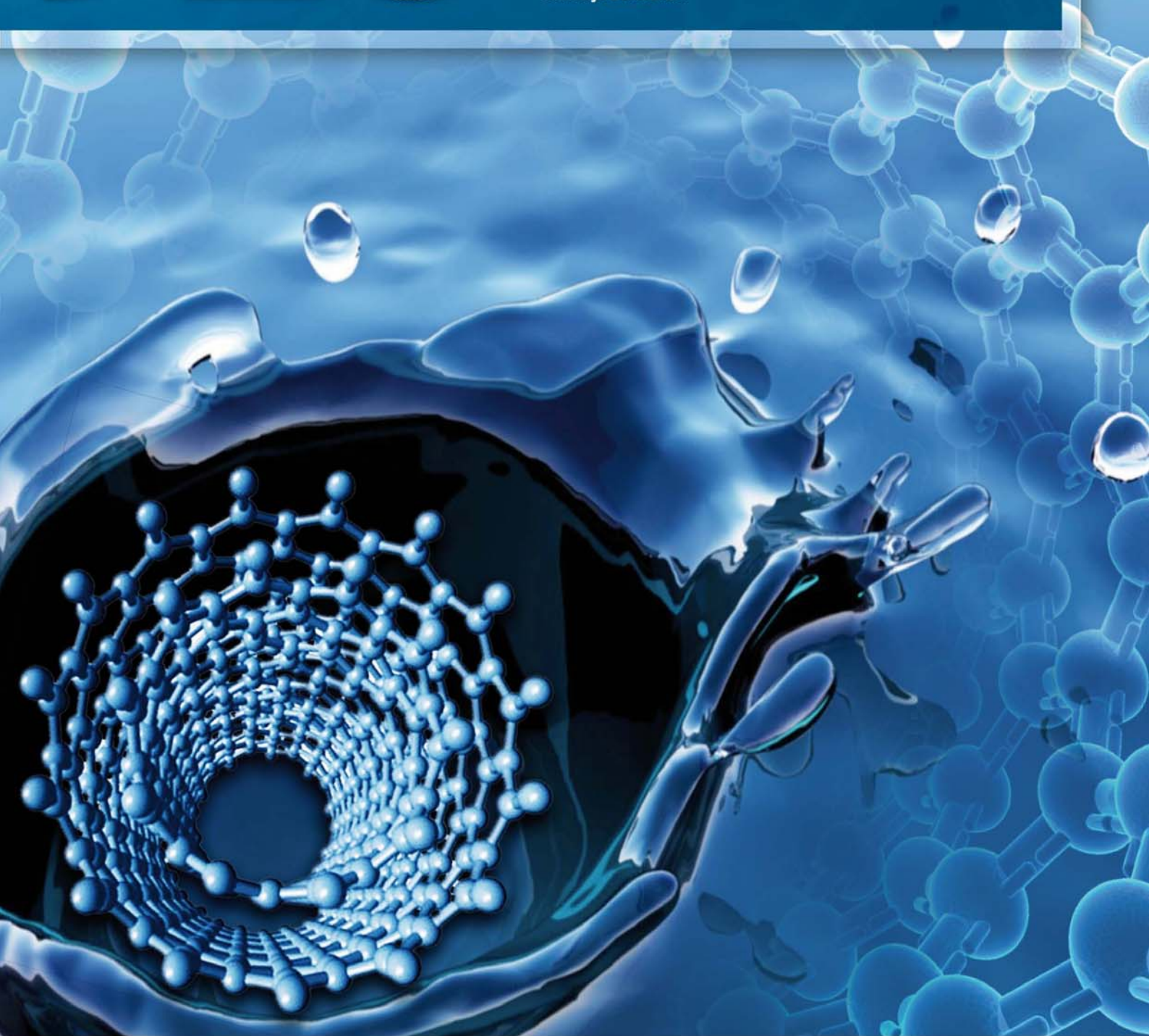


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Effect of phosphogypsum and dicyandiamide as additives on NH_3 , N_2O and CH_4 emissions during composting

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Abstract

A laboratory scale experiment of composting in a forced aeration system using pig manure with cornstalks was carried out to investigate the effects of both phosphogypsum and dicyandiamide (DCD, $\text{C}_2\text{H}_4\text{N}_4$) as additives on gaseous emissions and compost quality. Besides a control, there were three amended treatments with different amounts of additives. The results indicated that the phosphogypsum addition at the rate of 10% of mixture dry weight decreased NH_3 and CH_4 emissions significantly during composting. The addition of DCD at the rate of 0.2% of mixture dry weight together with 10% of phosphogypsum further reduced the N_2O emission by affecting the nitrification process. Reducing the phosphogypsum addition to 5% in the presence of 0.2% DCD moderately increased the NH_3 emissions but not N_2O emission. The additives increased the ammonium content and electrical conductivity significantly in the final compost. No adverse effect on organic matter degradation or the germination index of the compost was found in the amended treatments. It was recommended that phosphogypsum and DCD could be used in composting for the purpose of reducing NH_3 , CH_4 and N_2O emissions. Optimal conditions and dose of DCD additive during composting should be determined with different materials and composting systems in further study.

Key words: composting; phosphogypsum; dicyandiamide; greenhouse gas; pig manure

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Introduction

Composting is considered to be an effective way to treat livestock manure, which provides environmental benefit by utilizing nutrients and organic carbon in organic wastes. However, the high range of ammonia nitrogen losses (20% to 70% of initial nitrogen) during manure composting restricts the efficiency of nutrient recycling and causes air pollution (DeLaune et al., 2004; Shen et al., 2011). Studies have been conducted on reducing nitrogen losses in composting process by different kinds of amendments, including additives with phosphate, such as superphosphate and ground rock phosphate, to adjust the pH of composting materials (Termeer and Warman, 1993; Predotova et al., 2010).

Furthermore, emissions in the forms of nitrous oxide (N_2O) and methane (CH_4) during composting have become another focus of research in recent years, since

N_2O and CH_4 are powerful greenhouse gases with global warming potential of 298 and 25 compared to CO_2 over a 100 year period (IPCC, 2007). It was reported that about 0.2% to 6% of nitrogen in compost materials was lost as N_2O from nitrification or denitrification during the composting process (Beck-Friis et al., 2001; Szanto et al., 2007), and CH_4 emission amounted to 0.8% to 14% of the initial carbon depending on composting conditions (Hao et al., 2004; Wolter et al., 2004).

Limited research has been conducted on the effects of mineral additives on greenhouse gas emissions during composting. It was reported by Hao et al. (2005) that phosphogypsum, as the main byproduct of phosphoric acid production, might be effective to reduce the CH_4 emission during cattle manure composting due to the increased SO_4^{2-} content in the compost. On the other hand, dicyandiamide (DCD, $\text{C}_2\text{H}_4\text{N}_4$) as a nitrification inhibitor, was proved to be effective in reducing N_2O emission from cropland (Di et al., 2007; de Klein et al., 2011; Linquist et

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al., 2012). But for composting systems, research studies have been scarce. The addition of phosphogypsum and DCD might impact the gas emissions during composting by adjusting the acid radical ion content and shifting the equilibrium between nitrification and denitrification.

The objective of this study was to investigate the effect of phosphogypsum and DCD as amendments on the emission of ammonia and greenhouse gases from a pig manure composting system.

1 Materials and methods

1.1 Raw materials

Fresh pig feces from a pig farm using the Ganqingfen system (cleaning the manure dryly) (Schuchardt et al., 2011) in Beijing, China, were thoroughly mixed with chopped corn stalks (< 80 mm length) as structural materials at a ratio of 1/7 of manure wet weight. The characteristics of the raw materials are shown in **Table 1**. The phosphogypsum was obtained from a phosphate fertilizer plant in Hubei Province, China. DCD used in this study was chemically pure reagent grade.

1.2 Composting experiment

A series of composting vessels with a volume of 60 L each were used to simulate the forced aeration system (**Fig. 1**). The aeration rate was fixed at 0.48 L/(kg dry matter-min) and programmed intermittent aeration (start 25 min, stop 5 min) was used according to Jiang et al. (2011). Besides a control, three treatments T1, T2 and T3 were amended with different additions of phosphogypsum and DCD with regard to dry matter weight of the raw materials (T1: amended with 10% of phosphogypsum; T2: with 10% of phosphogypsum and 0.2% of DCD; T3: with 5% of phosphogypsum and 0.2% of DCD) with three replications. The phosphogypsum was added in treatments T1, T2 and T3 at the beginning. Half the mass of DCD (0.1% of mixture dry weight) was mixed with the raw materials at the beginning in treatments T2 and T3. The other half part was added during the turning activity on day 12. The composting process commenced on Dec 2, 2011, and was monitored for 30 days. The composting vessels were settled indoors and the piles were turned manually on day 6, 12, 18 and 24.

Table 1 Properties of pig feces and corn stalks.

	Moisture content (%)	TOC (g/kg DM)	TN (g/kg DM)	C/N
Pig feces	74.1 (0.09) ^a	375 (6.8)	27.7 (0.67)	13.5
Cornstalks	8.5 (0.05)	413 (2.3)	9.8 (0.08)	42.1

TOC: total organic carbon; TN: total nitrogen; DM: dry matter.

^a Values in parentheses are standard deviation ($n = 5$).

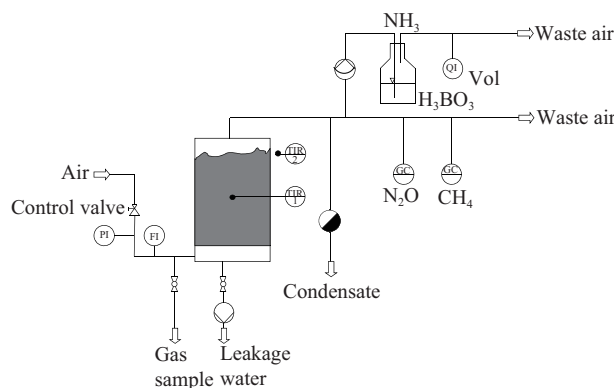


Fig. 1 Structure of composting system. PI: pressure indicator; FI: flow indicator; TIR: temperature information recorder; QI: quantity indicator; Vol: volume; GC: gas chromatograph.

1.3 Sampling and analyses

Temperature in the center of the composting material was measured and recorded by computerized online temperature sensors. Gas samples for greenhouse gases and ammonia were taken from the top of the vessels three times per day from 8:00 am to 2:00 pm. The ammonia emissions were measured by washing bottle with boric acid (2%) according to Ren et al. (2010). CH₄ and N₂O were analyzed by a gas chromatograph (3420A, Beifen, China), equipped with flame ionization detector and electron capture detector. The gas chromatograph parameters and analysis method were according to Jiang et al. (2011). The average flux obtained in the sampling hours was assumed to be representative for the whole day.

In addition to the start point and the end point, solid compost samples were taken during every turning for analyses. The moisture content was determined after drying at 105°C to constant weight. Total nitrogen (TN) and total organic carbon (TOC) was measured respectively by the Kjeldahl method and potassium dichromate titrimetric method according to the Chinese national standard of organic fertilizer (NY 525-2002). Masses of nitrogen and carbon were determined (N or C concentration × weight of DM), and the losses were calculated from the differences between the initial and the final masses (Tiquia et al., 2002; Ren et al., 2010). Ammonium nitrogen (NH₄⁺-N) and nitric-nitrogen (NO_x⁻-N) were analyzed by a segmented flow analyzer (Auto Analyzer 3, Seal, Germany) using 2 mol/L KCl extract (1:10, *m/V*). The pH, electrical conductivity (EC) and germination index (GI) were measured in 1:10 (*m/V*) compost water extract, and defined as the pH, EC and GI of the materials. *Brassica napus* seeds were used in GI tests to assess phytotoxicity of the compost, according to the method of Shen et al. (2011).

The mean and standard deviation of the three replicates were reported. Statistical Analysis System 8.1 for Windows was used for the variance analysis.

2 Results and discussion

2.1 Temperature and pH profile

The temperature of composting increases simultaneously with the decomposition of available organic materials by microorganisms during the composting process (Raut et al., 2008). As shown in **Fig. 2a**, the temperature of all the treatments increased quickly in the first 3 days and achieved the thermophilic phase ($> 55^{\circ}\text{C}$). The temperature profiles of the four treatments were similar. It was evident that the addition of phosphogypsum and DCD caused no adverse effect on the degradation of organic materials during composting. The thermophilic condition during composting was established easily and lasted for about two weeks. After the breakdown of available organic matter and nitrogenous compounds, the temperature of the piles declined gradually and approached ambient air temperature after 25 days.

The pH values of the four treatments remained at 7.5 to 8.2 during the composting process (**Fig. 2b**). The pH of all treatments increased in the first week, corresponding with the high ammonia emission rate, and then decreased, possibly due to the accumulation of organic acids in the piles. After 18 days, as the pile temperature declined,

the pH values of the four treatments increased slightly in accord with the high content of NH_4^+ in the compost and remained within a certain range. Compared with the control, adding 10% of phosphogypsum (based on mixture dry matter) decreased the pH value significantly in treatment T1. Similar results were reported by Hao et al. (2005) with an addition rate at 10% to 30% of manure dry matter. Adding DCD increased the pH value of compost in the treatments T2 and T3, compared to T1. It was supposed that DCD had an inhibitory effect on nitrification, which could be responsible for the higher NH_4^+ content, leading to the higher pH. The final compost had a pH level of 7.7 to 8.0 for the four treatments; statistical analysis results showed that there were significant differences between treatments control and T1, control and T2, T1 and T3, respectively ($p < 0.05$), but no significant difference between the others.

2.2 Gaseous emissions

The cumulative emissions of NH_3 , CH_4 and N_2O of different treatments during composting are shown in **Fig. 3**. In the first two weeks, all the treatments had clear NH_3 emission peaks. It was known that high temperature could increase the NH_3 emission (Pagans et al., 2006), but it was not the only definitive factor when the NH_4^+ absorbance

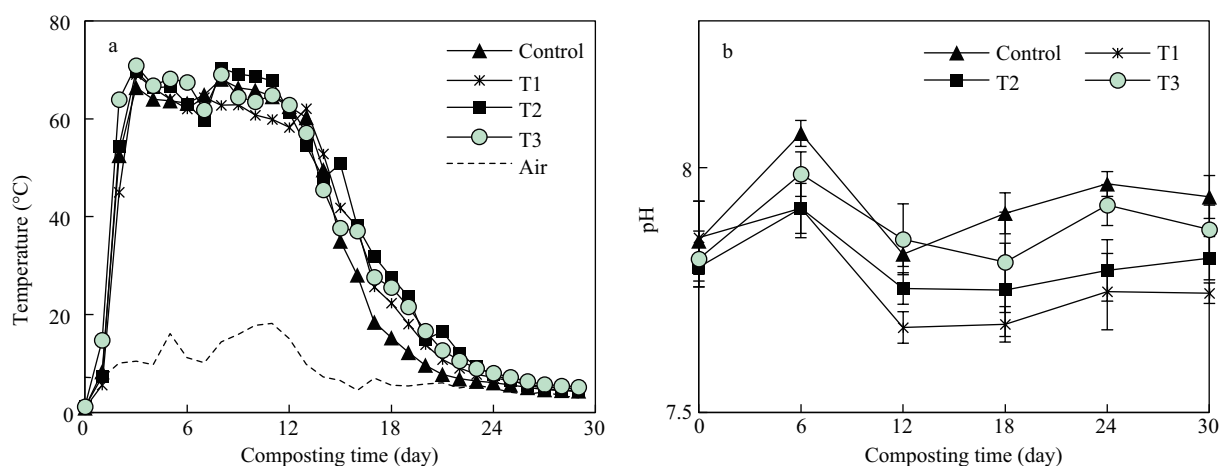


Fig. 2 Changes of temperature (a) and pH (b) during composting. T1: 10% phosphogypsum; T2: 10% phosphogypsum + 0.2% DCD; T3: 5% phosphogypsum + 0.2% DCD.

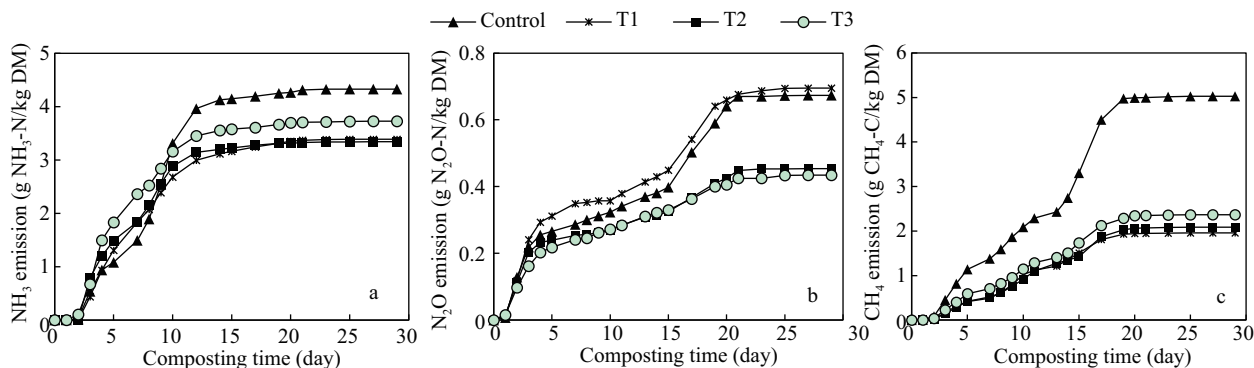
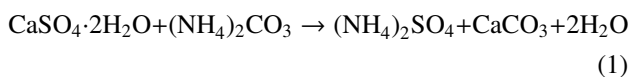


Fig. 3 Cumulative emissions of NH_3 (a), N_2O (b) and CH_4 (c).

of piles was modified by amendments, structure additives, pH value or frequency of turning, etc (Mahimairaja et al., 1994; Beck-Friis et al., 2001; Pagans et al., 2006; El Kader et al., 2007). Adding phosphogypsum reduced the NH₃ emission in treatment T1 during the whole composting process. In treatments T2 and T3, with the addition of DCD at the beginning and day 12, higher NH₃ emissions compared to control were detected on day 3 and 4 possibly due to the higher NH₄⁺ content in these treatments. It was reported in previous studies that the application of DCD led to increased NH₃ emission from arable land because of the additional nitrogen in DCD and the inhibitory effect on nitrification (Zaman and Blennerhassett, 2010; Soares et al., 2012). Part of the surplus NH₄⁺ was released in the form of free NH₃, as the temperature and pH value were high in the first week. In the second week, the peak values of the amended treatments became significantly lower than the control mainly because of the adjusted pH value and the enlargement of the ammonium retention capacity of the composting materials by phosphogypsum addition. As the main ingredients of phosphogypsum are CaSO₄·2H₂O and a small amount of free phosphoric acid, the simplified chemical reactions could be represented by Eqs. (1) (Burnett et al., 1996) and (2) (Hu et al., 2007).



As shown in **Fig. 3a**, adding DCD on day 12 made no significant difference to the NH₃ volatilization rate in the following week in treatments T2 and T3. After 15 days, the NH₃ emission flux of all treatments remained at a low level (below 0.05 g NH₃-N/(kg dry matter·day)) till the end. Compared to the control, the total NH₃ losses of treatments T1, T2 and T3 after 30 days were respectively reduced by 21.7%, 22.8% and 13.9%. It was indicated that addition of phosphogypsum decreased the NH₃ emission during composting, and the addition of DCD made no significant difference to cumulative NH₃ losses in this study.

N₂O emission, as the result of nitrification and denitrification, took place during composting. High accumulations of N₂O emission for all treatments were observed in the first 4 days and during the cooling phase (day 15 to day 24) of composting (**Fig. 3b**). The high N₂O emission that occurred at the beginning of composting was presumed to be derived from nitrification of ammonium in the raw materials while the pile temperature was suitable for nitrobacteria (El Kader et al., 2007; Fukumoto et al., 2003), and released gradually during the next days. It was reported that the activity of nitrobacteria might be suppressed while the temperature was above 40°C (Andersen et al., 2011; Fukumoto et al., 2003). A similar tendency was found in this study. As the pile temperature climbed up, the N₂O emission rate of all four treatments declined. After two

weeks, while the pile temperature decreased to around 40°C, the cumulative N₂O emission increased sharply in treatments control and T1. Adding phosphogypsum made no significant difference to N₂O emission during composting. On the other hand, the addition of DCD in treatments T2 and T3 brought about significant reduction of N₂O emission, especially during the cooling phase of composting. According to the previous studies, DCD could inhibit the metabolic turnover of *Nitrosomonas* bacteria (Zacherl and Amberger, 1990), which are responsible for the conversion of NH₄⁺ to NO₂⁻, and slow the nitrification process in soil by interfering with the cytochrome oxidase in the respiratory electron transport system of *Nitrosomonas* bacteria (O'Connor et al., 2012). O'Callaghan et al. (2010) stated that DCD did not have a significant effect on bacterial populations or ammonium-oxidizing archaea in agricultural soil, but the ammonium-oxidizing bacteria were significantly affected, with reductions in population size and altered activity. Nicol et al. (2008) noted that the pattern of changing ammonia oxidizer bacterial gene copies and transcriptional levels correlated more closely with nitrification rates, which indicated the dominant role of ammonium-oxidizing bacteria in ammonia oxidation. The reduced activity of ammonium-oxidizing bacteria in composting materials and the retarded rate of nitrification affected by DCD additives were supposed to be the reason for N₂O emission reduction in treatments T2 and T3. The statistical analysis *t*-test results of N₂O emission flux during 30 days showed significant differences between control and T2 (*p* = 0.008), control and T3 (*p* = 0.029). The total N₂O losses of treatments T2 and T3 were reduced by 33% to 35% compared to the control. Kelliher et al. (2008) found that the degradation rate of DCD in soils was accelerated in high temperature conditions. It was evident that the effect of DCD in composting process lasted for at least 8 days (from day 12 to day 20), since the DCD was added in treatments T2 and T3 both at the beginning of composting and on day 12. Further studies would be needed to determine the degradation pattern of DCD in different composting conditions.

Corresponding to the decomposition rate of available organic matter, most of the CH₄ emissions were released in 20 days (**Fig. 3c**). The CH₄ emission tendency was similar in the four treatments. High CH₄ emission fluxes were detected on days 3, 9, 15 and 17 in all treatments, which were 3 to 5 days after turning. The decrease of oxygen content in the piles and the high moisture content of composting materials should be responsible for the high CH₄ emission. Similar results were found by Kuroda et al. (1996) and Fukumoto et al. (2003). It was reported in previous studies that sulfur compounds could have a toxic effect on methanogens (Hao et al., 2005; Linquist et al., 2012). Pennock et al. (2010) also found that the annual CH₄ emissions from a freshwater wetland were significantly inversely correlated with the mean SO₄²⁻

concentration in the water. Possible mechanisms related to the suppression effect of sulfate on methanogenesis were summarized by Segers (1998). Sulfate-reducing bacteria could compete for the same organic carbon and energy source with methanogens, which would result in a low substrate concentration insupportable for methanogenesis, or the presence of SO_4^{2-} could lead to an improper high redox potential for methanogenesis in spite of its toxicity for methanogens. Furthermore, the high concentration of NH_4^+ could decrease the activity of methanogens (Sung and Liu, 2003). Adding phosphogypsum reduced CH_4 emissions significantly in all three amended treatments in this study. The possible reason was the high content of SO_4^{2-} in phosphogypsum (Hao et al., 2005) and the increased NH_4^+ concentration in amended treatments compared to the control. The statistical analysis *t*-test results of CH_4 emission flux showed significant differences between the control and the amended treatments ($p < 0.003$), but no significant difference was found among treatments T1, T2 and T3. The total CH_4 losses of treatments T1, T2 and T3 were reduced by 61.1%, 58.5% and 52.9%, respectively, compared to the control.

Overall, the gaseous emission results indicated that the addition of phosphogypsum was effective to reduce the NH_3 and CH_4 emissions during pig manure composting with cornstalks as bulking material. The same tendency was found in other composting processes with different raw materials such as cattle manure (Hao et al., 2005). Gabhane et al. (2012) reported that adding 5% of phosphogypsum in green waste (consisting of grass cuttings and fallen leaves collected from a garden area) on a dry weight basis for composting resulted in a decreased pH value for the composting materials, which could lead to a reduction of NH_3 emission. But the CH_4 emission had not been determined in their study. It was supposed that the phosphogypsum additive affected the NH_3 and CH_4 emissions in composting processes generally because of its acidic nature and the SO_4^{2-} concentration, which might not be easily affected by different raw materials. On the other hand, few previous studies have been conducted

to investigate the impacts of DCD on gaseous emissions in composting processes, although it was found to be effective in decreasing the N_2O emission from cropland in different conditions, such as a grazed pasture system with deposited animal urine (Smith et al., 2008; Zaman and Blennerhassett, 2010), irrigated soil with application of pig slurry (Vallejo et al., 2005) or flooded rice system (Linguist et al., 2012). It was determined that DCD had a toxic effect on nitrifying bacteria (Zacherl and Amberger, 1990), which was correlated to N_2O emission. Results in this study showed that DCD might be able to be used in pig manure composting for the purpose of reducing N_2O emission. But in view of the limited research that has carried out in composting systems, further studies are required to quantify the effect of DCD on gaseous emissions from different composting materials and different composting systems.

2.3 Chemical conditions and germination index

As shown in **Table 2**, after 30 days, the TOC of compost in the four treatments were around 310 g/kg DM. The TN in the final compost of treatments T2 and T3 were significantly higher than the control due to the additional nitrogen in the DCD additives. The addition of phosphogypsum reduced the TN mass losses in the amended treatments mainly due to the decreased ammonia emissions, but no significant difference of TN per kg DM was found between the treatments control and T1. During the composting process, the C/N ratio of the four treatments decreased from 18 to about 12.

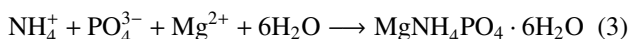
The NH_4^+ content in compost usually increases at the beginning due to the conversion of organic N to $\text{NH}_4^+\text{-N}$ via ammonification, and decreases afterward with the further conversion of $\text{NH}_4^+\text{-N}$ to NH_3 or $\text{NO}_x^-\text{-N}$ (Tumuhairwe et al., 2009; de Guardia et al., 2010). Similar results were obtained in this study, but adding phosphogypsum made the NH_4^+ content of final compost in treatments T1, T2 and T3 significantly higher than the control. It was indicated that the addition of phosphogypsum might have enlarged the capacity of the compost material to adsorb NH_4^+

Table 2 Characteristics of initial and final compost materials

		Control	T1	T2	T3
TOC (g/kg DM)	Initial	387.6 (8.5) a	367.2 (3.6) b	365.3(4.4) b	374.0 (7.6)ab
	Final	309.3 (8.4) a	312.4 (10.2) a	313.1 (7.9) a	308.0 (5.5) a
TN (g/kg DM)	Initial	21.7 (0.7) a	19.6 (1.3) a	20.6 (1.1) a	21.0 (0.6) a
	Final	24.5 (0.6) b	26.2 (0.9) ab	27.6 (1.1) a	27.1 (0.9) a
$\text{NH}_4^+\text{-N}$ (g/kg DM)	Initial	0.81 (0.13) b	1.07 (0.11) b	1.58(0.04) a	1.45 (0.25) a
	Final	1.19 (0.18) b	3.81 (0.21) a	3.77 (0.47) a	3.44 (0.17) a
$\text{NO}_x^-\text{-N}$ (g/kg DM)	Initial	0.05 (0.01) a	0.04 (0.02) a	0.03 (0.01) a	0.04 (0.01) a
	Final	0.23 (0.04) a	0.27 (0.03) a	0.09 (0.02) b	0.11 (0.03) b
EC (mS/cm)	Initial	2.23 (0.11) c	2.92 (0.01) a	2.87 (0.03) a	2.60 (0.04) b
	Final	1.71 (0.01) c	2.79 (0.08) a	2.78 (0.05) a	2.29 (0.05) b
GI (%)	Initial	22.1 (2.8) b	43.2 (7.3) a	45.4 (4.0) a	37.1 (11.0) a
	Final	100.7 (6.7) a	105.9 (6.5) a	112.4 (9.8) a	107.9 (8.9) a

EC: electrical conductivity, GI: germination index. Values in parentheses are standard deviation ($n = 3$); values followed by different letters within a horizontal line differ significantly at the 0.05 probability level.

and maintain the equilibrium between NH₃ and NH₄⁺ at a proper pH value. Furthermore, the phosphogypsum additive could contribute to the binding states of NH₄⁺ with phosphate radical, or other stable compounds (e.g. struvite, reflected in Eq. (3)) (Jeong and Kim, 2001; Ren et al., 2010).



To the contrary, the NO_x⁻-N ((NO₃⁻+NO₂⁻)-N) content reached the maximum at the end, and the NO_x⁻-N content of the final compost in treatments control and T1 without DCD addition were significantly higher than other treatments. The NO_x⁻-N content results confirmed that adding DCD affected the nitrification process during composting in this study.

Adding phosphogypsum significantly increased EC of the compost, which reflected the salinity levels (Table 2). But this would not be a problem since Ca²⁺ in phosphogypsum should be the reason for a high EC level, and applying compost with Ca²⁺ may be beneficial for soil (Hao et al., 2005; Himanen and Hänninen, 2009). The phosphogypsum and DCD as additives of composting caused no adverse effects on GI in this study. The GI of the four treatments was higher than 100% after 30 days, which was generally considered to indicate that the phytotoxicity was minimal (Bernal et al., 2009).

2.4 Nitrogen and carbon mass balances

Adding phosphogypsum decreased the total nitrogen losses in amended treatments mainly by reducing the ammonia emissions (Table 3). For all treatments, after 30 days composting, about 68% to 72% of TN losses were in the form of NH₃-N emission, and about 6% to 10% losses as N₂O-N emission; the rest were losses that could not be detected, including a small amount of N₂ (Andersen et al., 2011) and the possible error in analyses or cumulative emissions estimation from discontinuous measurements (El Kader et al., 2007). The N₂O emissions in treatments T2 and T3 with the DCD addition were about 2% of initial total nitrogen, lower than other treatments in this study and the previous studies by Szanto et al. (2007) and Jiang et al. (2011) under similar composting conditions. On the other hand, the differences in total organic carbon losses among the four treatments were small. All treatments lost

more than 55% of TOC during composting. About 1% to 3% of TOC losses were in the form of CH₄, and other losses were mainly CO₂ (Wolter et al., 2004; Jiang et al., 2011). Similar to the research results of Hao et al. (2005), the amount of CH₄ emitted during composting accounted for less than 1% of initial carbon in the phosphogypsum-amended treatments in this study.

3 Conclusions

The effects of phosphogypsum and DCD as additives on the emissions of greenhouse gases and ammonia during composting of mixtures of pig manure and cornstalks were investigated. The addition of phosphogypsum affected the emissions of both NH₃ and CH₄ during composting. Lower NH₃ emissions and higher NH₄⁺ content were detected in the treatments with phosphogypsum additives than in the control. There was a significant decrease in CH₄ emissions with phosphogypsum addition rate at 5% and 10% of mixture dry matter, but no significant effect on the emission flux of N₂O throughout the composting process in this study. Moreover, adding DCD at the rate of 0.2% of mixture dry matter brought about a remarkable reduction of N₂O emission during composting (especially during the cooling process of composting) in this study. It was concluded that DCD had the potential to be used in composting for the purpose of reducing N₂O emissions. Further study is needed to determine the degradation pattern of DCD in different composting conditions.

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Table 3 Total nitrogen and total organic carbon losses

	Control	T1	T2	T3
TN losses (%)	40.4	32.7	31.6	34.2
NH ₃ -N emission (%)	29.1	22.8	21.6	24.6
N ₂ O-N emission (%)	3.1	3.2	2.0	2.0
Other N losses (%)	8.1	6.6	8.0	7.6
TOC losses (%)	58.4	56.6	56.3	58.0
CH ₄ -C emission (%)	1.6	0.6	0.6	0.7
Other C losses (%)	56.9	56.1	55.7	57.3

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