Evaluation of sewage sludge-based compost by FT-IR spectroscopy

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Abstract

The aerobic batch composting fermentations of sewage sludge with wood chips and maturity compost as co-composting additives were carried out in an open type lab-scale reactor. Fourier transform infrared spectroscopy (FT-IR) spectroscopy was used to monitor the composting process, evaluate the degradation rate and thus determine the maturity. Although the composition of the input mixture strongly affects the shape of the infrared (IR) spectra, typical bands of components can be selected and used to follow the composting process.

The appearance, shape and intensity of the nitrate band at 1384 cm^{-1} was well pronounced and evident for a sewage sludge-based compost maturity. An increase of the peak ratio 1384/2925 and decrease of 2925/1034, 1034/1384 correlated with the degree of decomposition. For the composting mixture under study the peak ratios 1034/1384 and 1384/2925 were more demonstrative. Considering the influence of the composting mixture (components and their ratio) on the shape of the FT-IR spectra the nitrate band at 1384 cm^{-1} can be overlapped by other absorption bands (e.g. lignin bands in $1300\text{–}1400 \text{ cm}^{-1}$ region) thus appearing in the spectra as a shoulder, and therefore the ratios 1384/2925 and 1034/1384 become unusable to evaluate the maturity.

FT-IR spectroscopy is a quick and useful method to monitor the composting process; however any particular composting mixture needs preliminary studies of the spectra. Consequently, the most appropriate criteria describing the process (e.g. band ratios, shape and intensity of nitrate band) should be selected and the time/peak ratio curves recorded for a compost mixture under study. Afterwards monitoring of the composting process can be based on the comparison of the controls and definite samples.
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Keywords: FT-IR spectroscopy; Sewage sludge; Composting

1. Introduction

One of the possibilities to convert sewage sludge is co-composting it with different bulking agents (sawdust, wood chips, rice husk, pig manure, etc.)

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thus obtaining a humus-like material by biological degradation. Composting is defined as a process of aerobic thermophilic microbial degradation or an exothermic biological oxidation of various wastes by many populations of the indigenous microorganisms which lead to a stabilized, mature, deodorized, hygienic product, free of pathogens and plant seeds, rich in humic substances, easy to store and marketable as organic amendment or fertilizer (Stevenson and He, 1990; Haug, 1993; Ouatmane et al., 2000). Suitable microbial population or association, aeration, temperature, moisture content, and carbon availability are the necessary factors in recent composting technology. Microorganisms involved in the biological degradation are bacteria, actinomyces and fungi, but in particular microbial associations are highly effective to form the organic matter and speedup the humification processes (Xi et al., 2002). Mature compost possesses low bulk density, high water holding capacity, an alkaline pH, and a high organic content. Many methods have been proposed to establish the degree of compost maturity (Emeterio and Garcia, 1989; Inbar et al., 1990; Hsu and Lo, 1999). The key methods are—1) physical analyses: temperature, odour, colour, particle size, water, air content; 2) chemical methods: C/N ratio in solid phase and in water extracts, pH, cation-exchange capacity and tests for ammonia, hydrogen sulphide, nitrates and nitrites, organic matter, crude fiber analysis; 3) microbiological methods: metabolic activity, cell count and the study of the easily biodegradable constituents, respirometric studies, ATP and hydrolytic enzyme activity, hydrolysable polysaccharide content, relation between total organic carbon and soluble glucides, and ratio of carbon in reducing sugars to total carbon; 4) studies of humified organic matter: content and ratios of humic and fulvic acids and non-humic fractions, the degree of polymerization of humic compounds; 5) biological methods: the germination index of seeds incubated in water extracts of the compost. Considering the heterogeneity of compost biomass the parameters generally used to determine the maturity of compost are often overestimated due to the presence of humic-like substances such as proteins, lipids and intermediate microbial metabolites (Grigatti et al., 2004). Maturity compost consists of stable organic matter,

water, minerals, and ash. Consequently the composting process directly involves the changes of the chemical composition and physico-chemical parameters of the composting mixture, and can be controlled by spectral methods. Fourier transform infrared (FT-IR) spectroscopy being a modern non-destructive analytical method more and more often is used for the structure elucidation and quantification of a large variety of organic, inorganic, and biological samples. Two main advantages of IR spectroscopy are: 1) the analysed samples are not subjected to any chemical treatment thus avoiding secondary reactions; 2) all the compounds present in the sample are measured simultaneously, thereby simplifying and speeding up the analysis. The content of the main biochemical components carbohydrates, proteins, fats, as well as lignin, cellulose, etc., can be determined from one anlysis/spectrum by IR quantitative analysis (Grube et al., 1999). Today FT-IR spectroscopy is more frequently applied in environmental analyses including soil organic matter (Olk et al., 1999; Mermut and Eswaran, 2001), humic and fulvic acids (Naidja et al., 2002; Filip and Bielek, 2002; Chien et al., 2003; Senesi et al., 2003), organic wastes (Reeves and Van Kessel, 2002; Reveille et al., 2003), and their compost (Tseng et al., 1996; Provenzano et al., 2001; Smidt et al., 2002). It was shown that the evolution of different forms of nitrogen during composting depended on the material which supplied nitrogen to the mixtures and the organic matter (e.g. sewage sludge, municipal solid waste, brewery sludge, cotton waste, pine bark) degradation rate during composting (Sanchez-Monedero et al., 2001). Considering that the IR spectra reflect the chemical composition, preliminary studies of a particular composting mixture are necessary to select the criteria indicating the decomposition processes of the organic matter.

In this study the maturity compost obtained at previous fermentation was used as a component and was not only a source of a microbial association but a nitrate-containing amendment as well. The co-composting process of sewage sludge, wood chips and maturity compost was studied by FT-IR spectroscopy. The aim of this study was to elucidate the typical IR absorption bands or band growth rates correlating with the compost maturity/degradation degree.

2. Materials and methods

The composting raw material was a mixture of sewage sludge (Min-Sheng Sewage Treatment Plant in Taipei, Taiwan), bulking agent (wood chips) and maturity compost (from the previous fermentation). Sewage sludge was as a sludge cake obtained after stabilisation, conditioning and dewatering of raw sludge. Two mixtures of sewage sludge, wood chips and maturity compost in ratio: 1) 1:0.36:0.26; 2) 1:0.5:0.28 were used. The initial moisture was 60% and 55% of the first and second composting mixture correspondingly. The content of moisture and volatile solids (VS) were determined by standard methods of NIEA of the Republic of China, NIEA R203.01T and NIEA R212.01C, respectively.

The pilot-scale composting experiments were carried out in an open-bin reactor (Fig. 1) with total volume of 110 L. A thin iron pipe with a uniform-holed loop tail was kept inside the reactor. The mixture (composting source) was fixed on a stainless disc mesh 10 cm above the bottom and aerated through the mesh covered by the loop tail at 5 L/min. Temperature was measured by a thermometer (TM-917) inserted 60 cm below the top of the composting reactor and connected with a computer to record the data.

The samples of initial sewage sludge, wood chips, mature compost and compost samples at different decompositions degrees were studied by IR spectroscopy. All samples were dried at 60 °C, ground in a coffee grinder to homogenise and 100 mg of powder ground in a Specamill (SPECAC, USA) to the particle size of 1 μm. Powdered, dry samples (7–10 mg) and 1g of dried KBr were ground to homogenize and pelted. The IR absorption spectra were collected on a FT-IR spectrometer Perkin Elmer GX FT-IR system, absorption mode 800–3100 cm^{-1}, resolution 4 cm^{-1}, 16 scans.

3. Results

3.1. Physico-chemical characteristics of compost samples

Fermentation of the first composting mixture continued for 40 days and that of the second one for 30 days. The composting process was monitored following the changes of the temperature and the VS content. The content of moisture and VS of the components (sewage sludge, wood chips and maturity compost) slightly differed in both composting mixtures. The VS content of wood chips used for the second composting mixture was about 5% higher, but the initial moisture of the composting mixture was by 5% lower (Table 1).

The temperature of the first mixture (60% moisture) increased rapidly from 32.2 to 59.2 °C and further decreased to 40 °C during 7 days, then reached another maximum (48.9 °C) on 12th day (Fig. 2.). The ambient temperature was 29.0–32.0 °C. The composting process stopped after 40 days reaching the maturity. The content of VS decreased by 4.29% (from 81.95% to 77.66%) during the composting process while the content of moisture decreased from 61.56% (day 0) to 51.03% (day 40), as shown in Fig. 3.

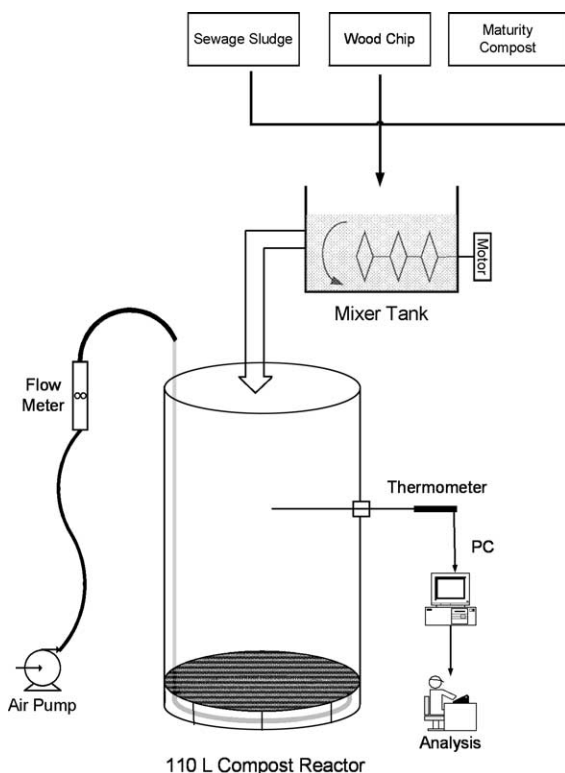During composting of the second mixture (55% moisture), temperature increased from 29.4 to 52.3 °C

Fig. 1. The composting reactor.

Table 1
Content of water and volatile solids of the co-composting sources

Co-composting source	1st mixture, initial moisture 60%		2nd mixture, initial moisture 55%	
	Moisture*, %	Volatile solids*, %	Moisture*, %	Volatile solids*, %
Sewage sludge	81.05 ± 0.63	57.51 ± 1.10	82.03 ± 0.47	56.82 ± 0.65
Wood chip	12.00 ± 0.21	82.00 ± 0.50	12.06 ± 0.09	87.67 ± 0.11
Maturity compost	50.82 ± 0.10	77.84 ± 0.19	51.03 ± 0.14	77.66 ± 0.13

* All results were the means of three replicates.

(Fig. 2.), the ambient temperature was 29.0–31.0 °C. The composting process was stopped after 30 days. During the composting process the content of VS decreased by 1.88% (from 82.10% to 80.22%), while the moisture content decreased from 57.94% (day 0) to 46.14% (day 30) (Fig. 3).

The results showed that the VS of the first mixture decreased more than that of the second mixture and the peak temperature of the first mixture was higher while the thermo phase (>50 °C) was more sustained (Fig. 2.). The total cell count and the activity of microorganisms of the first mixture were higher than that of the second mixture (data not shown). It was established that 60% of moisture was more appropriate for efficient composting.
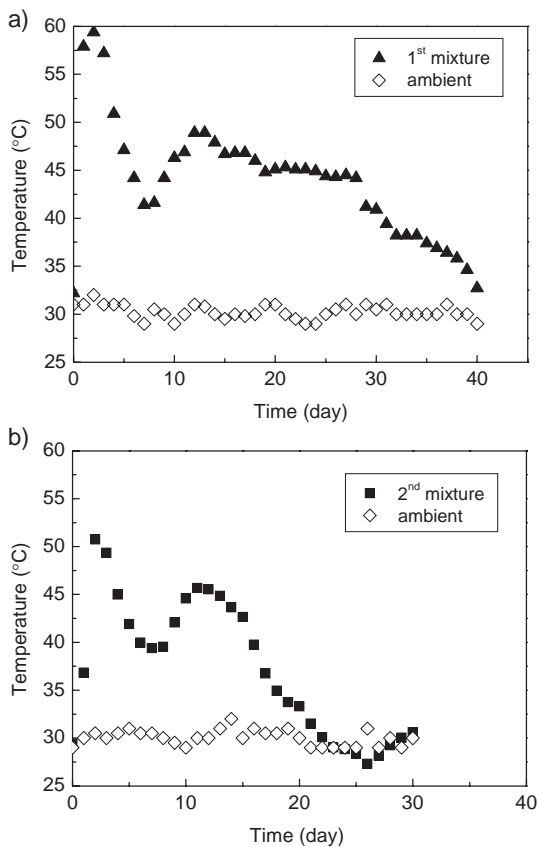

Fig. 2. The temperature curve of composting process a) 1st mixture (initial moisture 60%); b) 2nd mixture (initial moisture 55%).
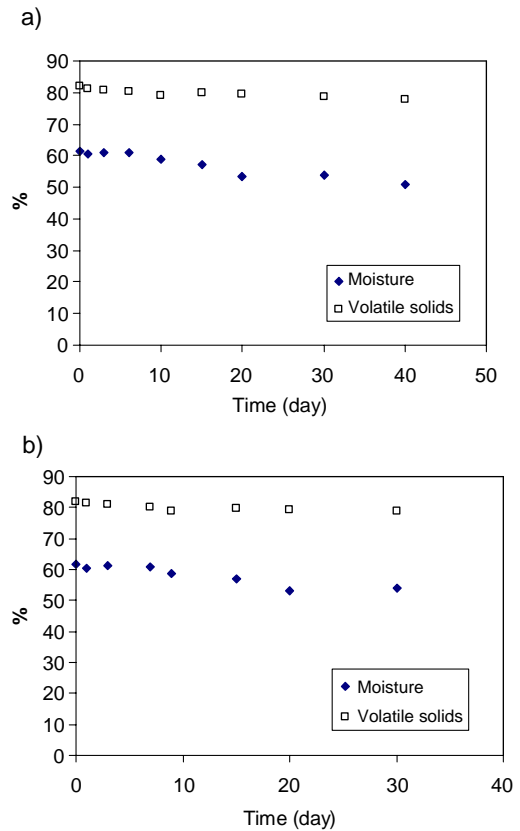

Fig. 3. Changes of moisture and volatile solid content during composting of the a) 1st mixture (initial moisture 60%); b) 2nd mixture (initial moisture 55%).

3.2. IR spectra of sewage sludge

The main absorbance in the FT-IR spectra of sewage sludge (Fig. 4.) in the region 1080–1030 cm^{-1} is assigned to C–O stretching of polysaccharides or polysaccharide-like substances, Si–O of silicate impurities, and clay minerals possibly in a complex with humic acids (Filip and Bielek, 2002; Senesi et al., 2003). Previous studies showed that the IR spectra of wastes containing sewage sludge differ from other organic wastes by an intensive, broad, typically asymmetric absorption band with maximum at 1034 cm^{-1} (Smidt et al., 2002). Two broad bands at 1540, 1654 cm^{-1} are of protein origin (Amide II and Amide I correspondingly) but the Amide I band disappears in a composted sewage sludge spectra. In the spectra of sewage sludge the absorption bands at 2925 and 2855 cm^{-1} are attributed to aliphatic methylene groups and assigned to fats and lipids. Lipids are an important fraction of sewage sludge that could influence the water retention capacity of amended soils, their structural stability and the biodegradation–humification balance in soils (Reveille et al., 2003).

The intensity changes of bands at 1034–1040 and 2925–2930 cm^{-1} testify the decomposition processes of organic components and can be used to evaluate the composting processes (Chefetz et al., 1998; Smidt and Lechner, 2001; Smidt et al., 2002). Comparison of the IR spectra of sewage sludge from the WTP's in Latvia (Daugavgriva), Lithuania (Raseiniai), Taiwan (Min-Sheng), and Austria, showed quantitative differences but not qualitative composition peculiarities (Grube et al., 2003a, 2003b; Smidt and Lechner, 2001).

3.3. IR spectra of wood chips

Comparison of the IR spectra of wood chips (Fig. 4) and the previously published IR spectra of sawdust, cellulose and lignin (Marchessault and Liang, 1960; Zbankov, 1964; Marchessault and Sundararajan, 1983; Tucker et al., 2001; Kacurakova et al., 2002;
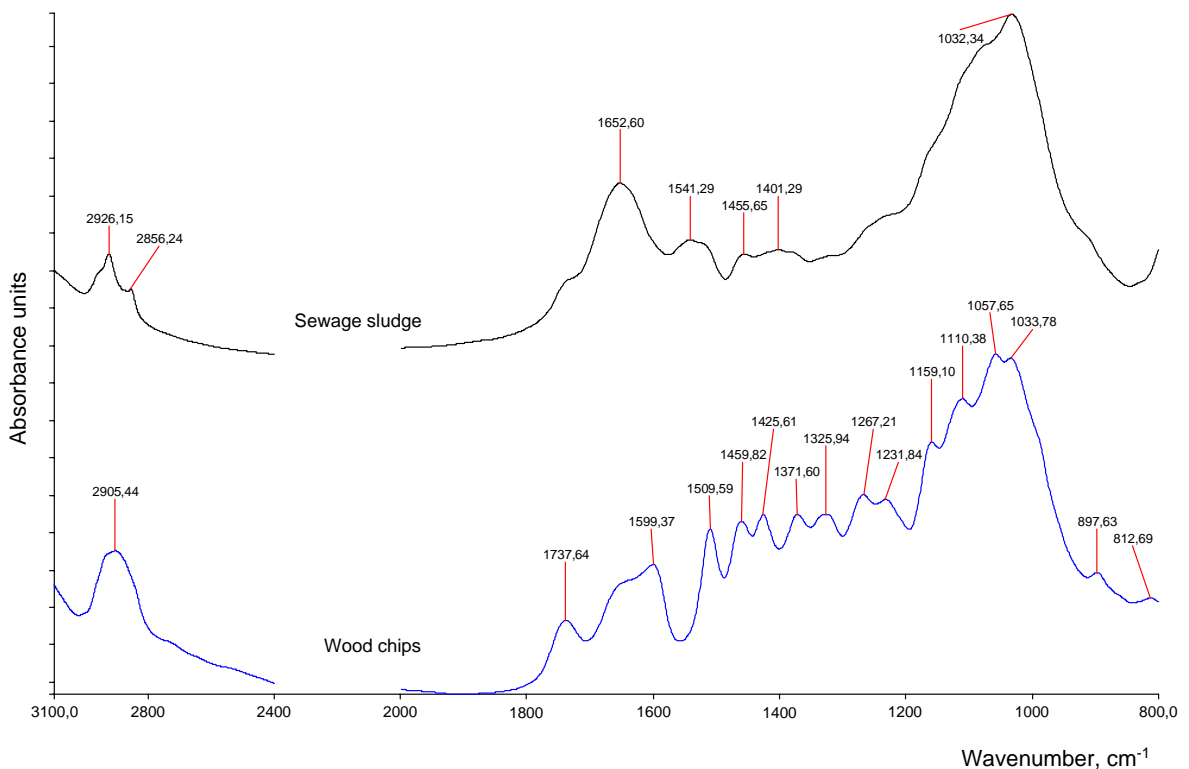

Fig. 4. FT-IR spectra of sewage sludge and wood chips.

Prachayawarakorn and Anggulalat, 2003; Dai et al., 2004) showed a strong similarity of the main absorption bands in the 900–1740 and 2800–3000 cm^{-1} regions.

The absorption bands in the in 950–1200 cm^{-1} region correspond to the valent C–O, C–C and deformation vibrations of ring structures of CH$_2$OH origin (Zbankov, 1964). The most intensive broad absorption band appears in the characteristic carbohydrate region with maximums at 1058 and 1033 cm^{-1} assigned to vibrations of C-3-H–O-3-H and C-6-H$_2$–O-6-H of cellulose groups and pyranosyl-ring vibrations, and 1160 cm^{-1} is assigned to asymmetric stretching of C–O–C of glycosidic link (Bower and Maddams, 1989). Absorption bands in the region 1500–1600 cm^{-1} could be assigned to the aromatic rings of lignin, 1371 cm^{-1} to deformation vibrations of CH$_2$ in xyloglucans, 1426 cm^{-1} to bending vibrations of CH$_2$ of cellulose, 1738 cm^{-1}—stretching vibrations of esters in pectins (Kacurakova et al., 2002). Peaks at 1510, 1460, 1420, 1270, 1230, 1130 cm^{-1} are typical for lignin (Matias et al., 2000;

Zancada et al., 2003). The broad band with maximum at 2905 cm^{-1} is assigned to CH stretching vibrations in cellulose (Marchessault and Liang, 1960).

Stone et al. (2001) showed that compositional changes during composting are similar to those observed during natural wood decomposition: carbohydrates were degraded more rapidly than lignin and the organic matter was degraded relatively non-selectively. Complex organic compounds like lignin are mainly degraded by thermophilic micro-fungi and actinomycetes. Tuomela et al. (2000) assumed that humus is formed mainly from lignin and is not totally mineralized during composting.

3.4. Evaluation of the composting process by FT-IR spectra

Previously we studied composting of sewage sludge with different additives: meat–bone meal, pig manure digestate, saw dust, cheese whey, brewer's yeast and cellulose splitting bacteria *Trichoderma lignorum, Trichoderma viride* (Grube et al., 2003a,

Table 2
Assignment of the principal IR absorption bands in the spectra of sewage sludge, wood chips and their compost

Wave number, cm^{-1}	Assignment	Characterisation	Reference
2925, 2855	Aliphatic methylene groups	Both bands are found in all waste samples and assigned to fats and lipids	Chefetz et al., 1998; Smidt and Lechner, 2002; Reveille et al., 2003
1650	Amide I (H-bonded C=O carbonyl stretch)	A band of protein origin	Bellamy, 1975; Filip and Bielek, 2002
1600–900	Cellulose and early decomposition products: aldehydes, ketones, esters, and carboxylic acids with short C-chains	These are characteristic bands of the input materials like wood, fruits, grass, and other plants	Marchessault and Liang, 1960; Tucker et al., 2001; Smidt et al., 2002; Dai et al., 2004
1540–1580	Amide II	A band of protein origin. The band is found in nitrogen-rich composts but not found in composted sewage sludge samples	Bellamy, 1975; Smidt et al., 2002
1510, 1460, 1420	Stem from waste components containing lignin	Wood and similar bulking agents additive to sewage sludge is evident by these characteristic bands	Ouatmane et al., 2000; Stone et al., 2001; Gonzalez et al., 2003
1384	Nitrate band	Sharp, stable, reproducible. Appears towards the end of the process when the material is well composted	Chefetz et al., 1998; Smidt et al., 2002; Reveille et al., 2003.
1080–1030	C–O streching of polysaccharides or polysaccharide-like substances. Si–O asymmetric stretch of silicate impurities	Sewage sludge and sewage sludge-based compost can be distinguished from others by the broad band with maximum at 1034 cm^{-1}	Bellamy, 1975; Filip and Bielek, 2002; Senesi et al., 2003; Smidt et al., 2002

2003b). The spectra of these samples were different—determined by different additives, but the absorption bands assigned to carbohydrates (1000–1100 cm^{-1}) and aliphatics (2850–2935 cm^{-1}) were common and evaluation of the composting processes can be based on the changes of the band relative intensity. This is in agreement with a conclusion made by Provenzano et al. (2001) that the FT-IR spectra of organic matter during composting are qualitatively similar but differ in the relative intensity of absorbance bands. The assignment of the principal absorption bands of sewage compost is shown in the Table 2. Comparison of the IR spectra of sewage sludge, wood chips and the composting mixture (Figs. 4 and 5) showed that the spectra of a composting mixture is a superposition of all components and the most intense was a broad absorption band at 1034 cm^{-1}. Occurrence of the characteristic bands of lignin: 1510, 1460, 1420 cm^{-1} (Ouatmane et al., 2000; Stone et al., 2001; Gonzalez et al., 2003) was evident for wood chips. The most obvious differences in the spectra of compost samples at different stages of degradation were in the 1350–

1400 cm^{-1} region where a nitrate band of characteristic shape appeared (Figs. 5 and 6). Usually the nitrate band is detected exclusively at later composting phase when the material is well composted (Chefetz et al., 1998; Smidt et al., 2002; Reveille et al., 2003) and indicates the state of decomposition at which nitrogen from decomposed components is oxidised. The wave number position is stable and highly reproducible at 1384 cm^{-1}. Later when plants absorb nitrate or it is washed out by leaching processes, this band disappears. Since in our study the maturity compost was one of co-composting amendments a weak nitrate band appeared in the spectra of the first day compost samples and disappeared after 6 days, probably because nitrogen was utilized by microflora of the composting mixture. After 15 days of composting the nitrate band appeared, increased until the end of composting, and was highest in the maturity compost sample. Koivula and Hanninen (2001) showed that the nitrogen content was higher in all humic fractions of the old bale and could be an indicator of microbial enrichment. As
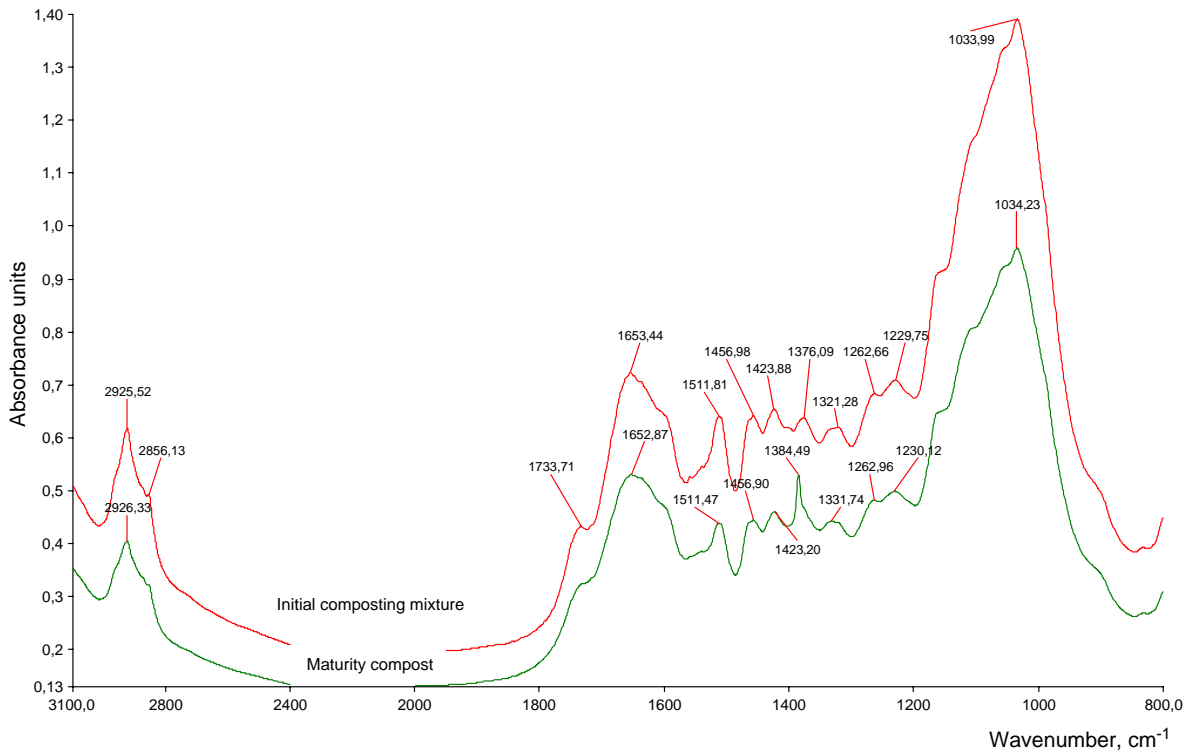

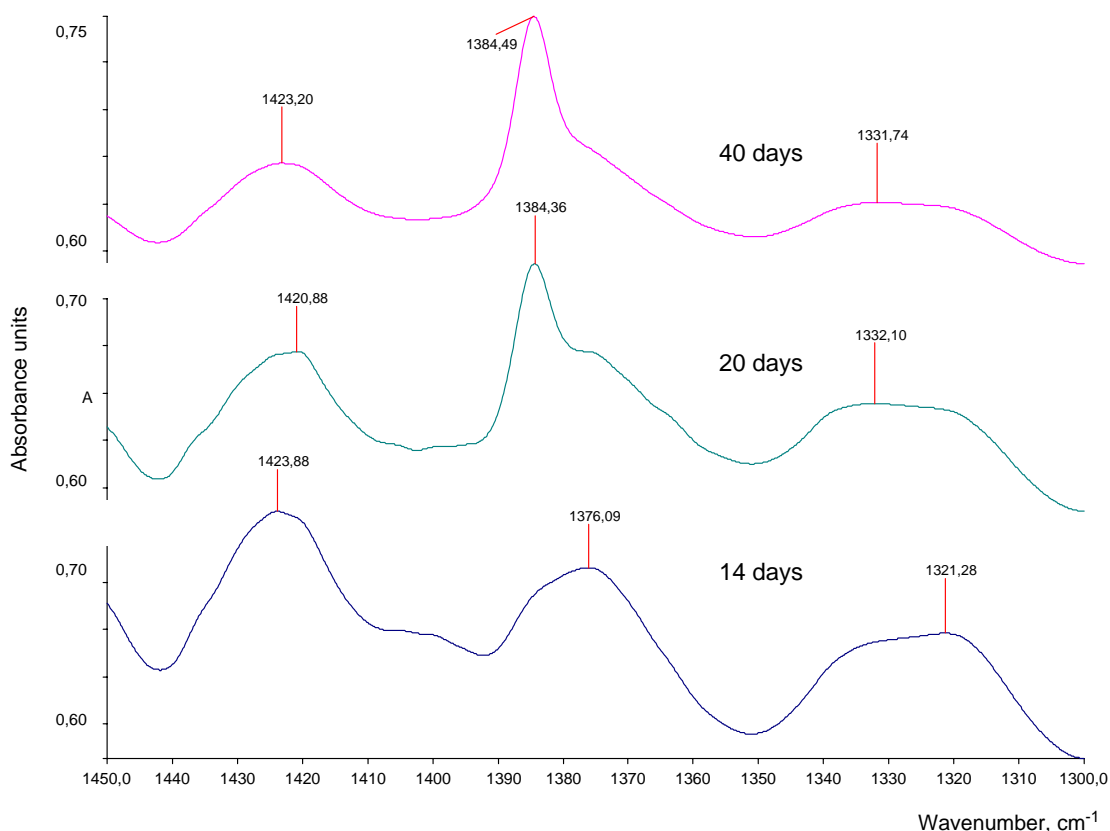Fig. 5. FT-IR spectra of initial composting mixture and maturity compost.

Fig. 6. The nitrate band at 1384 cm^{-1} after 14, 20 and 40 days of composting.

microbes die, their nitrogen becomes available to living microbes, and microbial utilisation of cellulose may intensify with time.

Quantitative evaluation of the composting process and maturity is based on a comparison of the relative intensity ratio of selected absorbance bands. Smidt et al. (2002) showed that decrease of the ratios 2925/1034 and 2925/1640 indicate the progress of composting and consequently higher decomposition degree. This is in agreement with the studies of the whole compost FT-IR spectra carried out by Hsu and Lo (1999) who also showed that easily degradable organic matter components, such as aliphatic chains, polysaccharides, alcohols and protein, are decomposed and therefore, the mature compost contained more aromatic structures of higher stability. Tseng et al. (1996) evaluated the degradation of organic matter by a quantitative FT-IR spectroscopy technique and demonstrated that the polysaccharide content

decreased approximately for 50% while the lignin content remained unchanged.

It is known that the C:N ratio decreases as compost becomes more mature or stable and con-
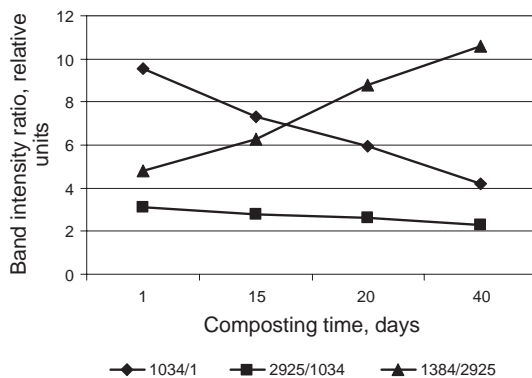

Fig. 7. Band intensity ratio time curves.

sequently, the C : N ratio is used as an indicator of the compost stability. In the IR spectra the C : N ratio can be assigned to the bands 1034 and 1384 cm^{-1}. On the other hand the development of band at 2925 cm^{-1} is used to indicate the decomposition of organic mater. Thus in this study the ratios of 1034/1384 and 1384/2925 were used and their time curves were more pronounced in comparison with the ratio 2935/1034 (Fig. 7). The decrease of the ratios 1034/1384 and 2925/1034 and increase of the ratio 1384/2925 indicated the progress of the decomposition process.

4. Conclusions

Although the IR spectra of organic waste compost is strongly determined by the qualitative and quantitative composition of the input material it allows to track the degradation of organic molecules and the concomitant generation of nitrate during the composting process and thus evaluate the degree of maturity. IR spectroscopy, being comparatively simple, quick and informative, can be used instead of several time consuming chemical methods especially for monitoring of routine composting processes. It also must be noticed that IR spectroscopy methods are environmentally friendly as produce remarkably less and "clean" wastes, and hopefully will be more estimated.

Acknowledgements

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