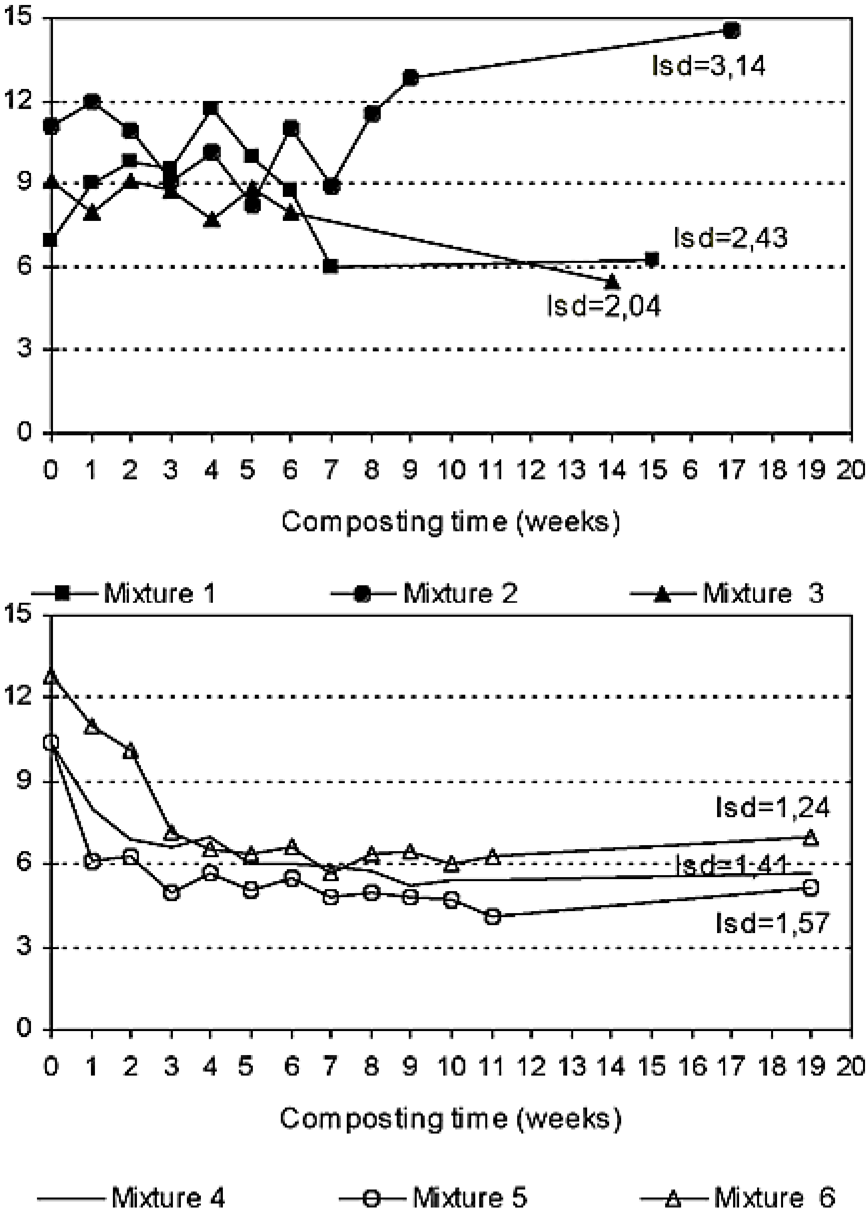

Figure 2. Changes in C_{OW}/N_{OW} ratio during composting for the different organic waste mixtures. lsd: Least significant difference at the probability level of $P<0.05$

4.2 Relationships between water-soluble organic fractions and compost maturity.

Although the results obtained were similar to those described by Chanyasak and Kubota, (1981), it is suggested that the C_{OW}/N_{OW} ratio should be used carefully as a maturity index. The calculation of this ratio requires the determination of this nitrogen form at very low concentrations, which means that the analytical error could be higher than 25%. Hue and Liu (1995) also criticised this stability index because of the analytical problems related to the determination of nitrogen at levels under 0.01 g/Kg and proposed a value of this ratio under 16 as a maturity index. They proposed using the C_{OW}/N_{OT} ratio instead of the C_{OW}/N_{OW} ratio to assess the maturity degree of composts.

Figure 3 shows the evolution of the C_{OW}/N_{OT} ratio and there was a similar pattern in all the mixtures. This value decreased progressively during the composting progress. The values varied from the initial 0.70 for mixture 3 and 1.51 for mixture 6, to reach final values for all the mixtures in the range 0.20-0.40. The values are below the limit established by Bernal *et al.*., (1998), which is more adequate than the limit of 0.70 suggested by Hue and Liu, (1995), as this value was found in some mixtures before at the termophilic phase of composting. According to the values measured for this index in this study all the mixtures reached a maturity degree after the composting process.

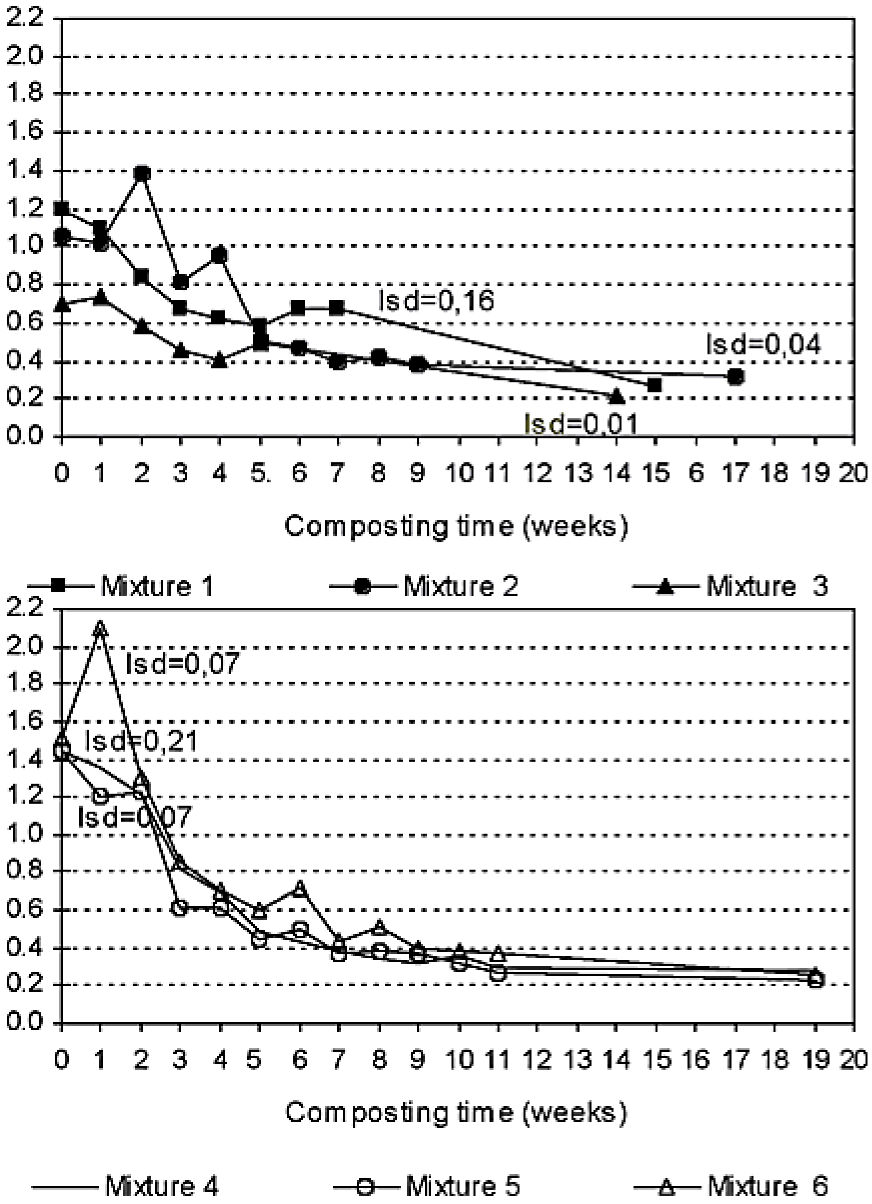

Figure 3. Evolution of C_{OW}/N_{OT} ratio during composting in the different organic waste mixtures. lsd: Least significant difference at the probability level of $P<0.05$

4.3. Germination index and study of phytotoxic substances.

Table 2 contains the germination index found for the six mixtures during the composting process. In mixtures 1, 2 and 3, prepared with high lignocellulosic content wastes, no phytotoxic effect was detected in any phase of the composting process, including the beginning of the process. In these three mixtures, organic matter degradation was not very intense which indicates that insufficient phytotoxins formed to cause phytotoxic effects on the *Lepidium Sativum* germination. In mixture 3, with the lowest organic matter degradation, the GI values were very closed to the control throughout the composting process.

The starting materials of mixtures 4, 5 and 6, prepared with MSW, completely blocked the germination. At the end of the thermophilic phase, mixture 4 had a GI under 60%, that is the limit proposed by Zucconi *et al.*., (1985) for compost without phytotoxic effects. Mixtures 4 and 5 had a GI of 74 and 92%, respectively. After the thermophilic phase, all the mixtures showed GI values over 60%. Therefore, the values of the GI in the six mixtures were in agreement with the values indicated for the C_{OW}/N_{OW} and C_{OW}/N_{OT} ratios, as maturity indices, since none of the end materials had phytotoxic effects.

The germination inhibition found for the starting materials of mixtures 4 and 5 could have been caused by a high content of low molecular weight organic acids (1.61% acetic acid, 0.74% propionic acid and 1.01% butyric acid) (table 2). The water extracts of these materials had a acetic acid concentration higher than 0.11%, that according to Devleeschauwer *et al.*., (1981) causes a germination inhibition greater than 90%. The high concentration of low molecular weight organic acids could have been the main cause for the germination inhibition because the initial concentration of the other phytotoxins (ammonium 0.16%, phenols: 0.22%, electrical conductivity: 4.6 dSm^{-1}) did not have any negative effects in the other mixtures. For example the GI of the mixtures 2 and 3, with ammonium values of 0.34 and 0.49%, respectively, did not show any phytotoxic effect.

5. CONCLUSIONS

The study of C_{OW}, carbohydrate and phenol in the water-soluble fraction of the organic matter during composting is not adequate to establish the degree of maturation of the compost since these compounds are not only involved in many degradation processes but also in formation processes from more complex starting compounds. These parameters could be used for materials with high concentrations of water-soluble compounds and which had also undergone strong degradation, as happened in the mixtures 4, 5 and 6 prepared with MSW. In all these materials the C_{OW} concentration reached final values between 0.31 and 0.48%.

The values of C_{OW}/N_{OW} and C_{OW}/N_{OT} ratios during the composting process could be used as maturity indicators for the organic matter because they were not dependent on the kind of materials used for the mixtures. Values of these ratios were found between the ranges established as a maturity index by other authors for other organic wastes, namely C_{OW}/N_{OW} values between 5 and 6, and C_{OW}/N_{OT} values under 0.55. Table 3 shows the maturity indices used by other authors and the values found in the mixtures used in the present work.

	GI (%)	NH_4-N (%)	phenols (%)	EC (dSm^{-1})	Acetic ac. (%)	Propionic ac. (%)	Butiric ac. (%)
Mixture							
1	77.8	0.14	0.37	3.90	--	--	--
1 I	65.4	0.34	0.34	4.31	--	--	--
1 T	71.1	0.08	0.23	5.03	--	--	--
1 A	69.4	0.04	0.08	6.74	--	--	--
1 M							
Mixture	87.5	0.49	0.44	3.17	--	--	--
2	82.7	0.27	0.44	4.07	--	--	--
2 I	82.2	0.02	0.12	5.81	--	--	--
2 T	81.8	0.02	0.19	6.25	--	--	--
2 A							
2 M	92.7	0.08	0.25	2.54	--	--	--
Mixture	101.7	0.04	0.15	2.43	--	--	--

3	95.5	0.04	0.12	3.21	--	--	--
3 I	94.4	0.02	0.07	3.06	--	--	--
3 T							
3 A							
3 M							
Mixture							
4	0.0	0.16	0.23	4.60	1.61	0.74	1.01
4 I	51.2	0.13	0.10	4.52	0.19	0.18	0.15
4 T	63.0	0.02	0.06	4.96	0.09	nd	nd
4 A	78.5	0.02	0.06	5.31	nd	nd	nd
4 M							
Mixture	0.0	0.16	0.23	4.60	1.61	0.74	1.01
5	73.6	0.10	0.12	5.82	0.04	0.09	nd
5 I	91.4	0.03	0.08	4.86	nd	nd	nd
5 T	87.4	0.02	0.04	5.64	nd	nd	nd
5 A							
5 M	0.0	0.19	0.25	5.50	--	--	--
Mixture	92.1	0.18	0.17	5.72	--	--	--
6	93.5	0.03	0.10	5.76	--	--	--
6 I	102.6	0.02	0.06	5.51	--	--	--
6 T							
6 A							
6 M							

--: not calculated; nd: not detectable.

Table 2. Values of the germination index (GI) and the main phytotoxic substances in water-soluble extract of the organic wastes during composting. Dry weight basis. (I: initial mixture, T: thermophilic phase, A: end of active phase, M: mature compost).

The stability degrees established by these indices were in agreement with the germination index determined using *Lepidium Sativum L.* and there were no phytotoxic effects for materials with a good maturation degree.

Maturity index	Authors	Values proposed	Values found	Comments
C_{OW}	Garcia *et al.*, 1992	< 0.5%	< 1%	Index valid only for MSW composts
C_{OW}/N_{OW}	Chanyasak and Kubota, 1981	5—6	5—6	Affected by analytical error and sludges
C_{OW}/N_{OT}	Hue and Liu, 1995 Bernal *et al.*, 1998	0.70 0.55	< 0.40	Valid for all kinds of materials
GI	Zucconi *et al.*, 1985	> 60	> 60	Valid for all kinds of materials. Fresh materials can have GI > 60

C_{OW}: water-soluble organic carbon. N_{OW}: water-soluble organic nitrogen. N_{OT}: total organic nitrogen. GI: germination index.

Table 3. Maturity indices used in other work.

6. ACKNOWLEDGEMENTS

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