

Changes in physicochemical properties and gaseous emissions of composting swine manure amended with alum and zeolite

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Abstract—Ammonia emissions from composted swine manure and the resulting physicochemical changes were monitored to determine the effectiveness of adding alum and zeolite during the composting process, as well as the most effective addition method. The two amendments reduced ammonia emissions 85-92%, with the finished compost retaining three-fold more $\text{NH}_4^+\text{-N}$ than the unamended control. The addition of zeolite sequestered 44% of the retained $\text{NH}_4^+\text{-N}$ at zeolite exchange sites. The addition of amendments did not appear to significantly affect microbial activity, because the patterns of CO_2 emissions, total organic carbon (TOC) reduction, and the ratio of humic acid to TOC of amended and unamended composts were very similar. The final respiration rates and Solvita[®] maturity index indicated that the finished compost was well matured and aged. Alum has a high potential to reduce ammonia emissions and concomitantly enhance fertilizer N value. Zeolite further reduces ammonia emissions, and improves fertilizer quality, by serving as a slow-release N source.

Key words: Composting, Ammonia Emission, Swine Manure, Alum, Zeolite

INTRODUCTION

Composting of animal manures is increasingly recognized as a viable treatment method that naturally recycles organic matter and improves the quality of compost in organic farming [1,2]. During composting, high ammonia emissions stem from the microbial decomposition of nitrogenous organic compounds in the manure, principally urea and uric acid, decreasing the fertilizer N value and leading to environmental pollution [3].

Ammonia emissions increase with increases of pH, moisture content, wind speed, ammonia concentration, and temperature [4]. Without proper control of these parameters, 33-62% of the initial total nitrogen of the manure may be lost during composting [5]. Thus, methods are needed to reduce ammonia emissions and nitrogen loss from the compost and to alleviate malodor from large-scale manure composting facilities [6]. Current ammonia emission reduction techniques include dietary manipulation, ventilation, chemical additives, dietary enzymes, odor removal filters, ozone, and various land application techniques [7]. Ammonia emissions are most efficiently inhibited by the use of chemical amendments that inhibit microbial decomposition, absorb ammonia, or acidify NH_3 to the ammonium ion, NH_4^+ [8]. However, the use of metabolic inhibitors is not desirable, as biodegradation improves the physical and biological properties of compost, including aeration, ease of seedbed preparation, water-holding capacity, and stimulation of soil microbial activity [4]. Alum (aluminum sulfate) and zeolite (clinoptilolites), the acidifying agent and ammonia absorbent, respectively, most commonly used in studies have been shown to greatly reduce ammonia emissions from animal manure in lab-scale and large-scale composting

processes [9]. However, few studies have investigated the effect of these amendments on the microbial decomposition of animal manure during the composting process. Compost is considered unstable if it contains a high proportion of biodegradable matter that may sustain high microbial activity. Stability is not only an important compost quality characteristic, but can also be used to monitor process performance and evaluate different composting strategies [10]. Parameters that effectively reflect the stabilization process of organic matter during composting can be traced and measured using various methods based on the physical (temperature, odor, optical density of water extracts), chemical (volatile solids, C/N ratio, total organic carbon, humic substances), or biological (respiration measured either as O_2 consumption or CO_2 production, enzyme activity) characteristics of compost [11]. Currently, compost stability evaluations usually use respirometric methods to monitor the degradation of organic matter [10,12,13].

In this study, we assessed the stabilization process of organic matter by monitoring CO_2 evolution, total organic carbon (TOC), and humic substances. Monitoring these parameters after the application of different amendments to the compost allowed us to optimize amendment addition strategies to minimize ammonia emissions, as well as produce compost with better characteristics for immediate use by plants. Our objectives were to (i) evaluate the evolution of ammonia and carbon dioxide after the addition of alum and zeolite, (ii) determine the proper timing of chemical amendment additions, and (iii) evaluate the effects of chemical amendments on the composting process and characteristics of the resulting compost.

MATERIALS AND METHODS

1. Preparation of Swine Manure for Composting

Swine manure (moisture content of 81%) collected from Yong-

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In Chukhyup Fertilizer Factory (Yong-In, Korea) was mixed with oven-dried sawdust and wood bark at a 7.6 : 1.7 : 1 weight ratio to adjust the moisture content of the mixture to about 60% and provide porosity for aeration. To quantify the effect of aluminum sulfate and clinoptilolite on the composting process, inorganic amendments were added to the swine manure mixture at four different application strategies. The control (R1) had no amendments. The three experiments with amendments (R2, R3, R4) are shown in Table 1.

As alum, we used a 51.0-57.5% range of aluminum sulfate ($\text{Al}_2(\text{SO}_4)_3 \cdot 14-18 \text{H}_2\text{O}$) with $\text{Al}_2(\text{SO}_4)_3$, as specified by the manufacturer (Daejung Chemicals and Metals Co.). Zeolite was 200-mesh clinoptilolite, (Na_2, Ca) $(\text{Al}_2\text{SiO}_{18}) \cdot 6\text{H}_2\text{O}$ (Rex Materials Co., Korea), with pH and electrical conductivity of 6.91 and 0.57 mS/cm, respec-

Table 1. Time schedule and amounts of alum (A) and zeolite (Z) added into the composting reactors as a source of aluminum sulfate and clinoptilolite, respectively

Reactors	Time and amounts added (w/w %)				
	Day 0	Day 1	Day 2	Day 3	Total
R1	0	0	0	0	0
R2	0	1.25 A	0.625 A	0.625 A	2.5 A
R3	1.25 A	0	0.625 A	0.625 A	2.5 A
R4	0	1.25 A 5.0 Z	0.625 A	0.625 A	2.5 A 5.0 Z

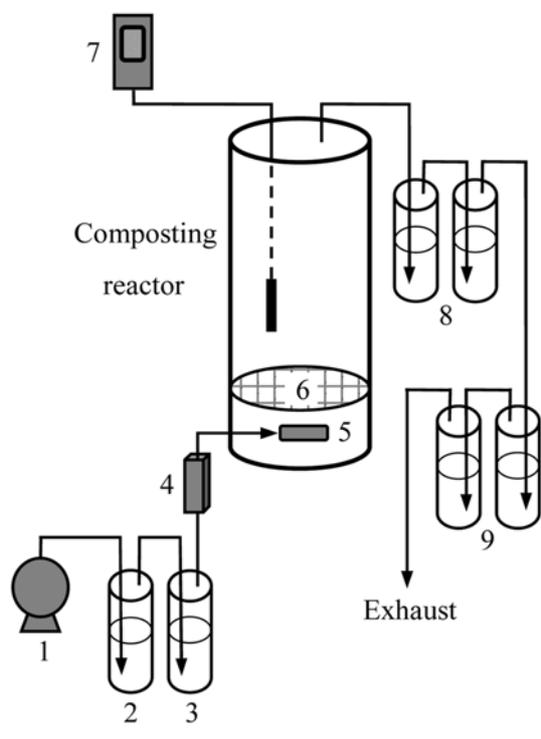


Fig. 1. Schematic diagram of acrylic reactor (18.5 cm diameter and 38 cm depth) containing 2,700 g of compost (manure/sawdust/wood bark) mixture.

1. Air blower
2. CO₂ trap (5 N KOH)
3. Deionized water
4. Flow meter
5. Air diffuser
6. Supporting screen
7. Temp monitor & probe
8. CO₂ trap (5 N KOH)
9. NH₃ trap (0.3 N H₂SO₄)

tively. The pore size, refractive index, cation exchange capacity, surface area, and hardness were 3-10 Å, 1.47-1.49, 100-160 meq/100 g, 80-100 m²/g, and 3.5-5, respectively.

2. Experimental Setup

As shown in Fig. 1, cylindrical composting reactors consisting of 1-cm-thick acrylic columns (18.5 cm diameter, 38 cm depth) received 2,700 g manure/sawdust/wood bark mixture. At the bottom of each reactor, a 9-cm-high space was allocated for aeration, and an 8-cm-high space at the top was maintained as headspace. The reactors were insulated by wrapping with 2-cm-thick cloth to minimize conductive heat loss. A temperature probe (multilogPRO, Fourier) inserted into the middle of each reactor logged temperatures every hour. Air, provided by a blower at a rate of 0.4 L/kg-manure mixture/min, was first passed through 750-mL 5 N KOH solution to remove ambient carbon dioxide, and then through 500-mL deionized water for humidification. The CO₂-free air was supplied to the reactor, and exhaust gases were passed through two consecutive impingers containing 100 mL 5 N KOH, and then through two more consecutive impingers containing 100 mL 0.3 N H₂SO₄ to trap CO₂ and ammonia, respectively. The CO₂- and ammonia-trapping solutions were replaced twice a day during high decomposition rate periods to ensure that the trapping solutions did not become saturated. To determine the various parameters, we sampled the compost once a day after the compost mixture was thoroughly mixed for homogenization.

3. Analytical Methods

Samples were collected at three randomized points from each reactor and well mixed for analysis. Moisture content was determined by drying a 2-3-g sample at 105 °C for 24 h. To determine pH, water-extractable TOC portion, NH₄⁺-N, NO₃⁻-N, and NO₂⁻-N, and electrical conductivity, a 3-g sample was added to 30 mL deionized water and shaken at 150 rpm for 2 h at 6 °C. The extract was then centrifuged (25,000 ×g, 20 min), and the supernatant was analyzed for the following parameters: pH, using a pH meter (Orion 430), organic carbon using a TOC analyzer (Shimadzu, TOC-5000A), NH₄⁺-N using an ammonia-selective electrode (Orion 95-12), NO₃⁻-N, and NO₂⁻-N using an Auto Analyzer 3 (Bran Luebbe), and electrical conductivity using an electrical conductivity probe (WTW). For humic acid determination, 1.5 g of compost was extracted with 15 ml of 0.1 M sodium pyrophosphate for 24 h at 60 °C. The resulting solution was centrifuged at 17,000 ×g for 20 min and 2 ml of the supernatant was mixed with 2 ml glycine-NaOH buffer (pH 10). The optical density of the solution was then taken at 465 nm and 665 nm with a spectrophotometer (Shimadzu, UV-1601PC). Standard solutions of humic acid (Sigma) were prepared in the range from 7.5 to 1,000 ppm. To determine the amount of NH₄⁺-N sequestered on zeolite, a 2 N KCl solution was used to extract NH₄⁺-N from the compost [5]. The concentration of CO₂ trapped in the 5 N KOH solution was determined by a double-titration method [14], and ammonia trapped in the 0.3 N H₂SO₄ solution was measured with an ammonia-selective electrode (Orion).

RESULTS AND DISCUSSION

1. Changes of Physicochemical Factors during Composting

The temperature of the manure mixture in the reactors began to rise soon after the establishment of composting conditions and reached

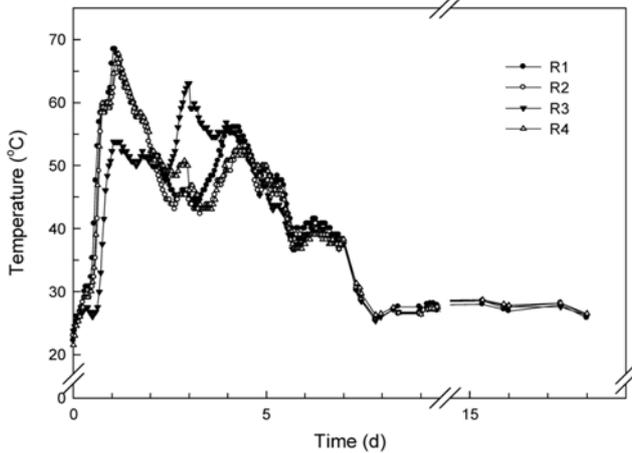


Fig. 2. Temperature profile of the composting reactors (R1-R4) prepared to identify the effects of alum and zeolite additions. Ambient air temperature fluctuated between 22.3 °C and 31.8 °C during the experiment.

the thermophilic phase after 1 day of aeration (Fig. 2). Several temperature peaks were observed during the composting period, with maximum temperatures of R1, R2, R3, and R4 being 68.4 °C, 66.1 °C, 63.1 °C, and 67.7 °C, respectively. R1, R2, and R4 reached maximum temperatures on Day 1 and R3 on Day 3, possibly due to the inhibiting effect of the low pH (<6.5) caused by the addition of 1.25% (w/w) alum to R3 on Day 0, as shown in Fig. 3. The inhibition of thermophilic microorganisms at low pH (<6.5) may explain the often-observed lag phase in the transition from mesophilic to thermophilic conditions in the initial composting phase [15]. Our preliminary experiments also showed that the addition of 2.5% and 5% (w/w) alum at the beginning of composting greatly affected microbial activity, due to abrupt drops in pH, from 7.5 to 4.7 and 4.0, respectively. Thus, the thermophilic phase was not achieved (data not shown). On the other hand, once microorganisms had created a thermophilic environment, the addition of 1.25% or 0.625% alum had negligible effect on thermophilic microbial activity.

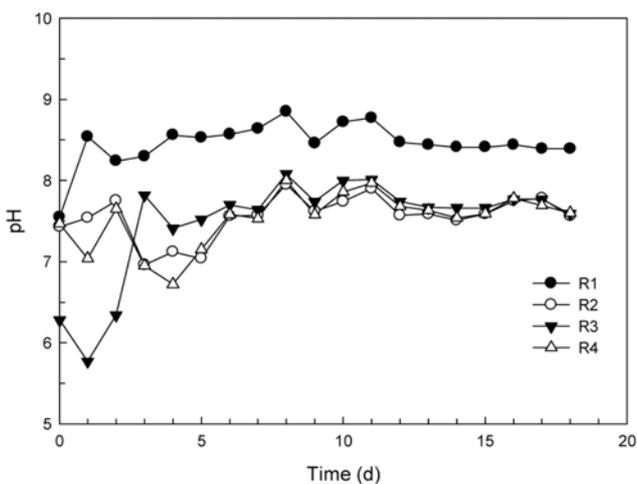


Fig. 3. Changes in pH of the composting reactors (R1-R4) during 18 days of composting of swine manure mixture.

During the initial phase of composting (Days 0-5), a pH decrease was observed in R2, R3, and R4 whenever alum or zeolite was added to the reactors (Fig. 3), as reported in other studies [4,9]. R3 recorded the most significant pH decrease with the addition of alum on Day 0, perhaps because of the absence of ammonia production to neutralize aluminum sulfate, the acidifying agent. Without this initial alum amendment, R2 and R4 did not register this extreme pH drop, although the pH was reduced slightly after alum was added. Multiple applications of alum in small doses, instead of a single large-dose application, also prevented abrupt changes in pH and ensured a successful composting process in all of the reactors. The pH changes in the reactors followed a similar pattern after Day 6, with the R1 range being 8.4-8.8 and that of R2, R3, and R4, 7.5-8.1.

The initial electrical conductivity (EC) of the manure mixture was 2.84 ± 1.40 mS/cm. The addition of alum increased R2, R3, and R4 EC, to 4.20, 4.27, and 3.83 mS/cm, respectively, on Day 4. Thereafter, R2, R3, and R4 EC slowly decreased, to 3.63, 3.94, and 3.29 mS/cm, respectively, by the end of the composting period (data not shown). The increase in EC with the addition of alum was associated with an increased SO_4^{2-} concentration, which also increased the concentration of NH_4^+ in the water phase, thus leading to a further increase in EC [16]. The EC of compost receiving zeolite (R4) was lower than that of R2 and R3 during the entire composting period. This indicates that zeolite and ions could exchange, leading to a decreased EC of the resulting compost. In contrast, R1 EC decreased rapidly, from 2.97 to 1.43 mS/cm in the Day 0-6 period, possibly because of high ammonia emissions at the high pH [17], which decreased the NH_4^+ concentration in the water phase.

2. Mitigation of Ammonia Emissions

The cumulative ammonia emissions from the reactors during the composting period (18 days) were 4.1, 0.7, 0.6, and 0.3 g- NH_3 -N/kg-dry compost for R1, R2, R3, and R4, respectively (Fig. 4). Although the pH and temperature profiles of R2 and R3 differed, depending on the alum application time, the addition of 2.5% alum to these reactors reduced ammonia emissions by 83.4-85.1%, compared with the control (R1). The application of 5% zeolite in addition to 2.5% alum to R4 reduced ammonia emissions by 92.4%, compared with the control. These reductions were somewhat higher

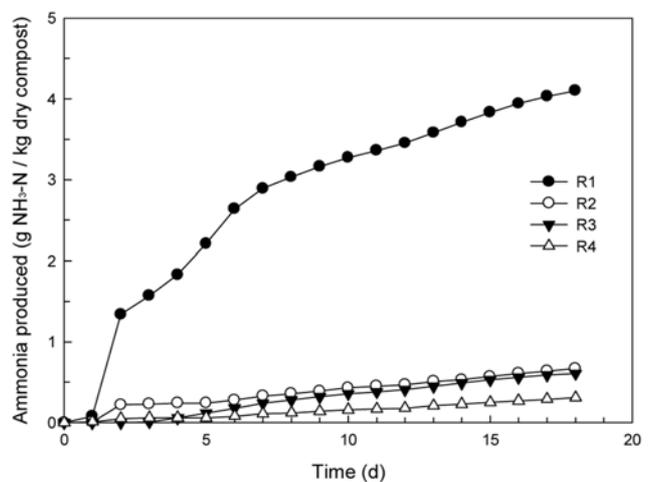


Fig. 4. Cumulative ammonia emission from the composting reactors (R1-R4).

than those reported in other animal manure composting studies: addition of 10% alum, 76% reduction [4]; 2.5% and 6.25% alum, $58 \pm 6\%$ and $57 \pm 10\%$ reduction, respectively [18]; 2.5%, 5.0%, and 10% zeolite, 20%, 50%, and 77% reduction, respectively [9]; and 2.5% and 6.25% zeolite, $22 \pm 6\%$ and $47 \pm 10\%$ reduction [18].

We attribute the reduction of emitted ammonia in all of the alum-treated reactors to simple acidification of the compost mixture, which is an effective way to conserve ammonia, as the percentage of total ammoniacal-N ($\text{NH}_4^+\text{-N} + \text{NH}_3\text{-N}$) dissolved as NH_3 gas is about 39%, 6%, 0.6%, 0.06%, and 0.006% at pHs of 9, 8, 7, 6, and 5, respectively [18]. However, most studies have reported that manure pH decreases to 4-6 after the addition of alum [4,18], which inhibits thermophilic microbial activity, as well as that of urease and uricase, which catalyze urea and uric acid decomposition to ammonia, respectively [9,16]. Thus, pH must be maintained near neutral to maximize ammonia solubilization and prevent the inhibition of thermophilic bacterial and enzyme activities involved in the degradation processes. The higher reduction of ammonia emission from R4, compared with R2 and R3, was most likely due to both a pH decrease by alum and NH_4^+ adsorption on NH_4^+ -exchange sites of zeolite [9,18], which decreased the quantity of dissolved ammoniacal-N ($\text{NH}_4^+\text{-N} + \text{NH}_3\text{-N}$) and, thus, the quantity of equilibrated $\text{NH}_3\text{-N}$ gas available for ammonia emission.

These modes of action of alum and zeolite were apparent when the water-extractable and KCl-extractable $\text{NH}_4^+\text{-N}$ were analyzed (Table 2). After the 18-day composting period of R1, R2, R3, and R4, the final pHs of the composting mixtures were 8.4, 7.6, 7.6, and 7.6, and water-extractable $\text{NH}_4^+\text{-N}$ values were 1.97, 6.51, 6.42, and 4.78 g $\text{NH}_4^+\text{-N}/\text{kg}$ -dry compost, respectively. The major loss of water-extractable $\text{NH}_4^+\text{-N}$ from the R1 manure mixture was due to ammonia-N emissions, but the ammonia-N loss of R4 requires further investigation, because its ammonia emissions were the lowest of the four reactors. 2 N KCl [19] successfully extracted most ammonium-N from the clinoptilolite, and the Day 18 values of 6.62, 6.58, and 6.31 g $\text{NH}_4^+\text{-N}/\text{kg}$ -dry compost for the KCl-extractable portion of R2, R3, and R4, respectively, were relatively consistent. These results are not inconsistent with those of Mulvaney [20], suggesting that the presence of $\text{NH}_4^+\text{-N}$ sequestered on the $\text{NH}_4^+\text{-N}$ exchange sites of zeolite could be replaced by K^+ . The concentrations of nitrite and nitrate in the four reactors never exceeded 0.03 g-N/kg-dry compost, as the high concentration of ammoniacal-N in the compost prevented nitrification, and nitrifying bacteria cannot thrive at thermophilic temperatures [21,22].

Our results suggest that the addition of alum alone or with zeolite

Table 2. Ammonium-N measured from the composting reactors (R1-R4) using either water or KCl solution

Reactors	Ammonia remained ^a (g $\text{NH}_4^+\text{-N}/\text{kg}$ dry compost)	
	Water-extractable	KCl-extractable
R1	1.94±0.40	2.36±0.39
R2	5.99±0.82	6.74±0.52
R3	6.20±0.54	6.83±0.89
R4	4.18±0.68	6.63±0.80

^aAverages of data obtained daily basis (n=18)

January, 2011

efficiently inhibited ammonia emissions during manure composting, thus alleviating odor problems near composting facilities and improving the fertilizer value of the compost by increasing its N content. Additionally, the form of ammonium-N sequestered in zeolite is a slow-releasing nitrogen source for plants, as it gradually releases NH_4^+ into the soil, thus avoiding the environmental problems of surface- and ground-water contamination by the extensive use of highly soluble NH_4^+ fertilizers [23].

3. Decomposition of Organic Matter during Composting

Composting is a natural aerobic process in which microorganisms transform organic matter into CO_2 , H_2O , and complex metastable compounds (e.g., humic substances). The final compost product should be stable, with the stability of compost defined as the degree to which the organic fractions have been transformed to recalcitrant or humus-like matter [10,11]. Many parameters with varying degrees of reliability and technical complication have been proposed for the evaluation of compost stability.

To evaluate compost stability as well as monitor the composting process performance, we conducted a respirometric analysis, based on CO_2 evolution during composting. The pattern of CO_2 emission in all the reactors was very similar, indicating that the addition of alum and zeolite did not significantly affect microbial activities involved in the decomposition processes (Fig. 5). More than 70% of the total CO_2 evolution occurred during the first week of the composting process, when ammonia emissions were most active (cf. R1 in Fig. 4). Throughout the composting process, CO_2 emissions from R4 were lower than those from the other reactors, which we attributed to the high adsorption capacity of zeolite for CO_2 [24]. After Day 8, CO_2 emissions from all of the reactors fell to a steady low value ($<1.3 \pm 0.3$ g $\text{CO}_2\text{-C}/\text{kg}$ dry-compost/day) in the range of final respiration rates under the mature condition [11,25]. The Solvita[®] maturity index based on CO_2 evolution was calculated as 7-8 in our experiments, indicating that the finished compost was well matured, aged, and cured, with few usage limitations [25,26].

The composting process occurs in two major phases. During the active first phase, readily degradable organic matter is decomposed by thermophilic microorganisms, with active production of CO_2 , NH_3 , and heat. Subsequently, as the decomposition rate decreases,

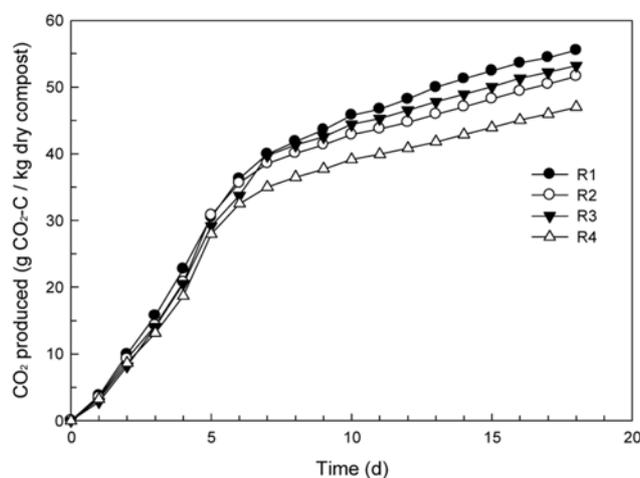


Fig. 5. Cumulative carbon dioxide emission from the composting reactors (R1-R4).

Table 3. Changes in water-extractable total organic carbon (TOC), carbon to nitrogen ratio (C/N, TOC/KCl-extractable ammonium-N), and the ratio of humic acid to TOC (HA/TOC) of the manure mixture

	Reactors	Day 1	Day 2	Day 4	Day 8	Day 10	Day 18
TOC (g/kg dry compost)	R1	30.09	17.53	10.95	11.64	10.76	10.03
	R2	32.03	20.36	17.31	13.87	13.27	10.55
	R3	35.09	26.58	19.84	13.91	15.16	10.50
	R4	37.02	16.83	15.91	14.23	12.60	10.74
C/N	R1	5.60	5.07	4.39	4.99	4.84	4.39
	R2	4.64	2.90	3.00	1.82	1.96	1.59
	R3	5.71	3.25	2.98	1.93	2.33	1.66
	R4	5.89	2.66	2.87	1.85	1.90	1.71
HA/TOC	R1	0.12	0.20	0.25	0.27	0.29	0.58
	R2	0.10	0.18	0.21	0.25	0.30	0.61
	R3	0.07	0.12	0.22	0.22	0.27	0.53
	R4	0.08	0.27	0.34	0.44	0.40	0.71

organic matter begins to mature and be stabilized in the form of humic substances, without a significant change in TOC concentration or CO₂ and NH₃ production [12]. At the end of this second curing phase, the ratio of humic acid to TOC (HA/TOC) can serve as an indicator of compost maturity [10]. In our study, the concentration of water-extractable TOC decreased as composting progressed in all reactors (Table 3), maintaining a steady low level after Day 8, concomitant with a slowdown in the daily CO₂ evolution rate (Fig. 5). Other studies [27,28] also have reported that water-soluble organic carbon content declines during the active degradation of organic matter, such as municipal solid waste and cow manure. The initial HA/TOC ratios of all reactors were in the 0.07-0.12 range and gradually increased to 0.53-0.71 on Day 18 (Table 3), which is similar to values obtained at the end of the curing phase in other studies [29]. The final C/N ratio of R1 on Day 18 was 4.39 (Table 3), which corresponds to the mature state of final compost reported in other studies [17]. However, the final C/N ratios of R2, R3, and R4 were 2.6-2.8 times lower than that of R1, due to the alum and zeolite amendments that trapped ammonia produced during composting. The lower C/N ratio indicates a higher N content of the compost, which improves its fertilizer value. Moreover, ammonium-N sequestered in zeolite is a slow-release N source for plants that gradually releases NH₄⁺ into the soil [23].

CONCLUSIONS

We investigated the effectiveness of four strategies of adding alum and zeolite systematically to composting swine manure to reduce ammonia emissions. The addition of alum efficiently reduced ammonia emissions and resulted in a higher N concentration and lower mass N loss at the end than the unamended control. Ammonia emissions were further reduced when compost was amended with zeolite in addition to alum. About half of the NH₄⁺-N retained in this compost was sequestered at NH₄⁺-exchange sites of zeolite, which improves fertilizer quality, because it acts as a slow-release N source. Multiple applications of alum in small doses and the addition time were critical for the optimum performance of the composting process with less fluctuation in pH and improved stability of thermophilic microorganisms. The amendment-adding strategies of this

study did not significantly affect the microbial activity of the decomposition process, and the final respiration rates and Solvita[®] maturity index indicated that the finished compost was well matured and aged. Thus, the systematic addition of alum and zeolite amendments to swine manure offers a high potential for reducing ammonia emissions with the least N-loss and high degradation and stability of the final compost.

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