



# Combining biochar, zeolite and wood vinegar for composting of pig manure: The effect on greenhouse gas emission and nitrogen conservation

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## ABSTRACT

The effect of enhancing wood vinegar (WV) with a mixture of biochar (B) and zeolite (Z) to compost pig manure (PM) in a 130 L reactor was evaluated to determine the levels of greenhouse gas (GHG) and ammonia emissions. Six treatments were prepared in a 2:1 ratio of PM mixed with wheat straw (WS; dry weight basis): PM + WS (control), PM + WS + 10%B, PM + WS + 10%B + 10%Z, and PM + WS with 0.5%, 1.0% and 2.0%WV combined with 10%B + 10%Z. These were composted for 50 days, and the results indicated that the combined use of B, Z, and WV could shorten the thermophilic phase and improve the maturity of compost compared to the control treatment. In addition, WV mixed with B and Z could reduce ammonia loss by 64.45–74.32% and decrease CO<sub>2</sub>, CH<sub>4</sub>, and N<sub>2</sub>O emissions by 33.90–46.98%, 50.39–61.15%, and 79.51–81.10%, respectively. Furthermore, compared to treatments in which B and B + Z were added, adding WV was more efficient to reduce the nitrogen and carbon loss, and the 10%B + 10%Z + 2%WV treatment presented the lowest loss of carbon (9.16%) and nitrogen (0.75%). Based on the maturity indexes used, nitrogen conservation, and efficiency of GHG emissions reduction, the treatment 10%B + 10%Z + 2%WV is suggested for efficient PM composting.

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## 1. Introduction

With the rapid development of intensive pig farming, a large quantity of pig manure (PM) is generated each year in China. In 2015, the production of PM had arrived approximately 490 million tons (NBSC, 2016). The effective management of the PM has become a significant issue for the Chinese government and livestock industry (Wang et al., 2016a). In the last decade, methods such as landfill, anaerobic digestion, and composting were tried as ways of disposing PM. Of these, composting has been widely

accepted as one of the preferred cost-effective methods for recycling organic waste. It not only reduces the volume of PM and destroys the weed seeds and pathogens, but also converts the organic waste into a humus-like, nutrient-enriched stable organic product (compost) that could improve soil fertility (Bernal et al., 2009; Li et al., 2012). However, the greenhouse gas (GHG; CO<sub>2</sub>, CH<sub>4</sub>, and N<sub>2</sub>O) and excessive ammonia (NH<sub>3</sub>) emissions associated with the composting process reduce the agronomic value of compost and cause secondary environmental pollution (Yang et al., 2015; Awasthi et al., 2016d).

Many practical approaches have been applied in order to promote progress in composting and reduce its adverse effect, including using different kinds of bulking agents (Chowdhury et al., 2014a), increasing the aeration rate (Tsutsui et al., 2013; Chowdhury et al., 2014b), and adding chemical agents and mineral additives (Li et al., 2012; Jiang et al., 2015; Awasthi et al., 2016c). To date, using mineral additives to improve the composting efficiency and quality of the end product, as well as reduce GHG emissions, are attracting increased interest from researchers. For example, Chowdhury et al. (2014b) reported that the addition of

**Abbreviations:** PM, pig manure; B, Biochar; Z, Zeolite; WV, wood vinegar; GHG, greenhouse gas; WS, wheat straw; GI, germination index; SS, sewage sludge; B+Z, 10%biochar + 10%zeolite; B + Z + 0.5%WV, 10%biochar + 10%zeolite + 0.5%wood vinegar; B + Z + 1.0%WV, 10%biochar + 10%zeolite + 1.0%wood vinegar; B + Z82.0% WV, 10%biochar + 10% zeolite + 2.0% wood vinegar.

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biochar could reduce 27–32% GHG emissions during the chicken manure composting. Yang et al. (2015) discovered that adding the phosphogypsum could decrease 85.8% CH<sub>4</sub> emission during the kitchen waste composting. Chan et al. (2016) demonstrated that co-compost 10% zeolite with food waste could reduce NH<sub>3</sub> emissions by 7.06% and improve the compost maturity compare to a struvite treatment. Meanwhile, our previous study indicated that composting PM with the addition of 10% medical stone could significantly reduce the nitrogen loss and N<sub>2</sub>O emissions (Wang et al., 2016b). Moreover, other researchers have revealed that a mixture of additives could reduce GHG emissions and improve the organic matter humification better than a single additive could (Chen et al., 2010; Awasthi et al., 2016b). However, adding a mixture of additives presents some drawbacks. For example, although adding 5% phosphogypsum and 0.2% dicyandiamide decreased N<sub>2</sub>O emissions, it lead to higher nitrogen losses and increase in salinity as compared to a treatment using phosphogypsum alone (Luo et al., 2013). Similarly, different amounts of dicyandiamide mixed with struvite reduced N<sub>2</sub>O emissions by 76–78%, but had an eligible effect on ammonia emissions from PM composting (Jiang et al., 2016a). In addition, our early researches showed that combinations of zeolite and biochar or lime could inhibit the GHG emissions and improve compost quality, but there is a potential for nitrogen loss when it is compared to the control treatment (Awasthi et al., 2016c, 2016d). Thus, the results of these findings indicate that adding a mixture of mineral additives or other chemical agents could effectively reduce GHG emissions and promote the composting process, but nitrogen conservation still needed to be improved.

Zeolite, as a natural porous mineral, has been widely used to reduce the various gas emissions, nutrients loss and the salinity during the composting process (Zorpas and Loizidou, 2008; Zhang and Sun, 2015; Chan et al., 2016). However, some previous researches indicated that the zeolite alone is not good enough to buffer against the low during the organic waste composting (Villaseñor et al., 2011; Singh et al., 2013). In recent years, Zhang and Sun (2015) and Awasthi et al. (2016c, 2016d) found that zeolite combined with other additives could further promote the composting process and reduce the GHG emissions. Wood vinegar (WV), a byproduct of the carbonization of wood or wood residues, contains large quantities of organic acids or components and has been widely used as an insect repellent, odor remover, and soil fertilizer (Chen et al., 2010; Wu et al., 2015). Recently, WV was used in composting to reduce NH<sub>3</sub> emissions (Chen et al., 2010; Zhang and Sun, 2015). However, to the best of our knowledge, the combination of biochar, zeolite, and different concentrations of wood vinegar as amendments to reduce GHG and ammonia emissions during PM composting has not been reported thus far. Therefore, this study investigated the effect, on GHG (CO<sub>2</sub>, CH<sub>4</sub> and N<sub>2</sub>O) emissions, nitrogen loss, and compost maturity, of combining different amounts of WV with biochar and zeolite during PM composting.

## 2. Materials and methods

### 2.1. Preparation of compost

Fresh PM and wheat straw (WS) were collected from a hogger and a farm in Yangling town, Shaanxi, China. Biochar (B) and WV were purchased from Yangling Yixing Energy Pvt. Ltd., China, while zeolite (Z) was obtained from the Zhejiang Shenshi Mining Industry Co., Ltd, China. The biochar was prepared from tobacco stalk via slow and dry pyrolysis at temperatures of 500–600 °C at atmospheric pressure for 24 h, initiated by the pyrolysis of feedstock from the bottom of a 5 m<sup>3</sup> kiln. The WS was chopped into 1 cm

lengths and used as a bulking agent to adjust the moisture content (~55%), bulk density (~0.5 kg/L) and carbon/nitrogen ratio (~25) of the initial compost substrate (Bernal et al., 2009; Awasthi et al., 2016d). B and Z were crushed into fine particles (2–5 mm) (Awasthi et al., 2016a) and applied to the composting mass as amendments. Selected physicochemical characteristics of the raw materials are presented in Table 1.

The compost was prepared in 130L PVC reactors, according to the reactor layout and composting process described in our previous researches (Li et al., 2012; Awasthi et al., 2017). A total of six treatments were designed, with fresh PM and WS mixed at a 2:1 ratio (dry weight), after which the other additives were added, as presented in Table 2. The temperature of the compost and the ambient were monitored thrice daily and the mean recorded. After the composting materials were mixed thoroughly, about 100L of each mixture was put into the reactors. The dosages of B, Z, and WV were based on the previous studies (Chen et al., 2010; Sonoki et al., 2013; Chan et al., 2016). Air was pumped into the reactor from the bottom of each vessel and an automatic device maintained a constant air flow rate of 0.3 L/kg (dry matter)/min (Li et al., 2012).

### 2.2. Sampling and analysis of compost

During the composting process, 500 g homogeneous compost samples were taken after 0, 4, 8, 15, 22, 29, 36, 43 and 50d. The samples were divided into two; one part was stored at 4 °C till analysis, while the other was air dried, powdered in an agate mortar, passed through a 0.1 mm sieve, and thoroughly mixed for further analysis. The fresh samples were used to detect pH, electrical conductivity (EC) at 25 °C, NH<sub>4</sub>-N, and NO<sub>3</sub>-N levels, and the seed germination index (GI), as per laboratory procedures (Zucconi et al., 1981; Lu et al., 2009; Li et al., 2012). An MP521 pH/EC meter (Shanghai, China) was used to measure the pH and EC according to methods in Li et al. (2015). To determine the levels of NH<sub>4</sub><sup>+</sup>-N and NO<sub>3</sub><sup>-</sup>-N, fresh samples were extracted with 50 mL 2 mol/L KCl solution (1:10 (w/v)) and analyzed using a segmented flow analyzer (Technicon Auto-analyzer II System, Germany). Ammonia gas was trapped in a boric acid solution and measured via titration with 1 mol/L hydrochloric acid (Komilis and Ham, 2006). The CO<sub>2</sub>, CH<sub>4</sub>, and N<sub>2</sub>O samples were collected daily in the first two weeks and two or three times weekly thereafter, while gas concentrations were determined using gas chromatography as described by Awasthi et al. (2016b), using an 6890N Network GC system (Agilent Technologies, United States of America) equipped with electron capture and flame ionization detectors. The temperature, pH, and GI were regarded as the maturity indexes in this research.

### 2.3. Statistical analysis

All physicochemical analyses were performed in triplicate. The data were subject to the one-way analysis of variance (ANOVA) and multiple other tests to compare the least significance difference (LSD) at *p* = .05 using SPSS v.18.0 for Windows. Redundancy analyses (RDA) were performed using Canoco 5 to determine correlations between physiochemical properties and GHG and ammonia emissions during the composting.

## 3. Results and discussion

### 3.1. Effect of amendments on maturity indexes

The variations of temperature in all the treatments are presented in Fig. 1a. During the experiment, our mixtures followed the three typical phases of composting: mesophilic, thermophilic,

**Table 1**

The physicochemical properties of raw materials.

Parameters	Pig manure	Wheat straw	Biochar	Zeolite	Wood vinegar
Moisture (%)	63.12 ± 1.10	6.45 ± 0.20	7.31 ± 0.21	0.60 ± 0.06	ND
pH	8.00 ± 0.06	6.57 ± 0.14	10.80 ± 0.02	9.39 ± 0.03	3.21 ± 0.01
EC (mS/cm, at 25 °C)	5.41 ± 0.11	2.72 ± 0.26	12.09 ± 0.40	0.06 ± 0.00	4.42 ± 0.02
OM (%)	73.04 ± 0.20	90.91 ± 0.88	65.12 ± 1.00	ND	21.63 ± 0.02
TP (g/kg)	29.38 ± 1.70	4.18 ± 0.37	5.47 ± 0.45	0.52 ± 0.06	0.05 ± 0.00
TKN (g/kg)	26.11 ± 0.09	1.03 ± 0.07	6.26 ± 1.24	0.93 ± 0.06	1.09 ± 0.10
TK (g/kg)	12.54 ± 0.39	0.80 ± 0.00	74.79 ± 4.74	0.69 ± 0.03	0.07 ± 0.00

ND (Not detected), EC (Electrical conductivity), OM (Organic matter), TP (Total phosphorus), TKN (Total kjeldahl nitrogen) and TK (Total potassium). Results are the mean of three replicates and error bars indicate standard deviation.

**Table 2**

Treatments and the amount of different additives used in each treatment on (dry weight basis).

Treatments	Percentage of additives amendment (dry weight basis)		
	Biochar (%)	Zeolite (%)	Wood vinegar (%)
Control	0	0	0
B	10	0	0
B + Z	10	10	0
B + Z + 0.5%WV	10	10	0.5
B + Z + 1.0%WV	10	10	1.0
B + Z + 2.0%WV	10	10	2.0

B (biochar), Z (zeolite), WV (wood vinegar).

and curing (Bernal et al., 2009). The temperature in all the treatments initially increased rapidly and reached the thermophilic phase ( $>50.0$  °C) within 2 d of incubation while maintaining high temperature ( $>50.0$  °C) above 5 days, which is necessary to destroy pathogens and weed seeds and ensure that the compost is sanitary (Li et al., 2012; Wang et al., 2016b; Jiang et al., 2014). The temperature trends indicated that the longest thermophilic phase was observed in the control treatment (10 d) and the shortest in the B + Z + 1.0%WV treatment (6 d), while the B, B + Z, B + Z + 0.5%WV, and B + Z + 2.0%WV treatments remained in the thermophilic phase for 7, 9, 7, and 8 d, respectively. Compared to the control treatment, the addition of B shortened the thermophilic phase of PM composting, possibly due to the B rapidly improving the degradation of available organic matter and increasing the temperature as composting starts. Then, as the bio-available organic matter is exhausted, the temperature gradually decreases until the end of the composting period, with the stabilization of the compost. Similar observations were also reported by Wang et al. (2013) and Czekala et al. (2016), who used biochar as an additive in PM and poultry manure composting. During the bio-oxidative phase, the peak temperatures in the control, B, B + Z, B + Z + 0.5%WV, B + Z + 1.0%WV, and B + Z + 2.0%WV treatments were 66.5 °C, 68.5 °C, 69.25 °C, 70.0 °C, 72.5 °C, and 72.6 °C, respectively. The results indicated that the adding these amendments could contribute to the rapid temperature rise and shortening of the thermophilic phase at the beginning of PM composting. Meanwhile, compare to the B + Z treatment, the temperature rose further with increasing WV dosages, possibly due to the WV containing some carbon sources that could be utilized by microorganisms, and the release of metabolic heat (Mu et al., 2003; Wu et al., 2015). Therefore, the addition of WV could increase temperatures; the maximum was observed in treatments with high dosages (1.0%–2.0%) of WV. The results of the present study were quite similar to those of Chen et al. (2010), who used bamboo biochar and its vinegar as additives for PM composting. After 30 d, the temperature of all the treatments gradually decreased and approached the ambient temperature at the end of composting. The variations in temperature of all the treatments were consistent with findings by Jiang et al. (2016a) and Wang et al. (2016b), who used struvite crystal-

lization and medical stone amendments to reduce  $\text{NH}_3$  and  $\text{N}_2\text{O}$  emissions during PM composting.

As shown in Fig. 1b, the addition of B significantly ( $p < .05$ ) increased the initial pH values of the compost matrixes, which was consistent with Li et al. (2015), who used B to improve nutrient transformation during PM composting. Furthermore, compared to the treatment using B alone, the addition of zeolite had no significant ( $p > .05$ ) effect on the initial pH. This is in line with Chan et al. (2016), who applied 10%Z to compost food waste. The acidic character of WV lowered the incipient pH value in all B + Z + WV treatments compared with the B + Z treatment, and with the increase in the amount of WV, the initial pH values became 8.02, 7.84, and 7.8, respectively. The results agreed with Chen et al. (2010). During the early stages of composting, the pH values of all treatments quickly rose as temperatures increased; pH values in the control, B, and B + Z + 0.5%WV treatments went up continuously towards the peak value for 8 d before decreasing gradually. However, for the B + Z, B + Z + 1.0%WV, and B + Z + 2.0%WV treatments, the pH reached the maximum values at the 4d and then decreased. The increase in pH may have resulted from the production and accumulation of ammonia, and the subsequent decreases could have been due to its volatilization (Chen et al., 2010; Jiang et al., 2016a). After 20 d, the pH values of all the treatments increased slightly at first and then decreased gradually. Apart from this, a significant increase in pH values was observed in all treatments during the curing phase, possibly due to the decomposition of organic acid, which later decreased with nitrification (Huang et al., 2004; Jiang et al., 2014; Li et al., 2015; Chan et al., 2016). At the end of the composting, the pH values were 7.96, 8.5, 8.60, 8.55, 8.64, and 8.56 for the control, B, B + Z, B + Z + 0.5%WV, B + Z + 1.0%WV, and B + Z + 2.0%WV treatments, respectively. The results indicated that the addition of B alone clearly ( $p < .05$ ) increased the pH values, likely due to its alkaline properties; this finding is in line with the previous studies by Li et al. (2015) and Awasthi et al. (2016a). However, the final pH values in all B + Z + WV treatments were similar to the B + Z treatment, and the differences among the B + Z + 0.5%WV, B + Z + 1.0%WV, and B + Z + 2.0%WV treatments were not significant ( $p > .05$ ). These results indicated that the addition of WV had no significant effect on the final pH as compared to B + Z treatment.

The germination index (GI) is a very important parameter that can indicate the maturity and toxicity of compost (Bernal et al., 2009; Li et al., 2012). The initial GI values of all the treatments were quite low (Fig. 1c) which could be attributed to the release of toxic ammonia and presence of low molecular weight volatile fatty acids (Chen et al., 2010; Jiang et al., 2016b). A similar result was also observed by Li et al. (2015), who co-composted biochar and PM. After 4 d, the GI values of the B, B + Z and B + Z + WV treatments gradually increased until the end of composting, while the GI value in the control treatment started to increase after 8 d. For mature compost, the GI value should be higher than 0.5 (Li et al., 2012; Wang et al., 2016a); the result in Fig. 1c shows that compost



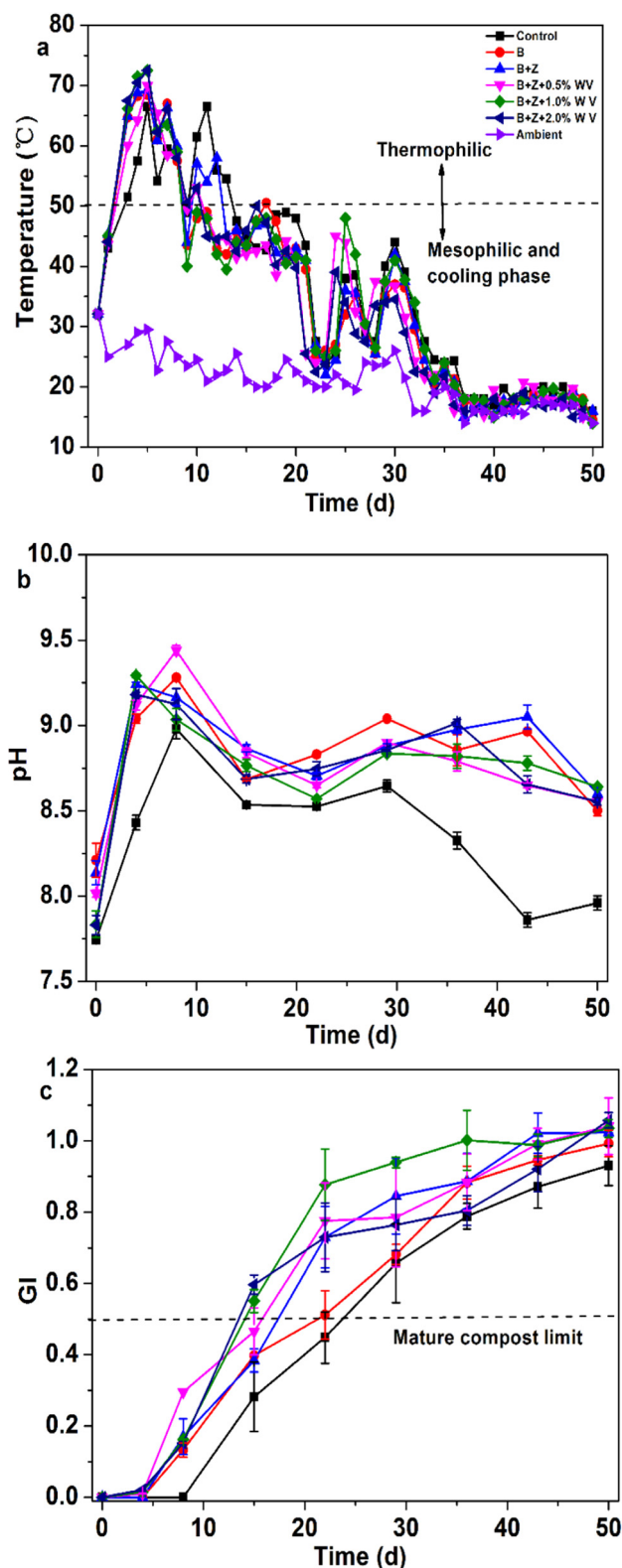


Fig. 1. Changes of temperature (a), pH (b) and GI (c) during pig manure composting. Results are the mean of three replicates and error bars indicate standard deviation.

from all treatments except the control had matured by 22 d. The control required a longer maturation period (25 d), possibly due to the longer thermophilic phase and late start to the stabilization of organic waste (Wang et al., 2016a, 2016b). Apart from this, the major drawback associated with the control treatment was higher

ammonia emissions, which considerably reduced the nitrogen content and quality of the compost. Among the all the treatments amended with B, the B + Z + 2.0%WV matured first, followed by B + Z + 1.0%WV, B + Z + 0.5%WV, B + Z, and B alone. And at the end of experiment, the GI value of the B and B + Z treatments were higher than that of the control by 6.66–9.96%, similar to findings by Li et al. (2015) and Lopez-Cano et al. (2016), who discovered that B could improve the maturation of PM and sheep manure during composting. In addition, Awasthi et al. (2017) indicated that, compared to B alone, a treatment using B and Z could further increase the GI value during the composting of sewage sludge (SS); compared to the B + Z treatment, the GI values in B + Z + WV treatments increased by 1.73–2.36%. This result indicated that the addition of WV further accelerated the GI, which was similar to the result achieved by Chen et al. (2010).

### 3.2. Effect of amendments on carbon dioxide and methane emissions profiles

The emissions of carbon dioxide ( $\text{CO}_2$ ) and methane ( $\text{CH}_4$ ) from the various treatments correlated well with the temperature of compost in this experiment (Fig. 2a and c). Similar results were also reported in previous researches (Wong and Fang, 2000; Chowdhury et al., 2014a).  $\text{CO}_2$  is the main gas produced during the composting process that could visually reflect the respiration rate of microorganisms and indirectly indicate the microbial activity (Awasthi et al., 2016d). The  $\text{CO}_2$  emissions of all the treatments initially increased dramatically and decreased thereafter. The maximum concentrations of  $\text{CO}_2$  emitted were 116.5, 164.5, 153.2, 139.0, 147.4, and 125.1 g/d for the control, B, B + Z, B + Z + 0.5%WV, B + Z + 1.0%WV, and B + Z + 2.0%WV treatments, respectively. Compared to the control, the treatment with B alone had higher ( $p < .05$ ) initial  $\text{CO}_2$  emissions, possibly due to the B amendment improving microbial activity and oxygen diffusion (Czekala et al., 2016). However, compared to B alone, the peak  $\text{CO}_2$  emissions of the B + Z treatment was lower, possibly due to the porous microstructure of Z reducing the  $\text{CO}_2$  emissions (Awasthi et al., 2016c; Kim et al., 2016). On the other hand, the addition of WV could have improved the adsorption capacity of B or Z and then decreased the  $\text{CO}_2$  emissions compared to the B + Z treatment (Chen et al., 2010; Kim et al., 2016; Xu et al., 2016). Following an initial rise, the  $\text{CO}_2$  concentrations of different treatments decreased sharply, likely due to the excessively high temperatures inhabiting the microbial activity and reducing easily available organic matter (Wong and Fang, 2000). After 20 d, two small  $\text{CO}_2$  emissions peaks were observed in all treatments, possibly due to the turning of the compost; fluctuations in temperature supported this suggestion (Fig. 1). At the end of composting, the  $\text{CO}_2$  emissions among all the treatments leveled off, indicating the stability of the compost. Compared to the control treatment (Fig. 2b), the cumulative  $\text{CO}_2$  emissions were reduced by 26.06, 30.15, 33.90, 45.91, and 46.98% in the B, B + Z, B + Z + 0.5%WV, B + Z + 1.0%WV, and B + Z + 2.0%WV treatments, respectively, and all the differences among them were significant ( $p < .05$ ). The results showed that the use of these additives could decrease  $\text{CO}_2$  emissions compared to control treatment. Sanchez-Garcia et al. (2015) also discovered a similar result while co-composting poultry manure with biochar. The shorter thermophilic phase and significant correlation between the temperature and pH profiles of the B, B + Z, and B + Z + WV treatments also support this conclusion (Figs. 1a and 6).

$\text{CH}_4$  is a major GHG generated during inefficient composting, and its production was attributed to methanogens deoxidize  $\text{CO}_2/\text{H}_2$  and acetic acid under anaerobic conditions (Yang et al., 2015; Sanchez-Garcia et al., 2015). The  $\text{CH}_4$  concentrations detected in all the treatments are presented in Fig. 2c. In this study,  $\text{CH}_4$  emissions mainly occurred during the initial stages of composting,

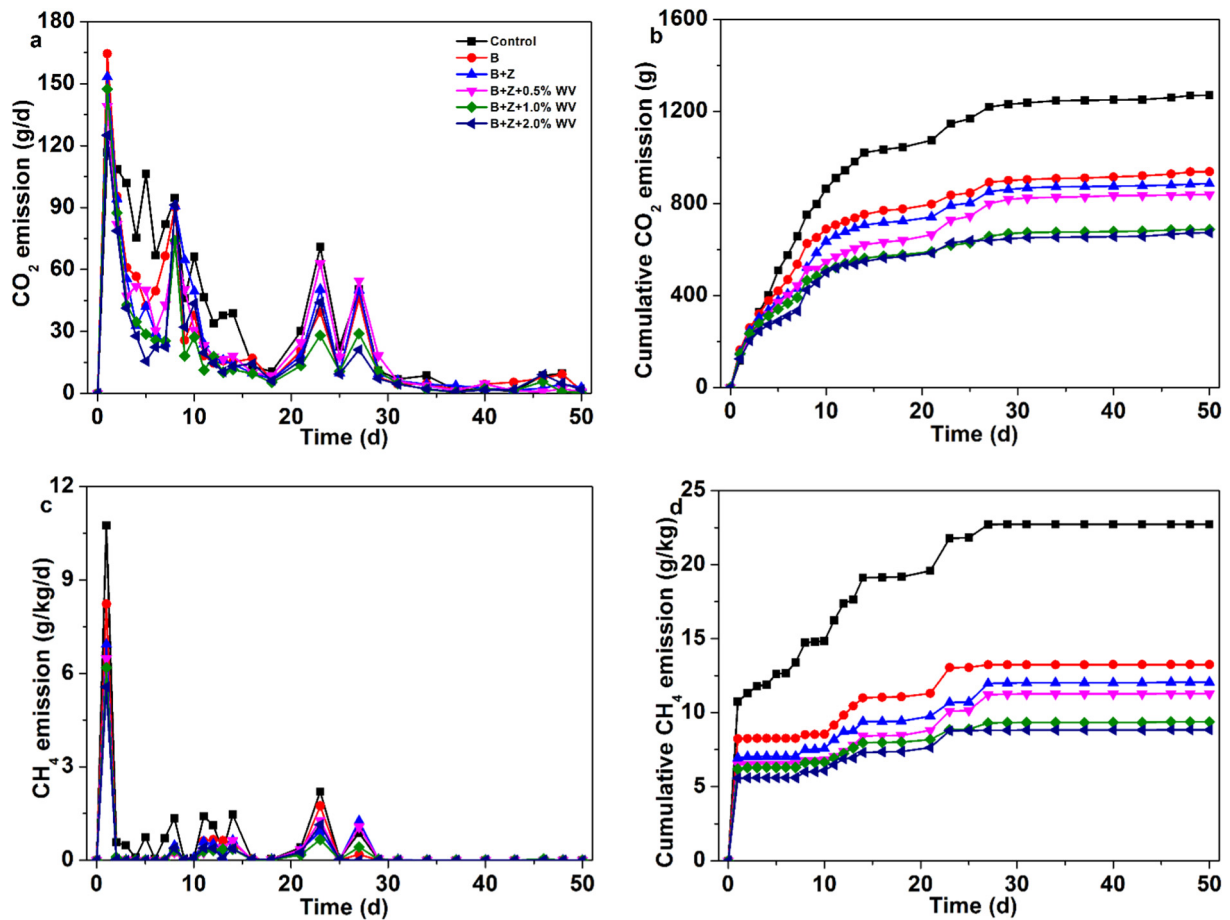


Fig. 2. Changes of CO<sub>2</sub> (a), cumulative CO<sub>2</sub> (b), CH<sub>4</sub> (c) and cumulative CH<sub>4</sub> (d) emissions during pig manure composting.

which coincided with findings from Awasthi et al. (2016a) and Jiang et al. (2016b) related to the composting of SS and PM. The CH<sub>4</sub> concentrations in all the treatments rapidly increased and reached the peak values on day 1, in line with the CO<sub>2</sub> emissions (Figs. 2a and 6). The initially large CH<sub>4</sub> emissions from all the treatments might have been due to the abundant nutrients and organic matter available in the compost substrates that stimulated microbial growth and rapidly depleted the oxygen concentrations (Sanchez-Garcia et al., 2015). On the other hand, the settling of the compost matrix during the early phase of composting could also have limited oxygen diffusion, thereby forming a partially anaerobic environment needed for CH<sub>4</sub> emissions (Luo et al., 2013; Chowdhury et al., 2014b). Thereafter, as organic matter decomposed and moisture was lost (data not show), the CH<sub>4</sub> emissions of all treatments declined sharply and fluctuated during the maturation phase. In this research, the CH<sub>4</sub> emissions of all treatments showed a similar tendency to those seen in previous reports by Chowdhury et al. (2014b) and Jiang et al. (2016b) for poultry and PM composting.

During the entire composting process, the cumulative CH<sub>4</sub> emissions in the treatments of the B, B + Z, B + Z + 0.5%WV, B + Z + 1.0%WV, and B + Z + 2.0%WV treatments were lower than that in the control (Fig. 2d); and were significantly ( $p < .05$ ) reduced by 41.73%, 46.96%, 50.39%, 58.75%, and 61.15%, respectively. Recent research has shown that the addition of B could effectively decrease the CH<sub>4</sub> emissions during composting. Sonoki et al. (2013) observed that the addition of a 10%B blend could increase the number of methanotrophs present while decreasing the methanogens, thus inhibiting CH<sub>4</sub> emissions during the ther-

mophilic phase of cattle manure composting. In addition, Awasthi et al. (2016d) discovered that the combined use of B and Z could significantly ( $p < .05$ ) inhibit CH<sub>4</sub> emissions during SS composting, possibly due to the B amendment increasing the porosity of the compost, while adding Z could have buffered the compost mass and boosted microbial activities. The cumulative CH<sub>4</sub> emissions decreased by 6.45%, 22.23%, and 26.76%, for the B + Z + 0.5% WV, B + Z + 1.0%WV, and B + Z + 2.0%WV treatments, respectively, compared to B + Z treatment. In this study, CH<sub>4</sub> emissions were high mainly during the initial phase of composting; therefore, mixing WV with B and Z could dramatically increase the ammonium contents (Fig. 4a) in compost, which would restrain methanogen activity and reduce ( $p < .05$ ) CH<sub>4</sub> emissions compared to the B + Z treatment (Luo et al., 2013; Jiang et al., 2016a). Thus, the B + Z + 2.0%WV treatment presented the lowest cumulative CH<sub>4</sub> emissions (8.83 g/kg), possibly due to having an initially higher ammonium content compared to the other treatments.

### 3.3. Effect of amendments on nitrous oxide and ammonia emissions profiles

N<sub>2</sub>O emissions during the composting process follow a complex mechanism mainly correlated to denitrification or nitrification processes, which are ways regulated by the NH<sub>4</sub><sup>+</sup>-N and NO<sub>3</sub><sup>-</sup>-N content, available carbon sources, and oxygen content in the composting matrix (Yang et al., 2015; Sanchez-Garcia et al., 2015). During the experiment period, the N<sub>2</sub>O discharge in all the treatments showed a similar trend (Fig. 3.a). The initial N<sub>2</sub>O emissions among all treatments were very low, possibly due to the fact

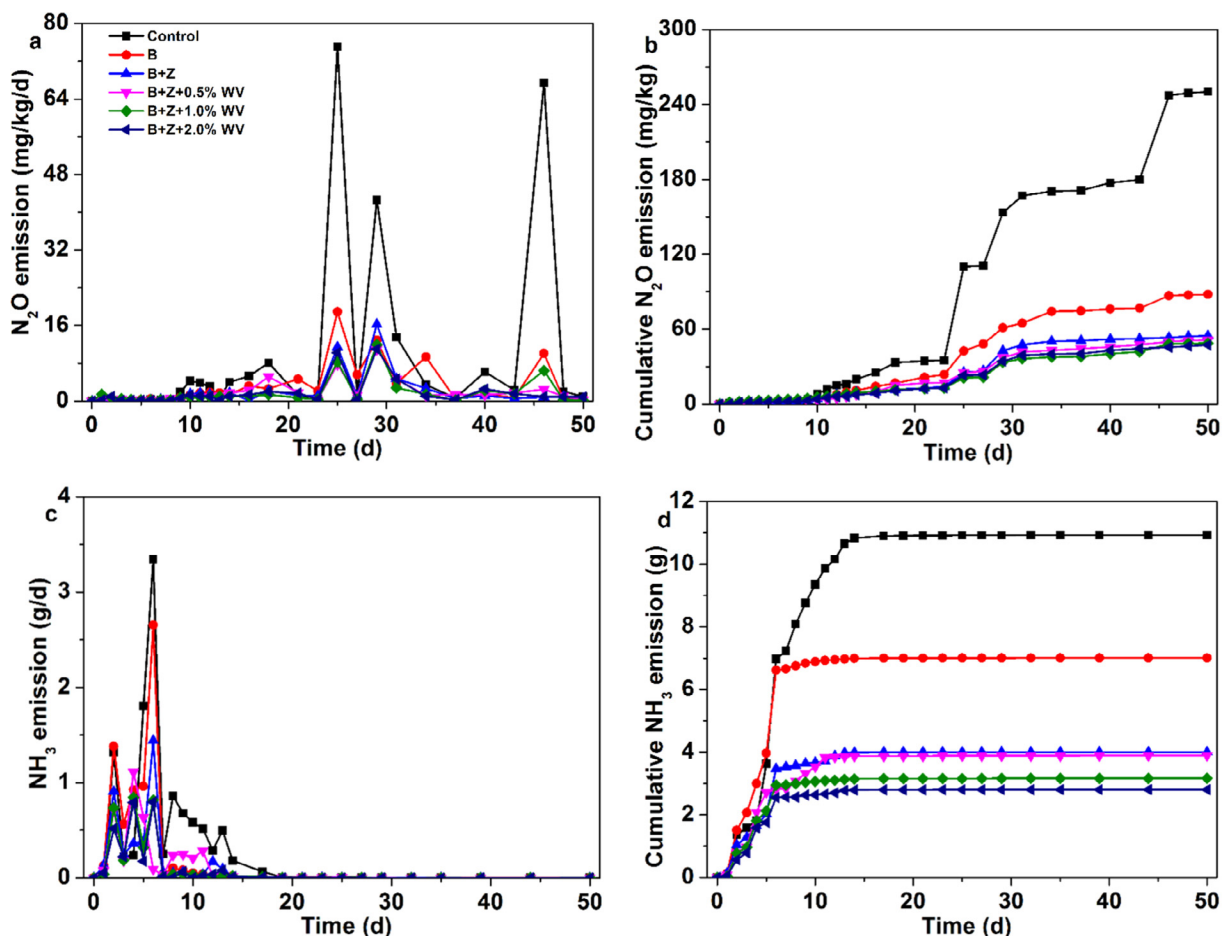


Fig. 3. Changes of  $N_2O$  (a), cumulative  $N_2O$  (b),  $NH_3$  (c) and cumulative  $NH_3$  (d) emissions during pig manure composting.

that optimum aeration and high temperatures could not support denitrification or nitrification. These  $N_2O$  emissions results corresponded with reports by Wolter et al. (2004) and Lopez-Cano et al. (2016), who composted PM and olive mill wastes. Sommer and Moller (2000) also reported that nitrifying and denitrifying microbes, which generally not survived at the early phase of composting, were responsible for the formation of  $N_2O$  that resulted in almost undetectable emissions. Furthermore, the initially low concentrations of nitrate (Fig. 4b) in all the treatments could be another reason for low  $N_2O$  emissions (Awasthi et al., 2016b).

In addition, some researchers have reported that high  $N_2O$  emissions were observed during the thermophilic phase when kitchen waste and SS were composted (Yang et al., 2015; Awasthi et al., 2016d). The variation in these results could be due to the different characteristics of the raw materials and experimental conditions. After 20 d, as temperatures decreased and nitrate concentrations increased, a large amount of  $N_2O$  was emitted from all treatments, likely due to the presence of  $O_2$ , low availability of degradable carbohydrate, and nitrate accumulation (Jiang et al., 2016a; Wang et al., 2016b). The results indicated that  $N_2O$  emitted during this phase could be due to nitrification (Jiang et al., 2016b). Similar patterns were also reported by Wang et al. (2013), when PM was composted with B, and Lopez-Cano et al. (2016), who composted olive mill wastes and sheep manure amended with B. Throughout the composting process, lower cumulative  $N_2O$  emissions (Fig. 3b) were observed in the B, B + Z, and B + Z + WV treatments compared to the control. Furthermore, compared to the control, the cumulative  $N_2O$  emissions from the B, B + Z, B + Z + 0.5%WV, B + Z + 1.0%WV, and B + Z + 2.0%WV treatments decreased

significantly ( $p < .05$ ) by 64.91, 78.14, 79.51, 80.52, and 81.10%, respectively. The lowest cumulative  $N_2O$  emission (47.29 mg/kg) was observed in the B + Z + 2.0%WV treatment. As stated in a previous study, the  $N_2O$  emissions and pH values were significantly negative correlated (Hao et al., 2005). In the current study, the B treatment presented a significantly higher ( $p < .05$ ) pH value and lower  $N_2O$  emissions compared to the control treatment, which also supported the above conclusion. In addition, Awasthi et al. (2016d) indicated that B mixed with Z could efficiently decrease the GHG emissions and improve enzymatic activities during SS composting. In comparison, the cumulative  $N_2O$  emissions in the B + Z was 37.69% lower than B treatment, possibly due to the fact that the added Z absorbed the  $N_2O$  in the compost (Kim et al., 2016) and consequently further reduced the  $N_2O$  emissions. Moreover, compared to the B + Z treatment, the cumulative  $N_2O$  emissions decreased by 6.28%, 10.91%, and 13.58% for the B + Z + 0.5% WV, B + Z + 1.0%WV, and B + Z + 2.0%WV treatments, respectively. The difference among these treatments was not significant ( $p > .05$ ). The result indicated that the combined use of WV, B, and Z could further decrease  $N_2O$  emissions compared to the B + Z treatment, which could be attributed to the lower nitrate levels in the B + Z + WV treatments (Fig. 4b) which reduced the  $N_2O$  emissions (Wang et al., 2013; Tsutsui et al., 2013; Jiang et al., 2016b). In addition, since mixing WV with B and Z could improve the adsorption capacity of B (Chen et al., 2010) and Z in compost (Awasthi et al., 2016c), this mixture of additives appears to act as a biofilter because porous micro-structure of B and Z combined with WV might be formed a unique net that efficiently reduced gas loss. Similarly, Awasthi et al. (2016d) reported that an amendment of

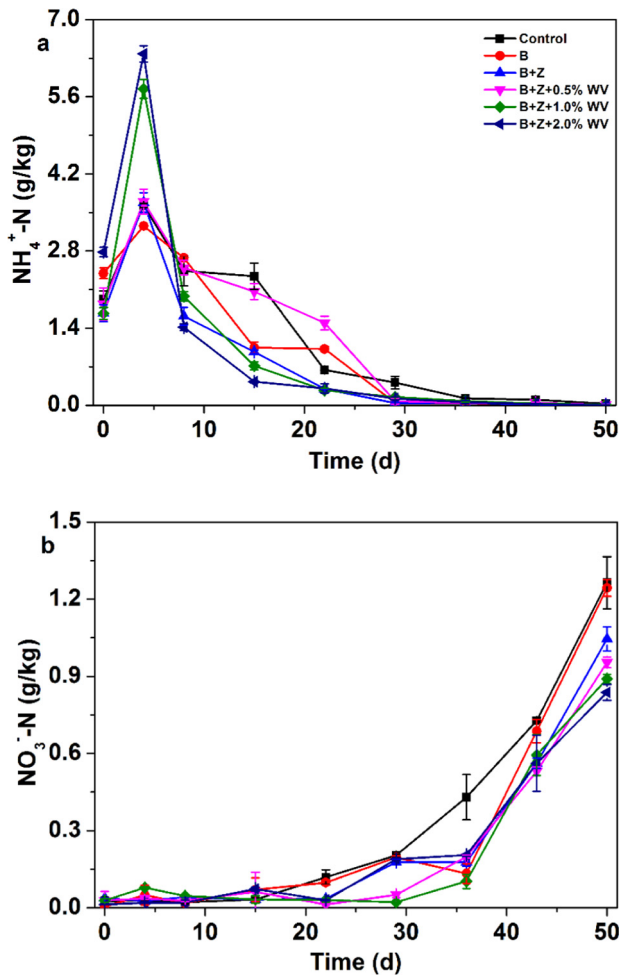


Fig. 4. Changes of  $\text{NH}_4^+\text{-N}$  (a) and  $\text{NO}_3^-\text{-N}$  (b) emissions during pig manure composting. Results are the mean of three replicates and error bars indicate standard deviation.

12%B + 10%Z considerably reduced GHG and ammonia emissions during SS composting. Therefore, the combined effect of WV, B, and Z further reduced the  $\text{N}_2\text{O}$  emissions and showed significant correlations with temperature and pH profile (Fig. 6). Subsequently, the  $\text{NH}_3$  and extractable ammonium profile change was also support this evidence which indicate that WV, B, and Z combined affect is more beneficial to improve the rate of composting and the same time reduced loss of nitrogen. This correlation is also reflecting in RDA (Fig. 6).

$\text{NH}_3$  volatilization is the main pathway for nitrogen loss during the composting process. It not only lowers the quality of final product, but also causes environmental pollution problems (Khan et al., 2014; Wang et al., 2016b). The variations in  $\text{NH}_3$  levels observed during the experiment period are presented in Fig. 3.c. Initially, as the temperature and pH rose,  $\text{NH}_3$  emissions from all treatments increased sharply and reached their maximum values within 4–6 d, while the maximum  $\text{NH}_3$  emissions were 3.35, 2.65, 1.44, 1.11, 0.84, and 0.79 g/d for the control, B, B + Z, B + Z + 0.5%WV, B + Z + 1.0%WV, and B + Z + 2.0%WV treatments, respectively. The results showed that the addition of B alone and B + Z could decrease  $\text{NH}_3$  emissions, in line with results from Khan et al. (2014) and Awasthi et al. (2016d), who used B and B + Z combinations to reduce  $\text{NH}_3$  emissions while composting chicken manure and SS. Increasing the WV amount significantly ( $p > .05$ ) lowered the  $\text{NH}_3$  observed in B + Z + WV treatments compared to the B + Z treatment, likely due to the fact that WV contained vari-

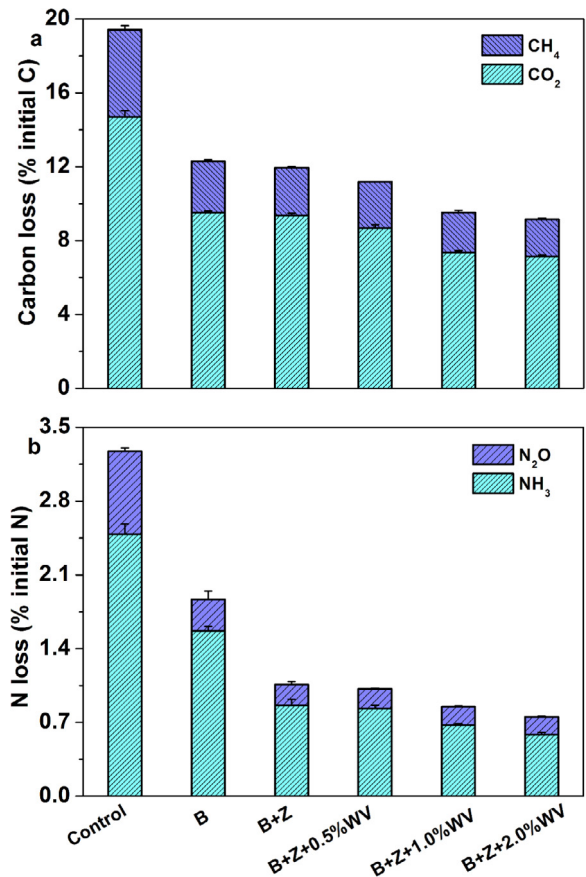


Fig. 5. Evaluation of carbon and nitrogen loss in different treatments at the end of composting. Results are the mean of three replicates and error bars indicate standard deviation.

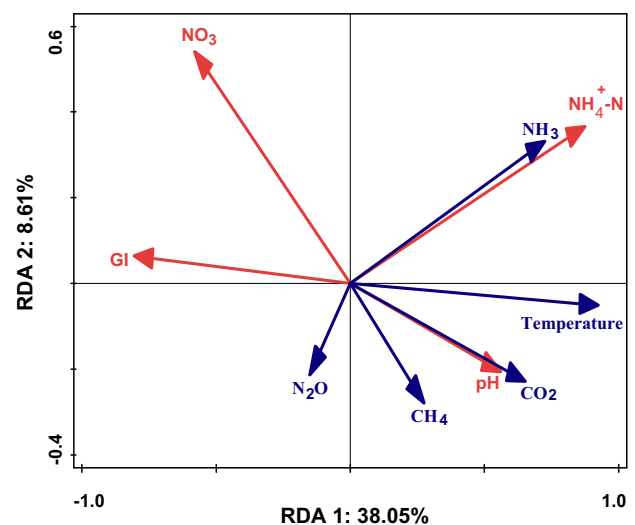


Fig. 6. Redundancy analysis (RDA) of physiochemical properties, greenhouse gas, ammonia emissions and germination index during the composting.

ous organic acids that could react with ammonium ions and reduce ammonia production (Chen et al., 2010). The initially high  $\text{NH}_3$  discharge could have been due to the rapid degradation of organic nitrogen and the higher temperature and pH (Li et al., 2015; Chan et al., 2016). After the first week,  $\text{NH}_3$  emissions were very low in the B, B + Z and all B + Z + WV treatments, but still relatively



high in the control, which could be explained by the longer thermophilic phase of the latter (Fig. 1). During the maturation phase,  $\text{NH}_3$  emissions in all treatments were nearly undetected because of the temperature decline and stabilization of organic matter. The  $\text{NH}_3$  emissions profile of our study was in line with that of Jiang et al. (2016a), who used nitrification inhibitor and struvite salts to reduce  $\text{NH}_3$  emissions during PM composting. Compared to the control (Fig. 3d), cumulative  $\text{NH}_3$  emissions were reduced by 35.88, 63.40, 64.45, 71.06, and 74.32% for the B, B + Z, B + Z + 0.5% WV, B + Z + 1.0% WV, and B + Z + 2.0% WV treatments, respectively, while the difference in cumulative  $\text{NH}_3$  emissions from all treatments was significant ( $p < .05$ ). Many previous studies indicated that B could effectively adsorb the  $\text{NH}_3$  and  $\text{NH}_4^+\text{-N}$  during composting, possibly due to its porosity and large specific surface area (Khan et al., 2014; Lopez-Cano et al., 2016; Awasthi et al., 2016a). Thus, the treatment using B alone presented lower  $\text{NH}_3$  emissions than control, likely due to the high adsorption ability of the former. However, compared to the B + Z treatment,  $\text{NH}_3$  volatilization in treatment using B alone was significantly higher ( $p < .05$ ), possibly attributed to the fact that Z can also trap the ammonia and lower  $\text{NH}_3$  emissions during the bio-oxidative phase of composting (Chan et al., 2016). A similar result was also observed by Awasthi et al. (2016d), who used B and Z to improve SS composting. Additionally, compared to the B + Z treatment, cumulative  $\text{NH}_3$  emissions reduced by 3.0%, 20.91%, and 29.90%, for B + Z + 0.5% WV, B + Z + 1.0% WV, and B + Z + 2.0% WV treatments, respectively. The differences in  $\text{NH}_3$  emissions in the B + Z and B + Z + 0.5% WV treatments were not significant ( $p > .05$ ), while the B + Z + 1.0% WV and B + Z + 2.0% WV treatments showed clear ( $p < .05$ ) reductions in  $\text{NH}_3$  emissions. Since WV could neutralize  $\text{NH}_3$  and improve the  $\text{NH}_3$  adsorption capacity of B or Z (Chen et al., 2010; Wu et al., 2015), the B + Z + WV treatments had lower ammonia losses than the B + Z treatment did, and the minimum cumulative emissions came from the B + Z + 2.0% WV treatment (2.81 g).

#### 3.4. Effect of amendments on $\text{NH}_4^+\text{-N}$ and $\text{NO}_3^-\text{-N}$ transformation

The changes in  $\text{NH}_4^+\text{-N}$  content in all the treatments presented a similar trend (Fig. 4a); they rose sharply initially, and peak values were observed at 4d. This initial increase was attributed to the rapid mineralization of organic nitrogen and ammonification with increased temperature and pH (Li et al., 2012; Awasthi et al., 2016c). A similar trend was observed by Huang et al. (2004) and Awasthi et al. (2016a), who co-composted PM and SS amended with B. In our study, the peak values of  $\text{NH}_4^+\text{-N}$  were 3.63, 3.25, 3.68, 3.70, 5.74, and 6.38 g/kg for the control, B, B + Z, B + Z + 0.5% WV, B + Z + 1.0% WV, and B + Z + 2.0% WV treatments, respectively. The maximum content was obtained in B + Z + 2.0% WV treatment, which was significantly ( $p < .05$ ) higher than the others. Some previous studies indicated that a high pH would improve the transformation of  $\text{NH}_4^+\text{-N}$  to  $\text{NH}_3$  during thermophilic phase (Chen et al., 2010; Li et al., 2015; Wang et al., 2016b). In contrast, in this study, the addition of B and Z increased the pH value while also keeping the  $\text{NH}_4^+\text{-N}$  content high during the first 4 d, likely due to the large number of micropores, negative ions, and the large surface area of B and Z that could adsorb  $\text{NH}_3$  or  $\text{NH}_4^+\text{-N}$  (Chen et al., 2010; Li et al., 2015; Chan et al., 2016). The results were consistent with those found by Awasthi et al. (2016d), who reported that the combination of wheat straw biochar and Z could reduce nitrogen loss and GHG emissions the most. In addition, compared to the B + Z treatment, mixing WV with B and Z could further trap  $\text{NH}_3$  and result in a  $\text{NH}_4^+\text{-N}$  increase (Chen et al., 2010). The addition of 0.5% WV could also slightly ( $p > .05$ ) raise the  $\text{NH}_4^+\text{-N}$  content, and as the amount of WV increased, more organic acid was introduced to the mixture, consequently increasing the  $\text{NH}_4^+\text{-N}$  content in the B + Z + 1.0% WV and B + Z + 2.0% WV treatments (Wu et al.,

2015). After 6 d, the  $\text{NH}_4^+\text{-N}$  content in all treatments gradually decreased and leveled off. This decline could be due to the transformation of  $\text{NH}_4^+\text{-N}$  into  $\text{NO}_3^-\text{-N}$  by nitrobacteria and the stabilization of compost mass (Li et al., 2012; Jiang et al., 2014). At the end of the composting period, the  $\text{NH}_4^+\text{-N}$  content in all treatments was within the permissible value (0.4 g/kg), showing that all the final compost was mature (Bernal et al., 2009).

The variations in  $\text{NO}_3^-\text{-N}$  levels in all the treatments are shown in Fig. 4b. The initial  $\text{NO}_3^-\text{-N}$  concentrations of all treatments were low, possibly due to the high temperature and pH inhibiting the growth of nitrifying bacteria (Li et al., 2012; Wang et al., 2016a). A similar trend was reported by Jiang et al. (2014) for PM composting with a nitrogen bacterial agent and Li et al. (2012), who composted PM amended with bentonite. Thereafter, as the temperature and pH decreased, the  $\text{NO}_3^-\text{-N}$  content in all the treatments gradually increased until the end of the composting period, and the final  $\text{NO}_3^-\text{-N}$  levels in the control, B, B + Z, B + Z + 0.5% WV, B + Z + 1.0% WV, and B + Z + 2.0% WV treatments were 1.26, 1.24, 1.05, 0.95, 0.89, and 0.83 g/kg, respectively. Compared to the control, the  $\text{NO}_3^-\text{-N}$  content in the treatment using B was slightly lower, in line with the results achieved by Wang et al. (2013) for composting PM with B. Nevertheless, some researchers stated that the addition of B improve denitrification and increased the  $\text{NO}_3^-\text{-N}$  content (Khan et al., 2014; Li et al., 2015), but we achieved the opposite result, possibly due to the different properties of substrates and B used. In addition,  $\text{NO}_3^-\text{-N}$  content in the control was significantly ( $p < .05$ ) higher than in the B + Z treatment, indicating that the further addition of Z could inhibit nitrification, which was in accordance with the results obtained by Li et al. (2012) and Jiang et al. (2016b), who applied bentonite and struvite to PM composting. Additionally, compared to the B + Z treatment, the  $\text{NO}_3^-\text{-N}$  content decreased by 8.74%, 14.85%, and 20.81% for the B + Z + 0.5% WV, B + Z + 1.0% WV, and B + Z + 2.0% WV treatments, respectively, as the amount of WV used increased. WV contained high concentrations of organic acids that could inhibit the antimicrobial activity (Wu et al., 2015); therefore, the combined effect of B, Z, and WV could influence the nitrification of compost and decrease its nitrate content.

#### 3.5. Carbon and nitrogen loss through greenhouse gas and ammonia emissions

The loss of carbon and nitrogen at the end of the experiment, via cumulative  $\text{CO}_2$  +  $\text{CH}_4$  and  $\text{NH}_3$  +  $\text{N}_2\text{O}$  emissions, are presented in Fig. 5. In this study, the major carbon loss (7.13–14.69%) was in the form of  $\text{CO}_2$ , while 2.02–4.73% of initial total organic carbon (TOC) was lost in the form of  $\text{CH}_4$  (Fig. 5a) due to the partially anaerobic conditions in the composting matrixes (Luo et al., 2013). For all treatments, the loss of carbon through  $\text{CO}_2$  and  $\text{CH}_4$  ranged from 9.16 to 19.42% of initial TOC levels; this was slightly lower than the range of 11.4–22.5% reported by Chowdhury et al. (2014b), who used wood biochar and adjusted the aeration flow rate to reduce GHG and ammonia emissions while composting manure. The discrepancy between these studies could be due to differences in compost materials, experimental conditions, and moisture content (Yang et al., 2015; Wang et al., 2016a). On the other hand, the lowest carbon loss was observed in the B + Z + 2.0% WV treatment, and the carbon loss significantly ( $p < .05$ ) reduced by 57.29% compared to the control, possibly due to the low  $\text{CO}_2$  and  $\text{CH}_4$  emissions in B + Z + 2.0% WV treatment (Wolter et al., 2004). After 50d of composting, the total  $\text{NH}_3$  emissions of all the treatments amounted to 0.58–2.48% of initial total nitrogen (TN); this was similar to the result reported by Jiang et al. (2014), who co-composted PM with apple pomace, bentonite, and calcium superphosphate. Among the treatments, the B + Z + 2.0% WV treatment had the lowest cumulative  $\text{NH}_3$  emissions, with only 0.58% of



initial TN lost as  $\text{NH}_3$ .  $\text{N}_2\text{O}$  emissions are also a pathway of nitrogen loss during composting (Yang et al., 2015; Jiang et al., 2016b). The cumulative  $\text{N}_2\text{O}$  emissions from all treatments accounted for 0.16–0.79% of initial TN loss, although Luo et al. (2013) and Yang et al. (2015) observed a higher level (2.0–3.1% and 1.97–2.16%) of  $\text{N}_2\text{O}$ -N loss while composting PM and kitchen waste.

#### 4. Conclusion

The results indicated that the B + Z + WV treatment could significantly ( $p < .05$ ) reduce GHG and  $\text{NH}_3$  emissions compared to treatments using B alone and B + Z. Meanwhile, compared with the control, the levels of  $\text{NH}_3$ ,  $\text{CO}_2$ ,  $\text{CH}_4$ , and  $\text{N}_2\text{O}$  decreased by 35.88–74.32%, 26.06–46.98%, 41.73–61.15%, and 64.91–81.10%, respectively, for the B, B + Z, and B + Z + WV treatments. Among all treatments, B + Z + 2.0%WV treatment presented the lowest GHG emissions and nitrogen losses, with a significant correlation between temperature and pH profile, and maximum GI at the end of PM composting. Therefore, the combination of B + Z + 2.0% WV could potentially improve the PM composting and the quality of the end product by mitigating GHG emissions and conserving nitrogen.

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