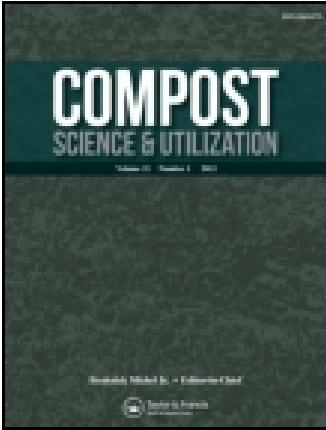


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# Composting of Source-Separated Household Organics At Different Oxygen Levels: Gaining an Understanding of the Emission Dynamics

B. Beck-Friis<sup>1</sup>, S. Smårs<sup>2</sup>, H. Jönsson<sup>2</sup>, Y. Eklind<sup>1</sup> and H. Kirchmann<sup>1</sup>

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Source-separated household organics were composted in a reactor at three oxygen levels, 16%, 2.5% and 1% in the compost gas. Short-chained fatty acids were initially present in the compost material, and were also produced during the mesophilic phase at all three oxygen levels. This indicated that partial anaerobic conditions existed. No NH<sub>3</sub> emissions occurred during the mesophilic phase due to acidic conditions. Composting at 2.5% and 1% O<sub>2</sub> concentrations prolonged the mesophilic phase and reduced the microbial activity as compared to 16% O<sub>2</sub>. This led to delayed and decreased emissions of NH<sub>3</sub>. Nitrous oxide was not formed during thermophilic conditions. Methane, which was measured at 2.5% and 1% O<sub>2</sub>, was only found during thermophilic conditions. The emission of methane indicates that anaerobic conditions occurred during the thermophilic phase. The main reactions regulating pH during composting were outlined involving the ion species VFA, NH<sub>4</sub><sup>+</sup>/NH<sub>3</sub> and CO<sub>2</sub>/HCO<sub>3</sub><sup>-</sup>/CO<sub>3</sub><sup>2-</sup>.

## Introduction

During composting of organic wastes, the gases NH<sub>3</sub>, N<sub>2</sub>O and CH<sub>4</sub> can be emitted to the atmosphere (Hellmann *et al.* 1997; Hellebrand 1998; Beck-Friis *et al.* 2000; Beck-Friis *et al.* 2001). The gases contribute to both soil acidification (van Breemen *et al.* 1982) and the greenhouse effect (Houghton *et al.* 1996; Prather *et al.* 1996). Concerning gaseous nitrogen emissions during composting, NH<sub>3</sub> has been found to be the dominant gas (Martins and Dewes 1992; Beck-Friis *et al.* 2001). Ammonia is released through mineralization of nitrogen rich organic material, and the amount of NH<sub>3</sub> emitted during composting is strongly related to pH, C/N ratio and temperature. Earlier compost studies with household organics have resulted in NH<sub>3</sub> emissions between 15 and 60% of initial nitrogen content (Kirchmann and Widén, 1994; Michel and Reddy 1998; Eklind and Kirchmann 2000; Beck-Friis *et al.* 2001). A strong reduction of O<sub>2</sub> concentration clearly decreases NH<sub>3</sub> emissions (Nakasaki *et al.* 1990; Michel and Reddy 1998).

Compost heaps of organics and manure can also emit N<sub>2</sub>O (Sibbesen and Lind, 1993; Hellmann *et al.* 1997; Hellebrand 1998; Beck-Friis *et al.* 2000; Sommer and Møller 2000), but to a much smaller extent than NH<sub>3</sub>. The processes known to produce N<sub>2</sub>O are nitrification and denitrification (Granli and Bøckman 1994) both of which may occur during composting (Diaz-Ravina *et al.* 1989). However, the very low amounts of NO<sub>3</sub><sup>-</sup> normally present during the initial phase of composting (Kirchmann and Widén 1994) would limit these processes. Furthermore, nitrifying bacteria are sensitive to high temperatures (> 40°C) and thus do not survive thermophilic conditions (Focht and Verstraete 1977). In other words, thermophilic conditions do not allow N<sub>2</sub>O to be formed and only at a later stage may these processes occur.

Extensive aeration of manure stacks and large compost heaps of household organics with low O<sub>2</sub> concentrations in compost air have been found to yield CH<sub>4</sub> emis-

sions (Husted, 1994; Lopez-Real and Baptista 1996; Beck-Friis *et al.* 2000; Sommer and Møller 2000).

Short-chained fatty acids are present in fresh compost, but are not found in mature composts (Keeling *et al.* 1994; Kirchmann and Widén 1994; Eklind *et al.* 1997; Eklind and Kirchmann 2000a; Beck-Friis *et al.* 2000). These acids affect the pH conditions in the compost and thereby  $\text{NH}_3$  emissions (Beck-Friis *et al.* 2001). In addition, short-chained fatty acids are toxic for microorganisms when pH is less than 5. This is due to their undissociated forms passing through cell membranes and thus causing an imbalance of protons. However, in their dissociated form, i.e., at pH higher than 6, volatile fatty acids are a readily decomposable substrate for microbes (Erickson and Yee-Chak Fung 1988; Brinton 1998).

### Objectives

In this paper, we present results from systematic composting experiments to gain a deeper understanding of the emission dynamics. The aim of this study was to find the influence of  $\text{O}_2$  concentration in the compost gas on the course of decomposition. In this context the following questions were considered:

1. Are thermophilic conditions reached with an  $\text{O}_2$  concentration of 1% in the compost gas?
2. Which  $\text{O}_2$  concentrations are required for mainly aerobic decomposition to be replaced by anaerobic digestion, as indicated by  $\text{CH}_4$  formation?
3. How does the  $\text{O}_2$ -gradient of gas contained in the compost macro pores affect emissions of  $\text{CO}_2$ ,  $\text{NH}_3$ ,  $\text{N}_2\text{O}$  and  $\text{CH}_4$ ?
4. What determines the length of the mesophilic period?
5. What role do short-chained fatty acids play during composting?
6. Which processes are involved in pH changes during composting?

In addition to answering the above questions, our results also provided information that enabled us to outline characteristics of the decomposition dynamics.

### Materials and Methods

The source-separated household organics used were collected on one occasion in Feb. 1995 from a central composting plant in Uppsala, Sweden, and thereafter frozen for storage at  $-24^\circ\text{C}$  (Eklind *et al.* 1997). Before composting, the waste was slowly thawed, and mixed with wheat straw and water in a free fall mixer (Universal 180 l, ATIKA, Germany) to obtain a C/N ratio of 22 and a water content of 65% wet weight. The compost material was then immediately milled in a cutting mincer (Mincing house diameter 200 mm, Palmia, Sweden) with hole dimension of 13 mm to further homogenise the mixture. The material was then placed in a 200 litre airtight cylindrical reactor. Although the water content was uniform in the substrate prior to composting, due to condensation on the reactor walls, the exterior of the substrate mass tended to develop a higher water content than the interior of the mass, see Smårs *et al.* (2001). In order to maintain small gradients in temperature and gas composition in the macro pores, the compost material was placed on a perforated metal sheet and an internal fan re-circulated the gases upwards through the compost. The oxygen level of the compost gas was regulated by a signal feed-back the gas analyser to a computer controlled valve for inflow of compressed air to the reactor. The reactor was cooled by pumping compost gas through a gas cooler in a separate loop in which the flow was roughly adjusted by a hand controlled valve. Finer temperature control was achieved by automatic feed-

back from the temperature signal to a channel heater, placed in the same loop as the above mentioned re-circulation fan. A detailed description of the compost reactor is given in Smårs *et al.* (2001).

The experiments were run at three oxygen levels, 16%, 2,5% and 1% in the compost gas. The temperature regulation system was not turned on until the compost had self-heated to 55°C, after which the temperature was kept at 55°C. Seventeen temperature sensors were installed in the reactor vessel registering the temperature every fifth minute. The experimental period varied between 25 and 31 days.

#### *Emission Measurements*

The contents of O<sub>2</sub>, CO<sub>2</sub>, NH<sub>3</sub> and N<sub>2</sub>O in the exhaust air were measured every fifth minute and logged by two computers. The O<sub>2</sub> concentration was monitored with a gas analyser (Servomex 1131, UK). Two different instruments were used for determining the CO<sub>2</sub> content, a gas analyser (Binos 4b.2 Germany) and a photoacoustic multi-gas monitor (Type 1312, Innova, Denmark) which was also used to measure NH<sub>3</sub> and N<sub>2</sub>O. In addition, an acid trap (150-200 ml 0,01M H<sub>2</sub>SO<sub>4</sub>) was also installed for measuring NH<sub>3</sub> emissions.

During the experimental runs, 3 to 6 samples of compost gas were taken every day from the exhaust air and put into evacuated glass vials for further analyses of N<sub>2</sub>O and CH<sub>4</sub> by gas chromatography (GC). Nitrous oxide was analysed using a Chrompack-CP 9001 equipped with a Ni<sup>63</sup> electron capture detector (250°C), injector (125°C) and oven (50°C). The column equipment consisted of two precolumns and a packed column (HEYSEP S 80-100 MESH). The carrier gas was He and the make up gas was N<sub>2</sub>. Methane was analysed using a Packard 428 equipped with a Porapack T column and Flame Ionization Detector (Packard-Becker BV, Delft, Netherlands)

#### *Analysis of Condensed Gas*

The outgoing compost gas was cooled from 20°C to 11°C to reduce the moisture content before analysis. The condensed liquid was collected and analysed for pH, NH<sub>4</sub>-N and NH<sub>3</sub>-N. The pH value was measured with a pHM 92 Lab pHmeter Radiometer, Copenhagen. The ammoniac concentration was analysed by flow injection analysis (FiaStar 5010 analyser, Tecator, Sweden) with spectrometric detection (ISO 11732: 1997) and the analysis method was modified according to Tecator application note 50.01/84. To avoid NH<sub>3</sub> losses from the condensate, the liquid was acidified.

#### *Analysis of Solid Samples*

Compost samples were taken daily during the first two weeks and then about every third day. The material was analysed with regard to dry matter content, pH, organic C, short-chain fatty acids, inorganic N (NH<sub>4</sub>-N, NO<sub>3</sub>-N) and total-N. To determine dry matter content, the material samples were dried at 105°C for 14 h. The pH value was analysed (pHM 92 Lab pHmeter) after the material was shaken with water (weight ratio of sample to H<sub>2</sub>O of 1:5) for 1 h at 20°C and then sedimented for 30 minutes. To determine the content of short-chained fatty acids, the samples were extracted (weight ratio of sample to ultra pure water 1:4) and shaken for 30 minutes at 10°C and then analysed on a HPLC (Waters, Milford, Mass., USA). To determine the amount of inorganic N, the samples were extracted with 2M KCl (weight ratio of sample to KCl 1:5) and measured on an autoanalyser (Technicon, TrAAcs 800, Germany). Because the

content of nitrate was very low, total-N was analysed by the regular Kjeldahl method. Finally, the amount of organic carbon in the compost material was determined with a dry combustion method using the instrument LECO CN-2000 (USA).

### Results and Discussions

#### *Gaseous Emissions of C and N: The Shift from the Mesophilic to the Thermophilic Phase Causes NH<sub>3</sub> and CH<sub>4</sub> Formation*

Composting household organics at different O<sub>2</sub> concentrations in the compost gas changed the length of the mesophilic phase from 7 to 14 days at 16 and 1% O<sub>2</sub> respectively, Figure 1. The less O<sub>2</sub> in the compost gas, the longer the mesophilic phase. In all the O<sub>2</sub> concentrations studied, 3 or 4 minor peaks of CO<sub>2</sub> emission were recorded during the mesophilic phase. At 16% O<sub>2</sub>, the first peak occurred on day 1, whereas with 1% O<sub>2</sub> the first peak occurred on day 8, Figure 2. The concentrations of short-chained fatty acids, both initially present in the material and formed during the mesophilic phase, were strongly reduced during the final part of the mesophilic phase, Figure 1.

Their disappearance coincided with a strong increase of CO<sub>2</sub> emission, Figure 2.

Maximum CO<sub>2</sub> emission rates occurred when the thermophilic phase started and reached 17, 11 and 9% CO<sub>2</sub>-C of initial C day<sup>-1</sup> when composting at O<sub>2</sub> concentrations of 16, 2.5 and 1%, respectively, Figure 2. Cumulative CO<sub>2</sub> emissions were 60, 49 and 42% of initial C after 25 days of composting at O<sub>2</sub> concentrations of 16, 2.5 and 1% respectively, Figure 3. The thermophilic phase started when approximately 10% of the initial C had been emitted, irrespective of the O<sub>2</sub> content in compost gas.

In addition to emissions of CO<sub>2</sub>, CH<sub>4</sub> formation occurred when the O<sub>2</sub> concentration was 2.5% and 1%. In total 0.9 and 1.5% CH<sub>4</sub>-C of initial C was formed with the 2.5 and 1% O<sub>2</sub> levels, respectively, after 25 days of composting. However, CH<sub>4</sub> was only formed during thermophilic conditions (Fig. 2). Under mesophilic conditions, CH<sub>4</sub>

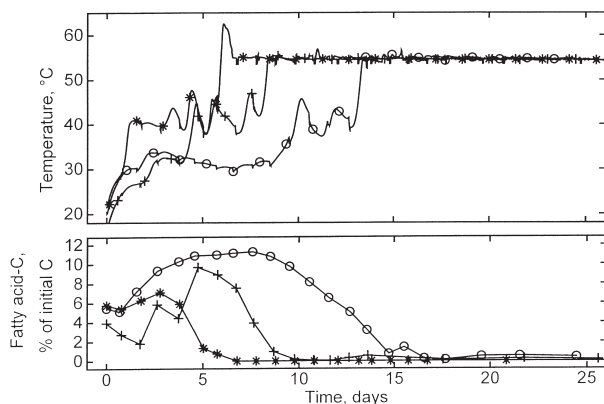


Figure 1. Compost temperatures, and content of fatty acid C in the compost material at the following O<sub>2</sub> concentrations in the compost gas: 16% (\*), 2.5% (+) and 1% (o).

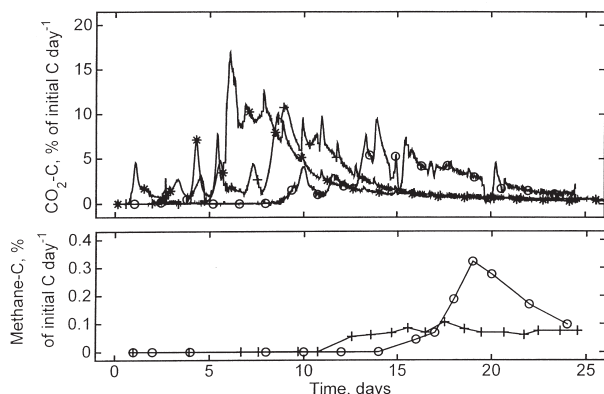


Figure 2. Formation rates of CO<sub>2</sub>-C and CH<sub>4</sub>-C as percentages of initial C, at the following O<sub>2</sub> concentrations in the compost gas: 16% (\*), 2.5% (+) and 1% (o). Methane analyses with 16% O<sub>2</sub> is only available from day 0 to day 6.

*Composting of Source-Separated Household Organics at Different Oxygen Levels:  
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was not detected at any of the O<sub>2</sub> concentrations. Unfortunately, in the experiment with 16% O<sub>2</sub> in the compost gas, CH<sub>4</sub> analyses were only made during mesophilic conditions. Therefore it was not possible to verify the effect on CH<sub>4</sub> formation of 16% O<sub>2</sub> in the compost gas on thermophilic phase. With reduced O<sub>2</sub> concentration, the emissions of CH<sub>4</sub> started during the thermophilic phase just after the maximum CO<sub>2</sub> emission peak (Fig. 2), which is in agreement with results by Sommer and Møller (2000). During the formation of CH<sub>4</sub>, the pH value in the compost material was higher than 8 and there was no lactic acid present and only low concentrations of acetic acid (<1% fatty acid C of initial C), Figure 4. This course of formation is in agreement with observations from landfills where the CH<sub>4</sub> emission started when the concentration of fatty acids had decreased (Barlaz *et al.* 1989).

In the case of nitrogen emissions, volatilisation of NH<sub>3</sub> started when composting shifted from mesophilic to thermophilic conditions. During this shift, the pH value in the material increased above 8, Figure 5. The highest NH<sub>3</sub> emission rate was measured during the most intensive period of the thermophilic phase. Furthermore, the amount of NH<sub>3</sub> emitted was highest with 16% O<sub>2</sub> in the compost gas.

The emissions of N<sub>2</sub>O were very low in all experiments. The concentration of N<sub>2</sub>O in the compost gas (16% O<sub>2</sub>), as measured by GC, was 8 ppm on the first day of composting and decreased to 1 ppm the following day. When the thermophilic phase started, N<sub>2</sub>O concentrations in the compost gas

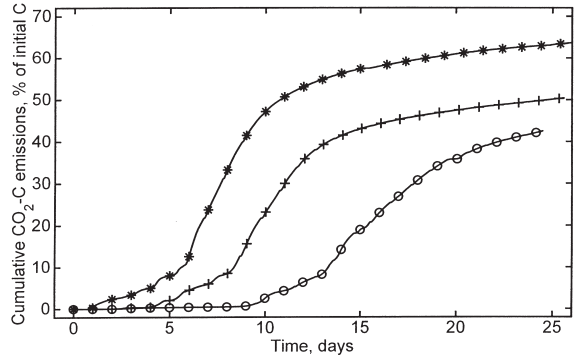


Figure 3. Cumulative CO<sub>2</sub>-C emissions at the following O<sub>2</sub> concentrations in the compost gas: 16% (\*), 2.5% (+) and 1% (o).

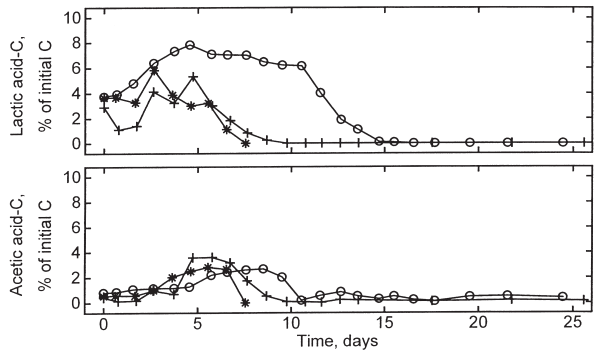


Figure 4. Amount of lactic acid and acetic acid in compost material at the following O<sub>2</sub> concentrations in the compost gas: 16% (\*), 2.5% (+) and 1% (o).

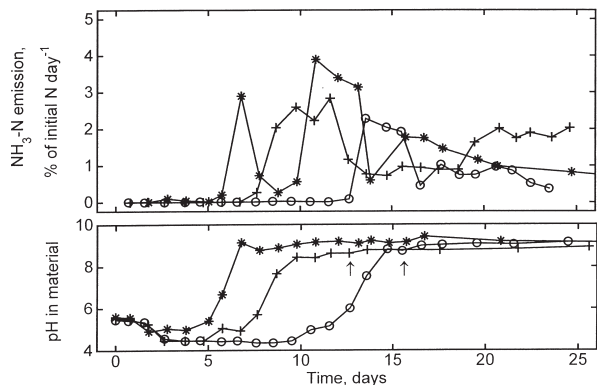


Figure 5. Emission rates of NH<sub>3</sub>-N in condensate and cooled gas and values of pH in compost material at the following O<sub>2</sub> concentrations in the compost gas: 16% (\*), 2.5% (+) and 1% (o). The arrows (≠) show the start of CH<sub>4</sub> emission when the O<sub>2</sub> concentrations were 2.5% and 1%.

were of the same order of magnitude as that of ambient air. The  $N_2O$  concentration was even lower at reduced  $O_2$  levels where at most about 2 ppm were measured in exhaust gas during the mesophilic phase. However,  $N_2O$  values in the exhaust gas recorded by the photoacoustic instrument were about 4 times higher. These are regarded as being less reliable since other gases seemed to interfere with the analysis. Osada *et al.* (1998) used a photoacoustic instrument for measuring  $CH_4$  and  $N_2O$  in pig houses and observed a slight overestimation of the gases, as compared to GC measurements, if the concentration of  $CO_2$  was more than 600 ppm. In our experiments, the compost gas contained in excess of 600 ppm  $CO_2$  and thus we judged an overestimation likely occurred.

#### *Both Aerobic and Anaerobic Conditions Coexist During Composting*

A question related to this study was if it is possible to reach thermophilic conditions through self-heating when reducing the  $O_2$  concentration in compost gas to as low as 2.5 or even 1%? The experiments showed that thermophilic conditions were reached through self-heating even at the lowest  $O_2$  concentration tested, although the mesophilic period was prolonged by a period of one week for the 1%  $O_2$  concentration compared with the 16%, Figure 1.

Fatty acids are regarded as a microbial product of anaerobic metabolism. During the mesophilic phase, production of fatty acids, and in particular of lactic acid was measured for all  $O_2$  concentrations tested, Figure 4. Smaller amounts of acetic acid were also measured (Figure 4) as were low concentrations of propionic acid, butyric acid and iso-butyric acid. This clearly indicates that partial anaerobic conditions exist even if the compost gas contains 16%  $O_2$  because of anaerobic zones between the air flow. However, although anaerobic conditions during the mesophilic phase no  $CH_4$  formation occurred probably due to the low pH.

Under thermophilic conditions, short-chained fatty acids cannot be used as an indicator for anaerobic conditions because they are quickly decomposed. Instead, we considered the production of methane during this phase as an alternative indicator of anaerobic conditions. Methane was detected, as most 2%, at both 2.5% and 1%  $O_2$  in the compost air and methane production was observed to increase with reduced  $O_2$  levels, Figure 2. By way of comparison, analyses of compost gas in large-scale compost heaps showed high concentrations of  $CH_4$  (2-47%) in the compost gas when the  $O_2$  level was below 7% (Beck-Friis *et al.* 2000). Increased amounts of  $CH_4$  have been observed even at  $O_2$  concentrations above 10% (Sommer and Møller 2000).

These observations that compost temperature increases, despite low oxygen concentration and  $CH_4$  formation, can also explain the results obtained from large scale compost heaps where methane emissions were measured under high composting temperatures (Beck-Friis *et al.*, 2000; Sommer and Møller 2000). In other words, aerobic and anaerobic conditions can co-exist leading to both high composting temperatures and formation of  $CH_4$ .

#### *Turnover of Short-Chained Fatty Acids, Nitrogen Causes the pH Changes During Composting*

The initial pH in the source-separated household organics was  $5.5 \pm 0.1$  and pH values decreased below 5 during the first days of composting. The pH values remained below 5 for periods of 5, 7 and 11 days, corresponding to  $O_2$  concentrations of 16, 2.5 and 1% respectively, Figure 5. Low pH values were only measured during the



mesophilic phase. Parallel with the shift to the thermophilic phase, pH increased to about 8. The pH increase was from 5.4 to 9.2 and took place within two days at 16% O<sub>2</sub> in the compost air and similar pH increase took place within three days for 2.5% and 1% O<sub>2</sub> respectively. During the following thermophilic phase the pH values remained high. Our results coincide with pH, temperature and CO<sub>2</sub> changes during composting as reported by Poincelot (1974).

The presence of short-chained fatty acids under acidic conditions, and their absence under alkaline conditions, indicates that fatty acids are a key factor regulating the pH during the mesophilic phase.

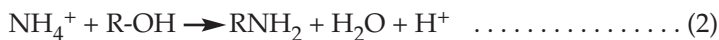
Studies on slurries show that pH values are mainly regulated by three groups, VFA (undissociated volatile fatty acids), NH<sub>4</sub><sup>+</sup>/NH<sub>3</sub> and CO<sub>2</sub>/HCO<sub>3</sub><sup>-</sup>/CO<sub>3</sub><sup>2-</sup>, and that a reduction of VFA concentrations is accompanied by an increase in pH (Georgacakis *et al.* 1982; Paul and Beauchamp 1989; Sommer and Husted 1995ab). Studies on composts showed that the same ion species are involved (Eklind and Kirchmann 2000a, 2000b; Beck-Friis *et al.* 2001). In addition to these factors, nitrification has been shown to reduce pH during late stages of composting (Tiquia *et al.* 1998), but this did not occur in our experiments. Compared with slurries, the concentrations of short-chained fatty acids in composts are about 4 times lower and of ammonium about 44 times lower (Kirchmann and Lundvall 1993). The main processes affecting pH during composting are outlined below.

#### *pH-Decrease During the Mesophilic Phase*

During the mesophilic phase, both aerobic and anaerobic processes occur. The anaerobic formation of short-chained fatty acids, here described by the production of lactic acid from sugar through the homo-fermentative pathway, produces two protons per sugar molecule (Gottschalk 1986).



This is believed to be the major acidifying process during this phase. The other acidification process is immobilisation of NH<sub>4</sub><sup>+</sup>-N into the microbial biomass. Ammonium immobilisation is an aerobic process in which one proton is released per molecule of NH<sub>4</sub><sup>+</sup>.



However, as no immobilisation could be detected during the mesophilic phase this process was not likely involved in the acidification, Figure 6.

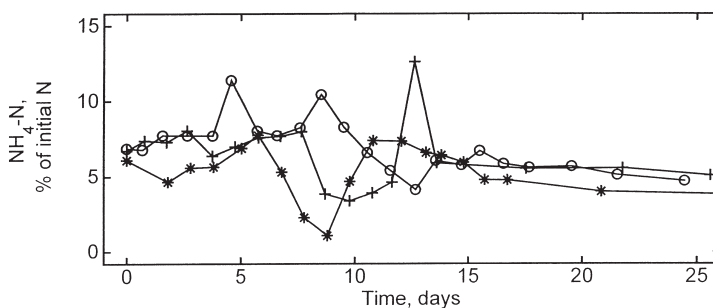


Figure 6. Amount of NH<sub>4</sub>-N in compost material at the following O<sub>2</sub> concentrations in the compost gas: 16% (\*), 2.5% (+) and 1% (o).

*pH-Changes During the Shift To the Thermophilic Phase*

At the end of the mesophilic phase, the content of short-chained fatty acids decreased markedly. The decomposition of fatty acids involves both decarboxylation and the assimilation of a proton which leads to a rise in pH.



Furthermore, organic N was mineralised during the thermophilic phase (Figure 6) which was most pronounced in the 16% O<sub>2</sub> treatment. This means that one proton was used per molecule of NH<sub>4</sub><sup>+</sup> formed.



One consequence of the pH increase will be that any remaining fatty acids dissociate and thereby become an available energy substrate for microorganisms.

At pH values above 8.5, precipitation of CO<sub>3</sub><sup>2-</sup> in the form of CaCO<sub>3</sub> will remove alkalinity. Carbonate precipitation is probably responsible for the fact that no higher pH values than approximately 9 are measured in composts.



**Conclusions**

When compost gases were regulated at 2.5% and 1% O<sub>2</sub>, methane was formed during thermophilic conditions after the short-chained fatty acids were decomposed.

Nitrous oxide was not formed during thermophilic conditions.

Composting at 2.5% and 1% oxygen concentration prolonged the mesophilic phase as compared to 16% oxygen. No ammonia emissions occurred during the mesophilic phase because of the acidic conditions.

During the mesophilic phase of composting, aerobic and anaerobic conditions co-exist, as indicated by temperature increase and the formation of short-chained fatty acids. During the thermophilic phase fatty acids disappear. Thus, the presence or absence of fatty acids cannot be used as a general indicator for anaerobic or aerobic conditions during composting.

Lactic acid is the main fatty acid formed during the mesophilic phase of composting of source-separated household organics.

Even low oxygen concentrations in compost air are sufficient to cause self-heating of the organic waste.

At the lower oxygen concentrations in the compost gas, rates of emissions of carbon dioxide and ammonia are also reduced. However, this does not necessarily mean that the total amount of gaseous emissions is reduced over the whole composting period, since the time taken to reach stability is prolonged at low oxygen concentrations.

The first change in pH during composting, early in the mesophilic phase, is an acidification caused by fatty acid formation. The second change is a rise in pH during the transition to thermophilic conditions, which results from the complete breakdown of fatty acids. Intensive mineralization of nitrogen is further responsible for increasing the pH, and finally, this pH increase is controlled when above 8.5 by the precipitation of carbonates.

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