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Assessment of the Reliability of a Commercial Maturity Test Kit for Composted Manures

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Stability significantly affects the potential for beneficial utilization of composts but is difficult to measure by farmers and compost producers. A simple four hour test (the Solvita® maturity test) that measures CO_2 evolution and ammonia emission from compost samples was compared to a traditional three-day, 25°C, CO_2 evolution rate procedure and to measurements of ammoniacal nitrogen concentrations in manure composts to assess the reliability of this test. Three composts — 1) Dairy manure amended with wheat straw, 2) The same dairy manure but amended with sawdust and 3) Swine manure amended with sawdust and ground wood pallets — were composted in windrows for 120 days. Samples were removed weekly to biweekly. CO_2 evolution rates of the three composts decreased from initial means (n=6) of 3.41, 3.42 and 9.35 to 0.63, 0.76 and 0.31 mg CO_2-C g^{-1} VS day^{-1}, for the dairy manure-straw, dairy manure-sawdust, hog manure composts, respectively. The corresponding mean Solvita CO_2 test values for these composts increased from 3.4, 3.0 and 3.2 to 6.8, 6.5 and 7.0, respectively. Correlation analysis between CO_2 evolution rates and Solvita CO_2 test values gave linear correlation coefficients (r) of -0.82, -0.78, and -0.87 for the straw-amended dairy manure, the sawdust-amended dairy and the hog manure composts, respectively. The Solvita NH_3 test gave highly significant correlations ($p<0.0001$) with ammoniacal-N concentrations (correlation coefficients (r) = -0.43, -0.64 and -0.65, respectively). The Solvita® maturity index, a combination of Solvita CO_2 and NH_3 values, correlated significantly with both CO_2 evolution rate and ammoniacal-N concentrations. However, the Solvita CO_2 index alone was the best predictor of compost CO_2 evolution rate or stability. The Solvita Maturity test, which combines the Solvita CO_2 and NH_3 tests, provided useful information about the potential for the development of a toxic response in plants due to excessive concentrations of ammoniacal-N present in some stable compost samples that would not have been detected if the CO_2 stability test were used by itself. We conclude that the Solvita maturity test provided a simple, inexpensive relative test of compost stability and NH_3 emission for diverse samples of composted manures. Even so, it did not accurately predict their CO_2 evolution rates measured by respirometry nor their ammoniacal-N concentrations. The test would be most useful for on-farm applications.

Introduction

Composting reduces the volume of manure to be applied to farmland (Pecchia *et al.* 2002) and offensive odor emissions during application, thus facilitating utilization on farms (Elwell *et al.* 2001; Louhelainen *et al.* 2001; Nicolai and Janni 2001; Park *et al.* 2001; Walker 1993). Composted manures can be stored easily and marketed as value-added plant disease suppressive products in the potting mix and golf course industries (Boulter *et al.* 2000; Craft and Nelson 1996; Hoitink *et al.* 1997).

The stability of composts, or the degree to which the organic fractions in composts have been stabilized during the process, critically affects compost quality. Unstable composts may be recolonized by fecal pathogens after peak heating (Bohnel and Lube 2000; Parmar *et al.* 2001). They may also generate odors (Elwell *et al.* 2001; Louhelainen *et al.* 2001; Nicolai and Janni 2001; Park *et al.* 2001; Walker 1993) and cause phytotoxic responses in plants during utilization (Hirai *et al.* 1993; Warman 1999). Operators of composting plants interested in process control and management as well as compost users, therefore, are very interested in the stability of composts.

Several types of compost stability tests have been developed. They range from elaborate tests which determine the rate of oxygen uptake under process control conditions (Tseng *et al.* 1995; Scaglia *et al.* 2000), to more simple batch tests that either measure O_2 uptake or CO_2 production at a constant temperature (Iannotti *et al.* 1994) or monitor metabolic heat produced by the compost microbiota during incubation in a nearly adiabatic chamber under controlled conditions (Dell'Abate *et al.* 1998). The rate of compost respiration determined over three days by CO_2 respirometry at 25°C, as described by Bartha and Pramer (1965), has been modified (T=37°C) and adopted by the U.S. Composting Council as a standard method of measuring compost stability (Thompson *et al.* 2002; TMECC 05.08-B). Rates of respiration are expressed as mg CO_2-C g^{-1} dry solids day^{-1} or mg CO_2-C g^{-1} -volatile solids day^{-1}. Even though these methods are simple, they are laborious and generally unsuitable for on-farm use.

A short duration, quantitative O_2 uptake procedure has been developed for testing compost stability at industrial scale facilities (Iannotti *et al.* 1994). The test is useful if compost samples are incubated at 37°C because the microorganisms in immature high temperature composts are not active at the lower temperature of 25°C that typically is used for soil respirometry. Low temperature preincubation of samples at 25°C avoids inhibition of microbial activity during the first 24-36 hr of a test in samples collected from high temperature immature composts and, therefore, it also avoids false stability readings (McKinley and Vestal 1984). Unfortunately, this stability procedure is too complex for on-farm use.

In some cases compost stability is determined on the basis of heat output by microorganisms in compost samples incubated in a nearly adiabatic Dewar flask (Jourdan 1982). The increase in temperature over time in the sample provides a measure of stability. Although this method seems simple enough and sound in principle (Finstein *et al.* 1983), it is a four-day test with several pitfalls that prevent it from serving as a reliable on-farm method unless many precautions are taken (Weppen 2002).

Compost maturity, which refers to the ability of composts to support plant growth, critically affects the value-added market potential of composts. It is much more difficult to define than stability. One aspect of compost maturity is compost ammoniacal-N concentration. Composts low in C/N ratio may contain phytotoxic concentrations of ammoniacal N (Hirai *et al.* 1983). This limits the application rate of composts in potting mixes and even in the field if it is applied directly before planting, thus severely limiting marketability of the product in value-added markets. Nitrate-N usually accumulates during curing of composts as a result of nitrification. Thus, a decrease in ammoniacal N concentration over time and an increase in nitrate-N concentration during curing is an indication of maturity (Ouatmane *et al.* 2000; Belete *et al.* 2001; Eggen and Oistein 2001). Thus, it is important to monitor stability based on respirometry as well as ammoniacal N in composts prepared for use in high value crops. This is one of the reasons why the U.S. Composting Council has adopted the Solvita® stability/maturity kit as a simple method for assessment of these properties of composts. It involves the use of colorimetric paddles sensitive to carbon dioxide and ammonia in a jar that contains a fixed volume compost sample. Although this test seems suitable for on-farm use, its accuracy to our knowledge has not been independently verified.

The objective of this research was to test the suitability of the Solvita® Compost Maturity Test for the determination of stability and ammonia toxicity potential of composted dairy and hog manures. The tests were evaluated by comparing Solvita CO_2, Solvita NH_3 and Solvita maturity index values to CO_2-C evolution rates and ammoniacal nitrogen concentrations measured throughout the composting process for three different types of composted manures.

Materials and Methods

Raw Materials and Composting Process

Three types of manures were used, 1) manure from an OSU dairy facility blended with a mixture of sawdust and wood shavings, 2) the same dairy manure blended with wheat straw, and 3) a partially composted hog manure mixed with shredded wood pallets (mostly red oak) and sawdust received from a High Rise Hog® facility (Keener *et al.* 2000; Michel *et al.* 2001). The ratio of dairy manure and bulking agents for the sawdust/wood shavings and wheat straw manure blends was approximately 3:1 (v/v) to produce an initial moisture content of approximately 65% and provide optimum conditions for composting as described previously (Pecchia *et al.* 2002). The partially composted hog manure was collected after three cycles of 1000 hogs and then blended with sawdust (hard wood, primarily red oak) to reduce its moisture content. Two equal sized batches of each of the three types of manures were composted from April 2001 to July 2001 in windrows with a height of approximately 1.5 m and width of 3 m to maintain process temperatures within the range of 55 to 70°C. Initially, the hog manure was turned daily to further reduce its moisture content, avoid leachate formation and reduce odor generation. Thereafter, all windrows were turned weekly or biweekly to maintain adequate porosity. Due to shrinkage of the compost windrow height was readjusted periodically to 1.5 m. Water was added if necessary to maintain optimum process conditions (Rynk *et al.* 1992).

Sample Collection and Preparation

Changes in physical and chemical properties and in stability and maturity of the three manure composts during composting were monitored using sampling protocols and analytical methods specified by the U.S. Composting Council (TMECC 2002). Three 9 L composite samples were collected from each compost type and batch at weekly or biweekly intervals within 1 hr after turning of the windrows. Ten subsamples were removed from five locations (2-3 m apart) within each windrow and composited. Half of these subsamples were removed from the high temperature center and the remainder from the outer zone of each windrow. Each composite sample was then mixed thoroughly to ensure maximum sample homogeneity and placed in a polyethylene bag. One 500 g subsample was cooled over ice and submitted for analysis of the chemical properties to the Service Testing and Research (STAR) Laboratory at The Ohio State University (Wooster, Ohio). A second 500 g subsample was placed in a partially closed polyethylene bag, preincubated overnight at 25°C and then used to determine compost stability based on CO_2 respirometry (Bartha and Pramer 1965) and on the Solvita® Compost Maturity test (TMECC method 05.08-E). The remainder of each sample (approximately 7 L) was stored at -15°C. Sampling of composts in windrows continued until after stability values based on CO_2 respirometry were less than 1.0 mg CO_2-C g^{-1} VS day^{-1}, a value recognized as stable for composts in general (TMECC 2002). Both tests were conducted at a temperature of 25°C.

Compost Stability

The rate of respiration (mg CO_2-C g^{-1} VS day^{-1}) for each compost sample was determined by the standard protocol (TMECC method 05.08-B) except that a temperature of 25ºC was used. Briefly, a 25.0 g subsample that had been preincubated overnight at 25ºC was transferred into a 1 L Mason jar containing 20 mL of 1 M NaOH in a 100-mL beaker. The lid was closed tightly and the jar was then incubated at 25°C. A jar without compost served as the control. Preliminary measurements of the oxygen concentration revealed that the air to compost volume ratio in the jar was sufficient to avoid O_2 limited respiration conditions for all compost samples used in this work. The rate of respiration expressed as mg CO_2-C g^{-1} VS day^{-1} was monitored for three days and the mean rate of respiration per day was then calculated.

Compost Maturity by Solvita® Kit Test

Manure compost maturity tests were performed with the Solvita® kit (Woods End® Research Laboratory, Inc., Mt Vernon, Maine) following the protocol specified in the Solvita® kit manual (Guide to Solvita® testing for compost maturity index) supplied by the manufacturer. The Solvita® kit measures carbon dioxide evolution and ammonia emission simultaneously. The moisture content of all the samples was within the optimum range (50-55%, w/w) for microbial activity. Samples were allowed to equilibrate at 25°C in partially closed plastic bags as described above and loaded into the Solvita® jars up to the fill line. Solvita® CO_2 and NH_3 test gel-paddles were carefully inserted into the compost without touching the gels after tearing the packs. The sample jars were then closed tightly thereafter. During this process the gel portion of the paddle did not come into contact with the samples and paddle positioning allowed easy viewing of gel color change. To determine Solvita® CO_2 and NH_3 kit values, the observed gel color change after 4-hr incubation at 25°C was matched with the color on charts supplied with the kit. The gel color change was used to determine Solvita® kit CO_2 values on a scale of 1-8 and NH_3 values on a scale of 1-5. Finally, the two values were used to determine the Solvita® maturity index on a scale of 1-8 which then represents the maturity level of the compost samples.

Chemical Properties of Composts

Changes in chemical properties of the three types of manures during composting were monitored according to standard protocols specified by the U.S. Composting Council (TMECC 2002). Samples were ground to the particle size specified by the analytical method for each chemical property. The number of subsamples (3-5) depended on the sensitivity of a particular assay, again as specified for each method in order to obtain means with coefficients of variation less than 30%. Percent dry solids (w/w) were determined after oven drying (60-80°C) to a constant weight. The pH was determined on a 1:5 dilution of compost with distilled water (TMECC method 04.11-A1:5). Electrical conductivity also was determined on this slurry with a solu-bridge conductivity meter (Beckman Instruments, Cedar Grove, New Jersey; TMECC method 04.10-A). Percent ash was determined after heating for 4 hr in a muffle furnace at 550°C (TMECC method 03.02-A). Percent volatile solids (VS) were determined by subtracting percent ash from 100. Total N analyses were performed using a Dumas combustion method (VarioMax N analyzer, Elementar Americas; TMECC methods 04.02-D and 04.01-A). The detection limit for this instrument was 200 mg N kg^{-1}. Total C was determined using coulometry.

This instrument converts C in the sample to CO_2 by oxidation at 1100°C. The detection limit was 1 mg C kg^{-1}. Inorganic C was determined by coulometry (model 5020, Coulometrics, UIC, Joliet, Illinois). Organic C was determined by subtracting inorganic C from total C. Total NO_3 –N and NH_4-N were determined by ion chromatography (TMECC method 04.02-B) and micro-Kjeldahl distillation-titration methods, respectively.

Statistical Analyses and Data Presentation

Means and standard deviations for all values presented in this work are based on two windrows per compost type with three replicates per windrow (n=3). Throughout the composting process, the two windrows of each type of manure used in this work were uniform in appearance and the chemical and biological properties were not significantly different. Therefore, the data for the tests from the two windrows of each compost type were combined (n=6). Because composting of all manures was started in the spring under similar weather conditions for each, mean values for the physical, chemical and biological properties of the manures during composting were plotted on the basis of days of composting rather than the julian date.

Statistical analyses of the correlations between Solvita® test results and the monitored manure properties during composting were performed using SAS statistical analysis software (SAS Institute Inc., Cary, North Carolina release 8.1 for Windows 98 platform) and Minitab (version 13 for Windows, Minitab Inc., www.minitab.com). The linear correlations between Solvita® test values of CO_2, NH_3-N and maturity and: 1) CO_2 respirometry, 2) compost age, 3) % VS, 4) C:N ratio, 5) organic C concentration, 6) total N concentration, 7) ammoniacal-N concentration, and 8) nitrate-N concentration were determined. Absolute Pearson linear correlation coefficients (r) were used to determine significance of correlations based on a level of probability (P-values). Correlation coefficients were declared significant for P-values ≤0.0005 and highly significant for P-values ≤0.0001.

The relationships between Solvita CO_2 value and CO_2-C respiration rate, Solvita Maturity Index and CO_2-C respiration rate and Solvita NH_3 index and ammoniacal-N concentration were analyzed by linear, log transformed linear, log transformed second order polynomial and polynomial second order regression analyses. The 90% prediction intervals and regression line equations were determined using Minitab software (Release 13.1).

Results

Compost Temperature and Moisture Content

Temperature profiles for the three types of composts differed considerably (Figure 1A). The dairy manure/sawdust compost reached temperatures of 60°C within ten days of window formation. In contrast, the dairy manure/straw compost did not reach this high temperature until after ten weeks of window formation. This may have been due to the very high initial porosity of this material which would be expected to lead to greater heat losses to the environment (Pecchia *et al.* 2002). The hog manure compost did not reach similarly high temperatures until after six weeks. Shortly after formation (1-2 days), leachate drained out of the base of this windrow indicating that its moisture content was too high and that this mixture could not support the same moisture content as the other two composts (65%). Therefore, it was turned several times to promote drying. Thus, the low initial temperature was probably due to low porosity and, there-

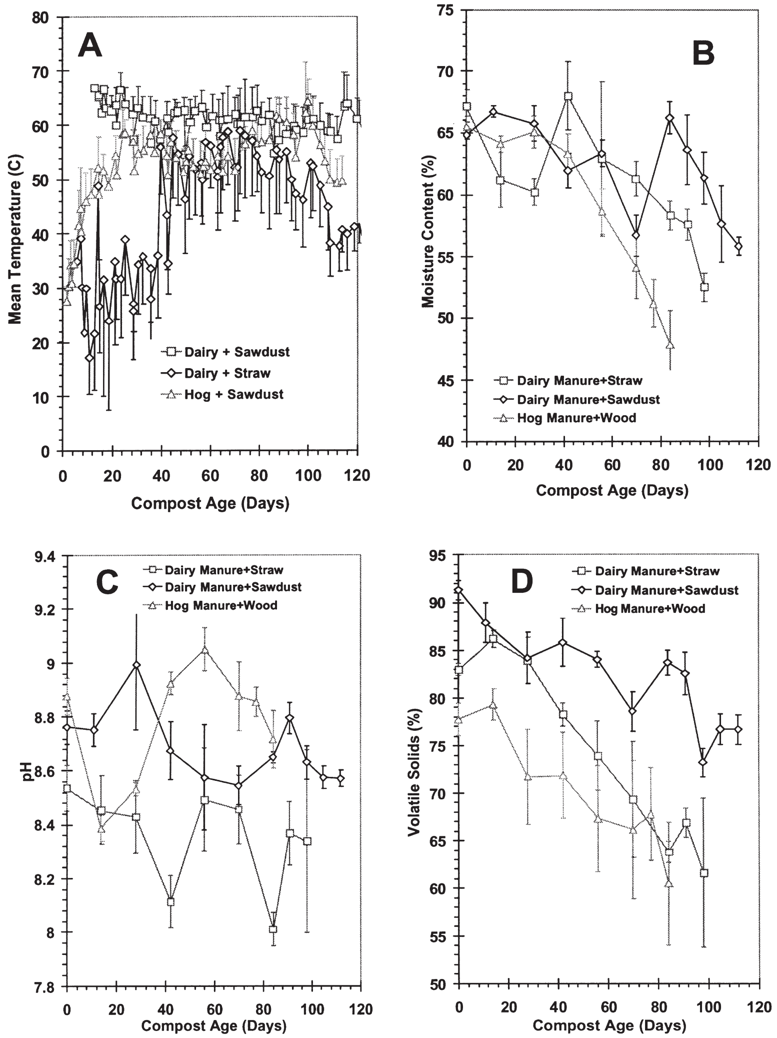

Figure 1. Trends in temperature (A), moisture content (B), pH (C) and volatile solids (D) during windrow composting of dairy manure mixed with straw or sawdust, and hog manure mixed with wood chips. Each value represents the mean of six randomly collected samples harvested at weekly or biweekly intervals from each of two windrows (3 replications per windrow).

fore, low oxygen availability in addition to the relatively resistant to decomposition ground wood used as bulking agent and carbon source in the hog manure compost. All windrows eventually reached mean temperatures within the optimum range (55-65ºC) for decomposition and pathogen destruction.

The mean moisture content of the dairy manure/straw compost decreased from an initial value of 67% to 52% after 98 days (Figure 1B). In the dairy manure/sawdust compost it decreased from 65% to 56% after 112 days and in the hog manure compost it decreased from 65% to 48% after 84 days.

Compost pH

In general, mean pH values of the hog and dairy manure/sawdust composts were higher (8.4-9.0) than those of the dairy manure/straw compost (8.0-8.5, Figure 1C). The pH of the hog manure compost fluctuated from 8.4 to 9 during the process. Large fluctuations in mean pH values also were observed in samples removed from the dairy manure/straw compost. Less variation in pH was observed in the dairy manure/sawdust compost.

Compost % Volatile Solids (VS) and Organic C

Trends in percent volatile solids for the three types of manures revealed significant losses in volatile solids during composting (Figure 1D). The greatest losses were observed for the dairy manure/straw compost where % VS declined from an initial mean value of 83% to 62% after 98 days representing a loss of 67% of the initial VS. Percent VS for the dairy manure/sawdust compost decreased from an initial mean value of 91% to 77% after 112 days representing a loss of 67% of the initial VS. VS% for the hog manure compost declined from 77% to 60% VS after 84 days which represented a 55% loss of the initial VS on a constant ash basis. Similar trends in destruction of dry solids were observed on the basis of changes in mean percent organic C (Figure 2a). In

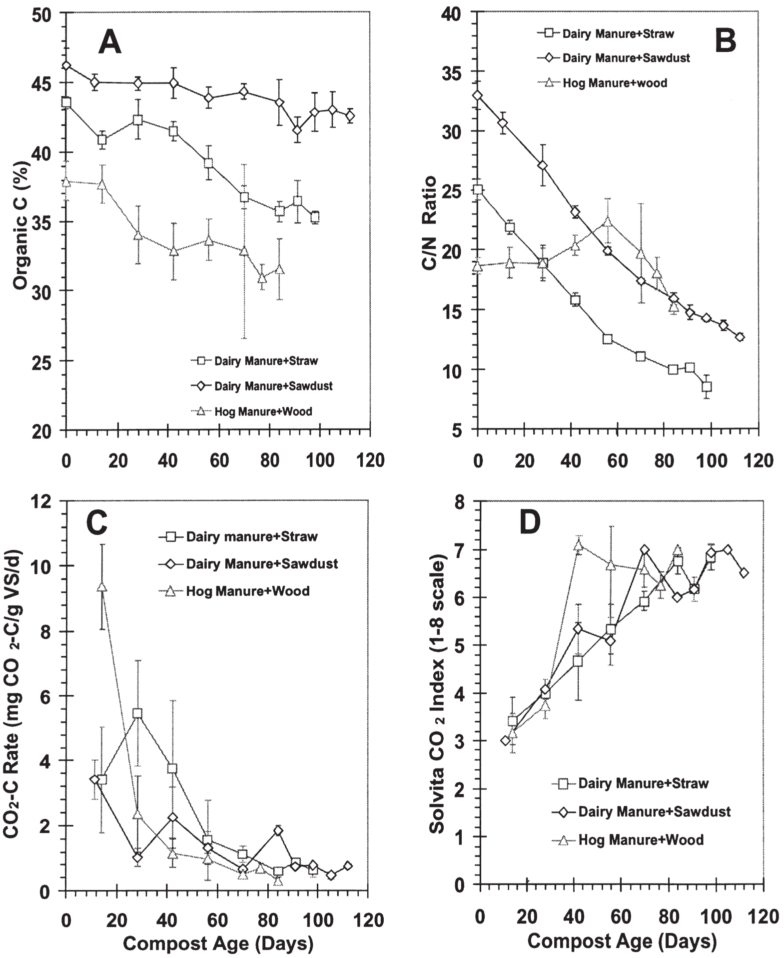

Figure 2. Trends in total percent organic C (A), C/N ratio (B), rate of CO_2 respiration (C) and Solvita® CO_2 index (D) during windrow composting of dairy manure mixed with straw or sawdust, and hog manure mixed with wood chips. Each value represents the mean of six randomly collected samples harvested at weekly or biweekly intervals from each of two windrows (3 replications per windrow).

the dairy manure/straw compost, percent organic C decreased from a mean value of 44% to 35% after 98 days. The organic C value of the dairy manure/sawdust compost declined from a mean value of 46% to 43% and that for the hog manure compost from a mean value of 38% to 32%. These represented losses of 64%, 63% and 52% of the initial carbon, respectively, on a constant ash basis.

Compost Total N, C/N, Ammoniacal N and Nitrate-N

The total percent N for the dairy manure/straw compost increased from an initial mean value of 1.8% to 4.2% after 98 days. Mean total percent N values for the dairy manure/sawdust compost were significantly lower than the sawdust compost and increased from an initial value of 1.4% to 3.4% after 112 days (Figure 3B). The high initial mean total percent N value of 2.1% for the hog manure compost was not

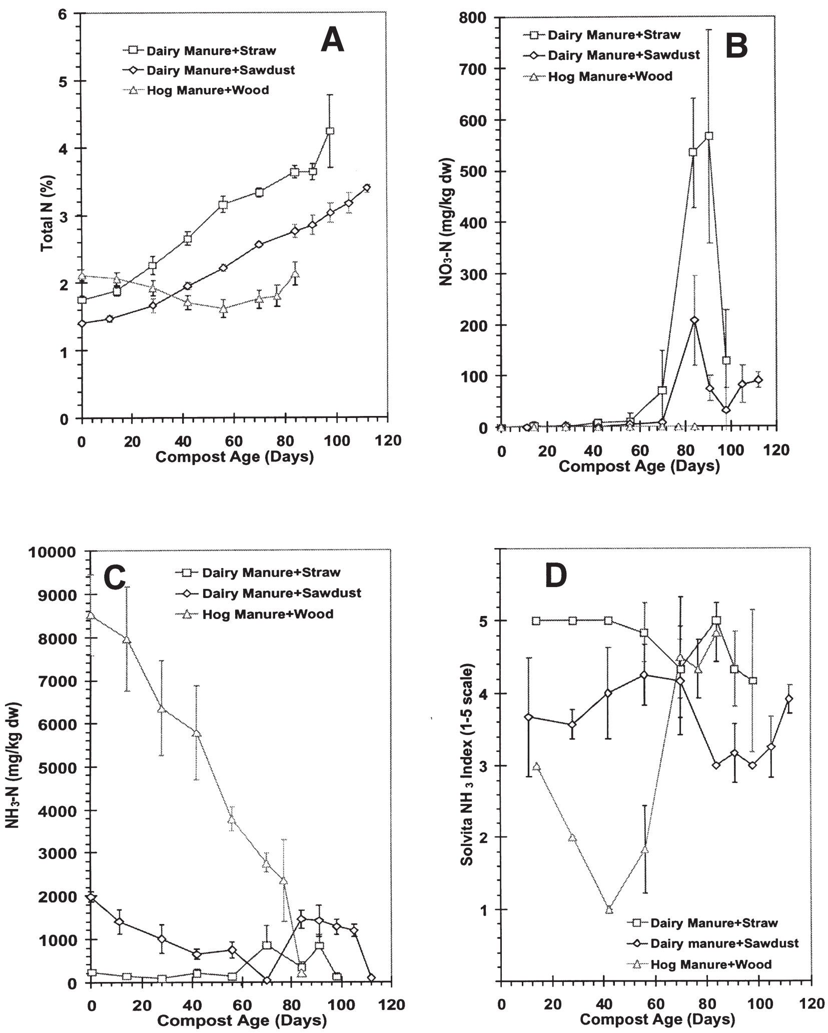

Figure 3. Trends in total nitrogen (A), nitrate-N (B) and ammoniacal-N (C) concentration, and Solvita® NH_3-index (D) during windrow composting of dairy manure mixed with straw or sawdust, and hog manure mixed with wood chips. Each value represents the mean of six randomly collected samples harvested at weekly or biweekly intervals from each of two windrows (3 replications per windrow).

significantly different from the value of 2.1% observed after 84 days, although a significant decrease followed by an increase was observed during the process. This suggests that significant losses of N occurred early during composting of this manure followed by concentration of N thereafter due to organic matter loss. The C/N ratio (Figure 2b) for the dairy manure/straw compost decreased from an initial mean value of 25 to 9 after 98 days. The mean C/N ratio values for the dairy manure/sawdust compost decreased from 33 to 13 after 112 days. The hog manure compost had a low initial C/N ratio. It decreased during composting from an initial mean value of 19 to 15 after 84 days.

The concentrations of ammoniacal-N early during composting of the dairy manure/straw were comparatively low, probably due to losses of ammonia from the porous mixture (Figure 3c). These concentrations increased after 56 days of composting when the straw had lost its physical structure and the bulk density of the compost increased (reported for these specific composts in Pecchia *et al.* 2002). The concentration of ammoniacal-N declined to a mean value of 116 mg kg^{-1} dw^{-1} after 98 days when temperatures in the windrow had declined also (Figure 1a). During this period late in the process, the concentration of nitrate-N increased to a value as high as 565 mg kg dw^{-1} on day 91 but declined to 128 mg kg dw^{-1} after 98 days (Figure 3a). The concentration of ammoniacal-N in the dairy manure/sawdust compost declined from an initial mean value of 1976 to 89 mg kg dw^{-1}after 112 days (Figure 3C). Nitrate-N was detected after 56 days and a mean concentration of 91 mg kg dw^{-1} was detected after 112 days. In the hog manure compost, extremely high ammoniacal-N concentrations prevailed throughout the entire 77-day composting period (Figure 3C). After 84 days, it had decreased to 196 mg kg dw^{-1} - but nitrate-N was not yet detectable (Figure 3A) in this still high in temperature compost (Figure 1A). Thus, the initial ammoniacal-N concentrations of the three composts ranged over two orders of magnitude (from 100 to nearly 10000 mg kg^{-1}) representing a wide range of initial ammoniacal-N concentrations.

CO_2 Respirometry and Solvita CO_2 Kit Analysis

The respiration rate based on CO_2 respirometry was measured after over night preincubation at 25°C followed by slight mixing of the samples to release trapped CO_2. The CO_2 evolution rates of the dairy manure/straw, dairy manure/sawdust, and hog manure plus wood shavings decreased from initial means (n=6) of 3.41, 3.42 and 9.35 to 0.63, 0.76 and 0.31 mg CO_2-C g^{-1} VS day^{-1}, respectively. This revealed significant changes in mean rates of respiration over time (Figure 2C). The greatest decrease in rate of respiration was observed during composting of the hog manure compost, particularly early during the process. A significant decrease in rate of respiration was also observed early during composting of the dairy manure/sawdust. The greatest decrease in rate of respiration activity in the straw-manure compost occurred between days 42 and 70 when the straw was losing its physical structure. The hog manure compost reached a low mean value of 0.31 mg CO_2-C g^{-1} VS d^{-1} after 84 days of composting. The dairy manure/straw compost reached a low value of 0.63 mg CO_2-C g^{-1} VS d^{-1} after 98 days. The dairy manure/sawdust compost reached a consistently low mean stability value after 91 days.

The rates of respiration measured on the first day of the three-day respirometry test were on average 50% higher than the CO_2 rate values averaged over the entire three day bioassay period (data not shown). This indicated that the microorganisms in the compost samples were not inhibited in activity due to initial conditions in the compost. Thus, a fourth day of measurement was not required for any of the respirometry bioassays performed in this work.

The mean Solvita® CO_2 test values increased from 3.4, 3.0 and 3.2 to 6.8, 6.5 and 7.0, respectively (Figure 2D). Overall, trends in stability observed with the respirometry procedure (Figure 2C) agreed inversely with those based on the Solvita® CO_2 test (Figure 2D). Since the CO_2 values measured by the Solvita® kit were made during the first 4 hours of incubation on the first day, they would be expected to be measuring a higher rate than the 3-day average values determined by the CO_2 respirometry procedure.

Solvita® Ammonia Kit and Maturity Analyses

The Solvita NH_3 index (1-5 scale) for ammonia emission by the three manures at various stages during the composting process showed that little ammonia was emitted by the dairy manure/straw compost (NH_3 index > 4.5 for all samples; Figure 3D). In general, more ammonia was lost from the dairy manure/sawdust than from the dairy manure/straw compost samples. The test indicated that the hog manure compost released much more NH_3 than the dairy manure composts and that the highest quantity of ammonia release occurred after 40 days. Interestingly, early during composting of this manure much less ammonia was detected by the Solvita NH_3 test (Figure 3D) even though the concentration of ammoniacal-N based on the extraction procedure was high at that time (Figure 3C). The Solvita Test measures the ammonia in the gas phase above the compost while the lab test determines ammoniacal N within the compost itself which may explain the differences.

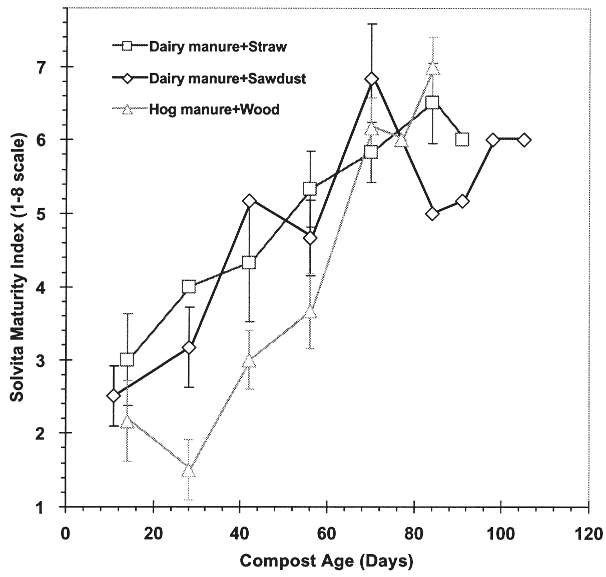

Figure 4. Trends in Solvita® maturity index during windrow composting of dairy manure mixed with straw or sawdust, and hog manure mixed with wood chips. Each value represents the mean ± one standard deviation of six randomly collected samples from each of two windrows (3 replications per windrow).

The Solvita CO_2 test and Solvita NH_3 test values were used to calculate the Solvita maturity index (scale 1-8). The Solvita maturity index values ranged from 2 to 3 initially, which indicates a "raw" or "very active" compost (Figure 4). The hog manure compost had the lowest maturity indices through day 56 (Figure 4) probably due to the contribution of the low Solvita NH_3 index values for this compost (Figure 3D). The two dairy manure composts showed similar Solvita maturity index values during composting (Figure 4). The Solvita maturity index for all three composts rose during composting. After 100 days all three exhibited Solvita maturity values of 6 to 7 indicative of "curing" or "finished" compost according to the Solvita test interpretive booklet.