

# Assessment of Maturity Degree of Composts from Domestic Solid Wastes by Fluorescence and Fourier Transform Infrared Spectroscopies

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Methods of assessment of compost maturity are needed so the application of composted materials to lands will provide optimal benefits. The aim of the present paper is to assess the maturity reached by composts from domestic solid wastes (DSW) prepared under periodic and permanent aeration systems and sampled at different composting time, by means of excitation–emission matrix (EEM) fluorescence spectroscopy and Fourier transform infrared spectroscopy (FT-IR). EEM spectra indicated the presence of two different fluorophores centered, respectively, at Ex/Em wavelength pairs of 330/425 and 280/330 nm. The fluorescence intensities of these peaks were also analyzed, showing trends related to the maturity of composts. The “contour density” of EEM maps appeared to be strongly reduced with composting days. After 30 and 45 days of composting, FT-IR spectra exhibited a decrease of intensity of peaks assigned to polysaccharides and in the aliphatic region. EEM and FT-IR techniques seem to produce spectra that correlate with the degree of maturity of the compost. Further refinement of these techniques should provide a relatively rapid method of assessing the suitability of the compost to land application.

**Keywords:** *Composting processes; compost maturity; spectroscopic characterization*

## INTRODUCTION

Recycling in agriculture of organic residues and wastes originated from various human activities, such as sewage sludges, municipal solid wastes, agro-food industry residues, wood-processing wastes, and agricultural crop residues, is recognized as a promising alternative to solid waste management. The principal contribution of organic waste utilization as soil amendment is that of restoring several fertility functions associated with “native” soil organic matter (1).

Soil application of organic wastes and residues of any nature requires, however, that these materials be properly subjected to appropriate treatments. In fact, the addition of fresh or immature organic material to soil is followed by its rapid decomposition with decrease in oxygen concentration and soil redox potential, which results in an increased mobility of reduced forms of some trace metals. The presence of simple organic acids in immature organic amendment may also be a cause of phytotoxicity. Other negative effects include a decrease of saturated hydraulic conductivity and water infiltration rate, an increase of electrical conductivity (soluble salt content) in the soil liquid phase, and modification of soil microflora (2). Therefore, the aim of these treatments is to provide a mature, stabilized organic material in which the humification process, which typically occurs under natural soil conditions, has started and has reached an adequate degree with the

production of “real” humic substances. The composting technique is an accelerated version of the processes involved in the natural transformation of organic debris in soil, which can be obtained through the provision of the most favorable conditions for microbial activity (3).

Recently, compost quality has improved due to the increasing demand for good quality compost and modifications achieved in the composting technique.

The maturity of compost, which can be defined as the degree of compost stability in physical, chemical, and biological properties, is an important factor affecting successful application in agriculture and its impact on the environment (2, 4–10). Despite the recognized importance of this aspect, no official or generally accepted regulatory standards to evaluate organic amendment quality are currently available.

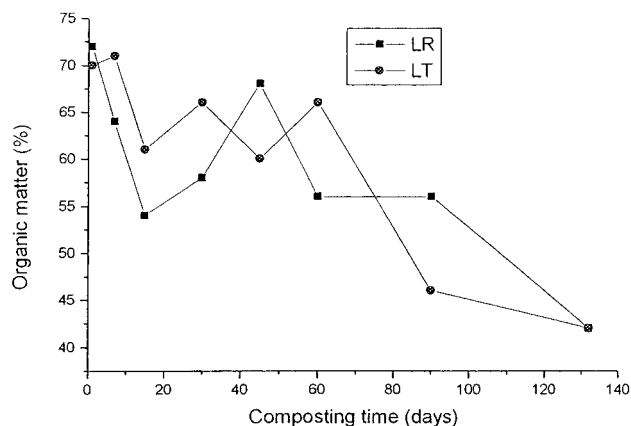
Fluorescence spectroscopy and Fourier transform infrared spectroscopy (FT-IR) have been widely applied to the characterization and differentiation of humic and fulvic acids of different origins and natures (11–14). Further fluorescence spectroscopy in the excitation, emission, and synchronous scan modes has been recently applied to the characterization of whole composts from domestic solid wastes sampled at different composting times (15).

The aim of this work is to assess the degree of maturity reached by a number of composted materials obtained from domestic solid wastes (DSW) sampled at different composting times by means of spectroscopic techniques such as excitation–emission matrix (EEM) fluorescence spectroscopy and FT-IR.

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**Figure 1.** Organic matter contents during composting of DSW.

## MATERIALS AND METHODS

Raw DSW samples were collected at a mill operating in Araraquara, São Paulo, Brazil. In this plant,  $\sim 100 \text{ Mg day}^{-1}$  of not selectively collected wastes are processed by the Sanecm System. In this system DSW is stored in a reception trench. Afterward, the material is put on a roller where metals, glasses, and other inorganic parts are removed by hand. The organic fraction (OF) is sent to a composting yard, where it is stacked.

In this experiment, the OF of the DSW was disposed in two piles of  $\sim 6 \text{ Mg}$  each. One pile was subjected to periodic aeration by manual mixing (LR samples), whereas the second pile was permanently aerated by five parallel PVC tubes penetrating the material (LT samples).

Samples were collected after composting for 1 day (LT1 and LR1), 7 days (LT2 and LR2), 15 days (LT3 and LR3), 30 days (LT4 and LR4), 45 days (LT5 and LR5), 60 days (LT6 and LR6), 90 days (LT7 and LR7), and 132 days (LT8 and LR8). To ensure sample homogeneity, 20 subsamples of each sample were randomly collected separately from the two piles at each above-mentioned composting time and thoroughly mixed and homogenized to obtain  $\sim 3 \text{ kg}$  of each final sample. Samples were then oven-dried at  $60^\circ \text{C}$  under forced air flow, ground, sieved through a  $0.5\text{-mm}$  sieve, and then stored in a refrigerator for subsequent analysis.

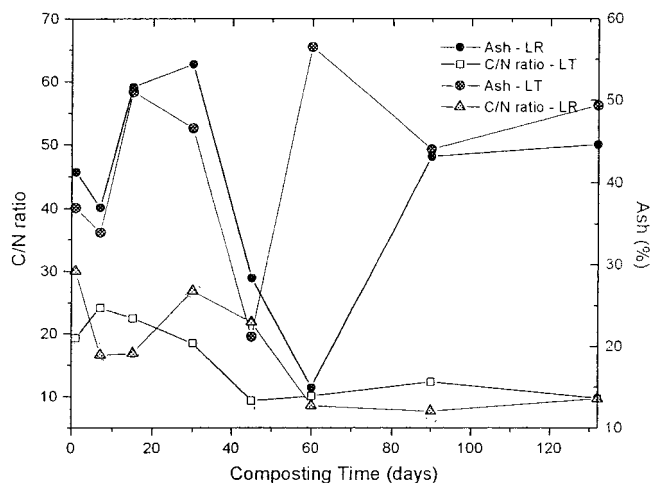
Organic matter (OM) was measured by ashing the samples in an oven at  $550^\circ \text{C}$  for 4 h. Ash content was determined by ignition of a 1-g sample at  $550^\circ \text{C}$  for 4 h. The contents of C and N were determined by an Elemental Analyzer Fisons Eager 200.

Contour maps of EEM spectra were obtained on water extracts of whole composts at a concentration of  $500 \text{ mg L}^{-1}$  after overnight equilibration at room temperature ( $\sim 20^\circ \text{C}$ ) and successive filtration through Whatman No. 2 paper. For comparison with previous works (11), the pH was adjusted to 8 with  $0.05 \text{ N NaOH}$ . A Hitachi model F-4500 fluorescence spectrophotometer was used, equipped with an F-4500 system program for data processing. The emission (Em) wavelength range was fixed from 400 to 600 nm, whereas the excitation (Ex) wavelength was increased from 300 to 500 nm in 5 nm steps.

FT-IR spectra were obtained with a Nicolet 5PC FTIR. Uniformly prepared mixtures containing 1 mg of sample and 400 mg of KBr, spectrometry grade, were formed into pellets under reduced pressure. Spectra were recorded in the  $4000$  to  $400 \text{ cm}^{-1}$  wavelength range.

## RESULTS AND DISCUSSION

The OM levels are high ( $\sim 70\%$ ) at the beginning of the process and then decrease dramatically during composting, reaching a value of  $\sim 40\%$  at the end of the process, thus indicating the occurrence of extended mineralization (Figure 1).



**Figure 2.** Ash content and C/N ratio during composting of DSW.

The C/N ratios and ash contents are presented in Figure 2 as a function of composting time. The trend of the C/N ratio is an important index used for the evaluation of the efficiency of the composting process and compost maturity. The decline observed for the C/N ratio from an initial value of 20–30 in the substrate to a final value of  $\sim 10$  in compost indicates an advanced OM decomposition and the achievement of an appropriate degree of OM stabilization. In this work, at the beginning of composting, C/N ratios were 30 for LR samples and 19 for LT samples. After 7 days, the LT value rose to 24 and then decreased constantly to a value of 9 at 45 days. LR ratios, after decreasing to 16 after 7 days and to 17 after 15 days, went up to 27 at 30 days and then decreased constantly to a value of 8 after 90 days. Afterward, values did not change significantly for either set of samples until the end of composting.

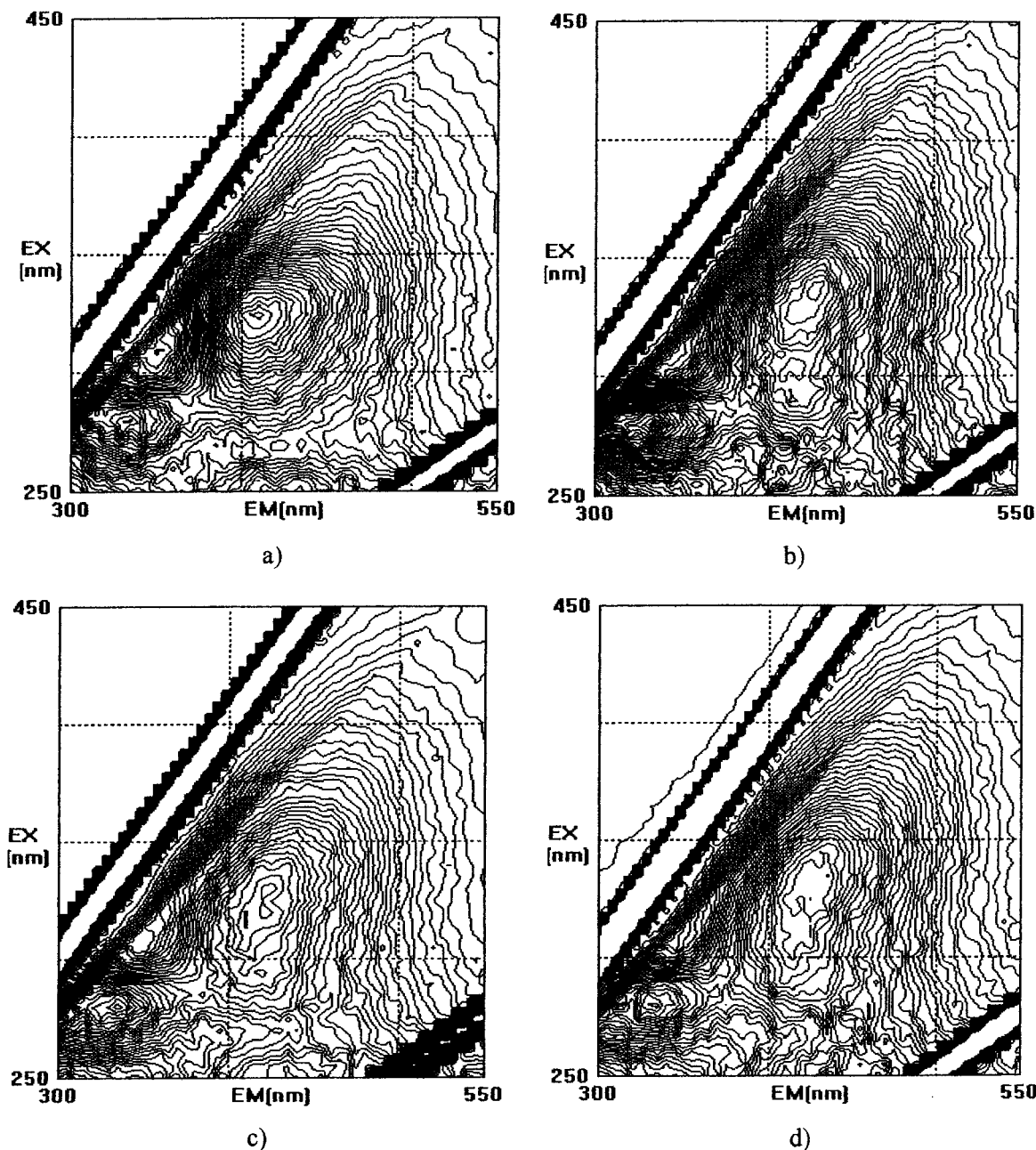
The pH of both sets of samples was 4 up to 15 days; it rose to 5.5–6 up to 90 days and became 7 at the end of composting time.

The initial ash content of the substrates was relatively high ( $\sim 40\%$ ), then decreased to a values of about 19% in sample LT5 and 15% in sample LR6, and finally increased to about 56 and 44%, respectively, in the samples collected at the end of composting.

Results of chemical analysis (pH; EC; total nitrogen,  $\text{NH}_4^+$ ,  $\text{NO}_2^-$ , and  $\text{NO}_3^-$  contents; total phosphorus; potassium) and temperature trends have been discussed in a previous paper (16).

**Contour EEM Fluorescence Spectra.** Contour EEM spectra of LR materials during composting are shown in Figures 3 and 4. The EEM spectra of the LT set of samples (spectra not shown) are similar to those of LR samples and do not exhibit any significant differences compared to those of the LR set. The spectra indicate the presence of two different fluorophores, each characterized by an Ex/Em wavelength pair. The first fluorophore is characterized by an emission/absorbance of energy at an Ex/Em wavelength pair of 330/425 nm, whereas the secondary peak is centered at an Ex/Em wavelength pair of 280/330 nm. The “contour density” of EEM maps appears to decrease strongly with composting days.

These results suggest that composted materials sampled at different composting time be constituted of



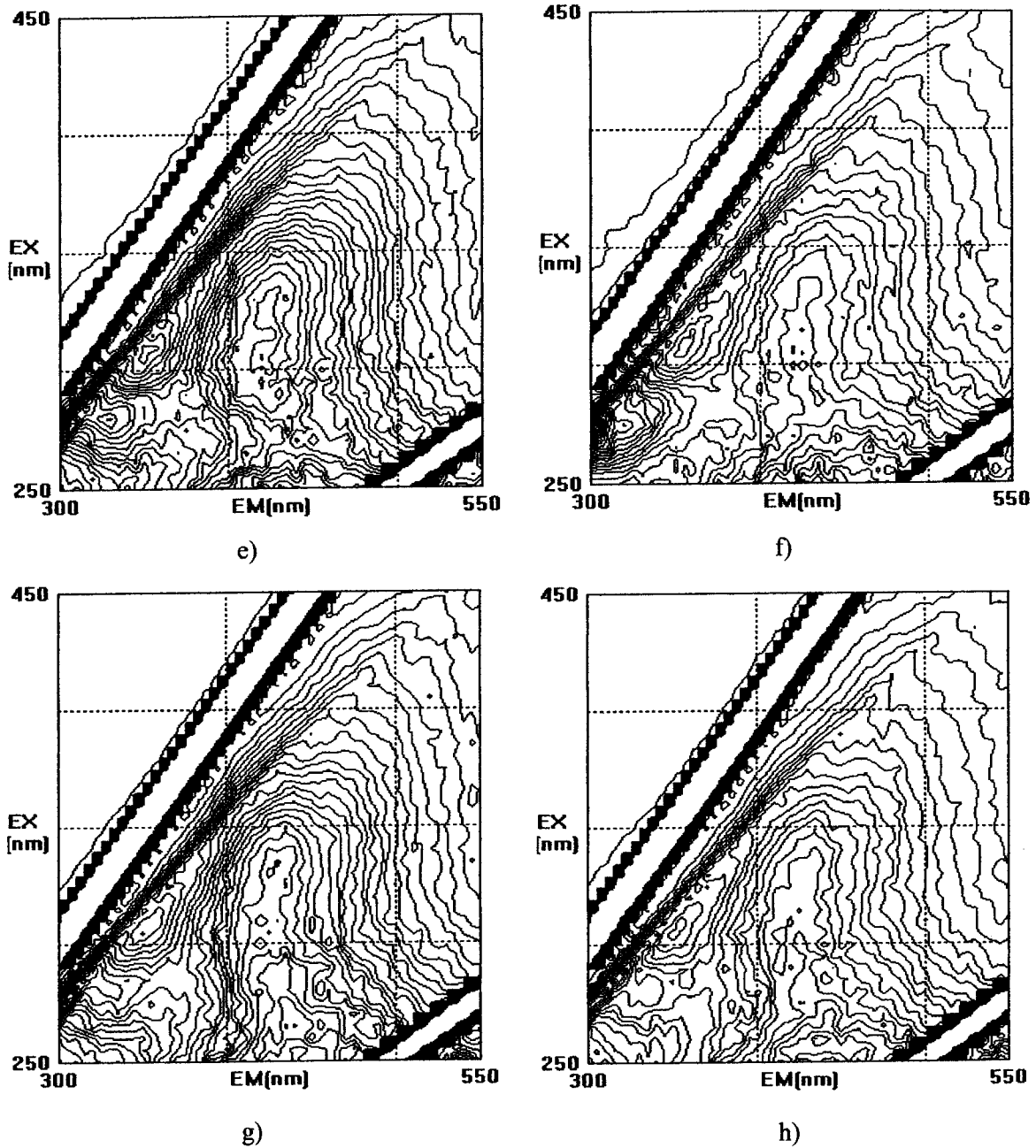
**Figure 3.** Contour EEM spectra of LR samples at 1 (a), 7 (b), 15 (c), and 30 days (d) of composting.

fresh organic matter and compounds of increasing complexity as the composting process proceeds. Thus, the relatively simple patterns of EEM spectra suggest the presence of fluorescing groups associated with simple structural units of low degree of aromatic polycondensation and/or conjugated chromophores. These results differ from excitation fluorescence spectra obtained on native soil humic acids that exhibit several peaks located at greater wavelengths due to their structural homogeneity and extended degree of aromatic conjugation and condensation (11).

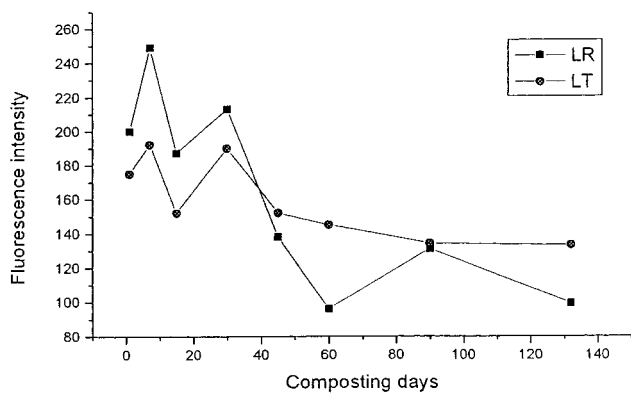
The fluorescence intensities (FI) of the primary peak ( $Ex/Em = 330/425$  nm) and of the secondary peak ( $Ex/Em = 280/330$  nm) for the two sets of composts are shown, respectively, in Figures 5 and 6 as a function of composting times. Fluorescence intensity appears to be greater for LR samples in the first 35 days of composting compared to LT samples.

The trend of the FI of the peaks provides some useful information on the evolution of OM during composting. Spectrographic results for both sets of composts are very similar and show a decreasing FI with increasing time of composting during the first 40 days of composting. Afterward, FI decreases dramatically until the final stage of composting. The initial increase of FI may be due to the increased probability of electron transition between the singlet and ground state, which occurs when decomposition occurs. This effect would result in an increased molecular complexity that produces an enlargement of the  $\pi$ -electron systems associated with aromatic structures and conjugated double bonds. The general decrease of FI with increasing composting time may be related to the extensive mineralization of OM that occurs during composting. This interpretation also accounts for the changes in contour density of EEM spectra observed during the process.

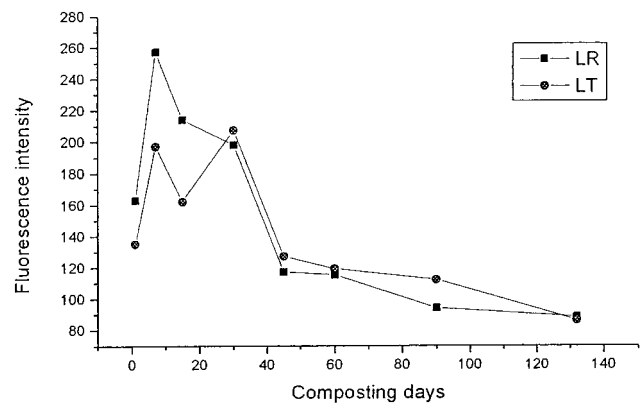




**Figure 4.** Contour EEM spectra of LR samples at 45 (e), 60 (f), 90 (g), and 132 days (h) of composting.



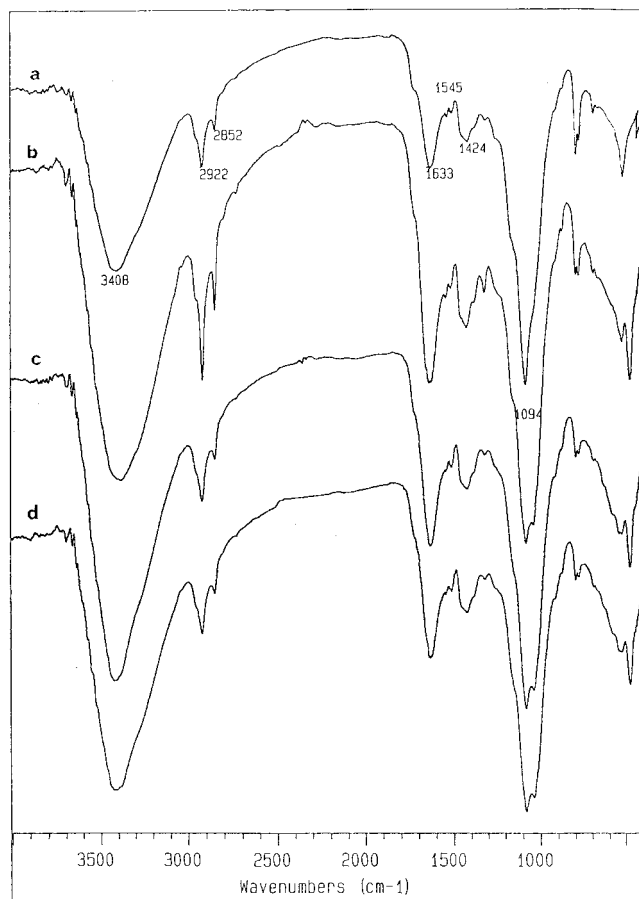
**Figure 5.** Fluorescence intensity of the primary peak of LR and LT samples.



**Figure 6.** Fluorescence intensity of the secondary peak of LR and LT samples.

**FT-IR Spectroscopy.** The FT-IR spectra of materials during composting (Figures 7 and 8) are qualitatively

similar one to another but differ in the relative intensity of absorption bands. They feature (a) a broad band at



**Figure 7.** FT-IR spectra of samples at 1 (a), 7 (b), 15 (c), and 30 days (d) of composting.

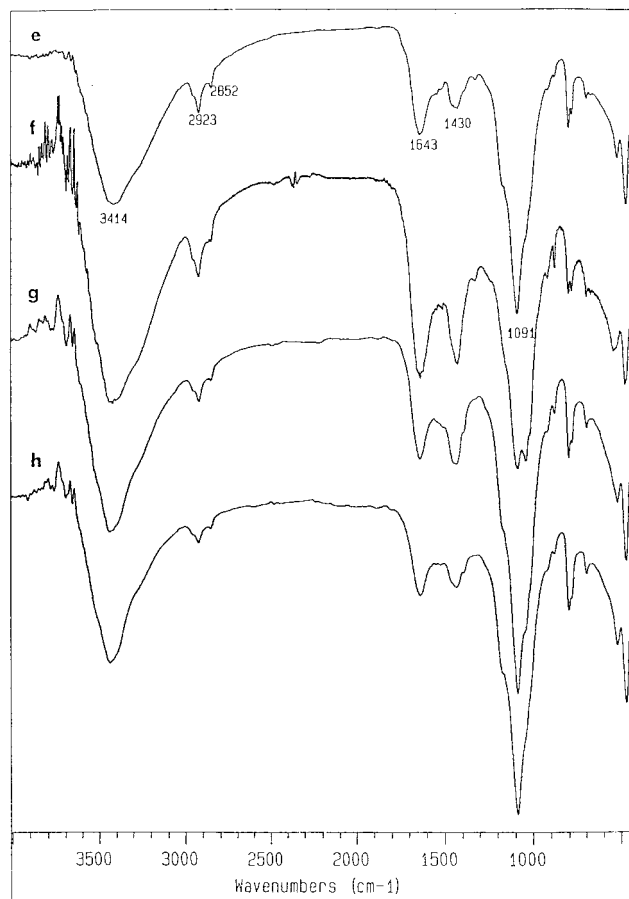
$\sim 3407\text{--}3439\text{ cm}^{-1}$  (OH groups stretching), (b) two distinct peaks at 2920 and 2850  $\text{cm}^{-1}$  (aliphatic C–H stretching), (c) a wide peak at 1640  $\text{cm}^{-1}$  (C=C in aromatic structures), (d) small peaks in the 1545  $\text{cm}^{-1}$  region (amide II vibrations), and (e) a strong band at 1090  $\text{cm}^{-1}$  (C–O stretch of polysaccharides, Si–O stretch).

Relative decreases of peak intensity in the aliphatic region at 2930 and 2850  $\text{cm}^{-1}$  and of polysaccharides at 1090  $\text{cm}^{-1}$  are particularly evident in FT-IR spectra of materials at 30 and 45 days of composting. These results suggest the occurrence of degradation and condensation reactions of organic structures. Furthermore, a strong reduction of relative peak intensity at 1643  $\text{cm}^{-1}$  and the disappearance of peaks in the 1545  $\text{cm}^{-1}$  region, particularly in samples at 90 and 132 days of composting, are also observed. These results would confirm the dramatic reduction of aromatic structures in the final stages of the composting process associated with extensive OM mineralization.

**Conclusions.** Compost maturity is difficult to assess from a single parameter; thus, several parameters should be taken into account.

This paper provides data on the characterization by EEM fluorescence and FT-IR spectroscopies of materials from DSW sampled at different composting times and subjected to two different treatments, that is, manual aeration and fixed aeration.

EEM spectra and FT-IR spectra of samples collected during and at the end of composting of DSW substrates show that periodic or permanent aeration has no relevant effect in the stabilization of OM and compost



**Figure 8.** FT-IR spectra of samples at 45 (e), 60 (f), 90 (g), and 132 days (h) of composting.

maturation. The major difference is related to the intensity of fluorescing groups, which appears to be greater in samples obtained with manual aeration in the first 35 days of composting. Thus, manual aeration seems to be more effective than the tube-permanent system.

Results provide evidence of progressive mineralization and humification of OM during composting. In the experimental conditions carried out in this work a shorter time of composting, up to 45 days, is advisable to limit severe loss of organic matter in the final product.

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