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Chemical and spectroscopic analysis of organic matter transformations during composting of pig manure

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Abstract

Composting of separated pig manure (SPM) was studied in an attempt to elaborate upon organic matter (OM) transformation during the process and define parameters for product maturity using both chemical and spectroscopical methods. Composting was performed in two piles and the following parameters were measured in 10 samples during 122 days of composting: temperature, ash content, C/N ratio, water-soluble organic C concentration, and humic substance content (humic acid, fulvic acid, and nonhumic fraction—HA, FA, and NHF, respectively). A spectroscopic method (Fourier-transform infrared [FTIR]) was used to study the chemical composition of the OM. The C/N ratio and ash content exhibited a typically high rate of change during the first 49 days and leveled off thereafter. Water-soluble organic C concentration rapidly increased to a maximum at Day 18 and declined thereafter. The increasing level of HA at various stages of composting indicated the progression of humification. The FTIR spectra revealed an increase in aromaticity and a decrease in carbohydrates as decomposition proceeded. Spectrometric measurements using FTIR provide information significantly correlating to conventional chemical parameters of compost maturity. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: Pig manure; Composting; Humic substances; Organic matter transformation; FTIR

1. Introduction

About 11 million heads of pigs are on feed at any time in Taiwan (Taiwan Agricultural Year Book, 1995). Approximately 80% of the feeding occurs in the country's southern six counties. Pig house manure is normally cleaned daily with groundwater down a sump where the solid fraction is separated from slurry through a mechanical sieve. Modern confinement livestock management systems generate large amounts of manure that are applied to limited land areas in proximity to the manure source. Inappropriate disposal of manure may pose environmental problems such as pollution of ground and surface waters due to leaching and run-off of organics, nutrients, and other pollutants.

Composting manure is a useful method of producing a stabilized product that can be used as a source of nutrients and soil conditioner in the field. The composted product has the advantage of improving soil structure, increasing soil organic matter (OM), suppressing soil-borne plant pathogens, and enhancing plant growth (Hoitink and Fahy, 1986; Chen et al., 1989). Noncomposted manure or immature compost applied to agricultural soil may cause phytotoxicity to plants and adversely affect the environment (Garcia et al., 1992).

Many tests have been proposed to assess compost maturity and stability. Changes in compost stability, or the degree to which composts have been decomposed, can be predicted with C/N in the solid phase (Saviozzi et al., 1992; Jimenez and Garcia, 1992), soluble organic C concentration in water extracts (Garcia et al., 1991; Leita and De Nobili, 1991; Inbar et al., 1993), humification indices (Jimenez and Garcia, 1992; Chefetz et al., 1996), oxygen and CO_2 respirometry (Iannotti et al., 1994), plant growth bioassays (Chen and Inbar, 1993), and NMR and IR spectroscopy (Chen and Inbar, 1993). Several authors have concluded that using a single parameter as a maturity index is insufficient and that crossbreeding several parameters is usually needed. Although several studies have investigated OM transformation using chemical and spectroscopic methods in relation to compost maturity during composting of municipal solid waste (MSW), municipal sewage sludge,

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and separated cattle manure (Chen and Inbar, 1993; Chefetz et al., 1996; Gennaro et al., 1993; Inbar et al., 1989) very few studies have been conducted on separated pig manure (SPM) compost. Since the contents, constituents, sources, and nature of the OM in SPM may be different from MSW, sewage sludge, and other animal wastes, differences in the humification and transformation of OM in SPM compost may be observed.

Understanding OM transformation throughout the composting process and proper evaluation of compost stability and maturity are essential for successful utilization of composts. The objective of this work is to study OM transformation during SPM composting using both chemical and spectroscopical methods. This research should provide information for the establishment of stability and maturity indices for composted SPM.

2. Materials and methods

2.1. Composting of SPM

The solid fraction or SPM obtained from separation of slurry was composted in two piles on an indoor concrete area at a pig farm where the pigs were fed with a typical corn/soybean ration. Fresh SPM was collected from a storage tank behind the mechanical solid–liquid separation sieve. The raw material was divided into two piles (about 1.5 m^3 each) without forced aeration and composted for 122 days. The compost was turned, mixed, and sampled at 0, 3, 7, 12, 18, 25, 33, 49, 80, and 122 days. Samples were taken immediately after turning to ensure maximum homogeneity among samples. Water was added immediately after the compost was turned to maintain a moisture content of 50–60% (w/w). At a depth of 0.30 m within the composting piles, the temperature was taken daily during the first 40 days and then once every 3 days until the end of the process . The samples (4 liters) were placed in partially closed polyethylene bags, transported to the laboratory, and then stored at 5°C. Smaller subsamples were air-dried and used for analyses.

2.2. Chemical analyses

The moisture content of air-dried composts was determined after drying to a constant weight at 105°C in a forced-air oven. Total C and N were analyzed using a Heraeus CHN-O-RAPID analyzer on composts ground to <0.25 mm. Ash measurements were determined at 400°C for 8 h in a furnace (NEY Model 2-525).

2.3. Water soluble organic C

Twenty grams of each compost was weighed into a 250-ml polycarbonate centrifuge tube and extracted with 200 ml of deionized water by shaking for 24 h. The extracts were centrifuged (Kubota Model 6800) at 10 K rpm for 25 min and filtered through 0.45-μm filter membranes. Water extracts were immediately analyzed for pH and stored at 5°C until analysis for organic C.

2.4. Humic substances

For studies on humic substances (HS), 20 g of compost was extracted with 200 ml of 0.1 N NaOH for 24 h. Supernatant solution containing soluble HS was separated by centrifugation at 10 K rpm and the residue resuspended in 0.1 N NaOH. This procedure was repeated eight times. The combined solutions were filtered through 0.45-μm filter membranes and acidified to pH 1 with 3 M H_2SO_4, allowed to stand at room temperature for 24 h, and centrifuged to obtain the fulvic fraction (FF) (humic acid [HA] fraction discarded). The FF was separated into fulvic acid (FA) and nonhumic fraction (NHF) by adsorption of the FA onto Amberlite XAD-8 resin (mesh size 20–60, Sigma, St. Louis). The Amberlite XAD-8 resin was cleaned before use according to the method described by Thurman and Malcolm (1981). The FF was passed through the column at a flow rate of about 12 bed volumes per hour. The column was then washed by one bed volume of distilled water. At this stage, the FA adsorbed onto the resin while the NHF was eluted. The combined solutions, FF and NHF, were stored at 5°C until analysis for organic C.

2.5. Organic C analyses

Organic C analyses of water extracts and combined solutions, FF and NHF, from NaOH extracts were conducted with a total organic carbon analyzer (O·I·Corporation Model 700). Organic C content in the HA fraction of NaOH extracts was not directly analyzed but calculated by the difference between total and FF values. The FA content was calculated as the FF minus NHF. The following humification parameters were calculated on NaOH extracts: (1) humification index (HI)=HA/FA; (2) humification ratio (HR) = HA/FF; and (3) HA percentage (HP) = 100×HA/HS.

2.6. Fourier-transform infrared (FTIR) spectroscopy

The FTIR spectra of SPM compost at 10 stages of the process were obtained for a wavenumber range of 4000 to 400 cm^{-1} in a Nicolet 550 Magna-IR spectrometer (Nicolet Instrument Corp., Madison, WI). Bulk materials were dried and finely ground. The samples were prepared for analyses by mixing 100 mg of KBr with about 2.4 mg of the material and then compressing the mixture to pellets. To obtain FTIR spectra, 400 scans were collected. Spectra of the residual ash obtained after ignition at 400°C for 8 h and loss of the

OM were recorded using the same methodology. To compare one spectrum with another, a linear baseline correction was applied using 4000 cm^{-1}, 2000 cm^{-1}, and 860 cm^{-1} as zero absorbance points. The major peaks (intensity and wavenumber) were found by using OMNIC software (Nicolet Instruments Corp.). The spectra of the ash-free OM was calculated by subtracting the spectrum of the ash from that of the bulk materials using the OMNIC software.

2.7. Statistical analysis

Ash content, C/N ratio, temperature, and water-soluble organic C concentration results are presented as the average value of two replicates of the two composting piles, while the data for each pile is the average of three replicates. The HA, FA, and NHF content are given as the average value of three replicates of a mixed sample from the two composting piles. Spectroscopic analyses were determined on a mixed sample from the two composting piles. Results show that coefficients of variation of all chemical analyses were less than 0.11. The *t*-test result reveals that there was no significant (95% confidence level) difference between the two piles.

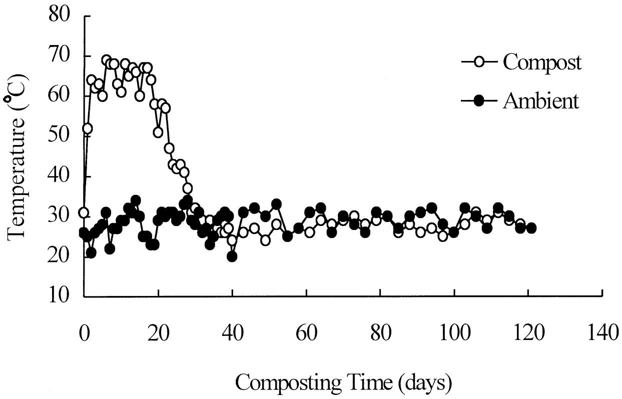

Fig. 1. Compost and ambient temperature during separated pig manure composting.

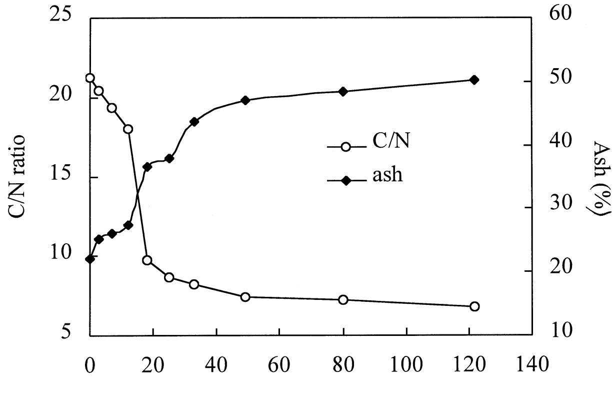

Fig. 2. The C/N ratio and ash content during separated pig manure composting.

3. Results and discussion

3.1. Composting of SPM

Temperature variation during composting (Fig. 1) followed a typical pattern exhibited by many composting systems (Jimenez and Garcia, 1992; Inbar et al., 1993; Tiquia et al., 1997). Three phases were observed during the process: (1) a thermophilic phase lasting for the first 18 days, during which the temperature rose from 31 to 52°C within 24 h and increased to a maximum of 69°C with a mean of 64°C; (2) a cooling phase, in which temperature began to drop at Day 18 and leveled off at Day 29; and (3) after 29 days of composting the compost temperature equaled that of the ambient with no measureable temperature changes. A temperature of 55°C for 3 days is necessary to destroy the pathogens in the compost, whereas the critical temperature for killing most weed seeds is 63°C (Rynk et al., 1992).

The change in the C/N ratio and ash content reflects OM decomposition and stabilization during composting (Fig. 2). The C/N ratio decreased rapidly from an initial value of 21 in the raw material to 10 after only 18 days. The ratio continued to decrease, but less sharply, to 7.4 after 49 days. From this point on the C/N ratio stabilized at a value of about 7 for the remainder of the process. The initial and final ash contents of the compost were 22 and 50%, respectively (Fig. 2). The change in ash content followed a reverse trend to that of the C/N ratio, exhibiting three phases: (1) Days 0–18, when most of the OM decomposed; (2) Days 18 to 49; and (3) Day 49 to the end of the experiment, during which the maturation stage began and the rate of OM decomposition was extremely low.

3.2. Water-soluble organic C

Temporal changes caused the water extract to exhibit slightly alkaline pH ranging from 7.35 to 7.98. The alkaline pH of the water extracts throughout the composting process was probably due to the release of NH_3 from the composts. The water extract from fresh SPM was yellow in color and, as composting proceeded, the intensity of this color increased to dark black at Day 18 and decreased to light brown at the end of composting. These changes in color imply changes in the type and concentration of water-soluble organics. Water-soluble organic C is the most readily biologically active compound in composts applied to soils. The water-soluble organic C level gradually increased from 981 mg $liter^{-1}$

in the raw SPM to 1640 mg liter^{-1} at Day 12, rapidly increased to 5560 mg liter^{-1} at Day 18, and then gradually decreased to 1020 mg liter^{-1} at the end of the process (Fig. 3). As C compounds that are highly available to microbes (e.g. sugars, hemicellulose, organic and amino acids, proteins) were degraded during the thermophilic stages of the decomposition, breakdown products were continuously released, resulting in an increase in water-soluble organic C. Meanwhile, dust gradually formed in the bulk samples at this stage due to microbial activity, as evidenced by dust emitted from the bulk samples during preparation. The maximum organic C value detected on Day 18 could be partly attributed to a release from dust, as evidenced by the highest dust level emitted from the bulk sample on that day. In the cooling and ambient temperature stages the substrate level was low and caused a reduction in soluble C, which was accompanied by the reduction and eventual disappearance of dust in the bulk samples. Water soluble fractions of organic C were relatively small in the raw material (2%) and mature compost (4%) but were substantial at the end of the thermophilic stage (up to 17.5%) (Fig. 3). Other investigators found similar water-soluble organic C trends for sludge-amended soil (Dudley et al., 1986) and composting of MSW (Iannotti et al., 1994). Leita and De Nobili (1991) and Inbar et al. (1993), however, reported that water-soluble organic C declined throughout the composting process for MSW and cattle manure, respectively. This suggests that the type of raw material and composting pattern greatly affect the water-soluble organic C concentration during composting.

A decline in water soluble organic C is often used as an indicator of compost maturity (Garcia et al., 1991; Saviozzi et al., 1992; Inbar et al., 1993). Organic C in the water extract of immature SPM composts consists of sugars, hemicellulose, phenolic substances, organic and amino acids, peptides, and other easily biodegradable substances (Garcia et al., 1991; Chen and Inbar, 1993). In mature compost most of the soluble organic C is present as humic substances, which are resistant to further decomposition, thus explaining its increased stability observed with time during composting. Leached dissolved organics were responsible for enhanced Zn migration through soil columns (Boyle and Fuller, 1987). Enhancement of heavy metal solubility and mobility in soil by dissolved organic C after pig manure application was observed (Japenga et al., 1992).

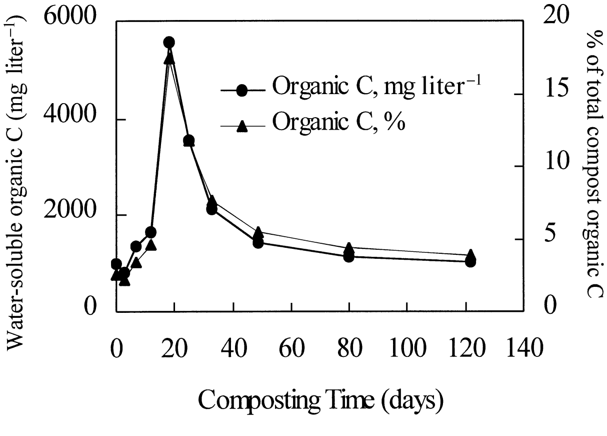

Fig. 3. Water-soluble organic C concentration and water-soluble organic C as a percentage of total compost organic C during separated pig manure composting.

3.3. Humic substance content

Humic substances comprise the most important fraction of OM because of their unique properties, such as the capacity to interact with metal ions, the ability to buffer pH, and the ability to act as a potential source of nutrients for plants. NaOH-extracted HS from composts can be separated into HA, FA, and NHF (Chen and Inbar, 1993). The levels of HA, FA, and NHF in SPM compost at various stages of the process represent the humification process (Fig. 4). Total HS increased from 28% of the OM in the raw material to 44% of the OM after 33 days, stabilizing at this value until the end of the process. The increasing-plateauing trend of the HS levels during SPM composting agrees with that of Inbar et al. (1989) for separated cattle manure but contradicts that reported by Chefetz et al. (1996), who showed that HS levels did not change during composting of MSW. This suggests that the type of raw material is of major importance to the humification process during composting. The FA level gradually decreased from 7.7% of the OM in the raw material to 6.3% in the mature compost with a peak value of 8.6% occurring at Day 18. The HA level increased during the composting process, gradually increasing from 4.6 to 6.0% for the first 18 days, sharply increasing to 15% at Day 25, and then gradually increasing to 21% in the mature compost. The increasing level of HA represents the degree of humification and maturity of compost. In general, fresh composts contain low levels of HA and higher levels of FA (Inbar et al., 1989; Ciavatta et al., 1993; Chefetz et al., 1996), a trend also shown in this study. As composting proceeded the HA content increased, whereas the FA level slightly decreased. Of the humic substances extracted with NaOH, the majority (43–64%) was recovered as NHF for the first 49 days and shifted to HA for the remainder of the process. Temporal changes of NHF exhibited a trend similar to that of water-soluble organic C. The NHF increased rapidly from 16 to 25% of the OM for the first 18 days of composting, probably due to a continuous release of breakdown products of the easily biodegradable substances during the thermophilic stage, and then decreased to 19% in the mature compost due to decomposition and humification of the breakdown

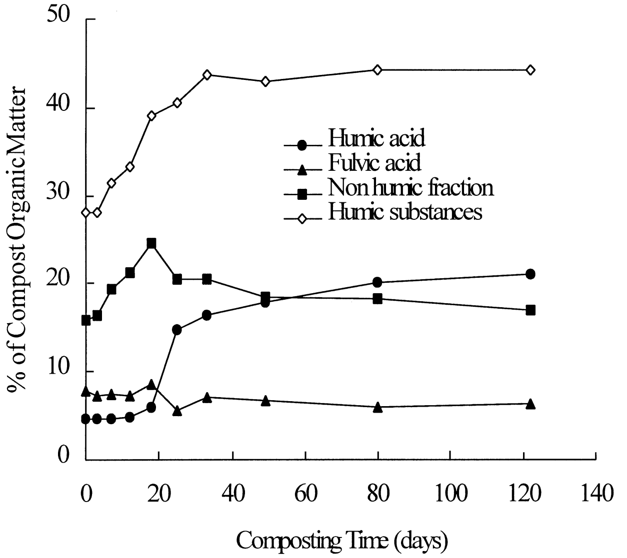

Fig. 4. Relative content of humic acid, fulvic acid, nonhumic fraction, and humic substances as a percentage of compost organic matter during separated pig manure composting.

products and not-easily biodegradable OM during the cooling and ambient temperature stages. All three humification parameters (HI, HR, HP) increased during the composting as follows (Table 1): (1) HI remained steady at 0.6–0.7 for the first 18 days, sharply increased to 2.63 at Day 25, and slowly increased to a final value of 3.33; (2) HR remained steady at 0.17–0.20 for the first 18 days, sharply increased to 0.56 at Day 25, and increased to 0.90 in the mature compost; and (3) HP values exhibited a similar trend to those exhibited by HI and HR. The increasing trend of these parameters indicates that HA became the main fraction of HS during composting. The trend of change of these values is similar in various wastes although specific values differ in other wastes. HI and HR are commonly used to analyze the humic fraction (Jimenez and Garcia, 1992; He et al., 1995; Chefetz et al., 1996). The changes in HI, HR, and HP values reveal that the FA and NHF extracted from SPM contain relatively high levels of biodegradable OM that was mostly decomposed during Days 18–25.

Table 1
Humification index (HI), humification ratio (HR), and percentage of humic acid (HP) during separated pig manure composting

Composting time (days)	HI	HR	HP
0	0.60	0.20	16.4
3	0.63	0.19	16.3
7	0.63	0.18	14.9
12	0.66	0.17	14.3
18	0.69	0.18	15.3
25	2.63	0.56	36.1
33	2.34	0.60	37.4
49	2.69	0.71	41.5
80	3.38	0.83	45.4
122	3.33	0.90	47.4

3.4. FTIR spectroscopy

The FTIR spectra of all six bulk SPM compost samples from different stages (raw, 7, 18, 33, 80, and 122 days) of composting exhibited similar peaks (Fig. 5). The main absorbance bands were a broad band around 3300–3400 cm^{-1} (H bonds, OH groups), two distinct peaks at 2930 and 2850 cm^{-1} (C-H asymmetric, C-H stretch of -CH), a well-pronounced peak at 1650 cm^{-1} (C$=$C in aromatic structure), a small peak at 1560 cm^{-1} (amide II bonds), a shoulder at 1420 cm^{-1} (C-H deformation of CH_2 or CH_3 groups), a rising narrow sharp peak at 1385 cm^{-1} (COO^-, CH_3), a small peak at 1250 cm^{-1} (aromatic C, C-O stretch), a slight shoulder around 1160 cm^{-1} (aliphatic CH_2, OH or C-O stretch of various groups) and a broad peak at 1080 to 1040 cm^{-1} (C-O stretch of polysaccharide, Si-O stretch) (Baes and Bloom, 1989; Inbar et al., 1989).

Distinct changes in the spectra resulting from the composting process were reduction of peaks in the aliphatic region at 2930 and 2850 cm^{-1}. The 1560-cm^{-1} peak became a shoulder possibly as a result of the relatively rapid biodegradation of the amino chain. Peaks in the polysaccharide region at 1050 cm^{-1} decreased, while the 1380 cm^{-1} peak became sharper. The relative height of the aromatic region at 1650 cm^{-1} rose as the composting process proceeded. Most peaks in the aliphatic, amino, and polysaccharide regions leveled off during composting, indicating that SPM

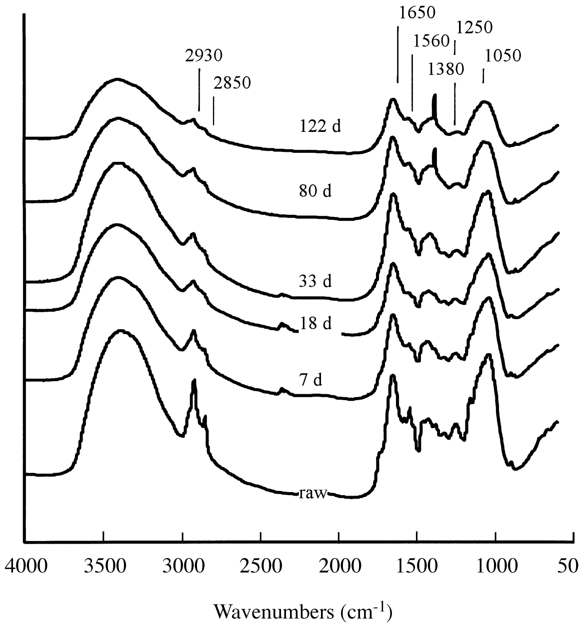

Fig. 5. Fourier-transform infrared spectra of bulk separated pig manure compost at six stages of composting (raw, 7, 18, 33, 80, 122 days).

material had become more uniform and stable at the end of the process.

In general, the spectra of the bulk SPM resemble spectra of composted separated cattle manure (Inbar et al., 1989) and soil HAs classified as Type III (Mud Lake HA) by Stevenson and Goh (1971). Samples of separated cattle manure and this type of soil HAs are distinguished by the presence of a strong absorption band near 1650 cm^{-1}, moderately strong absorption at 1540 cm^{-1}, strong absorption near 1050 cm^{-1}, and relatively pronounced absorption near 2900 cm^{-1}. A unique feature of these spectra is the presence of bands indicative of proteins and carbohydrates.

The spectra of SPM compost (wavenumbers 4000–400 cm^{-1}) were different from spectra of hardwood bark, pine bark, winery solid waste, and MSW composts (Inbar et al., 1991; Niemeyer et al., 1992; Chefetz et al., 1996). The main differences between the spectra of SPM compost and those of other composts were the following: (1) the aromatic and polysaccharide peaks had become the main peak in the spectra of SPM, bark, and winery waste composts as opposed to the 1450 cm^{-1} peak in the MSW spectra; (2) a narrow sharp peak at 1800 cm^{-1} in MSW spectra that appears as a small peak at 1710 to 1720 cm^{-1} in bark and winery waste compost spectra, does not appear in the SPM spectra; (3) a sharp peak at 2520 cm^{-1}, which can be attributed to S-H stretch of aromatic or nonaromatic mercaptans and sulfides in the MSW spectra, does not appear in the SPM spectra; and (4) an intense and sharp peak at 870 cm^{-1} in the MSW spectra that does not appear in spectra of SPM and other compost.

Following Inbar et al. (1989), changes in the SPM spectra were monitored by calculation of the ratio between the intensity of major peaks. The peaks at 2930, 2850, 1650, 1560, and 1050 cm^{-1} were chosen for these calculations. The 1650/2930 ratio (aromatic C/aliphatic C) increased from 1.04 to 1.68, the 1650/2850 ratio (aromatic C/aliphatic C) increased from 1.49 to 2.33, the 1650/1560 ratio (aromatic C/amide II bond) increased from 1.36 to 1.67, and the 1650/1050 ratio (aromatic C/polysaccharide) increased from 0.86 to 1.11. The changes in these ratios indicate that easily degradable OM constituents, such as aliphatic and amide components, polysaccharides, and alcohols, are chemically or biologically oxidized and, therefore, the mature compost contained more aromatic structures of higher stability. The linear correlation of the aromatic to aliphatic peak ratios (1650/2930 and 1650/2850) and aromatic to polysaccharides peak ratio (1650/1050) to the C/N ratio (Fig. 6) exhibited $R^2 = 0.93$, 0.87, and 0.82, respectively. This correlation indicates that FTIR is a useful and reliable tool in the analysis of OM decomposition during composting.

Spectra of residual ash samples were recorded to verify whether these peaks were related to OM or mineral forms in the compost. The residual ash spectra were similar to that of calcite ($CaCO_3$), exhibiting major absorption bands at 2350, 2210, 1620, 1430, and 874 cm^{-1}. The ash also exhibited peaks in the 1100 to 950 cm^{-1} region that can be attributed to Si-O components (Kodama, 1985). Thus, mineral matter is responsible for the unusual peaks observed in the spectra of the SPM compost.

To correct and reduce the effects of mineral components, the residual ash spectrum was subtracted from the bulk spectrum. The spectra of the ash-free SPM compost at three stages (raw, 33, 122 days) of composting are presented in Fig. 7. The spectrum of the mature compost is generally similar to that of the ash-free

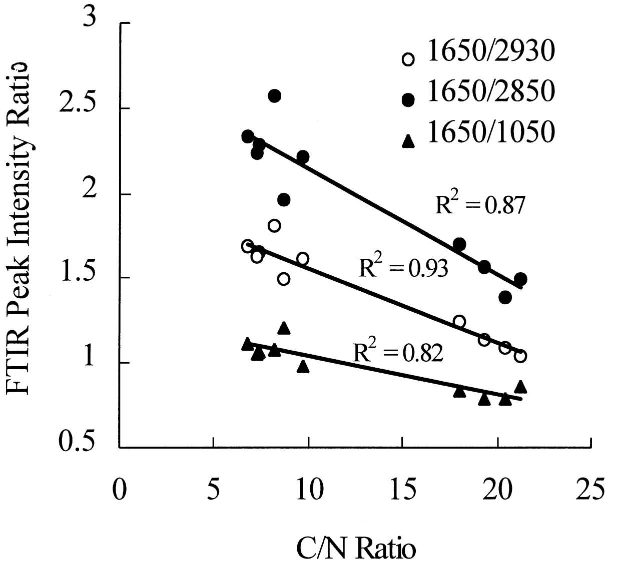

Fig. 6. Fourier-transform infrared (FTIR) peak intensity ratios (1650/2930, 1650/2850, 1650/1050 cm^{-1}) versus C/N ratio during separated pig manure composting.

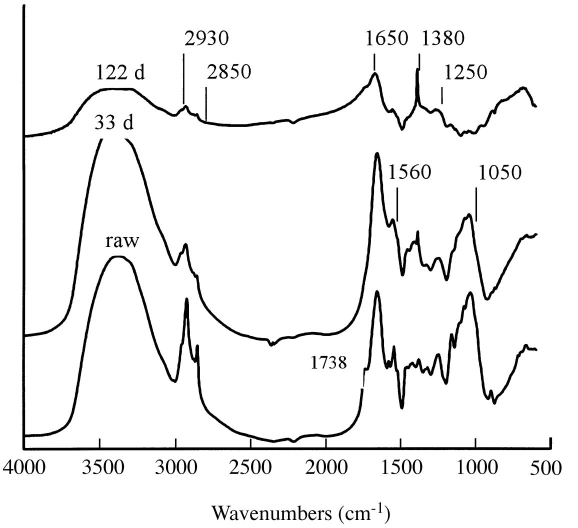

Fig. 7. Fourier-transform infrared spectra of ash-free organic matter in separated pig manure compost at three stages of composting (raw, 33, 122 days). Ash spectra were subtracted from those of the bulk materials.

MSW compost (Chefetz et al., 1996) with specific differences in (1) the polysaccharide region peak (1050 cm^{-1}), which could be attributed to the ash residual leveling off in SPM while remaining broad in MSW compost; and (2) the 1385 peak, which was prominent in the SPM spectra but small in MSW compost spectra. Other differences between the raw SPM materials and mature compost as revealed by comparing the subtracted spectra can be pointed out: (1) leveling off in the aliphatic region (2930 and 2850 cm^{-1}); (2) reduction of the 1738-cm^{-1} shoulder (CO_2H groups) in the mature compost; (3) a relative increase in the aromatic region (1650 cm^{-1}); (4) reduction and formation of a shoulder of the 1540-cm^{-1} peak (amide II bond); (5) a sharp increase in the 1380-cm^{-1} peak (COO^-, CH_3); and (6) leveling off in the polysaccharide region (1050 cm^{-1}). The leveling off of the polysaccharide region peak (1050 cm^{-1}) in the ash-free mature SPM compost indicates that the broad 1050-cm^{-1} peak in the bulk mature compost was attributed to Si-O components.

4. Conclusions

Decomposition of SPM was characterized using various chemical methods. The C/N ratio, ash content, and HS content, along with temperature, changed rapidly during the first 30 days and reached a plateau thereafter. In addition, the amount of water-soluble organic C was substantial at the end of the thermophilic stage. These parameters were all good indicators of SPM compost stability and maturity. FTIR spectroscopy was also applied to bulk SPM compost samples without extraction. The findings in FTIR spectra in this research indicate that easily degradable OM constituents, such as aliphatic chains, polysaccharides, alcohols, and protein, are decomposed and, therefore, the mature compost contained more aromatic structures of higher stability. OM transformation, as analyzed by FTIR, also suggests that the composting process transforms heterogeneous raw SPM OM to a compositionally uniform product at the end of the process. The high correlation between the 1650/2930, 1650/2850, and 1650/1050 FTIR peak ratios and C/N ratio indicate that these ratios can be used as maturity indices. All chemical and spectroscopic parameters exhibited three phases: (1) rapid decomposition during the first 30 days; (2) stabilization until Day 50; and (3) maturation from Day 50 on. The SPM compost, described in this study was mature and ready for use as an agricultural substrate after about 80 days of composting.

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