

# Characterization of Waste Organic Matter by FT-IR Spectroscopy: Application in Waste Science

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A series of experiments has shown that FT-IR (Fourier transform infrared) spectroscopy is a helpful tool for characterizing waste organic matter, its decomposition, and stabilization in rotting processes. A specific set of differently treated input materials, originating from various composting plants, was chosen to reflect a wide range of spectroscopic properties. The approach to FT-IR spectra interpretation is presented. Changes of relative absorbances of the band at  $2925\text{ cm}^{-1}$  (methylene groups of aliphatics) reflect the progress and dynamics of composting processes. Different processes can be compared by the specific development of their  $2925\text{ cm}^{-1}$  band. Nitrate was quantified by calibrating nitrate band heights with added amounts of  $\text{KNO}_3$ . The concentrations and band heights (absorbances) were linearly correlated ( $R^2 = 0.9968$ ,  $SD = 0.001$ ). Bands of inorganic components are useful to assess the decomposition process because they also indicate the development of organic matter. Different wastes can be distinguished by their fingerprint region ( $1500\text{--}900\text{ cm}^{-1}$ ). This region also reveals fresh and undecomposed materials. The presence or absence of specific bands provides information about the decomposition status of materials.

Index Headings: FT-IR spectroscopy; Waste materials; Organic matter; Decomposition; Stabilization.

## INTRODUCTION

Assessing the characteristics and stability of organic matter is an important issue in waste management. Before deposition, mechanically-biologically pretreated municipal solid wastes should be as "stable" as possible to ensure that ecologically harmful emissions are avoided and that material flows are adapted to natural ones. Composted wastes that are applied as fertilizer or recultivation substrates must meet the necessary requirements of stability concerning the slow release of nutrients and the decomposition of inhibiting metabolic substances. Some interesting questions in waste science focus on parameters and methods that adequately reflect the state of organic matter and its future behavior. The present practice is to use certain chemical parameters describing the quantity of organic matter (ignition loss, organic carbon, and nitrogen) or the maturity of composts (C/N ratio). Biological aerobic or anaerobic tests are more meaningful because they represent the current or potential reactivity.<sup>1</sup>

Different research methods have been applied to characterize organic matter in wastes or soils. Differential scanning calorimetry and spectroscopic methods were used for characterizing composts from municipal solid wastes.<sup>2</sup> Due to favorable properties of humic substances,

most interest was focused on them. Nuclear magnetic resonance (NMR) spectroscopy, pyrolysis-field ionization mass spectrometry (Py-FIMS), FT-IR spectroscopy, and gel permeation chromatography (GPC) also reveal structural details of the molecules and their size.<sup>3-5</sup>

The need to gain further insight into the composition of organic matter and its state of decomposition or stabilization led to the use of IR spectroscopy. This method offers some advantages. The spectrum summarizes numerous parameters to provide a more holistic view representing the complexity of the sample. Since the entire sample can be investigated, chemical preparation steps are avoided and changes in the sample are minimized. Beyond identifying relevant bands to characterize the sample, a comparison of different levels of decomposition to the spectroscopic characteristics seems possible. A comparison of the sample spectra with the spectra of the ignition residue or soils can help to assess the status of decomposition and mineralization. In order to adapt this tool to complex samples, spectra interpretation is carried out based on chemical and biological properties. Certain experience in soil and composting science improves the applicability of this method to such complex questions. The change of organic matter due to deforestation was investigated by means of FT-IR spectroscopy.<sup>6</sup> The development of organic matter in different soil layers has been described by their spectroscopic properties.<sup>7</sup> FT-IR spectroscopy was shown to be a helpful tool for characterizing decomposition processes of forest litter.<sup>8</sup> Changes of organic matter in differently managed soils were investigated using diffuse reflection infrared Fourier transform (DRIFT) spectroscopy.<sup>9</sup> This method was also applied to characterize composts, peat, and humic acids.<sup>10</sup> The addition of manures and sewage sludge to soils influences their humic acid quality. Infrared analysis of soil organic matter contributed to the knowledge of their chemical characteristics.<sup>11</sup> The humic acids in composts from different origins<sup>12</sup> and from wastes<sup>13,14</sup> have been characterized spectroscopically. Chemical properties of different waste materials are reflected by specific functional groups' contributions in the spectrum. The progress of the composting process is followed by varying absorbances of relevant bands.<sup>15</sup> Humic substance functional group composition was also investigated by means of FT-IR spectroscopy.<sup>16</sup>

The objective of our study was to apply FT-IR spectroscopy to the characterization of waste organic matter and to gain a more holistic insight into its modification during decomposition.

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## MATERIALS AND METHODS

Biowastes from different plant material, sewage sludge, and mechanically-biologically pretreated (MBP) wastes were investigated. All samples were characterized by specific chemical, physical, and biological parameters according to the ÖNORM S 2023,<sup>17</sup> which concerns methods and quality control of composts.

Apart from nitrate and ammonium, which were determined in fresh materials, all analyses were carried out on materials that were air-dried, ground in an agate mill, and sieved (<0.63 mm).

Humic acids were determined by extraction and precipitation.<sup>18</sup> Respiration activity was determined by the oxygen demand of the material.<sup>1</sup>

For spectroscopic characterization, 2 mg of the sample were thoroughly mixed with 200 mg KBr (FT-IR grade) and pressed to a pellet. The pellet was immediately measured in transmission mode under ambient conditions. The resolution was set to 4 cm<sup>-1</sup> and 32 scans were recorded, averaged for each spectrum, and corrected against the spectrum with ambient air as background. The measurement was carried out in the mid-infrared area (4000–400 cm<sup>-1</sup>) with a Bruker Equinox 55 FT-IR Spectrophotometer at the Institute of Chemistry (University of Agricultural Sciences, Vienna).

Certain characteristic bands that represent the input material and the composting process were selected to determine the relative absorbances.<sup>7</sup>

The relative absorbance (%) is the height of a distinct band multiplied by 100 and divided by the sum of all compared band heights. Band heights were measured and corrected referring to the chosen baseline by the Perkin Elmer Software "Spectrum lite". The chosen baseline for calculating a distinct band height was the same for each sample. Figure 1 shows the procedure of band height measurement.

For biowastes, ten bands were used to calculate relative absorbances: 2925, 2850, 1640, 1430, 1320, 1260, 1160, 1030, 910, and 875 cm<sup>-1</sup>. For quantitative nitrate-N determination, the calibration was carried out by adding KNO<sub>3</sub> to a biowaste sample corresponding to 0.1, 0.2, 0.5, and 1% of NO<sub>3</sub>-N. Nitrate is indicated as nitrate-N according to the ÖNORM S 2023.<sup>17</sup> Relative absorbances were calculated using the ten band heights indicated above and the nitrate band (1384 cm<sup>-1</sup>) height.

## RESULTS AND DISCUSSION

**Spectral Characteristics of Different Wastes. Sewage Sludge (SL).** Figure 2 shows the development of sewage sludge (SL) from fresh to composted material. Different stages of fresh, anaerobically stabilized, and composted SL are reflected by their spectroscopic characteristics. The stretching vibration of bonded OH groups and water causes the broad band that is found at around 3400 cm<sup>-1</sup> in each waste material. At 3695 and 3620 cm<sup>-1</sup>, two bands appear when SL is composted (Fig. 2c). They can be assigned to stretching vibrations of silanol SiO-H.<sup>19</sup> They are not found in fresh or stabilized SL. They are therefore a good indicator for aged materials. The band of the Si-O-Si asymmetric stretch is found at around 1030 cm<sup>-1</sup>. The silanol Si-O stretch at around 915 cm<sup>-1</sup> only appears at an advanced stage of mineral-

ization. Wastes containing SL compost can be distinguished from other wastes by the huge band at 1030 cm<sup>-1</sup>. Carbonates absorb at 1430 and 875 cm<sup>-1</sup>, and phosphate absorbs between 600 and 500 cm<sup>-1</sup>.<sup>19</sup> The sharp nitrate band appears exclusively at a later phase. Its presence indicates the state of decomposition at which nitrogen from decomposed components is oxidized. Its wavenumber position is stable and highly reproducible at 1384 cm<sup>-1</sup>. Later, when nitrate is absorbed by plants or washed out by leaching processes, this band disappears again. This was observed in spectra of aged SL deposits and confirmed by wet-chemical analyses. Inorganic compounds (silica, silanol, carbonate, nitrate, phosphate) exhibit very prominent bands whose positions hardly change. Inorganic bands can easily be distinguished from organic bands by their size and shape. The silica band at 1030 cm<sup>-1</sup> typically has an asymmetric shape at the top.<sup>19,20</sup> Due to the relative increase of the inorganic part during the composting process, the corresponding bands enlarge as well. Apart from the stable wavenumber position, this serves as a further hint to their identification.

The bands at 2925 and 2850 cm<sup>-1</sup>, which are attributed to aliphatic methylene groups, were found in all waste samples. Due to their general presence and the relation of their decrease to the decomposition of waste materials, these bands are very useful for comparing composting processes. The band at around 1640 cm<sup>-1</sup> can be attributed to vibrations of C=C bonds and C=O in carboxylates, amides, and OH bending vibrations from water,<sup>7,11</sup> which is adsorbed by functional groups in organic matter and inorganic constituents.<sup>21,22</sup> In consequence of decomposition, their ratio changes and the contribution of each component can not be ascertained.<sup>23</sup> However, a considerable decrease of the band at 1640 cm<sup>-1</sup> takes place when SL is composted. The band at 1540 cm<sup>-1</sup>, which is very large initially, can be assigned to amide II (Figs. 2a and 2b). This band was found in nitrogen-rich composts from spent coffee and domestic solid wastes.<sup>15,24</sup> It is not found in composted SL samples. The band at around 1240 cm<sup>-1</sup>, which can be attributed to C-O stretch vibrations of carboxylic acids or C-N stretch vibrations by amide II,<sup>19,25</sup> decreases to a shoulder or disappears completely when the compost reaches maturity.

**Mechanically-Biologically Pretreated (MBP) Municipal Solid Wastes.** The spectra of SL and mechanically-biologically pretreated (MBP) materials are very similar, but those of the latter are less differentiated. MBP solid wastes show the same inorganic bands that SL does. Both have an intense carbonate band at 1430 cm<sup>-1</sup>, although that of MBP wastes is higher. This information can be used to differentiate MBP from SL. During decomposition, inorganic bands increase. Again, nitrate appears at a later stage of decomposition. In the 23-week-old sample, nitrate becomes clearly visible.

Figure 3 shows the development of MBP material. The spectra reveal the characteristics of organic matter after 1, 12, and 23 weeks of decomposition (process I, see Fig. 3A) and after 2, 5, 10, and 13 weeks of decomposition (process II, see Fig. 3B). As mentioned above, the decrease of the 2925 cm<sup>-1</sup> band indicates the progress of decomposition. In process II, the band at 1640 cm<sup>-1</sup> does not distinctly diminish. The 1540 cm<sup>-1</sup> band is only visible in process II after 5 weeks. It disappears during the

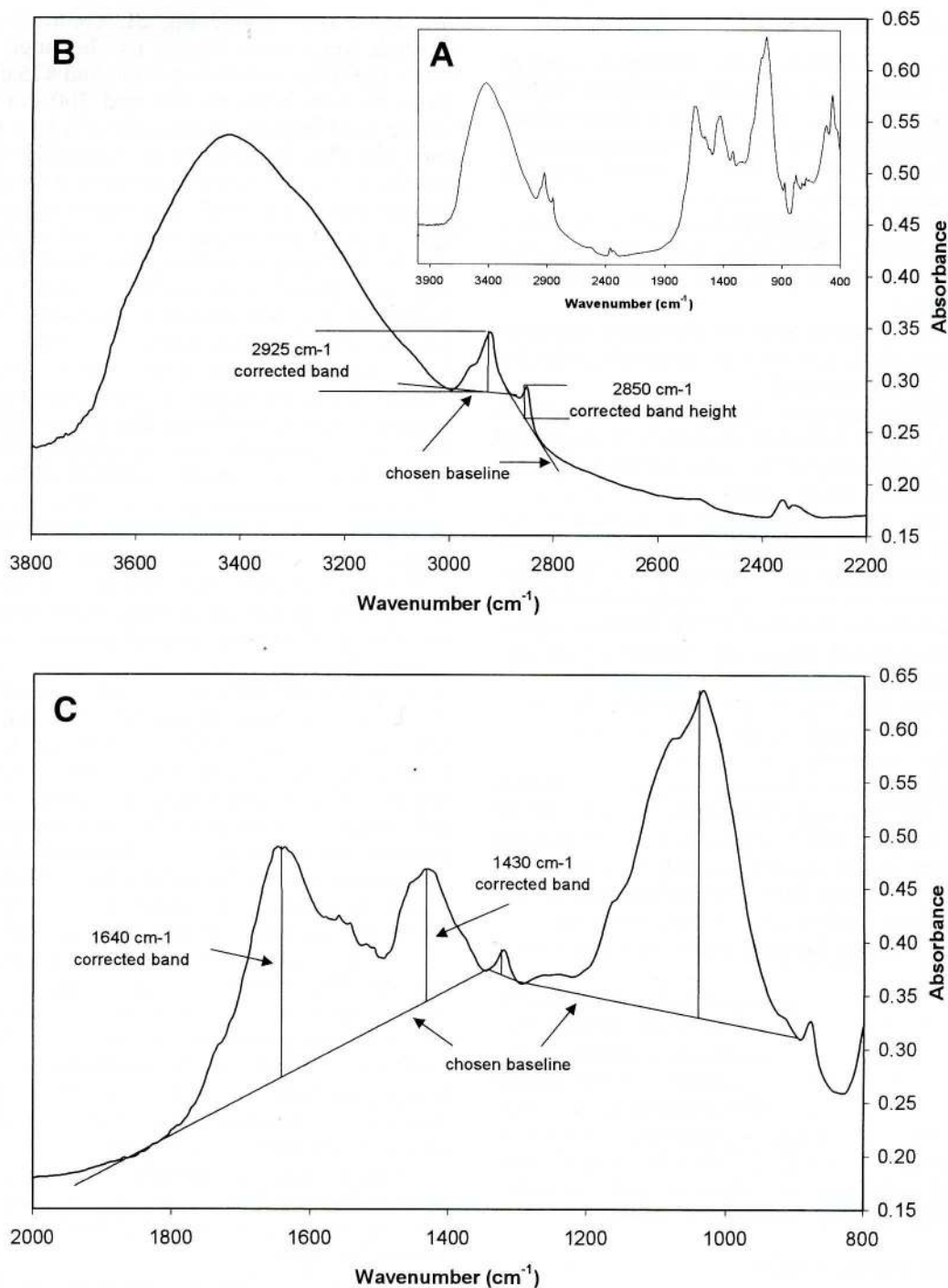


FIG. 1. Band height measurement is demonstrated for several relevant bands. The band height is corrected by the Perkin Elmer Software "Spectrum lite" referring to the chosen baseline. (A) The whole spectra; (B) measurement of the corrected band height of the bands at 2925 and 2850  $\text{cm}^{-1}$ ; (C) measurement of the corrected band height of bands.

composting period. The 1320  $\text{cm}^{-1}$  band is still present after 13 weeks (process II), whereas it has disappeared after 12 weeks in process I. Shoulders at the left side of the huge silica band can be assigned to polysaccharides that absorb in the area between 1170 and 950  $\text{cm}^{-1}$ .<sup>20</sup> Two shoulders at the left side of the silica band are still found in aged samples: one shoulder at 1160  $\text{cm}^{-1}$ , the other at 1080  $\text{cm}^{-1}$ . They are caused by stretch C–O–C group vibrations in cyclic structures.<sup>24,26,27</sup> They are stronger in the material from process II. Table I provides back-

ground information about conventional parameters of both materials. The higher content of organic matter (ignition loss in % of dry matter) and ammonium and the higher respiration activity (=RA as  $\text{mg O}_2/\text{g dry matter}$ ) after 10 and 13 weeks in process II indicate slower decomposition progress. This is reflected by spectroscopic characteristics. Nitrate becomes visible after 23 weeks (process I), when the concentration exceeds a certain value.

**Biowaste.** As far as inorganic bands are concerned,

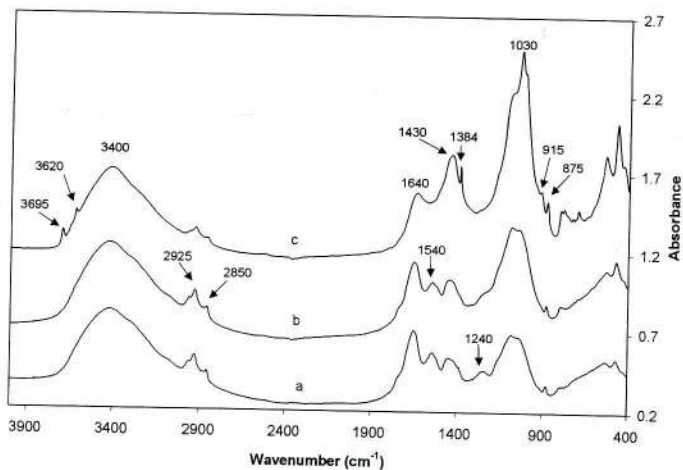


FIG. 2. Development of sewage sludge (SL) from fresh to composted material. (a) Fresh material, (b) anaerobically stabilized material, and (c) composted material.

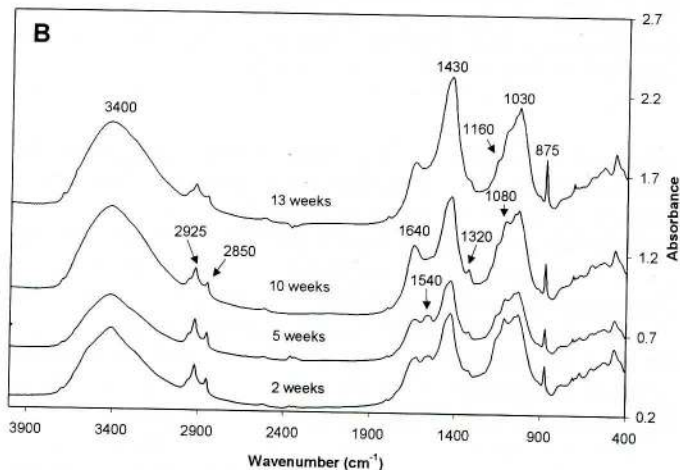
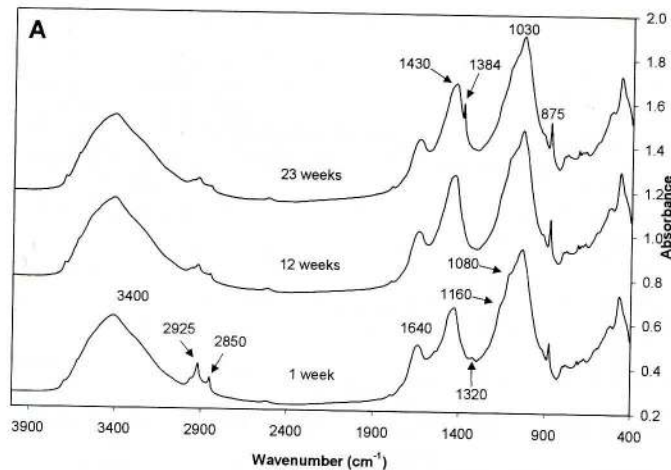


FIG. 3. Comparison of two composting processes of MBP material ((A) process I, (B) process II) proceeding at different speeds. Particular stages of decomposition and stabilization can be distinguished by spectral characteristics.

each band is not possible and not necessary. But the variety of bands reveals immaturity or, in other words, undecomposed material. Most of the bands observed at the beginning disappear during the composting process. Samples therefore become more similar to one another and less distinguishable. This phenomenon reflects the chemical development of uniform metabolic products. Thus, the similarity of compost spectra can be interpreted as an indicator of compost maturity.<sup>15</sup>

**Measurement of Band Heights and Calculation of Relative Absorbances.** Two bands are very useful for comparing relative absorbances because they are found in every sample: the band at 2925 cm<sup>-1</sup>, which can be

biowaste spectra (Fig. 4) do not differ distinctly from SL and MBP materials. The carbonate bands at 1430 and 875 cm<sup>-1</sup> and the silica band at 1030 cm<sup>-1</sup> are regularly found in biowastes. Carbonates can contribute significantly to the inorganic content of a sample. Inorganic bands are less prominent in biowastes than in SL and MBP materials. Figure 4 shows the different characteristics of fresh (Fig. 4a) and composted (Fig. 4b) biowaste material. The sharp nitrate band appears when the material is well composted.

The 2925, the 2850, and the 1640 cm<sup>-1</sup> bands decrease during decomposition. A small band at around 1710 cm<sup>-1</sup> can be related to the C=O stretch of aldehydes and ketones.<sup>22,28-30</sup> It is present only briefly. Some small bands at around 1510 cm<sup>-1</sup> stem from waste components containing lignin.<sup>15</sup> These bands are identified in biowastes due to their content of wood and plants that are rich in lignin. The addition of wood and similar bulking agents to sewage sludge or MBP wastes is evident by these characteristic bands. In composts from biowastes, the 1320 and 1240 cm<sup>-1</sup> bands are reduced to a shoulder.

In fresh, undecomposed biowaste materials many bands are visible in the fingerprint region from around 1600 to 900 cm<sup>-1</sup>. The bands can be attributed to cellulose and early decomposition products such as aldehydes, ketones, esters, and carboxylic acids with short C-chains. Biowastes can be distinguished from other wastes by the contribution of these characteristic bands of the input materials like wood, fruits, grass, and other plants. Bulking agents added to wastes can be recognized by these spectroscopic properties. Initially, the exact identification of

TABLE I. Comparison of two MBP-composting processes (I and II) concerning ignition loss, nitrate-N and ammonium-N content, and respiration activity (RA) expressed as oxygen demand per gram dry matter (DM). n.d.: not determined.

Weeks		Ignition loss (% of DM)		RA (4 days) (mg O <sub>2</sub> /gDM)		RA (7 days) (mg O <sub>2</sub> /gDM)		NO <sub>3</sub> -N (ppm)		NH <sub>4</sub> -N (ppm)	
I	II	I	II	I	II	I	II	I	II	I	II
1	2	33.2	45.9	30.0	49.0	47.0	78.0	90	n.d.	550	n.d.
	5		46.0		36.0		58.0		n.d.		n.d.
	10		41.1		21.0		31.0		130		150
12	13	27.2	38.9	2.2	15.2	3.5	22.2	100	130	20	160
23		25.5		2.1		2.9		450		24	

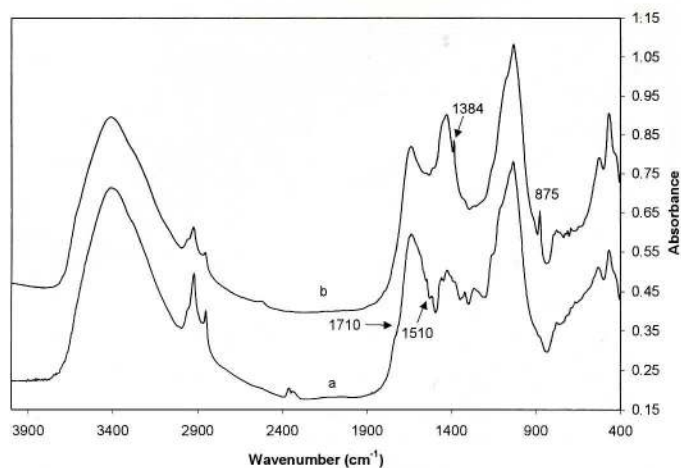


FIG. 4. The presence of distinct bands in biowaste spectra like the 1510 and the 1710  $\text{cm}^{-1}$  bands indicates an early stage of decomposition. The nitrate band ( $1384 \text{ cm}^{-1}$ ) appears at a later phase, when the material is well composted.

assigned to aliphatic methylene groups, and the silica band at  $1030 \text{ cm}^{-1}$ . The first band indicates decomposition by its decrease, the second by its increase.

Relative absorbances of the  $2925 \text{ cm}^{-1}$  band were applied to compare two composting processes of biowaste. The comparison during the first 42 days is shown in Fig. 5. The content of organic matter in biowaste B was higher than in biowaste A, which is reflected by a higher relative absorbance. The slope of the decreasing relative absorbances indicates the progress of the process.

The slow progress of process A is confirmed by chemical and biological parameters. No remarkable decrease of the ignition loss was recorded during the first six weeks. Respiration activity (RA) is lower at the beginning, but does not reach the low level of process B after 42 days of decomposition. A great oxygen demand indicates high metabolic activity and therefore the advancing decomposition. The pH value of process A samples does not change during this period, whereas it increases in material B. The increasing pH value indicates the progress of the composting process, in particular the phase when acidic metabolic substances of an early stage have been transformed. The formation of humic acids is faster in process B. Table II shows selected conventional parameters characterizing the composting processes A and B. The humic acid content is expressed by optical densities at 400 nm per gram organic dry matter (OD/g oDM).

TABLE II. Comparison of two biowaste composting processes (A and B) regarding ignition losses, respiration activity (RA expressed as oxygen demand per gram dry matter (DM)), pH value, and the formation of humic acids expressed as OD (optical density) per gram oDM (organic dry matter).

A	B	A	B	A	B	A	B	A	B	A	B
Day		Igniton loss (% of DM)		RA (4 days) (mg $\text{O}_2/\text{g DM}$ )		RA (7 days) (mg $\text{O}_2/\text{g DM}$ )		pH value		Humic acids (OD/g oDM)	
1	1	45.1	67.7	50.0	100.0	67.0	177.0	6.5	5.4	...	275
7	6	47.4	66.2	47.0	86.0	65.0	111.0	6.2	6.5	504	276
16	13	40.9	66.3	23.5	89.0	37.3	120.0	6.5	6.1	506	317
21	21	43.3	55.2	22.5	19.2	31.0	28.6	6.7	7.0	432	950
36	36	43.3	61.6	13.1	17.9	19.5	31.3	6.6	8.7	835	1800
42	41	45.4	46.3	12.6	7.9	18.4	10.3	6.4	8.8	823	2300

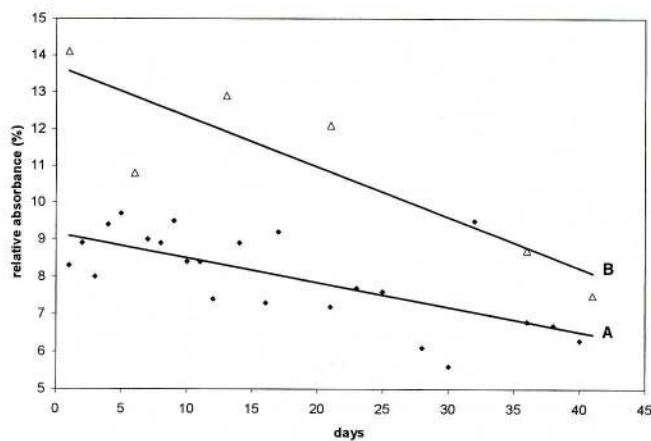


FIG. 5. The progress of two different biowaste composting processes ((A)  $\blacklozenge$ ; and (B)  $\triangle$ ) is compared by their slopes of relative absorbances at  $2925 \text{ cm}^{-1}$ .

**Quantification of the Nitrate-N Content.** Adding precise amounts of  $\text{KNO}_3$  to biowaste enabled calibration of the FT-IR method. A linear correlation between the concentration ( $\text{NO}_3\text{-N}$ ) and the relative absorbance ( $R^2 = 0.9968$ ,  $\text{SD} = 0.001$ ) provides information about the actual amount of nitrate-N in the sample. Figure 6 shows the increasing band heights at  $1384 \text{ cm}^{-1}$  according to increasing amounts of nitrate added to biowaste material. Band heights are influenced by the matrix, and thus, individual calibrations should be performed for different waste materials. This method provides additional information about the actual nitrate content in the entire sample, independent of its freshness, as demanded by the  $\text{ONORM S 2023}^{17}$  for the extraction and photometric determination of nitrate.

## CONCLUSION

Several series of waste samples representing different materials and stages of degradation have shown that FT-IR spectroscopy is a suitable method for describing complex properties such as the status of decomposition and stabilization of organic waste matter. FT-IR characterization adds much more information about a specific sample than conventional parameters such as ignition loss or total organic carbon and nitrogen, albeit there is no simple correlation between chemical and biological properties and spectroscopic characteristics. However, conventional parameters are still needful for spectra interpretation. Spectra of the entire sample especially provide more

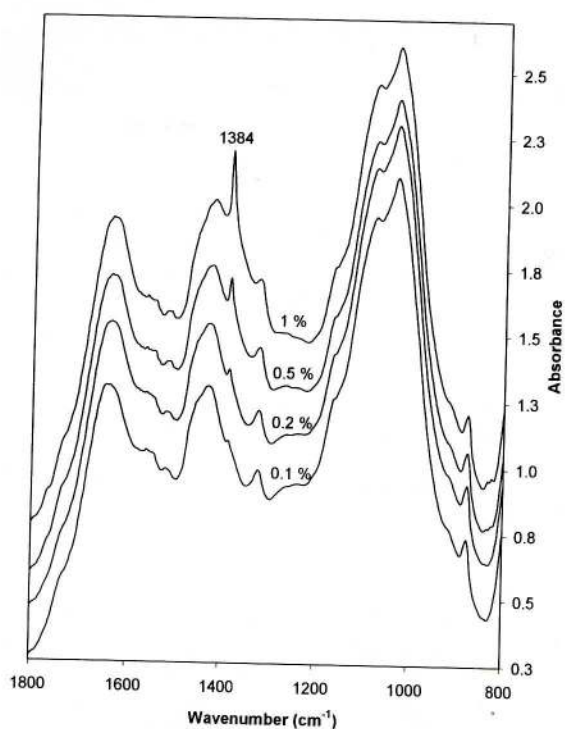


FIG. 6. Increasing nitrate band at  $1384\text{ cm}^{-1}$  due to addition of increasing amounts of  $\text{KNO}_3$  (0.1, 0.2, 0.5, and 1.0% nitrate-N) for calibration.

qualitative information about the organic matter in treated waste materials as they also include bands of inorganic compounds, reflecting their interdependency, which influences the kinetics of processes.

State and stability of organic matter can be assessed based on the appearance and disappearance of distinct bands such as those at  $1540$  or  $1320\text{ cm}^{-1}$ . Composting processes with different treatments, input materials, and storage and environmental conditions can be compared by the development of relative absorbances at  $2925\text{ cm}^{-1}$ . The run of relative absorbances over time can yield information about the velocity of decomposition, as well as about the progress and kinetics of the process.

A collection of spectra from samples representing the desirable quality will be used as a benchmark for organic matter stabilization and, due to its simple and fast practicability, the adaptation of this tool to practical application is an interesting near term possibility.

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