# Effect of natural zeolite (clinoptilolite) on ammonia emissions of leftover foodrice hulls composting at the initial stage of the thermophilic process

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#### Abstract

This study investigated ammonia reduction by natural zeolite (clinoptilolite) on leftover food-rice hulls composting in seven enclosures containing composting fermenters. Amounts of 1.6%, 3.1%, 13.7%, 21.8%, 31.5% and 47.2% of clinoptilolite were added to six portions of the feedstock mixture on a dry mass basis, respectively, and 0% of clinoptilolite as a control. The ammonia emission pattern in the thermophilic stage of composting with the presence of clinoptilolite was investigated dependently of the process temperature. The addition of 31.5~47.2% clinoptilolite to leftover food composting was the optimum dose for the reduction of ammonia emission in leftover food composting. It was demonstrated that clinoptilolite acted as an adsorbent in the composting of leftover food in granular form.

Key words: Ammonia, Clinoptilolite, Composting, Leftover food, Thermophilic.

# 1. Introduction

Urban agriculture would not only provide food production and employment, but also help to create an improved microclimate, promote soil conservation, improve nutrient recycling, improve water management and biodiversity, balance O2-CO2, and increase the environmental awareness of city inhabitants (Deelstra and Girardet, 2000). Certainly, urban-agriculture requires an organic source that is not provided in abundance from animal husbandry; therefore, other resources such as food waste can be an important organic source in urban agriculture. The best way is to utilize the organic source from food waste in the composting process. Organic natural resources are used for recycling through biochemical processes into compost, which can be used as a soil improver or organic fertilizer. However, waste processing can contribute to odor pollution, especially in urban areas. If not managed and utilized properly, odor problems cause the closure of some composting facilities and it becomes a bottleneck for urban agriculture to continue their activity and even more to expand their utility program. Therefore, odor management is required to minimize the impact and make composting more environmentally acceptable.

Chemicals that commonly translate to odors in composting processes include ammonia gas (NH<sub>3</sub>) and some volatile organic acids (VOA). Ammonia is one of the main compounds responsible for the generation of offensive odors and atmospheric pollution (Pagans *et al.*, 2006) when composting organic waste such as household waste, meat and many types of food processing waste

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(Beck-Friis et al., 2001; Rynk, 1992) with high nitrogen content. The most important parameter to be considered in the ammonia control of composting management is temperature, as temperature is an excellent indicator of the biological activity in the composting process (Haug, 1993). Ammonia emission starts when the thermophilic period is greater than 45°C in the composting environment. It is widely reported that high temperatures cause the high possibility of ammonia emission (Kirchman and Witter, 1989b). Therefore, it is necessary to take measures against ammonia gas emission under the composting process especially at the beginning of the thermophilic stage.

Zeolite is one material that may reduce ammonia emission. Originally zeolite is used for soil improvement, but it is also used as a moisture control agent in composting. Some papers reported that the release of ammonia gas has been successfully reduced by zeolite in composting piles of municipal solid waste (Turan and Ergun, 2007), sewage sludge (Villasenor et al., 2011; Witter and Lopez-Real, 1988), waste water (Emadi et al., 2001; Cooney et al., 1999) and animal rearing (Witter and Kirchmann, 1989a; Venglovsky et al., 2005). Natural zeolite is used commonly because of its unique adsorption, ion exchange, molecular sieve, and catalytic properties. Clinoptilolite is the most abundant source of natural zeolite and has the chemical formula  $Na_{0.1}K_{8.57}Ba_{0.04}$  $(A_{19.31}-Si_{26.83}O_{72})_{19.56}H2)$  (Galli *et al.*, 1983). Earlier studies have shown that clinoptilolite, and certain other natural zeolites are effective in removing ammonia from wastewater (Emadi et al., 2001; Zorpas et al., 1999). However, there are few studies on the effects of natural zeolites on ammonia reduction during the thermophilic stage of food waste composting.

In the study the influence on ammonia emission was investigated under the composting process especially in the early thermophilic stage at different doses of clinoptilolite using a small-scale composting reactor. In particular, the study focused on the correla-

tion between ammonia emission and temperature at different doses of clinoptilolite. Moreover, this study used food waste such as leftover food from school lunches that was widely available and easy to collect in urban areas. Although its content was relatively safe, other ingredients such as bulking agents and odor management were required.

#### 2. Materials and methods

#### 2.1 Materials

Leftover food waste was collected from sixteen primary schools of Fuchu city in Tokyo, and the lunch was managed by the school lunch center of Fuchu city. The test materials were sent to the laboratory and stored in a cooler container at 5°C. As there was no hard organic debris, the materials were cut into smaller sizes (<10mm) manually using a shovel. Japanese rice hulls were added to achieve a bulk density of less than 496 g L<sup>-1</sup> as recommended by Oshins, 2006 and have the nitrogen content of 0.34. Hydrated lime (Ca(OH)<sub>2</sub>) was used to prepare the acidity of the composting. In this experiment, in order to observe the property of clinoptilolite clearly, only 0.25% of Ca (OH) 2 was added as it was alkaline. About 60 L (60.25 kg) of leftover food from the lunch center was mixed with 120 L (12.5 kg) of rice hulls (volume ratio 1: 2) and added with 0.25% (0.18 kg) of hydrated lime. The initial characteristics of the feedstock mixture were a moisture content of 65.0%, volatile solid of 89.8%, a pH of 5.2, electrical conductivity of 4.6 mS cm<sup>-1</sup> and a C/N ratio of 18.6. A natural zeolite (clinoptilolite) that has the commercial name "Itaya Zeolite-13," was used in this experiment, which was registered by the Japan Fertilizer and Feed Inspection Association. The clinoptilolite was in granular form. It was passed through a sieve to achieve uniformity of less than 4 mm. The properties of "Itaya Zeolite-13" are shown in Table 1 (data provided by the manufacturer). The feedstock mixture was rich in organic matter, nitrogen and carbon. The moisture content reached the general optimum values of less than 70% at the initial state of composting. The results obtained suggested that these materials were available for use in the composting process.

#### 2.2 Experimental setup

The composting processes were conducted in the laboratory in seven closed fermenters of 10-L cylindrical reactors 250 mm in

**Table 1.** Properties of clinoptilolite provided in the experiment.

Component	Concentration
Total Silicic Acid (SiO <sub>2</sub> )	69.7 %
Aluminum Oxide (Al <sub>2</sub> O <sub>3</sub> )	10.0 %
Total Potassium $(K_2O)$	3.06 %
Sodium Oxide (Na <sub>2</sub> O)	2.36 %
Iron Oxide (Fe <sub>2</sub> O <sub>3</sub> )	1.23 %
Total Unslaked Lime (CaO)	0.91 %
Total Magnesia (MgO)	0.17 %
Ignition Loss	3.76 %
Cation Exchange Capacity (CEC)	170 cmol kg <sup>-1</sup>
Moisture Content (MC)	5.92 %
pН	7.5

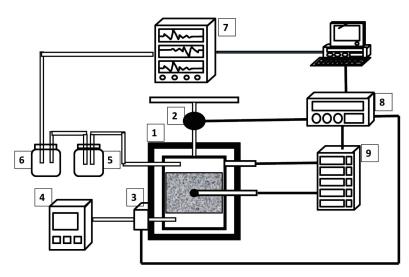
diameter and 270 mm deep. The reactors were made of stainless steel material with a well enclosed structure and a double-chamber system with high insulation to maintain a constant atmospheric condition. The amount of feedstock mixture and clinoptilolite in each experimental run (hereafter Run) is shown in Table 2. After being well mixed by a power mixer (WPM-70A; Minato Electric Industry Co., Ltd, Japan) for 5 minutes, about 4.5 kg of the total amount of feedstock mixture plus clinoptilolite was added to each fermenter while the amount of application was limited by the fermenter size. All of the seven closed fermenters of 10-L cylindrical reactors were set under controlled conditions.

As shown in Fig. 1, one thermocouple was placed horizontally at the center of each reactor at a depth of 135 mm from the top. It was used for monitoring the material temperature and controlling the reactor temperature. The cylinder was insulated with a chamber to minimize the conduction heat loss through the fermenter. The temperature in the chamber was controlled to follow the material temperature within 1°C accuracy. Temperature and gasses were monitored and recorded using data loggers connected to a personal computer. Two bottles of trapped gas were connected to a photoacoustic field-gas monitor (Type 1412; Innova Air Tech Instrument, Denmark) with data loggers to monitor the ammonia gas concentration. One night before the experiment was used for

**Table 2.** Mixture composition of test materials in each run.

	Run Name -	Feedstock m	ixture (FM)	Clinoptilolite			
Run		mass kg	ratio %	mass kg	ratio %	per FM %	
1	CL 0.0%	4.500	100.0	0.000	0.0	0.0	
2	CL 1.6%	4.429	98.4	0.071	1.6	1.6	
3	CL 3.1%	4.360	96.9	0.140	3.1	3.2	
4	CL 13.7%	3.883	86.3	0.167	13.7	15.9	
5	CL 21.8%	3.520	78.2	0.980	21.8	27.8	
6	CL 31.5%	3.084	68.5	1.416	31.5	45.9	
7	CL 47.2%	2.376	52.8	2.124	47.2	89.4	

Name of experimental plot CL#.#% implies clinoptilolite (CL) and its mixing ratio #.# % in dry basis.



- 1. Reactor (ESPEC, LU-112)
- 2. Load cell (KYOWA, LTZ-50KA)
- 3. Flow meter (Panasonic, FM-253-4)
- 4. Compressor (ANEST IWATA, CLD15B-8.5D)
- 5. Water trap (As one, UM sample bottle)
- 6. Gas trap (As one, UM sample bottle)
- 7. Gas monitor (INNOVA1412)
- 8. Data logger (KYOWA, UCAM-20DC)
- 9. Temp. controller

**Fig. 1.** Schematic of test composting reactor.

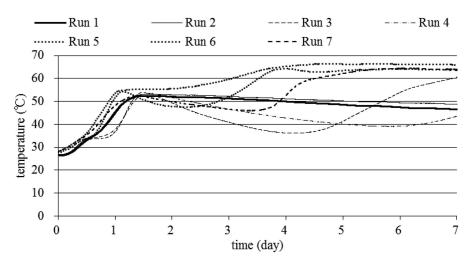


Fig. 2. Temperature records in the reactor during the composting test.

the experimental preparation as the acclimation time less than  $25^{\circ}\mathrm{C}$  required for the activity of microorganisms in the material to adapt to the mesophilic condition. Temperature and ammonia data were monitored and recorded continuously from the first day at 1-minute intervals and at 9-minute intervals during the following 7 days of composting, respectively. The short period of 7 days was considered necessary to observe the ammonia emission pattern that developed starting from the thermophilic stage (>45  $^{\circ}\mathrm{C}$ ). Compressed air of 100 kPa was introduced to the bottom of each reactor and distributed to the material mixture through a perforated plate at a flow rate of 0.4 L min $^{-1}$ .

## 2.3 Data Sampling and Analysis

Moisture content (MC), volatile solid (VS), pH, C/N ratio, ammonium nitrogen (TA-N) and electrical conductivity (EC) were measured to determine the initial conditions of the feedstock mixture. MC was determined by an oven dry method at 105°C for 24 hours and VS was obtained by determining the loss of mass

after ignition on triplicate samples at  $600^{\circ}\text{C}$  for 2 hours. And VS is regarded as the organic matter content. Values for pH and EC were measured by the glass electrode method D-24, Horiba, Ltd, Japan at a ratio of 1:10 (feedstock mixture: water). The C/N ratio was measured using the C/N analyzer, Sumika Chemical Analysis Service. Ltd, Japan. TA-N was measured by indophenol blue absorptiometry using the supernatant of extract by 2 mol  $l^{-1}$  potassium chloride solution. The ammonia emission rate was calculated by formula (1).

$$E = \frac{\sum (C_{\text{amo}} \times f) \times 17.03 \times 60}{D_{\text{org}} \times 22.4}$$
 (1)

*E*: ammonia emission rate [mg kg<sup>-1</sup> VS],  $C_{\rm amo}$ : ammonia gas concentration [ppm = mL L<sup>-1</sup>], f: air flow rate (= 0.4) [L min<sup>-1</sup>],  $D_{\rm org}$ : mass of decomposed organic material [kg], 17.03: molecular weight of ammonia [g mol<sup>-1</sup>], 22.4: unit volume [L mol<sup>-1</sup>]

Correlation analyses between temperature and ammonia emis-

<b>Table 3.</b> Properties of test composting materials before and after the experim
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			Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 7
Clinoptile	olite	%	0	1.6	3.1	13.7	21.8	31.5	47.2
Weight	Start	kg	4.50	4.51	4.50	4.50	4.50	4.50	4.50
	Finish	kg	4.15	4.17	4.11	4.16	3.95	3.79	3.95
	Decreasing Ratio	%	0.08	0.08	0.09	0.08	0.12	0.16	0.12
MC	Start	%,w.b.	65.0	64.1	63.1	56.2	51.6	43.4	34.3
	Finish	%,w.b.	65.0	62.7	62.4	55.2	44.8	38.8	26.3
	Decreasing Ratio*	%	7.6	9.6	9.8	9.3	23.8	24.6	32.7
VS	Start	%TS	89.8	84.6	82.3	60.7	48.0	36.8	22.5
	Finish	%TS	88.5	84.3	80.4	60.0	44.9	33.3	20.2
	Decreasing Ratio*	%	9.0	4.3	9.1	6.6	6.4	17.4	11.8
рН	Start	-	5.2	5.4	5.3	5.3	4.8	4.7	4.9
	Finish	-	4.5	4.4	5.2	4.7	4.8	7.6	6.4
EC	Start	mS cm <sup>-1</sup>	4.5	4.3	4.3	3.1	2.9	2.2	1.4
	Finish	$mS cm^{-1}$	4.9	4.8	4.4	3.6	2.6	1.8	1.2
C/N	Start	-	18.6	25.9	21.7	25.5	24.5	25.2	23.2
ratio	Finish	-	20.8	23.3	19.2	23.7	25.8	18.6	21.3
TA.N	Start	mg kg <sup>-1</sup>	254	216	213	210	170	144	11
	Finish	mg kg <sup>-1</sup>	280	270	354	187	280	431	230

<sup>\*</sup>Decreasing rate of MC and VS were calculated from total amount of them.

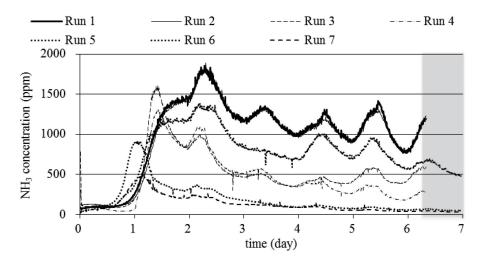


Fig. 3. Records of emitted ammonia gas concentration during the composting test.

sions were performed using Microsoft Excel.

#### 3. Results

Figure 2 shows temperature changes under all test conditions. Significant differences in the temperature profiles were confirmed among the conditions of the thermophilic stages of each treatment. Temperature development in all treatment reactors on Day 1 ranged from 36 to 64°C. However, the temperature in each treatment did not tend to decrease especially for the high clinoptilolite addition groups (21.8%, 31.5% and 47.2%) due to a weak fermented state by a lower moisture content if compared with Run 1~4. The maximum temperatures were 52.3, 53.1, 60.5, 53.5, 66.0, 64.0, 64.0°C for 1.6%, 3.1%, 13.7%, 21.8%, 31.5% and 47.2% of

clinoptilolite content, respectively.

The characteristics of composting materials before and after the 7-day composting test are shown in Table 3. The initial MC of each run varied with the dose of the clinoptilolite and the effect was maintained until the end of the 7 days of composting. After 7 days of composting, Run 1 showed no change in the MC, VS, pH and C:N ratio. The amount of clinoptilolite increased in line with the decrease in the moisture content in Run  $2\sim7$ . The VS loss varied from 9.0, 4.3, 9.1, 6.6, 6.4, 17.4, and 11.8% for Run  $1\sim7$ , respectively; a possible effect identified was that the clinoptilolite improved volatile solid degradation, especially for Run 6 (CL 31.5%) and 7 (CL 47.2%). Values for pH tended to increase with time due to the alkalinity of clinoptilolite that has a pH value of

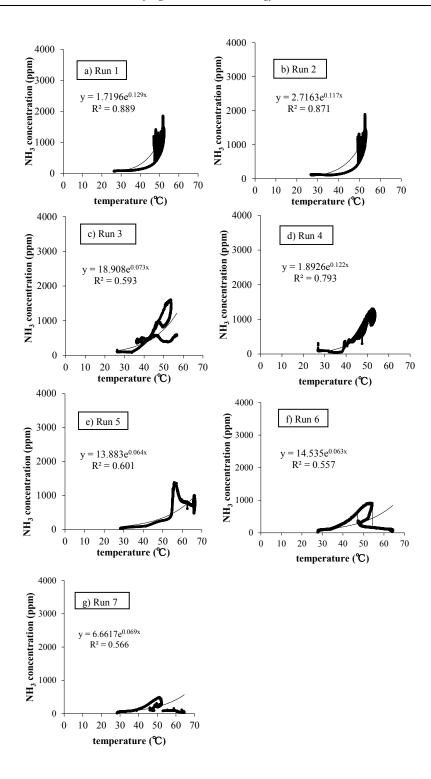


Fig. 4. Effect of reactor temperature on ammonia emission.

7.5. In contrast, the pH decreased in the group of low concentrations of clinoptilolite addition (Run 2~5). Increased doses of clinoptilolite affected the decrease of ammonium nitrogen TA-N at the start in all treatments as shown in Table 3 by the dilution effect.

Figure 3 illustrates the transition of ammonia gas concentration. In Run 1~4, because of a defect of the measuring device, the data from 6.3 days onwards was lost. Ammonia gas emission data was calculated using only 6.3 days of data. A gradual decrease was

observed in ammonia gas concentration by different doses of clinoptilolite. The averages of ammonia gas concentrations were 997, 979, 520, 448, 744, 193, 127 ppm for Runs 1 to 7, respectively. The results clearly showed that ammonia emissions reached higher levels in the non-added and the lowest doses of clinoptilolite (CL 1.6%). The ammonia concentration decreased with the increase in the dose of clinoptilolite, except for Run 5. Run 3 showed the highest temperature range (55~66 °C) during the composting process. Gradual decreases of ammonia were ob-

	Ammonia Gas (ppm)								
$^{\circ}\! C$	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 7		
25-30	86.6	122.8	102.5	112.2	43.0	76.7	46.8		
30-35	95.1	113.7	95.1	59.6	68.6	109.9	67.1		
35-40	115.2	165.3	398.3	231.7	86.4	258.8	98.2		
40-45	182.9	947.4	506.5	367.2	131.5	327.8	199.4		
45-50	393.3	1208.3	748.8	633.4	231.6	452.7	178.1		
50-55	956.0		878.9	1028.8	496.0	141.7	275.3		
55-60			559.9		1128.7	78.0	92.1		
60-65					773.4		46.6		
65-70					726.2				

**Table 4.** The average of ammonia gas in 5 degree temperature interval.

served in CL 31.5% clinoptilolite for Run 6 and CL 47.2% clinoptilolite for Run 7.

Figure 4 shows the effect of temperature on ammonia gas concentration under the respective experimental conditions. Correlation curves of temperature and ammonia gas concentration were calculated from the rising-up to the maximum temperatures. During the period, ammonia gas concentration exponentially increased with temperature. The increasing rate of ammonia emission was moderated by the increase of the clinoptilolite dose. The correlation coefficients between the ammonia emission and temperature are estimated by more than 0.5 for all treatment. Table 4 also shows average of ammonia in 5 degree temperature interval. The beginning of ammonia emission was observed at 45°C, and the peak of ammonia emission appeared at 50~55°C. The low average values of ammonia emission were observed in the groups of high concentrations of clinoptilolite addition (Run 6~7).

Figure 5 illustrates the effect of temperature on the ammonia gas  $(NH_3)$  concentration and clinoptilolite mixing ratio. The ammonia emission rate was calculated by formula (1). In this study, the mass of emitted ammonia per degraded organic matter was calculated at 27.6, 48.3, 11.2, 16.9, 31.1, 3.27, 3.03 g kg<sup>-1</sup> VS for Runs  $1\sim7$ , respectively.

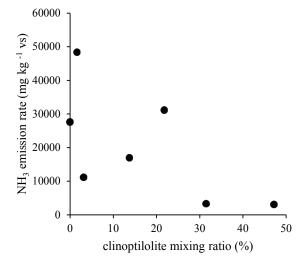


Fig. 5. Effect of clinoptilolite mixing ratio on ammonia emission rate.

#### 4. Discussion

Temporal variation of ammonia emission rate occurred because of the different uses of clinoptilolite doses and the experiment focused on the first rise of thermophilic stage of the full composting process. Ammonia emission starts when thermophilic >45°C and adequate pH coexist in the composting environment. And, it is widely reported that high temperatures inhibit the nitrification process (Grunditz and Dalhammar, 2001), and thereby, the possibility of ammonia emission is high (Kirchman and Witter, 1989a). The initial pH 5.2 of the feedstock mixture still remained at lower levels of acidity of the test materials even when Ca (OH)<sub>2</sub> was added, in order to reach a pH value of 5.5~8.5 suggested as the optimum by Bertoldi et al. (1983). The material temperature in all treatments successfully reached the level at which the fermentation process was activated. Excessive low moisture less than 30% was used to restrain the fermentation; however, the temperature evolution results showed that the successful composting process continued (Fig. 2). Hwang and Neculita (2013) stated that zeolite had the effect of amendment for increasing pH in Food Waste-Based Compost in South Korea. In the case of low pH material composting, the addition of seeding compost (Miyatake et al., 2008) or pH adjusting material (Kofujita et al., 2007) achieved the composting process successfully. The ammonia emission rate decreased with the clinoptilolite dose. In the group lower doses of clinoptilolite (~CL 21.5%) show inconsistent in reduction of ammonia emission rate. The 21.8% clinoptilolite treatment showed a higher level of ammonia emission rate, and also showed less stability of volatile solid degradation; this probably due to unchanged into optimum value of pH and reached  $>65^{\circ}$ C. In the case of Run 6 (CL 31.5%) and 7 (CL 47.2%), ammonia emission rates were less than 20% of Run 1 that had no clinoptilolite application. There was a tendency for the increase of clinoptilolite to accelerate ammonia reduction. Stentiford (1996) suggested that a temperature between 35~40°C maximized the microbial diversity in the composting process. Conversely, Beck-Friis et al. (2001) reported that ammonia emissions started when the thermophilic temperature was higher than 45°C. Pagans et al., (2006) stated that the ammonia emission pattern in the composting of five types of waste (source-separated organic fraction of municipal solid waste (OFMSW); dewatered raw sludge (RS); dewatered anaerobically digested sludge (ADS); animal

by-products (AP); and partially hydrolyzed hair (HH)) investigated strongly depended on the process temperature. As shown in Fig. 4, increasing clinoptilolite had the effect of moderate ammonia emission with an increase temperature. Especially, the addition of 31.5% (Run 6) and 47.2% (Run 7) of clinoptilolite was successful in reducing the ammonia gas emitted from compost as lower levels of the ammonia emission rate were reached. On Table 4, we can see clear the response of interval temperature (5 degree) toward decreasing ammonia gas (NH<sub>3</sub>) emission under the respective experimental conditions.

Decomposition of organic nitrogen in compost produced the TA-N. Furthermore, clinoptilolite adsorbed part of the ammonium nitrogen that was emitted from compost and ammonia emission decreased which is detected by a photoacoustic field-gas monitor. These results reflected the statement of Wang et al. (2007) that clinoptilolite exhibits high selectivity for the ammonium ion (NH<sub>4</sub><sup>+</sup>). Witter and Lopez-Real (1988) reported that the retention of ammonium and ammonia by clinoptilolite was due to the ion exchange and adsorption processes, respectively. Similarly, Villasenor et al., (2011) reported that as the concentration of clinoptilolite increased, the retention of ammonium increased and emissions of ammonia gas decreased. The water vapor from compost that contained ammonia gas through ionization was adsorbed easily by clinoptilolite microspores as water molecules are smaller than carbon dioxide and oxygen molecules. Therefore, it can be stated that clinoptilolite added enough adsorbed ammonium nitrogen that was emitted from food waste composting successfully.

Frequently, zeolite as clinoptilolite was used as a soil conditioner. Although it depends on the soil characteristics, the application rate of zeolite was several tons per 0.1 ha. In contrast, compost was applied to farms at the same levels as zeolite. In this study, almost the same amount of clinoptilolite as the feedstock mixture was mixed in Run 7, which observed the least ammonia emission.

# 5. Summary and Conclusions

The ammonia emission pattern in the thermophilic stage of composting with the presence of clinoptilolite was investigated dependently of the process temperature. Clinoptilolite application successfully supported the thermophilic stage of the composting process, as there was no indication of inhibition of the microbial activity in the hemophilic stage identified from temperature evolution. Clinoptilolite adsorbed excess nitrogen and retained ammonia during the thermophilic stage of composting. The application of CL 31.5~47.2% clinoptilolite to leftover food composting was the best doses for reducing ammonia emission. The results of this study demonstrated that clinoptilolite acted as an adsorbent and had great potential for removing ammonia from the composting of leftover food especially in granular form.

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