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Assessment of maturity of vineyard pruning compost by Fourier Transform Infrared Spectroscopy, biological and chemical analyses

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Abstract

Maturity and organic matter transformation of vineyard pruning residues characteristics composted in open wooden rotting boxes (*V*=0.25 m^3) for 122 days at initial C:N ratios of 60 (origin), 52, 47 and 29, adjusted by urea addition, were assessed by combinative means of FT-IR spectroscopy, chemical and biological analyses. The self-heating test (22 °C), the pH (7.7 to 7.9) and the germination index (93 to 105) indicate a mature compost, the C:N ratio (16 to 32) is higher than the recommended value in literature. Decreases in the C:N ratio, organic matter and phytotoxicity, an increase of the pH value, rotting degree, a strong decrease of the aliphatic compounds and an increase of aromatic carbon occurred during composting. The FT-IR spectroscopy showed a new peak at 1265 cm^{-1} appeared as P=O groups relative to phosphatic salts in treatments with urea. The addition of urea as a N source can speed the rotting process of vineyard pruning residues to get mature compost, but results in higher degradation of organic matter and losses of gaseous nitrogen in form of ammonia. FT-IR spectroscopy combined with other chemical and biological analyses could be a valuable tool for testing organic matter conversion and compost maturity of vineyard pruning residues. Ultimately, only the utilisation of compost and its effect on the plant growth can give the final answer about the maturity and quality of compost, even the effect of the compost depends on several criteria as time of utilisation and kind of crop.

Keywords: vineyard pruning; compost, maturity, FTIR

Zusammenfassung

Einschätzung der Reife von Rebschnitt-Kompost durch Fourier-Transform-Spektroskopie, biologische und chemische Analysen

Bei der Kompostierung von Rebschnitt in offenen Rottebehältern (V=0,25 m^3) während eines Zeitraumes von 122 Tagen wurden die Reife und der Umbau der organischen Verbindungen durch FT-IR-Spektroskopie und chemische und biologische Methoden untersucht. Durch Zugabe von Harnstoff wurde C:N Verhältnis auf 60 (Ausgang), 52, 47 und 29 eingestellt. Die Temperaturen beim Selbsterhitzungstest (22 °C), die pH-Werte (7,7 bis 7,9) und die Keimtests (93 bis 105) zeigten in allen Varianten einen reifen Kompost, während die C:N Verhältnisse (16 bis 32) über den Empfehlungen der Literatur lagen. Während des Rotteprozesss verringerte sich das C/N-Verhältnis, der Gehalt an organischer Substanz und die Phytotoxizität, der pH-Wert und der Rottegrad nahmen zu und es gab eine starke Abnahme der aliphatischen Verbindungen und eine Zunahme der aromatischen Kohlenstoffe. Bei den Komposten mit Harnstoffzugabe erschien bei 1265 cm^{-1} ein neuer Peak als P=O Gruppe (Phosphatsalz). Die Zugabe von Harnstoff als Stickstoffquelle kann den Rotteprozess von Rebschnitt beschleunigen, führt aber zu Stickstoffverlusten durch gasförmiges Ammoniak. Eine Kombination von FT-IR-Spektroskopie mit anderen biologischen und chemischen Analysen kann ein wertvolles Instrument sein zur Bewertung des Konversion organischer Verbindungen und der Kompostreife. Letztendlich kann aber nur die Anwendung von Kompost und seine Auswirkungen auf das Pflanzenwachstum eine Antwort geben auf die Frage nach dessen Reife und Qualität, auch wenn der Effekt von Kompost von verschiedenen Kriterien wie z. B. dem Zeitpunkt der Anwendung und der Pflanzenart abhängt.

Schlüsselworte: Rebschnitt, Kompost, Reife, FTIR

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1 Introduction

Grapes (*Vitis vinifera* L.) are the most widely grown commercial fruit crop in China. Approximately 392,400 ha of vineyards were operated in 2002, yielding approximately 3.5 million tonnes of grapes. With a share of 5.4 % of total world output of grapes, China ranked fifth in the world for the same time period (Li et al., 2003). Pruning practices are conducted annually for selected fruiting wood. This maintains vine shape and form, and regulates the number of buds retained per vine for current and sustainable yields and quality of fruits. Consequently, vineyard pruning residues are largely generated as an organic by-product during the annual dormant management season of grapevines. Based on conservative estimates, the amount of vineyard pruning residue in China was more than two million tonnes on a fresh weight basis, although the actual amounts were influenced by climatic conditions, varieties and cutting regimes (Fischer, 1982; Sanchez et al., 2002).

In the past, most vineyard pruning residues in China were burned, causing either the waste of agricultural renewable resources or a potential endangerment of human health and the environment. As agricultural sustainability and intensive production practices have been increasing in recent years, producers are also looking for alternative methods for dealing with the large volume of vineyard pruning residue generated. Composting has become a preferred method of recycling organic wastes worldwide, and by-products are applied as soil conditioners and organic fertilizer (McConnell et al., 1993; Wong et al., 1996; Butler et al., 2001). However, if unstable or immature compost is applied, it can induce anaerobic conditions since the micro-organisms utilize O_2 in the soil pores to break down the material (Mathur et al., 1993). Another problem is associated with the immaturity of organic acids during the early stages of the composting process (He et al., 1995).

The maturity of compost, which can be defined as the degree of decomposition of phytotoxic organic substances produced during the active composting stage, is one of the key factors affecting the successful use of compost in agricultural and horticultural production and its impact on the environment (Inbar et al., 1990; Huang et al., 2001; Bentio et al., 2003). As such, numerous parameters in the chemical, physical, spectroscopic, biochemical and microbiological properties of compost have been widely studied (Chen, 2003). Among the methods applied, Fourier transform infrared spectroscopy (FT-IR) and other spectroscopic techniques for assessing compost maturity have been shown to be of significance. These are powerful and simple tools to identify the molecular character of compost and track the formation of humic substances (Gerasimowicz et al., 1985; Gerasimowicz et al., 1986; Benny et al., 1996; Inbar et al., 1989; Provenzano et al., 1998; Ouatmane et al., 2000; Zhan et al., 2001; Provenzano et al., 2001; Rynk, 2003).

To date, relatively few investigations have been made on the composting of pure vineyard pruning residues. The objective of this work was to investigate the changes in organic matter during the composting of vineyard pruning residues at different initial C:N ratios over a period of four months with the combined use of FT-IR, biological and chemical analyses to qualitatively assess the maturity characteristics of vineyard pruning compost.

2 Material and Methods

Composting procedure: Four open wooden rotting boxes (50x50x100 cm) (Schuchardt, 1985) were constructed in the Academy of Agricultural Research for Gansu State Farms (AARGSF). The boxes were insulated with 100 mm of mineral wool to reduce the heat losses. The material used for composting was fresh vineyard pruning residue cut at the end of October of 2003 from vineyards on the Huang Yanghe Farm, Gansu, in the northwest of China. The fresh vineyard pruning residue was shredded with a chopping machine to an average particle size of 7.5 mm. For each rotting box, 70 kg of the fresh vineyard pruning residue was mixed with the required amount of nitrogen in the form of urea, mixed thoroughly and wetted repeatedly to obtain a moisture content of 65 %. Table 1 shows the additions of urea and water and the resulting C:N ratios of the mixtures. The boxes were placed indoors at an average ambient temperature of about 19 °C. A plastic foil with 10 holes (Diameter: 5 mm, spacing 10-12 cm) covered the material on the top of the boxes, allowing aeration from above. Fresh air aerated through the bottom of the box, supported by the chimney effect of the warm rotting material. The temperature during composting was measured daily at a central point in the box. The composts were turned to facilitate the aeration, wetting, sampling and measurement of mass, volume, and bulk density after 0, 15, 60, and 120 days of composting. Samples (about 100g each) were sent to the laboratory for analysis.

Table 1:
Summary about the trials

Treatment	C:N (-)	N addition (kg/t DM)	Water addition[1] (l/t)
B1	60	0	1168
B2	52	1.0	1280
B3	47	3.6	1495
B4	29	7.5	1406

[1] within the total composting time of 122 days
bulk density: 288 kg/m^3

Chemical analysis: The aqueous compost extracts were obtained by mechanically shaking the samples with double distilled water (DDW) at a solid:DDW of 1:10 (w/v, DM) for 1 h, centrifuged at 3000 rpm for 10 min and filtered through No. 202 quantitative filter paper (Xinhua, China). The pH was measured directly by an Orin 920 ISE pH meter with the above filtrates. Organic matter (OM) was measured by determining the loss on ignition at 550 °C for 6 h (Provenzano et al., 2001). The carbon content was calculated on the basis of a C content of polysaccharides of 44.4 % of OM. Total N was determined with the Kjeldahl method (Benito et al., 2003). All chemical analyses were made in triplicate.

Phytotoxicity test: Seed germination and radicle length tests were performed in plastic germination boxes (11.5x11.5x4.5 cm, ZAU, China) with selected Chinese cabbage seeds (*Brassica rapa*). Five millilitres of each water extract (the same as used in pH analysis) was pipetted onto a sheet of filter paper kept inside the germination box. Thirty seeds were distributed evenly on the filter paper and incubated at 25 °C in the dark for 48 h. Triplicates were analysed for each treatment. The germination index (GI) for evaluating phytotoxicity was calculated according to the following formula (Huang et al., 2001):

$$GI\ (\%) = Gc \times Lc \times 100 / Gt \times Lt$$

Where:
Gc = mean seeds germinated in treatment
Gt = mean seeds germinated in control
Lc = mean length radicle in treatment
Lt =mean length radicle in control

Rotting degree: The rotting degree was determined in a self-heating test in Dewar vessels (V=1.5 l) adapted to the method described in the Methods Book for the Analysis of Compost (FCQAO, 1994).

FT-IR spectroscopy: FT-IR was conducted with a Nicolet NEXUS 670 FT-IR spectrophotometer using the KBr method. Pellets were prepared by mixing about 1 mg of ground compost and 100 mg of KBr and compressing the mixture under vacuum at 10 tons for ten min. To limit moisture interference, both KBr and samples were dried separately at 105 °C before making pellets. Spectra were recorded in the 4000 to 400 cm^{-1} wavelength range.

3 Results and Discussion

3.1 Temperature

The temperature profile is a good indicator of the heat generation by the microbial metabolism and biochemical activity during the composting process. In general, the temperature profile indicates that the lower the C:N ratio, the greater the maximum temperature attained and the longer the duration of the thermophilic range (=50 °C). After two days of composting, all four treatments reached 50 °C and entered the thermophilic range, indicating the quick establishment of microbial activities associated with respiratory metabolism in the composting boxes. There were significant differences in the maximum temperature and the length of thermophilic range among the four treatments. The maximum temperature values of B4, B3, B2 and B1 reached were 76, 70, 68, and 65 °C, respectively, which appeared on the third day of composting for treatments of B4, B3 and B2, but on the fourth day for B1. The length of thermophilic ranges of B4, B3 B2 and B1 were 20, 20, 6 and 6 days, respectively. Thereafter, these temperatures began a gradual decline and were near the ambient temperature at the end of composting trials. Trial B1 did not reach a temperature as high as the others with urea addition, this might be due to the lack of nitrogen. Increasing N with urea to decrease the initial C:N ratio in vineyard pruning residues improved microbial activity despite nitrogen losses. The heat generation reached a maximum at C:N 29 in Trial B4. This supports the general literature which states that at C:N ratios <30, nitrogen does not limit the microbial activity, but can increase nitrogen losses as ammonia gas.

3.2 OM, C/N ratio and pH

The changes in OM content and pH are given in Table 2. The OM content in vineyard pruning residues was rela-

Table 2:
Changes of C:N ratio, pH value and OM content of vineyard pruning during composting at different initial C:N ratios

Time of composting (days)	B1 C/N (-)	OM (%)	pH (-)	B2 C/N (-)	OM (%)	pH (-)	B3 C/N (-)	OM (%)	pH (-)	B4 C/N (-)	OM (%)	pH (-)
0	60	97.5	7.5	52	97.5	7.5	47	97.5	7.5	29	97.5	7.5
14	56	96.7	7.8	47	96.0	7.9	32	96.4	8.0	29	96.1	8.2
53	53	96.7	7.9	37	95.5	7.9	29	95.0	7.5	23	94.9	7.5
122	32	95.4	7.7	32	94.7	7.7	22	94.2	7.8	16	93.6	7.9

tively high (97.5 %) at the beginning of the composting process. The OM contents of four treatments decreased gradually during composting. By day 122, the OM contents of B1, B2, B3 and B4 had dropped by 2.2 %, 2.9 %, 3.4 % and 4.0 %, respectively, corresponding to an OM mass degradation of 36, 52, 56 and 59 %. As with temperature, the trend indicates that the lower the C:N ratio, the greater the decline of the OM content of the composting material. Compared with composts of straw, household waste and pig manure, the decline of the OM content of vineyard pruning compost is very low (Provenzano et al., 2001; Ouatmane et al., 2000; Sadaka et al., 2003; Huang et al., 2001). This is because carbon compounds in vineyard pruning are bound by lignin which is highly resistant to biological degradation. Our results are in general similar to those reported by Garcia et al. (1991) and Ouatmane et al. (2000) for different kinds of wastes including ligneous material.

The decline of the C:N ratio was observed in all four treatments from an initial value of 29 to 56 in the substrates to a final value of 16-32 in compost due to an advanced OM decomposition, of which the minimum C:N final value was >12, the limit accepted for mature compost (Bernal et al., 1998; Iglesias-Jiménez and Pérez-García, 1992). For vineyard pruning residues with a high quantity of wooden compounds, the high initial C:N ratio does not make a good index to evaluate its maturity. This result is in agreement with Benito et al. (2003).

The changes in pH of B1, B2, B3 and B4 followed a similar trend with a rise to pH 7.8, 7.9, 8.0 and 8.2 at Day 14 from the original pH 7.5 for all trials on Day 0, and then a decrease to pH 7.7, 7.7,7.8 and 7.9 at the end of composting, respectively. The pH rise during thermophilic range can be explained by the generation of ammonia from ammonification of the added urea and mineralization of organic nitrogen through microbial activities during composting (Huang et al.). Thereafter the decrease in pH is probably caused by volatilisation loss of ammonium and the increase in H^+ from the nitrification process (Eklind et al., 2000). The microbial decomposition of organic matter and the production of organic and inorganic acids would also be responsible for the decrease (Mathur, 1991). There was no significant difference in pH between the four treatments during the composting period.

3.3 Germination index

As shown in Figure 1, the GI values of all treatments of C:N ratios increased significantly as the composting process proceeded. The GI values fluctuation in all treatments was between 46.0 and 76.3 % before Day 53. This may be attributed to the release of toxic concentrations of ammonia and low molecular weight organic acids (Huang, et al., 2001). The GI values of B1, B2, B3 and B4 increased to >80 % at Day 65 and finally reached to 94.0 %, 95.1 %, 103.1 % and 102.4, respectively. A germination index of 80 % to 85 % has been considered an indicator of mature compost free of phytotoxins (Li et al. 2000). According to this GI criteria, all vineyard pruning compost reached maturity after Day 65 under the experimental conditions without regard to the addition of urea.

Table 3:
The assignment of absorption bands in FT-IR spectra of vineyard pruning

Band position (cm^{-1})	Assignment*	Representative compounds
3400~3356	ν(OH)	carbohydrates, protein, amides
2925	ν_{as} (CH_2)	carbohydrates, aliphatic compounds
1740~1735	ν(C=O)	carboxylic ester, ketones
1638~1600	ν_{as}(-COO^-), ν(C=C),δ(N-H) ν(C=O),	carboxylic ion, aromatic ring in lignin, amines and amine salts, lignin, amides
1510~1512	ν_{ring}	lignin, the compounds containing aromatic ring
1460~1445	δ(CH_2,CH_3)	carbohydrates, aliphatic compounds, amino acid salts
1430~1420	ν(C-O),δ_s(CH_2), ν_{ring}, ν_s(-COO^-),δ(OH)	lignin, aliphatic compounds, carboxylic ion
1377	δ_s(CH_3)	aliphatic compounds
1332~1320	δ(OH)	carbohydrates
1265	ν(P=O)	phosphatic ion
1264~1245	ν(C-O), ν(C-O-C)	phenols, aryl ethers, esters
1160~1105	ν(C-O), ν(C-N), ν(C-O-C), ν_{ring}	carbohydrates, aliphatic compounds, amino acids
1053~1047	ν(C-O), δ(OH)	carbohydrates
897	ν(-C-C-)	carbohydrates

*ν stretching; δ bending (in-plane); s symmetric; as asymmetric

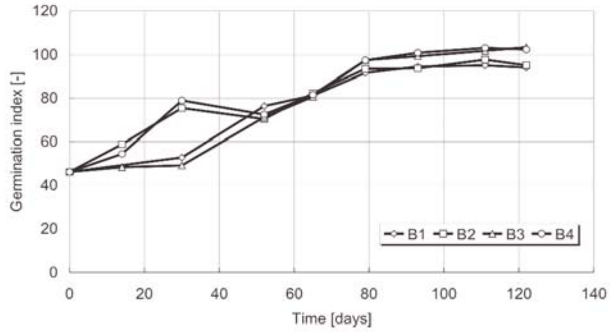

Fig. 1:
Germination index of the compost during rotting time

3.4 Self-heating evaluation

The self-heating test based on Dewar flask measurement has merit as a general technique for evaluating compost maturity. The temperature maximums of final composts of B1, B2, B3 and B4 in the Dewar flask were 25, 25, 25 and 24 °C, respectively on day 3, indicating a fall to Rotting degree V, which is considered an indicator of finished compost (FCQAO, Methods Book, 1994).

3.5 FT-IR Spectroscopy

The FT-IR spectroscopy provides information on the chemical structure of the material. Band assignments for the spectrum of the fresh vineyard pruning residue and its compost and compounds related to them were summarized in Table 3. The table indicates that the vineyard pruning residue contains a number of atomic groups and structures (OH, CH_2, C=O, C=C, C-O-C, and so on) related mainly to carbohydrates, protein, amides, aliphatic compounds, hemicellulose, cellulose and lignin, etc.

The FT-IR spectra of vineyard pruning during composting at different initial C:N ratios are given in Figure 2. Comparison between spectra before composting shows great qualitative similarity among the different initial C:N ratios. Common series of bands were: a very broad band at around 3410 cm^{-1} (H-bonded OH groups), a sharp peak centered at 2925 cm^{-1} (aliphatic C-H asymmetric stretching), a peak in 1740 cm^{-1} (C=O of bonded carboxylic ester, ketones), a broad band at 1600~1638 cm^{-1} (C=C in aromatic structures), a small peak at around 1510 cm^{-1} (skeleton vibration of aromatic rings, relative to lignin), peaks at 1460~1420 cm^{-1} (C-H deformation of CH_2 or CH_3 groups, COOH), a peak at 1377 cm^{-1} (CH_3 groups), a peak at near 1330 cm^{-1} (O-H deformation), peak at the 1246 cm^{-1} range (C-O stretch of phenols, aryl ether, C-O and OH of COOH groups, amide), a peak at around 1050 cm^{-1} (C-O stretch of polysaccharides).

FT-IR spectrum of vineyard pruning ash was also recorded for verifying whether the peaks observed related to organic matter or to mineral forms (Figure 3). The spec-

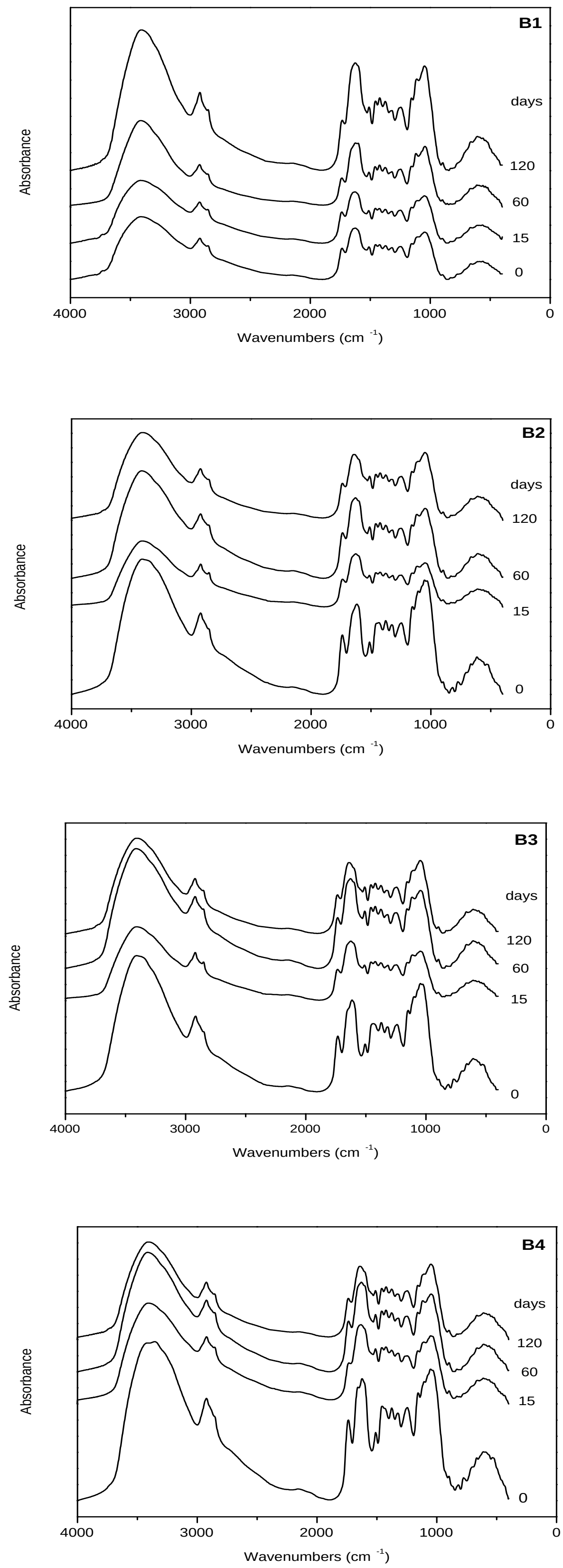

Fig. 2:
FT-IR spectra of vineyard pruning compost at different initial C:N ratios at the start and after 15, 60 and 120 days

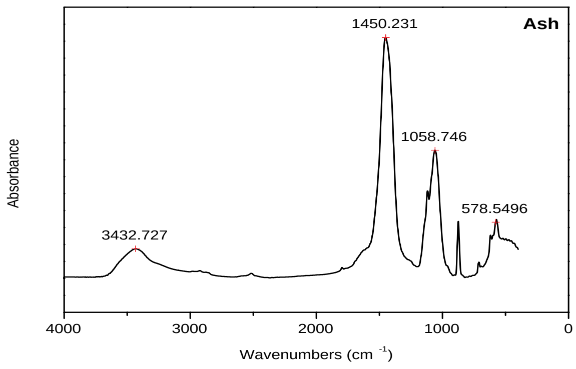

Fig. 3:
The FT-IR spectrum of vine pruning ash

trum exhibited peaks that could be attributed to calcite (1450 cm^{-1}, 1059 cm^{-1}, 578 cm^{-1}, and 496 cm^{-1}) and to phosphate minerals. The result indicates that the unusual peaks observed could be related mostly to carbonate or phosphate rock residues in which the ash was rich.

For the four C:N ratios, the spectra recorded at different stages of composting exhibit the same peaks indicating that no noticeable qualitative changes have occurred during composting. FT-IR spectra differ in the intensity of some peaks that levelled off or down during the decomposition progress. The trend exhibits that the lower the C:N ratio, the greater the intensity of changing of some peaks in FT-IR spectra of the composting material. Relative decreases of peak intensity at 3410~3356 cm^{-1} indicate the decomposition of carbohydrates as a result of the decrease of atomic groups and structures of OH and CH_2; and decreases of peak intensity at 2925 cm^{-1} and 1377 cm^{-1}, caused by the decreases of CH_3 and CH_2 groups, suggest the decomposition of aliphatic compounds. In particular, there were strong reductions of peak intensity at 1740 cm^{-1} in the FT-IR spectra of all treatments after 60 days of composting. This result suggests the occurrence of the decarboxylation of organic matter during the decomposition process, which agrees with those reported by other authors for composted municipal wastes (Benny et al., 1996; Provenzano et al., 1998), spent coffee and domestic solid wastes (Ouatmane et al., 2000). Furthermore, there was no significant change of the peak intensity at around 1610 cm^{-1} before 15 days of composting, but a new peak at around 1630 cm^{-1} appeared after 60 days, which was likely due to the decomposition of lignin and the formation of humic matter (Wu et al., 1997; Ouatmane et al., 2000). On the other hand, all spectra showed a relative increasing in the peak intensity at around 1420 cm^{-1}, providing evidence of the reduction in aliphatic compounds and the increase in aromatic compounds during composting (Wu et al., 1997). Another new peak at 1265 cm^{-1} appeared as PO groups in treatments with urea, indicating probably the formation of phosphatic salts. The results from FT-IR spectra above summarized that the FT-IR spectroscopy can provide information on the chemical structure of the material, and thus can be applied for the assessment of the maturity of composts. During the composting of vineyard pruning residues, the relative decrease in OH, CH_3 and CH_2 groups, the increase in CO groups, the appearance of COO groups in the form of carboxylic salts, and PO groups in the form of phosphatic salts, indicate the decreases of aliphatic compounds, the increases of aromatic compounds, and the OM mineralization during composting of vineyard pruning residues.

4 Conclusions

The maturity criteria C:N ratio, pH, self-heating temperature, germination index and FT-IR spectra compost from vineyard pruning residues after 122 days show no definite results (Tab. 4), whereas the self-heating temperature, the pH and the germination index indicate a mature compost, the C:N ratio is higher than the recommended value in literature. From the FT-IR spectra it is difficult to conclude whether a compost is mature or not, even if some criteria as the degradation of carbohydrates, proteins, amides, etc., and the synthesis of humus compounds show a clear change during the rotting time. The FT-IR spectra indicate a strong decrease of the aliphatic compounds and an increase of aromatic carbon. A new peak of 1265 cm^{-1} appeared as P=O groups relative to phosphatic salts in treatments with urea. The addition of urea as an N source can speed the rotting process of vineyard pruning

Table 4:
Overview about the results for compost maturity

Criteria	Maturity of compost		
	Reference value	Actual value	Reference value fulfilled
C:N ratio	<12	16 to 32	no
pH	>7	7.7 to 7.9	yes
Self-heating test	<30 °C	22 °C	yes
germination index	>80 to 85	93 to 105	yes
FTIR spectra	none or unknown	see figure 2	unknown
plant test with tomato	superior to zero or mineral fertilizer	started in May 2004	

residues to obtain a mature compost, but results in higher degradation of organic matter and losses of gaseous nitrogen in form of ammonia. From this work, it may be concluded that FT-IR, as a non-destructive and a relatively rapid spectroscopic technique, can give some valuable information about the maturity of compost, but a definite determination is not yet possible. A combination with other chemical and biological analyses could be necessary to test organic matter conversion and compost maturity. Finally only the utilisation of compost and its effect on the plant growth can give the final answer about the maturity of compost. And the effect of the compost depends on several criteria including time of use and type of crop. Trials on the effect of vineyard compost on tomato plants begun in May 2004 will give additional information about maturity.

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