



Influence of zeolite and lime as additives on greenhouse gas emissions and maturity evolution during sewage sludge composting



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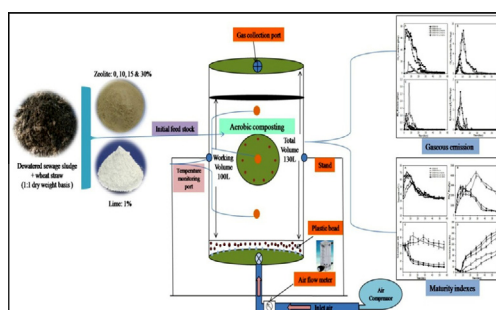
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HIGHLIGHTS

- Zeolite + 1% lime addition significantly increases CO₂ and decreases NH₃ emission.
- N loss was reduced to 50% through zeolite + 1% lime composting process.
- Zeolite + 1% lime addition can alleviate the low pH and reduce the NH₃ loss.
- Methane emission is very low in 30% zeolite with 1% lime amended treatment.
- Addition of 30% zeolite with 1% lime gave the least N₂O emission.

GRAPHICAL ABSTRACT



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ABSTRACT

This study aimed to evaluate the role of different amount of zeolite with low dosage of lime amendment on the greenhouse gas (GHGs) emission and maturity during the dewatered fresh sewage sludge (DFSS) composting. The evolution of CO₂, CH₄, NH₃ and N₂O and maturity indexes were monitored in five composting mixtures prepared from DFSS mixed with wheat straw, while 10%, 15% and 30% zeolite + 1% lime were supplemented (dry weight basis of DFSS) into the composting mass and compared with treatment only 1% lime amended and control without any amendment. The results showed that addition of higher dosage of zeolite + 1% lime drastically reduce the GHGs emissions and NH₃ loss. Comparison of GHGs emissions and compost quality showed that zeolite amended treatments were superior than control and 1% lime amended treatments. Therefore, DFSS composting with 30% zeolite + 1% lime as consortium of additives were found to emit very less amount of GHGs and gave the highest maturity than other treatments.

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1. Introduction

In China, approximately 30 million tons of sewage sludge (SS) are generated per year from different wastewater treatment plants (WWTPs); and its production rapidly increasing with the

population and industrialization, which grew by more than 13% per year from 2007 to 2015 (Cai et al., 2016). Meanwhile society is urging for better environmental conditions, thus increasing the demand for upgrading and expansion of wastewater treatment plants in China. As a consequence, huge quantities of SS are produced and its management is an expansive as well as environmentally sensitive problem, because this waste contains high load of toxic heavy metals (HMs), organic micropollutants and pathogenic

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microbes, which have led to strict legislation for SS applications (Villasenor et al., 2011; Maulini-Duran et al., 2013), and therefore urgent need for sustainable ecofriendly management of SS or biosolids.

However, numerous chemical and biological methods for SS management have been applied since long time, including landfilling, incineration, anaerobic digestion and aerobic composting. In the above methods, composting is one of best alternative method for SS management, which allows their reuse as soil conditioners or organic fertilizers at agricultural land for food and feed crops without toxic effects (Villasenor et al., 2011; Li et al., 2013; Awasthi et al., 2016). And the expenditure of the application of this technology for SS management is relatively modest as compare to other methods (Szanto et al., 2007; Tsutsui et al., 2013; Shao et al., 2014). An unfortunate occurrence during SS composting is the loss of nitrogen through ammonia emission and bioavailability of HMs (Sanchez-Monedero et al., 2010; Villasenor et al., 2011; Yang et al., 2013), which reduces the agricultural value of the end product. Consequently there are some other environmental impacts associated with composting, such as GHGs or odors emissions are one of the most important problems (Beck-Friis et al., 2000; Manios et al., 2007; Luo et al., 2013). The report of the International Panel on Climate Change (IPCC) stated that continues increasing emission of GHGs are mainly methane (CH₄) and nitrous oxide (N₂O), which have a high warming potential and responsible for climate change, and is 30 and 210 times higher than CO₂ emission, respectively (IPCC, 2007).

Many researchers have reported the global warming potential of GHGs emissions from different waste management strategies and also highlighted the adverse effects during the composting process (Lou and Nair, 2009; Sanchez-Monedero et al., 2010; Maulini-Duran et al., 2014). As we know, CO₂ is the main gas generated during early stage of composting but its global warming influence is accounted as neutral for the evaluation of the impact of waste management practices since these CO₂ emissions are of biogenic origin (IPCC, 2007). Beside this, microbial transformations and initial feed stock characteristics of the composting mass mainly responsible in formation of GHGs during composting and similarly to those taking place in other process such as wastewater treatments plants, soil and landfilling (Yang et al., 2013; Shao et al., 2014). However, a great deal of research has been performed last few years and numerous methods have been also suggested to reduce the GHGs emission by increasing aeration (Beck-Friis et al., 2000; Li et al., 2013), inoculation of microbial enriched waste or pure bacterial consortium (Awasthi et al., 2015) or the addition of alkaline materials (Wong et al., 2009; Luo et al., 2013). Consequently, lots of previous studies demonstrated the effect of additives such as coal fly ash, wood biochar, kaoline, bentonite and lime as suitable amendments for the SS composting (Fang and Wong, 1999; Wong et al., 2009; Li et al., 2012; Wang et al., 2016) and/or alkaline stabilization of SS and pretreatment of bio-mass (organic waste) (Zhang et al., 2011).

But limited research has been focused on the effects of mixture of mineral additives on GHGs emissions reduction during DFSS composting. Therefore, addition of zeolite combined with lime into DFSS may provide numerous benefits during the composting, because porous micro-structure of zeolite leads to reduce the emission of mixture of gases and act as biofilter (Villasenor et al., 2011; Maulini-Duran et al., 2014), while addition of lime alleviate the initial acidic pH during the thermophilic stage of composting (Fang and Wong, 1999; Wong et al., 2009). Villasenor et al. (2011) reported that zeolite not only work as additive, but also used as bulking agents to increase the sludge porosity. Previous studies have been revealed that the addition of phosphogypsum and dicyandiamide as additives significantly reduce the GHGs emission and increase the microbial activity (Luo et al., 2013); and increase

the macronutrient concentration in mature compost through ion exchange (Li et al., 2013). Last few years numbers of scientific papers are published on composting of different types of organic waste using either zeolite or lime as amendments, but they all were mainly focused on unidirectional prospects either nitrogen conservation or HMs immobilization (Wong et al., 2009; Villasenor et al., 2011; Chan et al., 2016), while very limited studies have been found regarding the comparison of different concentration of zeolite combined with lime of low loading to reduce the GHGs emission and assess the maturity parameters.

Therefore, the objective of this research work was to study: (1) the influence of mixture of additives amendments on GHGs emissions; (2) the influence of mixture of additives amendments on microbial population and maturity properties during DFSS composting. Hence the present study was designed to provide the baseline on role of additives consortium amendment on evolution of GHGs emission and maturity index for DFSS composting, which is not clear in previous available composting literature.

2. Materials and methods

2.1. Composting feed stock collection and processing

The DFSS used in this study was collected from a local municipal wastewater treatment plant (Yangling, Shaanxi Province, China) and wheat straw (WS) was obtained from local market (Shaanxi Province, China), which is used as bulking agent to increase the porosity of the composting mass and adjust the C/N ratio. While before using WS was cut into 1 cm length using a mechanical chopper to obtain uniform particle size. The natural zeolite was purchased from Zhejiang Shenshi Mining Industry Group Co., Ltd., China, which has a diameter of 3.5–4.0 (Ai) and cation exchange capacity of 120–160 meq/100 g; and lime from local market. The selected physicochemical properties of each raw material are listed in Table 1.

2.2. Experimental design and methods

Composting was done in a Polyvinyl chloride (PVC) composter with a total volume of 130 L under controlled room temperature and aeration conditions; and its detailed description already reported in our previous study (Li et al., 2012). The outer wall of reactor was 4 cm thick and coated with thermal insulating material. Fresh air was supplied from the bottom of the composter through a perforation plate and gas-flow rate maintained at 0.35 L h⁻¹ kg⁻¹ (dry weight basis) during beginning of composting process and at early maturation phase the air flow was slightly increased when the temperature was below 55 °C according to the previous work in our group (Li et al., 2012; Wang et al., 2016). Initially 35 kg of fresh DFSS was mixed with 7.8 kg WS with the ratio of 1:1 (dry weight basis) to adjust the C/N ratio to ~25 as

Table 1
Characteristics of composting materials used in the present experiments.

Parameters	DFSS	WS	Zeolite
Moisture content (%)	81.53 ± 2.10	14.20 ± 0.32	0.05 ± 0.01
pH (solid:water = 1:5)	8.17 ± 0.14	5.03 ± 0.13	8.14 ± 0.24
EC (ms cm ⁻¹) (solid:water = 1:5)	3.05 ± 0.03	1.74 ± 0.05	1.14 ± 0.10
Total organic matter (% dw)	76.14 ± 2.01	96.03 ± 3.15	ND
Total organic carbon (% dw)	38.25 ± 1.04	56.61 ± 1.43	ND
Total Kjeldahl nitrogen (% dw)	3.12 ± 0.20	0.82 ± 0.04	ND
C:N ratio (dw)	12.25 ± 1.02	69.03 ± 1.23	ND

ND – not detected, EC – electrical conductivity, DFSS – dewatered fresh sewage sludge and WS – wheat straw, dw – dry weight basis. Results are the mean of three replicates ± standard deviation.

recommended by Wong et al. (2009) for efficient composting. Then zeolite was mixed with 1% lime at 0%, 10%, 15% and 30% of DFSS dry weight basis and then amended into the initial composting feed stock, while 1% lime selected on the basis of Fang and Wong (1999) recommended for SS composting. In addition, 1 kg of plastic spheres was mixed with initial feed stock to adjust the initial bulk density to ~ 0.5 kg/L according to our previous work experience (Awasthi et al., 2015). The initial moisture content was also adjusted to 55% through addition of water on dry weight basis. About 100 L of composting mixture was filled in each reactor and composted for 56 days, while treatment with 1% lime amended and without additives as control were also carried out for comparison purpose. Periodically composting mass was turned and mixed properly on sampling days 0, 3, 7, 10, 14, 21, 28, 35, 42, 49 and 56; meanwhile moisture content was maintained to 55% and kept constant throughout the experiment. The composting biomass temperature was monitored every day four times (8 h) by the using of thermocouple probe inserted into the center of the composting materials and averaged temperature was reported, respectively. Despite this the room temperature was also recorded. The thermocouple, air inlet and outlet pipes were disconnected from the reactor when the composting mass was turned and mixed. At the time of GHGs collection, the introducing fresh air was stopped because it may dilute the concentration of air; beside this the exhausted ammonia gas was trapped by absorbing ammonia into boric acid and analyzed as per the standard methods (Hao et al., 2001; Komilis and Ham, 2006).

2.3. Gas sampling and analysis

GHGs samples were collected in three times a week (during the thermophilic phase) and twice a week (during the maturation phase) by the using of 1-LTedlar[®] PLV gas sampling bag w/Thermo-green[®] LB-2 Septa (sigma-aldrich) according to Maulini-Duran et al. (2014). From each reactor one sample was withdrawn and then air samples were transferred into disposable syringes within the space at 30 ml fitted with rubber stoppers, while GHGs concentration (CO_2 , CH_4 and N_2O) were analyzed within 12 h using gas chromatography (Agilent Technologies 6890N Network GC system, China) as described by Colon et al. (2012). All samples were analyzed in triplicate for each gas and the emission fluxes were used to calculate the experimental data by second order polynomial equation (gas concentration vs time) (Hao et al., 2001), and the results were expressed as grams of C or N/kg/time (d). Ammonia emission from the composter was measured from the exhaust gas by adsorbing NH_3 into boric acid and concentration were determined by titrated against 1 mol L^{-1} hydrochloric acid (Komilis and Ham, 2006).

2.4. Compost sampling and physicochemical analysis

A homogenous 250 g compost samples were collected from each treatment after proper mixing of composting mass as mentioned above. The collected compost samples moisture content was determined based on the initial and final weight after drying in an oven at 105°C for about 24 h, until a constant weight was achieved. A 1:5 aqueous extract (dry weight basis) of fresh compost was used for the analysis of pH, electrical conductivity (EC), extractable ammonium ($\text{NH}_4^+\text{-N}$) and seed germination. The pH and EC were measured using pH meter with a glass electrode (INESA PHSJ-3F, China) and conductivity electrode (INESA DDS-307, China). Extractable ammonium ($\text{NH}_4^+\text{-N}$), total Kjeldahl nitrogen (TKN), total organic carbon (TOC) and total organic matter (TOM) were determined following the standard methods for testing compost materials (TMECC, 2002). TOM was determined by incineration of air dried samples at 550°C for 24 h in a muffle fur-

nace, while seed germination test was performed with *Lepidium sativum* L. seeds as described by Zucconi et al. (1981).

2.5. Statistical analysis

All the chemical analyses were performed in triplicate samples to determine the difference between physic-chemical properties of between each treatment. For total bacterial population, a homogenous composite sample was prepared and the isolation was carried out in triplicate agar plates. While data were subjected on the basis of one-way analysis of variance (ANOVA) and multiple comparison tests were also performed to compare the least significance difference (LSD) at $p = 0.05$ values using SPSS 13.0 software (SPSS for Windows, Version 13.0, USA).

3. Results and discussion

3.1. Gaseous emissions during composting

The evolutions of CO_2 , CH_4 , NH_3 and N_2O emissions during DFSS composting are shown in Figs. 1a, c and 2a, c. Because the CO_2 emission is provide the information about the presence of carbon source and rate of composting; and from NH_3 emission was asses the loss of nitrogen, while CH_4 and N_2O emissions are directly related to the presence of anaerobic condition during composting. In Figs. 1b, d and 2b, d the cumulative CO_2 , CH_4 , NH_3 and N_2O emission are also summarized for different treatments in terms of each GHGs per gram of composting mass.

3.1.1. Changes of carbon dioxide and methane emissions profile

The CO_2 and CH_4 are two main gases generated by microbial degradation of TOM during DFSS composting, but the higher CO_2 emission is indication of rapid degradation and active microbial activity, while increasing concentration of CH_4 showed the improper aeration and porosity between feedstock particles. Microbial activities are mainly responsible for the CO_2 production and its maximum emission was observed in zeolite with lime amended treatments and comparatively very low CO_2 was emit from lime amended and control treatments (Fig. 1a). The higher CO_2 generation was observed in all zeolite with 1% lime amended treatments from the beginning of the process and reached the maximum CO_2 production in DFSS + 1% lime + 10% zeolite on day 3; and DFSS + 1% lime + 15% zeolite and DFSS + 1% lime + 30% zeolite on day 4. Consequently, high temperatures and microbial activity also support this evidence (Figs. 3a, S1). Thereafter CO_2 emissions gradually decreased in all zeolite amended treatments by the end of the long thermophilic phase (3 weeks); and after day 22 down to levels lower than $10 \text{ g C m}^{-2} \text{ d}^{-1}$, which is reflecting the stability of the mature compost. While when compare with low microbial activity and late start of thermophilic phase (Fig. 3a), the CO_2 emission was significantly very low in the control and 1% lime amended treatment (Fig. 1a, b). In all zeolite amended treatments irrespective of 1% lime concentration, the evolution of the CO_2 emissions was almost similar throughout the composting process, which reflected the high stability degree was achieved by the end product after 56 days of composting process. Although, CO_2 emission profiles in all zeolite + 1% lime amended treatments are quite similar with temperature profiles and total aerobic bacterial population (Fig. S1a), but CO_2 emissions decrease earlier than temperature, might be due the rapid degradation of organic matter and insulation of reactors that prevent the rapid heat loss as previous reported by (Wong et al., 2009; Sanchez-Monedero et al., 2010; Chan et al., 2016).

Therefore, no significant difference was observed in the cumulative CO_2 emission profile among the different concentration of

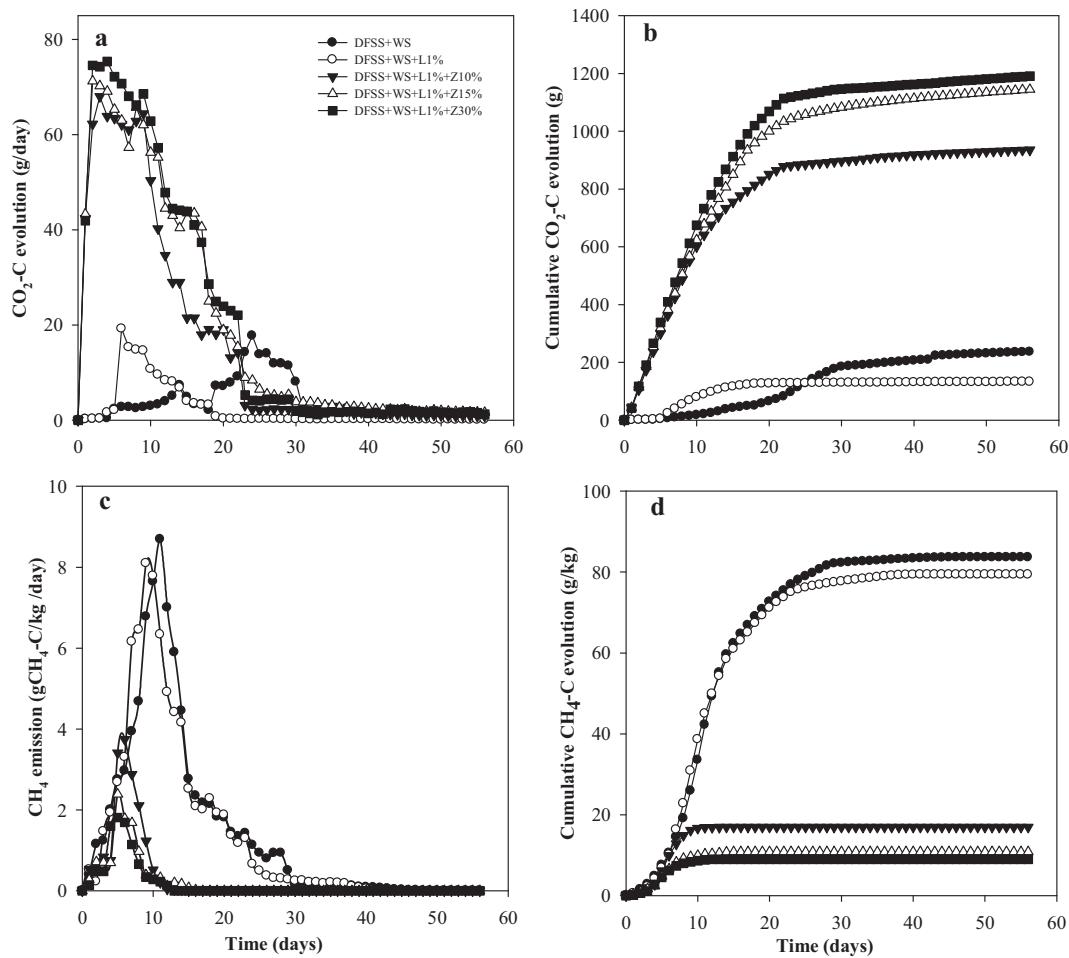


Fig. 1. Carbon dioxide (a), cumulative carbon dioxide (b), methane (c) and cumulative methane (d) emission from different treatments during composting of dewatered fresh sewage sludge. DFSS + WS: dewatered fresh sewage sludge + wheat straw (Control); DFSS + WS + L1%: dewatered fresh sewage sludge + wheat straw + lime 1%; DFSS + WS + L1% + Z10%: dewatered fresh sewage sludge + wheat straw + lime 1% + zeolite 10%; DFSS + WS + L1% + Z15%: dewatered fresh sewage sludge + wheat straw + lime 1% + zeolite 15%; and DFSS + WS + L1% + Z30%: dewatered fresh sewage sludge + wheat straw + lime 1% + zeolite 30%. Results are the mean of three replicates and error bars indicates standard deviation.

zeolite addition combined with 1% lime amended treatments (Fig. 1a), similarly total aerobic bacterial population, specific substrate degrading aerobic bacterial population and rate of degradation were also supporting this evidence (Fig. S1). Despite this, cumulative CO_2 evolution in the control and 1% lime amended treatment were significantly also lower ($P < 0.05$) than that of others treatments, because the acidic condition did not favor the microbial activities as evidenced of short thermophilic phase and total aerobic bacterial population (Fig. S1). The results implies that the addition of 1% lime combined with different dosages of zeolite efficiently promoted the degradation of organic carbon, especially when 1% lime combined with 30% of zeolite. In contrast, only 1% lime addition is not enough for buffering the initial acidic pH and stimulating the microbial activity to immediate decomposition. Several earlier researchers have also reported similar result for different types of organic waste composting such as for food waste Wong et al. (2009) and Chan et al. (2016), SS Li et al. (2013) and olive mill wastes Sanchez-Monedero et al. (2010). Hence, the higher amounts of zeolite addition were significantly buffer the pH and provide the proper aeration for aerobic microbes' proliferation, which is most probable cause to improve the composting efficiency.

Methane emission is indicator of deoxidization of produced CO_2 and acetic acid during composting by methanogenic bacteria under the anaerobic conditions (Yang et al., 2013; Santos et al., 2016). As

shown in Fig. 1c, the statistical analysis of their variance showed that there is no significant difference ($P < 0.05$) on CH_4 emissions among different amount of zeolite + 1% lime amended treatments. While, the major difference on CH_4 emissions was observed between 1% lime and control treatments as compare with zeolite mixed with lime amended treatments. In control without lime and zeolite addition, CH_4 production was the maximum due to the slow degradation of TOM, which may related to the low microbial activity and pH (Figs. S1 and 3b) as observed by Maulini-Duran et al. (2014) for municipal solid waste and source selected organic fraction of municipal solid wastes (OFMSW). In addition, the late thermophilic stage start, low indigenous aerobic bacterial population and pH were also responsible for higher CH_4 emission in control and 1% lime amended treatments (Fig. 1c-d). However, previous studies reported that long duration of anaerobic condition, high moisture, initial composition of the mixtures (C/N ratio) and insufficient porosity between composting mass or improper aeration also responsible for excessive CH_4 generation during the composting (Amlinger et al., 2008; Luo et al., 2013; Santos et al., 2016). Beside this, moisture content was optimally ranged around 55% during composting (data not shown) and initial C/N ratio was also adjusted to ~25 (Table 1) in composting feed stock. In addition, the air flow rate was always constant from the first day of composting process. Meanwhile the rapid biodegradable TOM could be also lead to oxygen depletion, creating anaerobic zones

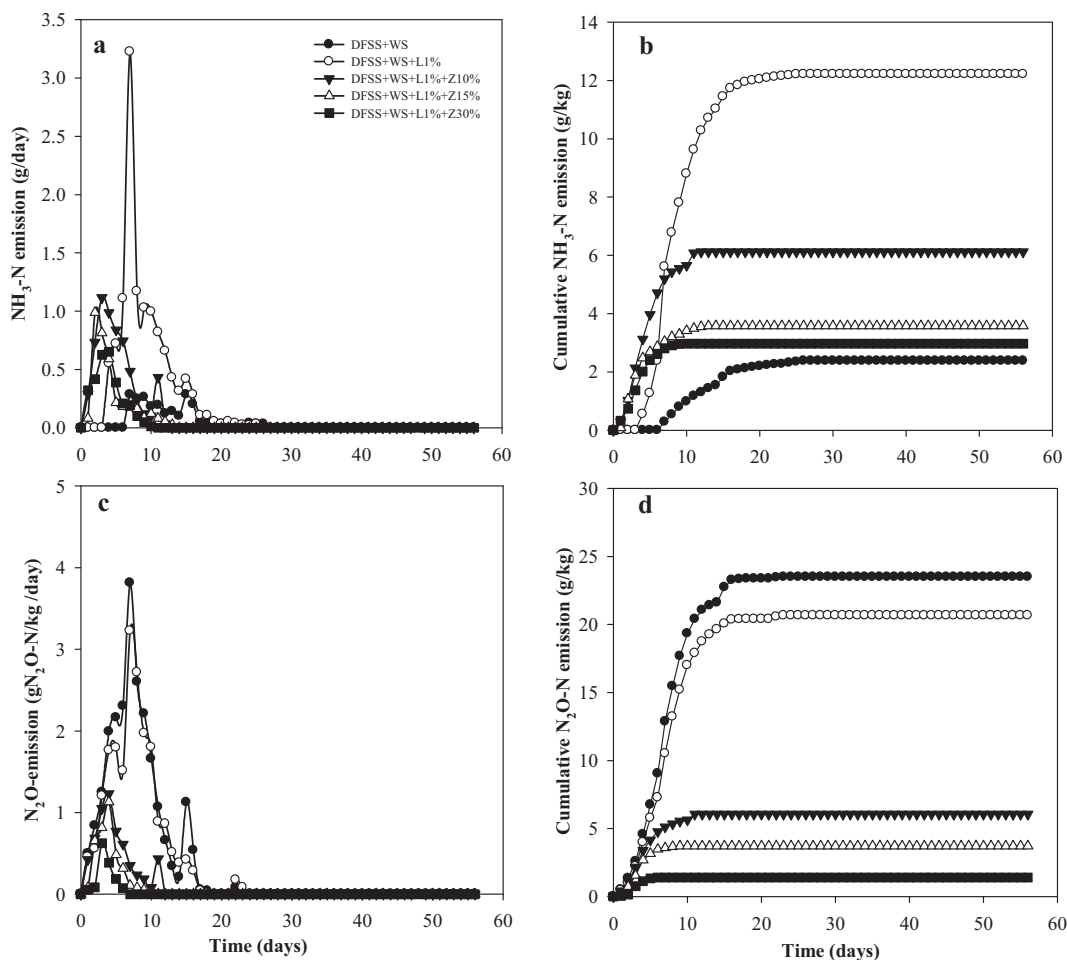


Fig. 2. Ammonia (a), cumulative ammonia (b), nitrous oxide (c) and cumulative nitrous oxide (d) emission from different treatments during composting of dewatered fresh sewage sludge. DFSS + WS: dewatered fresh sewage sludge + wheat straw (Control); DFSS + WS + L1%: dewatered fresh sewage sludge + wheat straw + lime 1%; DFSS + WS + L1% + Z10%: dewatered fresh sewage sludge + wheat straw + lime 1% + zeolite 10%; DFSS + WS + L1% + Z15%: dewatered fresh sewage sludge + wheat straw + lime 1% + zeolite 15%; and DFSS + WS + L1% + Z30%: dewatered fresh sewage sludge + wheat straw + lime 1% + zeolite 30%. Results are the mean of three replicates and error bars indicates standard deviation.

in the composting matrix. Maximum CH_4 emission was observed in control on day 7 to day 13 and in 1% lime amended treatment on day 6 to 14 (Fig. 1c-d), when the microbial activity and pH both are also lower. He et al. (2000) and Li et al. (2013) reported that composting wastes with high moisture and small particle size such as SS could lead to the formation of anaerobic zones due to compaction and porosity as well as low aerobic microbial population effects. Although material compaction could be detected at the end of the process as the composters were neither opened or composting material was not turned during the process composting. But, it cannot be easily observed whether the compaction occurred at the beginning or maturation stages of the composting process, because there is a marginal difference between initial and final porosity of composting matrix.

In contrast, our results indicate that excess water content was easily removed from the composting mass and provide efficient porosity for microbial growth, when higher dosage of zeolite was added. The addition of 30% zeolite + 1% lime (Fig. 1c-d) raised the early pH, promoted total aerobic bacterial population and increased the rate of composting. Some earlier authors reported that the addition of excess dosage of zeolite caused the SS and food waste to retain water, increased the porosity between composting matrix and optimum moisture content were observed in the final compost (Villasenor et al., 2011; Chan et al., 2016). However, water

retention capacity of zeolite was reduced the excess water in composting mass, but its bulking effect may have extra-ability to decrease the CH_4 emission during the thermophilic stage of DFSS composting in present investigation. Because gases and water vapor could be removed more easily from the waste into the atmosphere. Furthermore, this extra-ability of zeolite would only be observed at high temperatures during the DFSS composting, meanwhile, the water retention capacity of zeolite can help to maintain the desired moisture level during the maturation stage at mesophilic temperatures. Therefore our study provided clear landmark on correlation between zeolite concentration and CH_4 emission, while previous available literature only give the information in respect of importance of zeolite for nitrogen conservation and HMs immobilization, respectively.

3.1.2. Changes of ammonia and nitrous oxide emissions profile

Ammonia evolution profile during DFSS composting is shown in Fig. 2a-b. As we know most of the ammonia emission occurred during the early thermophilic phase with the decomposition of organic nitrogen-containing materials. Numerous researcher reported that instantaneous losses of ammonia during the composting causes odor problems and reduce the nutrient quality of end product, because ammonia is not water soluble under high temperature (Tsutsui et al., 2013; Awasthi et al., 2016); and about

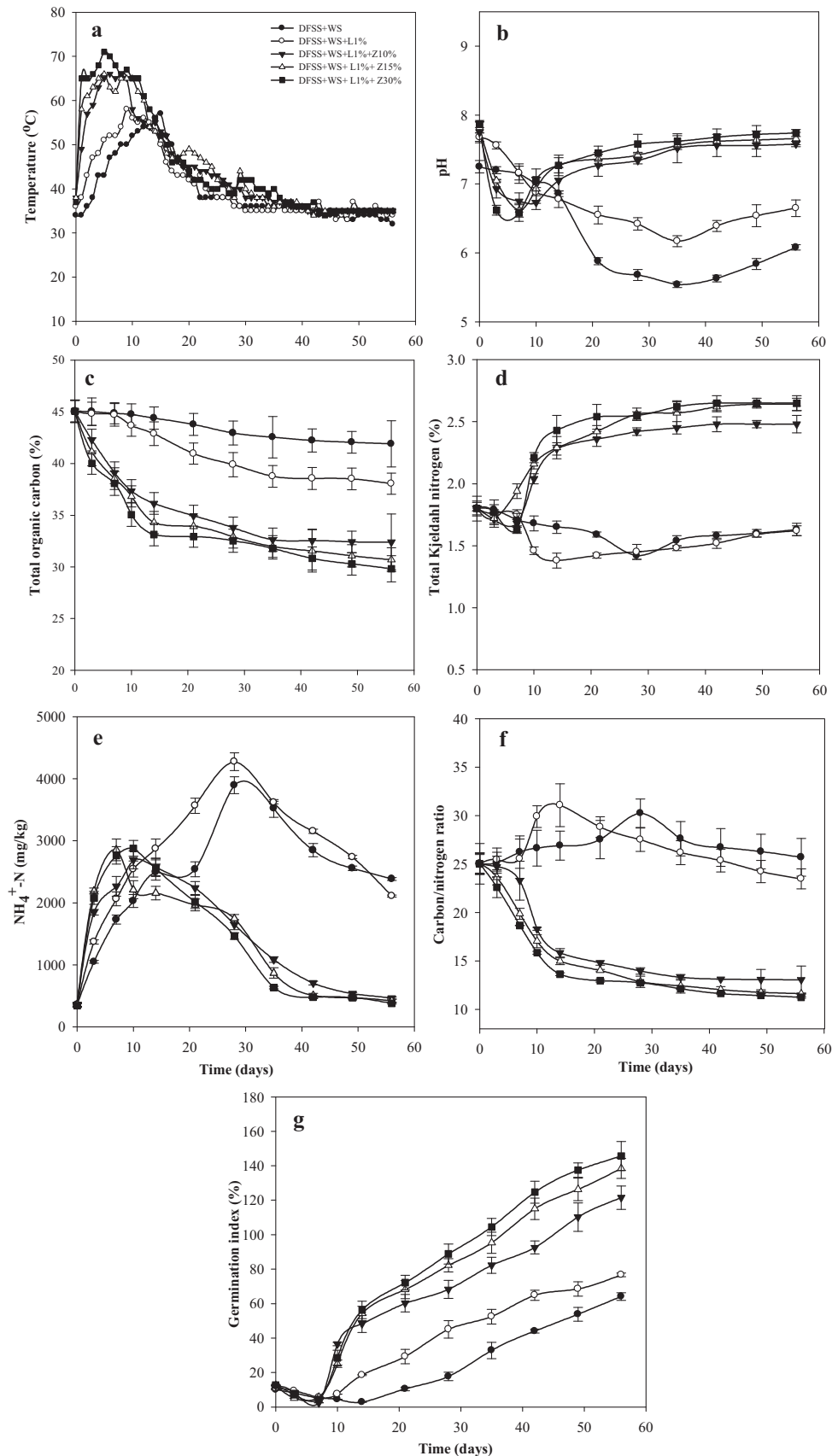


Fig. 3. Changes in maturity index; temperature (a), pH (b), total organic carbon (c) and total Kjeldahl nitrogen (d), extractable ammonia (e), carbon/nitrogen ratio (f) and seed germination index (g) in different treatments during composting of dewatered fresh sewage sludge. DFSS + WS: dewatered fresh sewage sludge + wheat straw (Control); DFSS + WS + L1%: dewatered fresh sewage sludge + wheat straw + lime 1%; DFSS + WS + L1% + Z10%: dewatered fresh sewage sludge + wheat straw + lime 1% + zeolite 10%; DFSS + WS + L1% + Z15%: dewatered fresh sewage sludge + wheat straw + lime 1% + zeolite 15%; and DFSS + WS + L1% + Z30%: dewatered fresh sewage sludge + wheat straw + lime 1% + zeolite 30%. Results are the mean of three replicates and error bars indicates standard deviation.

65% nitrogen could be volatilized in the form of ammonia (Komilis and Ham, 2006; Wong et al., 2009; Li et al., 2013; Chan et al., 2016). From the beginning of composting, very low concentration of ammonia emitted from control, because of the low degradation and pH that stimulated the volatilization of ammonium into NH_3 , while it would not be considered as N conservation (Fig. 2a). In contrast, 1% lime amended treatment was showed a maximum peak on day 7, due to the higher temperature and pH on these days promotes the ammonia emission, thus it can be attributed to the loss of nitrogen.

While addition of different concentration of zeolite with 1% lime was reduced more than 50.43% nitrogen loss, when compared with 1% lime and control treatments. Whereas among the all zeolite amended treatments, 30% zeolite with 1% lime addition reduced the maximum nitrogen loss (50.43%) followed by 10% (41.12%) and 15% (43.17%) zeolite amended treatments. However, the reduction capacity of NH_3 loss is very marginal among the zeolite with 1% lime amended treatments, and the difference was not very significant. It is interesting the cumulative NH_3 loss in 1% lime amended treatment was maximum when compared with the zeolite amended treatments, while cumulative ammonia emission significantly low in control as shown in Fig. 2b. Some researcher reported that high bulk density of the initial feed stock is likely to be responsible for the low NH_3 emissions, as reported by Li et al. (2013) showing that compacting or high moisture content and C/N ratio of initial composting matrix could reduce the free air space and thus decreased the NH_3 emission by 30–70% (Wong et al., 2009; Li et al., 2013; Chan et al., 2016).

In addition, our results clearly indicate that as the zeolite amount increased with 1% lime, consequently the NH_3 emission was reduce, might be excess amount of zeolite addition could be behave as bulking agent and enhance the activity of ammonia-assimilating microorganisms and provide a nutrient source as well as proper aeration. However, 25.02% and 23.3% reduction in the ammonia loss during swine manure, SS and food waste composting with struvite salts, zeolite and different bulking agents amended treatments reported by the several earlier researchers (Fukumoto et al., 2003; Li et al., 2013). Considering the total ammonia loss during the SS composting about 21–23% (Li et al., 2013), ammonia emission reduction value very high achieved in this experiment, while very limited information is available about the influence of addition higher dosage of zeolite combined with 1% lime. The NH_3 emission profile of our study is consistent with previous findings employing zeolite addition (Manios et al., 2007; Szanto et al., 2007; Villasenor et al., 2011; Li et al., 2013). When compare with the other studies (Luo et al., 2013; Chan et al., 2016), very low amount TKN was loss in the form of NH_3 in zeolite with 1% lime amended treatments, respectively (Fig. 2a–b). Thus addition of higher amount of zeolite combined with lime to DFSS composting had great impact on NH_3 emissions. Because the NH_3 emission was decrease as the concentration of zeolite increase, due to retention of ammonium and ammonia or ion exchange and adsorption processes. Similarly, Tsutsui et al. (2013) observed that ammonia losses were reduced by increasing the concentration of clinoptilolite, while they used 5% and 10% of clinoptilolite and obtained only 11–9% reduction in TKN loss, respectively.

The N_2O emissions profiles of all five treatments are listed in Fig. 2c and d. In our study, rapidly N_2O emissions began from day first in all treatments; while control and 1% lime amended treatments have relatively very high N_2O emissions immediately after the composting started (Fig. 2c–d), might be due to incomplete nitrification/denitrification processes that normally convert NH_4^+ into N_2 and a non-polluting gas. There are some controversies in the literature about N_2O emissions profile, Maulini-Duran et al. (2013) reported that most of N_2O might have been synthesized from denitrification, when the materials were stored and dis-

charged at the start of composting because of the anaerobic condition/or a nitrate or nitrite accumulation. Similarly, El Kader et al. (2007) also reported that a high concentration of N_2O at the start of composting may actually have been produced before the composting process started. Most of the N_2O in all five four treatments was emitted during the thermophilic phase. This emission pattern is similar to those previously reported by El Kader et al. (2007) for farm manure composting, by Yang et al. (2013) for kitchen waste composting and by Maulini-Duran et al. (2014) for OFMSW composting. However, our result completely opposite to the observations reported by Fukumoto et al. (2003) and Li et al. (2013) that the activity of nitrifying bacteria would be inhibited at high temperature ($>40^\circ\text{C}$), meanwhile N_2O emission is negligible during the thermophilic phase, which normally occur during the first week of the composting process. Beside this, Szanto et al. (2007) reported that methanogenic and methanotrophic microbes were capable of ammonium oxidation under thermophilic conditions, while Sommer and Moller (2000) reported that nitrification could also occur on the surface of the composting matrix, where the temperature and aeration were suitable for nitrifying bacteria such as *Nitrosomonas* and *Nitrobacter*. After early thermophilic phase, as the temperature gradually dropped and the porosity increased, this was appropriate for nitrification, but our results opposite to previous study (Fukumoto et al., 2003; Szanto et al., 2007).

In our study nitrification decreased because of the reduction of NH_4^+ emissions, which is a produced ammonification process during TOM degradation at early stages of the composting. Meanwhile, rate of denitrification process also decreased with the reduction in nitrification, thus reducing the amount of N_2O produced from both nitrification and denitrification (Sanchez-Monedero et al., 2010). The small peaks were observed from day 4 to 11 in the zeolite combined with 1% lime amended treatments, might be due to rapid degradation of TOM and ammonification process stimulate the NH_4^+ production and as results nitrification processed increase, consequently the N_2O emission. In spite of this, it was observed that higher N_2O emission reported in other studies (Beck-Friis et al., 2000; Ermolaev et al., 2014; Santos et al., 2016), while our study indicated that excess amount of zeolite addition important role play for rapid degradation of OM because it will provide proper aeration and reduce the loss of nitrogen as well as N_2O emission (Fig. 2c–d). Consequently, several composting studies reported that poor aeration and maximum ammonia emission is responsible for higher N_2O production (Hao et al., 2001; Szanto et al., 2007; Tsutsui et al., 2013). Furthermore, Sanchez-Monedero et al. (2010) and Ermolaev et al. (2014) reported that different mechanisms and multiple factors throughout the composting process responsible for N_2O production from in-vessel composting; such as nitrification, nitrifier denitrification and denitrification.

But all these processes are depending on the ammonia transformation into nitrate or nitrite or presence of mineral nitrogen and available carbon sources and oxygen concentration in the composting mass. Apart from the availability of mineral nutrients and ammonia emission, the aeration between the composting feedstock has an important role in N_2O emission (Amlinger et al., 2008), because of the heterogeneity of the composting materials, while aerobic and anaerobic zones also coexist to create different gradients of aeration through the composting process (Hao et al., 2001). In addition, Beck-Friis et al. (2000) reported that nitrifying bacteria can enhance the N_2O emission at very low aeration; and Hao et al. (2001) and Santos et al. (2016) reported that pure culture of nitrifying bacteria responsible for the early ammonia oxidation and then significantly N_2O emission start under limited aeration. Therefore, the statistical analysis of N_2O emission and cumulative N_2O emission (Fig. 2c,d) clearly indicate that excess of zeolite combined with 1% lime addition drastically reduce the loss of nitrogen and N_2O production from in-vessel composting.

3.2. Maturity indexes

3.2.1. Changes in temperature, pH and total organic carbon profile

The result indicate that temperature rapidly increase in all treatments during the first week of composting, when zeolite amended treatments DFSS + WS + L + Z10%, DFSS + WS + L + Z15% and DFSS + WS + L + Z30% achieved values more than 60 °C within four days (Fig. 3a). Such rapid increasing trend of temperature showed repaid degradation of feed stock, due to the presence of readily-available organic matters (Manios et al., 2007; Li et al., 2012; Ermolaev et al., 2014). In the control and 1% lime amendment treatments, temperature increased slowly and maximum temperature was observed during the first 7 days being 52 °C, while the maximum temperature of zeolite amended treatment DFSS + WS + L + Z10% (66 °C) on day 6, DFSS + WS + L + Z15% (65 °C) on day 6 and DFSS + WS + L + Z30% (71 °C) on day 5 (Fig. 3a). These high temperatures in zeolite amended treatments were maintained for two weeks and then gradually decreased to 40–50 °C after four weeks of composting, which is indicating the end of thermophilic phase. Beside this, in control and 1% lime amended treatments were showed lower temperature as compare to zeolite amended treatments, while maximum temperature 57 °C and 58 °C were observed on day 15 and day 9. Meanwhile, temperature of control and 1% lime amended treatments dropped rapidly after a very short thermophilic phase, because in control no alkaline amendment and 1% lime amendment not enough to buffer the volatile fatty acids (VFAs) produced during the rapid degradation of organic matter and as consequence decrease in pH executing unfavorable condition for microbial activity. Simultaneously, lot of heat and GHGs are produced during microbial degradation of organic matter. Therefore, the CO₂ profile in the exhaust gas of zeolite amended treatments exhibited the similar trends as that temperature, as shown in Fig. 1a, while very low emission observed in control and 1% lime amended treatments, might be due to slow degradation which is also responsible for delayed the thermophilic phase. The delayed CO₂ emission correlated well with the thermophilic temperature regimes of the composting process in control and 1% lime amended treatments. Wong et al. (2009) and Awasthi et al. (2016) reported that temperature in compost is directly influenced by the overall rate of the microbial growth and its enzymatic activity for degradation process, which is dependent on availability of energy in the material and favorable environmental conditions such as pH and aeration. These factors are influenced in terms of porosity between particles and buffering capacity of added additives, both of which were found to be significant correlation with zeolite combined with 1% lime amended treatments, when explaining variations in GHGs emission and alleviation of early low pH in the original and transformed data sets.

The pH is one of the critical parameters influenced the microbial activity and GHGs emission during composting; and optimal pH 7.0–8.0 was suggested by several earlier authors for efficient composting (Szanto et al., 2007; Wong et al., 2009; Ermolaev et al., 2014; Chan et al., 2016). The decreasing trend of pH was observed in all treatments at the early stage of composting as reported previously (Villasenor et al., 2011; Yang et al., 2013). As shown in Fig. 3b, the pH of zeolite + 1% lime amended treatments rapidly decreased from 7.75 to 6.73 (DFSS + WS + L + Z10%), 7.82 to 6.93 (DFSS + WS + L + Z15%) and 7.87 to 6.58 (DFSS + WS + L + Z30%) and then gradually increase until the end of composting, which was likely due to the rapid degradation of organic matter and accumulation of organic acid. Consequently, the continuous organic carbon and nitrogen mineralization by microorganism, and the carbonate formation pH decreased, but as organic acids were decomposed and release of ammonium and volatile ammonia, which increases pH profile. Thus, the pH values increased and

reached to 7.58 (DFSS + WS + L + Z10%), 7.66 (DFSS + WS + L + Z15%) and 7.74 (DFSS + WS + L + Z30%) at the end of composting. However, Chan et al. (2016) reported that the addition of 10% zeolite did not significantly buffer the pH by alleviating the initial VFAs, while our previous study with 10% zeolite for gelatin industry sludge (GIS) composting (Awasthi et al., 2016) indicated that zeolite amended treatments showed slightly higher pH compared with the control and 1% lime amended treatments.

In contrast, lower dosage of 1% lime combined with zeolite drastically buffer and provide the optimal pH for microbial activity during the first two weeks of composting (Figs. 3b and S1), while control and with 1% lime amended treatments initially buffer the pH, but later on did not show additional pH enhancement. In addition, the reduction in pH during the beginning of SS composting was also observed in control as reported previously (Villasenor et al., 2011; Ermolaev et al., 2014), but decreasing trend of pH would maintained until day 35 and then slightly increased; and decreasing trend of pH was due to the accumulation of VFAs, which inhibit the microbial activity and organic matter degradation. Such influence was also reflecting in the temperature and CO₂ emission profiles of control treatment (Figs. 3a and 1a). Similar results regarding the pH changes with zeolite and lime amendments in SS composting were obtained in a previous studies (Villasenor et al., 2011; Li et al., 2013), but no literature available to its combined addition for composting. Beside this, several earlier authors also reported that high dosage of zeolite could pose a dilution effect that would also affect the pH, meanwhile it can provide a good physical structure such as retaining water and high moisture contents, while the effects may not be instantaneous compared with struvite salts and other additives (Villasenor et al., 2011; Li et al., 2012; Chan et al., 2016). At the end of composting, the result indicated that zeolite with 1% lime amended treatments were in the range of satisfactory values of pH (7–8.5) and end product was well stabilized (TMECC, 2002), while control and 1% lime amended treatments was not well matured. The final compost statistical analysis results showed that there were significant differences between zeolite with 1% lime amended treatments and control and 1% lime, respectively ($p < 0.05$), while no significant difference among the all three zeolite + 1% lime amended treatments.

The influence of different amount of zeolite + 1% lime and compare with control and 1% lime amended treatments on TOC was listed in (Fig. 3c), as the organic matter (OM) mineralization leads to decrease the TOC content in all treatments compost matrix, due to the net loss of organic carbon in the form of CO₂ and minimum TOC content was found at the end of the process (Wong et al., 2009; Sanchez-Monedero et al., 2010; Chan et al., 2016). With the composting process, the zeolites + 1% lime addition significantly facilitated TOC reduction, and the lowest TOC were observed 29.82 ± 1.26 , 30.69 ± 1.15 and 32.40 ± 1.10 in DFSS + WS + L + Z30%, DFSS + WS + L + Z15% and DFSS + WS + L + Z10% treatments, while highest TOC were found in control (41.90 ± 1.23) and 1% lime amended treatments (38.05 ± 1.02) at the end of composting. The maximum TOC content in the control and 1% lime amended treatments were mainly due to the lower microbial population (Fig. S1), decomposition and the low pH (Fig. 3b), which are not allow efficient transformation of complex organic compound and inorganic nitrogen as nitrifiers. A similar trend for TOC reduction was also observed by Wong et al. (2009) for food waste, Awasthi et al. (2015) for OFMSW, Villasenor et al. (2011) for SS and Li et al. (2012) and Wang et al. (2016) for pig manure composting. However, TOC is not important parameter but most of researchers reported that TOC value reduction is directly involved to CO₂ emission and maturity parameters like C/N ratio (TMECC, 2002; Chan et al., 2016).

3.2.2. Changes in extractable ammonia and total Kjeldahl nitrogen profile

Microbial activities mainly responsible for the variations in the concentration of extractable ammonium nitrogen ($\text{NH}_4^+\text{-N}$) due to the transformation of organic nitrogenous compounds into ammonium and ammonia gas, which are generated during the composting of initial feedstock (Szanto et al., 2007; Li et al., 2013; Chan et al., 2016). The results revealed that the concentrations of extractable $\text{NH}_4^+\text{-N}$ rapidly increased in all the zeolite amended treatments during early phases and then gradually stabilized at the end of the composting (Fig. 3e), except control and 1% lime amended treatments. Several earlier authors reported that ammonia is formed during the active degradation of organic compounds at optimum pH due to precipitation of ammonia to ammonium ion equilibrium (Wong et al., 2009; Villasenor et al., 2011; Ermolaev et al., 2014; Chan et al., 2016). In control and 1% lime amended treatments, maximum concentrations of $\text{NH}_4^+\text{-N}$ (3895.22 ± 143.53 mg/kg and 4274.29 ± 138.06 mg/kg) were observed on day 28 and then slightly decrease until the end of composting, which were comparatively higher than the $\text{NH}_4^+\text{-N}$ contents in 10% (2693.14 ± 146.43 mg/kg, on day 10), 15% (2853.46 ± 173.32 mg/kg, day 7) and 30% (2878.02 ± 125.39 mg/kg, on day 7) zeolite + 1% lime amended treatments, respectively. This higher $\text{NH}_4^+\text{-N}$ concentrations in control and 1% lime treatments were mainly due to low pH did not proliferate the microbial activity and then slowed down mineralization of organic nitrogenous compounds. Our result of $\text{NH}_4^+\text{-N}$ is in line with the previous findings (Yang et al., 2013; Chan et al., 2016) for food and kitchen waste, (Villasenor et al., 2011; Li et al., 2013) SS and GIS composting (Awasthi et al., 2016). In addition, 1% lime cannot significantly increase the pH substantially (Fig. 3b); and hence the low pH influenced the microbial activity and ammonification as well as composting process. Furthermore, higher dosage of zeolite mixed with 1% lime (DW) addition was mainly responsible of buffer the pH to an optimum level for the efficient microbial activity (Fig. S1), which resulted in higher ammonification and subsequently demonstrating a great affinity of zeolite for $\text{NH}_4^+\text{-N}$; and also showed the pH as crucial physicochemical factor influence the affinity. Beside this, at low pH ammoniac nitrogen had to compete with hydrogen ion for the adsorption site, while in an alkaline condition, ammoniac nitrogen would be promoted to rapid ammonia emission.

Therefore, the additions of zeolite combined with 1% lime are crucial for exploiting the extra-benefits of zeolite such as alleviate the initial low pH, reduce the nitrogen loss and GHGs emission from the composting reactor. Hence, when the pH is neutral the ammonium adsorption onto zeolite will be higher with the rate of organic waste degradation increase under optimum pH (6.5–7.5) (Villasenor et al., 2011; Maulini-Duran et al., 2013; Awasthi et al., 2016). After the second week, the $\text{NH}_4^+\text{-N}$ concentration significantly decreased, might be due volatilization of ammonia ion and nitrification, while in control and 1% lime amended treatment did not significantly buffer the pH and hence above the permissible limit of $\text{NH}_4^+\text{-N}$ concentrations were observed at the end of composting (Table S1), which indicate the immaturity of the compost (TMECC, 2002).

However, the TKN content profiles were totally different between zeolite + 1% lime and 1% lime as well as control treatments as listed (Fig. 3d). In zeolite + 1% lime amended treatments, the TKN content initially slightly decrease in first week and then sharply increase until the day 42, thereafter almost steady state values were observed. While when compare with the 1% lime and control treatments, a decreasing trend of TKN were observed until the day 14 and 28; and then slightly increase until the end of composting process that make significant differences ($P < 0.05$) from zeolite + 1% lime amended treatments (Fig. 3d). The initial

decreasing trend of TKN in zeolite + 1% lime amended treatments may be attributed due to the loss of ammonia, while long decreasing trend in control and 1% lime amended treatments because of no zeolite addition showed slow decomposition of organic matter and excessive loss of ammonia as indicated in ammonia emission profile (Fig. 2a). But during the maturation stage the TKN gradually increase, due to the net loss of the composting mass and nitrogen fixation (Wong et al., 2009; Li et al., 2013; Wang et al., 2016). The results clearly indicated that zeolite amendment offers potential way to facilitate the total aerobic bacteria and specific substrate degrading bacterial activity (Fig. S1a–e), which involved in nitrification, organic nitrogen mineralization and in the same time improves nitrogen conservation, respectively.

3.2.3. Changes in C/N ratio and seed germination index profile

As shown Fig. 3f, the solid C/N ratio in all zeolite + 1% lime amended treatments gradually decreased from the beginning to the end of composting process, due to the loss of TOC and nitrogen in the form of CO_2 and ammonia (Figs. 1a–b and 2a–b). Among the all zeolite + 1% lime amended treatments the trend of C/N ratio profile similar, while in the control and 1% lime amended treatments behaved totally different, and the increasing trend of C/N ratio were observed that could directly correlate with the rapid loss of ammonia and slow mineralization of organic matter during the composting. In addition, several earlier scientists also reported that CO_2 and ammonia emission directly interlinked with mineralization of organic matter coupled with the increasing TKN and decreased TOC content of the compost (Wong et al., 2009; Chan et al., 2016). Thus, in zeolite + 1% lime amended treatments, C/N ratio values reached maturation within 28 days of composting, which indicated that addition of higher dosage of zeolite combined with 1% lime significantly reduced the duration of maturation for DFSS compost. Despite control and 1% lime amended treatments compost have been not mature after 56 days indicating longer time need for maturation (Fig. 3f and Table S1).

Moreover, seed germination test was employed to evaluate the compost maturity and affect of phytotoxic substances. As listed in Fig. 3g, germination index (GI) value in all treatments were very low during the early phase of composting, this was due to the rapid degradation of organic matter produced huge quantity of VFAs (data not shown) and ammonium, which are toxic to seed germination. The GI for the 15% and 30% zeolite + 1% lime amended treatments were rapidly increased and reached over 80% within 28 days; and 10% zeolite + 1% lime amended treatments also achieve the maturity value on day 35, while in control and 1% lime amended treatments the GI value only reach $64.12 \pm 2.16\%$ and $76.65 \pm 1.04\%$ until the end of composting, respectively. Result indicate that higher dosage of zeolite with 1% lime have good buffering capacity that alleviate the concentration of toxic VFAs compounds, immobilize the HMs (Table S1), which provide good texture and reduce the nitrogen loss. Beside this, when compared with zeolite amended treatments, long maturity period was required (~56 days) for control and 1% lime amended treatments, probably due to slow degradation, high extractable ammonia and HMs contents (Table S1) (TMECC, 2002). Despite this, Fang and Wong (1999) reported that lime amendment did not reduce the maturation period and higher dosages of lime adversely affect the GI. In previous studies also observed that 10% zeolite addition with GIS composting reduce the maturation period to 20 days (Awasthi et al., 2016), while other studies reported longer period of maturation with various types of composting matrix (Wong et al., 2009; Wang et al., 2016). Thus our finding is similar with the some previous authors, who reported that zeolite addition not only reduce the nitrogen loss, but it could also alleviate the most of phototoxic compounds during the composting and offer

high nutrient compost (Villasenor et al., 2011; Chan et al., 2016; Awasthi et al., 2016).

4. Conclusion

Results indicate that composting of DFSS supplemented with zeolite + 1% lime drastically reduced the CH₄, N₂O and NH₃ emissions and reach the compost maturity within 35 days. Taking into account compost quality, adding zeolite + 1% lime was superior compare to the other two treatments for reach the thermophilic phase, reducing GHGs emission as well as alleviate low pH and achieve compost maturity. While control and 1% lime amended treatments were not mature after 56 days. Therefore, the best strategy by addition of 30% zeolite + 1% lime can reduces the GHGs emissions and facilitates nitrogen conservation during DFSS composting.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at <http://dx.doi.org/10.1016/j.biortech.2016.05.065>.

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