

# Nitrogen Isotope Compositions of Synthetic Fertilizer, Raw Livestock Manure Slurry, and Composted Livestock Manure

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To investigate the difference in N isotope ratio ( $^{15}\text{N}/^{14}\text{N}$ , expressed as  $\delta^{15}\text{N}$ ) among N sources (synthetic fertilizer, livestock manure, and manure compost), eight synthetic fertilizer, four livestock manure, and thirty-seven compost samples were collected and analyzed for  $\delta^{15}\text{N}$ . The mean  $\delta^{15}\text{N}$  values of N sources were  $-1.5 \pm 0.5\text{‰}$  (range:  $-3.9$  to  $+0.5\text{‰}$ ) for synthetic fertilizer,  $+6.3 \pm 0.4\text{‰}$  ( $+5.3$  to  $+7.2\text{‰}$ ) for manure, and  $+16.0 \pm 0.4\text{‰}$  ( $+9.3$  to  $+20.9\text{‰}$ ) for compost. The lower  $\delta^{15}\text{N}$  of synthetic fertilizer was attributed to its N source, atmospheric  $\text{N}_2$  of which  $\delta^{15}\text{N}$  is  $0\text{‰}$ . Meanwhile, more  $^{15}\text{N}$ -enrichment of compost than manure was assumed to be resulted from N isotopic fractionation (faster loss of  $^{14}\text{N}$ -bearing compound than  $^{15}\text{N}$ ) associated with N loss particularly via  $\text{NH}_3$  volatilization during composting. Therefore, our study shows that  $\delta^{15}\text{N}$  values could successfully serve in discriminating two major N sources (synthetic fertilizer and compost) in agricultural system.

**Key words:** Livestock manure compost, Nitrogen isotope ratio, Nitrogen isotopic fractionation, Nitrogen source, Synthetic fertilizer

## Introduction

The N isotope ratio ( $^{15}\text{N}/^{14}\text{N}$ , expressed as  $\delta^{15}\text{N}$ ) of nitrogenous compounds have been intensively used in tracing the fate of synthetic fertilizer in soil (Broadbent et al., 1980; Karamanos and Rennie, 1980; Karamanos and Rennie, 1981a, b), plant (Kohl et al., 1973; Meints et al., 1975; Shearer and Legg, 1975; Broadbent et al., 1980), and groundwater (Kreitler, 1979; Flipse and Bonner, 1985; Komor and Anderson, 1993; Feast et al., 1998). These works are possible due to considerably lower  $\delta^{15}\text{N}$  values of synthetic fertilizer than those of natural soil N. Since synthetic N fertilizer is produced through the Haber-Bosch process, which uses atmospheric  $\text{N}_2$  (the  $\delta^{15}\text{N}$  is  $0\text{‰}$ ) as the reactant, the  $\delta^{15}\text{N}$  values of synthetic fertilizer are close to  $0\text{‰}$ . For groundwater, contribution of livestock feedlot to  $\text{NO}_3^-$  contamination has been estimated through

$\delta^{15}\text{N}$  investigation as  $\delta^{15}\text{N}$  values of livestock manure N are higher than those of soil-N via N isotope fractionation (faster reaction of  $^{14}\text{N}$ -bearing compounds than  $^{15}\text{N}$ -bearing N compounds) during N loss such as ammonia volatilization (Gormly and Spalding, 1979; Heaton, 1984).

More recently, the use of the  $\delta^{15}\text{N}$  of plant tissues as a potential technique to identify the use of synthetic fertilizer in cultivating has been extensively studied in an effort to develop a technique for certification of organic produce that was grown without application of synthetic fertilizers (e.g., Choi et al., 2002; 2003; 2006; Bateman et al., 2005). The principle of this technique is quite similar to that used in identifying  $\text{NO}_3^-$  contamination sources of groundwater (Choi et al., 2007); the  $\delta^{15}\text{N}$  of synthetic N fertilizer manufactured from atmospheric  $\text{N}_2$  using the Haber-Bosch process is usually lower than that of organic source of N fertilizers (e.g. livestock manure and its compost) that can be used in organic farming (Choi et al., 2003). However, up to now, there is limited information on the distribution of  $\delta^{15}\text{N}$  amongst synthetic fertilizer, manure, and compost that is basic data for utilization of  $\delta^{15}\text{N}$  in

identification of N sources (Bateman and Kelly, 2007). In this study,  $\delta^{15}\text{N}$  values of different N sources (synthetic fertilizer, manure, and compost) were investigated to confirm their differences in N isotopic compositions.

## Materials and Methods

**Sample collection** Urea  $[(\text{NH}_2)_2\text{CO}]$ , ammonium sulfate  $[(\text{NH}_4)_2\text{SO}_4]$ , and six types of compound fertilizers (N-P<sub>2</sub>O<sub>5</sub>-K<sub>2</sub>O ratio: 11-12-10, 12-8-12, 13-0-13, 15-15-15, 16-0-12, and 21-17-17) were collected. Four livestock manure samples (two pig, one cattle, and one poultry) were taken from the livestock feedlots around Gyonggi and Chonnam Provinces. Thirty-seven samples of commercially available composts were collected from compost manufacturing factories. Manure type of the compost samples are provided with their N concentration and  $\delta^{15}\text{N}$  in Table 1.

**Chemical analyses** Synthetic fertilizers were digested by permanganate-reduced iron modification of Kjeldahl method to include nitrate and nitrite and steam-distilled with 10 N NaOH (Bremner, 1996). The liberated  $\text{NH}_3$  was

trapped in 0.01 N  $\text{H}_2\text{SO}_4$  and titrated with 0.01 N NaOH to determine the amount of  $\text{NH}_4^+$ . The pH of the titrated solution was adjusted to 2–3 using 0.1 N  $\text{H}_2\text{SO}_4$  or 0.1 N NaOH, and then water in the solution was evaporated to dryness under an infra-red lamp (Hauck, 1982). Nitrogen gas was prepared through the reaction of  $(\text{NH}_4)_2\text{SO}_4$  and alkaline LiOBr under vacuum (Hauck, 1982). The  $\delta^{15}\text{N}$  values of  $\text{N}_2$  were determined using a stable isotope ratio mass spectrometer of dual inlet type (VG OPTIMA, Micromass, UK).

The manure slurry samples were passed through a Whatman #42 filter paper under suction to remove coarse solid particles. The samples were processed following the same procedure as synthetic fertilizer analysis.

Composts samples were dried with a freeze drier to avoid  $\delta^{15}\text{N}$  changes via N isotopic fractionation associated with  $\text{NH}_3$  volatilization during drying and the dried samples were ground to a fine powder with a ball mill. The N concentrations and the corresponding  $\delta^{15}\text{N}$  values of the compost samples were determined using a continuous-flow stable isotope ratio mass spectrometer linked with a CN analyzer (IsoPrime-EA, Micromass, UK).

Nitrogen isotope compositions were calculated as

**Table 1. Manure type, N concentration, and  $\delta^{15}\text{N}$  of livestock manure composts (n=37).**

ID	Manure type	N concentration (%)	$\delta^{15}\text{N}$ (‰)	ID	Manure type	N concentration (%)	$\delta^{15}\text{N}$ (‰)
1	Pig	1.44	+16.7	21	Pig, Cattle	1.46	+15.8
2	Poultry, Pig	2.10	+17.4	22	Pig, Cattle	1.45	+19.8
3	Pig	1.57	+17.7	23	Pig	1.14	+17.9
4	Pig	1.09	+18.2	24	Pig	1.20	+19.2
5	Pig	1.88	+16.8	25	Pig	1.23	+20.9
6	Poultry, Pig	2.45	+16.6	26	Pig, Cattle	1.60	+16.6
7	Pig	1.35	+15.4	27	Pig, Cattle	1.71	+15.2
8	Poultry, Pig	2.69	+18.8	28	Poultry, Pig	3.20	+12.0
9	Pig, Cattle	1.91	+19.4	29	Poultry, Pig	3.50	+9.3
10	Pig	1.88	+17.3	30	Pig, Cattle	1.60	+13.7
11	Pig, Cattle	2.09	+17.2	31	Pig, Cattle	1.80	+13.8
12	Pig, Cattle	2.15	+14.8	32	Pig	1.50	+16.6
13	Pig	1.66	+14.8	33	Pig, Cattle	1.90	+15.8
14	Pig	1.51	+16.0	34	Pig, Cattle	2.30	+14.6
15	Poultry, Pig	2.41	+17.2	35	Pig	1.40	+13.8
16	Pig	1.04	+15.3	36	Pig, Cattle	1.80	+17.2
17	Pig, Cattle	1.51	+14.5	37	Pig, Cattle	1.90	+15.1
18	Pig	1.19	+12.3				
19	Pig	1.06	+18.9				
20	Pig, Cattle	1.89	+10.0				

$$\delta^{15}\text{N} (\text{‰}) = [(R_{\text{sample}}/R_{\text{standard}}) - 1] \times 1000$$

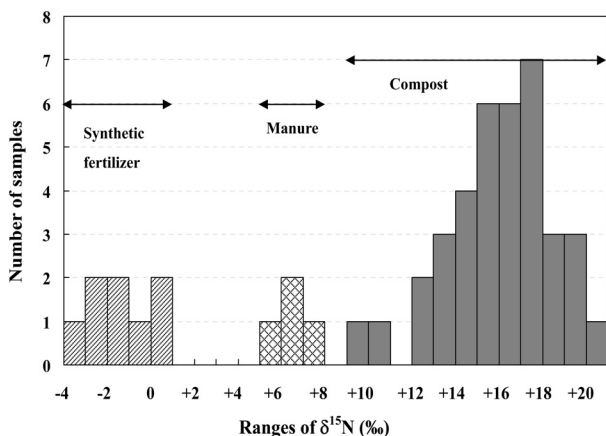
where  $R_{\text{sample}}$  and  $R_{\text{standard}}$  are the ratio of  $^{15}\text{N}/^{14}\text{N}$  for sample and standard (atmospheric  $\text{N}_2$ ), respectively. The accuracy and reproducibility of measurement checked with a reference material (RM 8548: IAEA-N2) from International Atomic Energy Agency were better than 0.4 and 0.2‰, respectively. All the chemical analyses were triplicated for each sample.

**Statistical analysis** Statistical analysis was carried out with PASW Statistics (SPSS Inc. Chicago, IL). We report the range, frequency distribution, and means (with standard errors) of  $\delta^{15}\text{N}$  values without statistically based comparison due to the inherent limitation of the study design (pseudo-replicated samples). Pearson correlation analysis was used to examine the relationship between concentration and  $\delta^{15}\text{N}$  of compost N.

## Results and Discussion

The mean values of  $\delta^{15}\text{N}$  of synthetic fertilizer, manure, and compost were  $-1.5 \pm 0.5\text{‰}$  (ranged from -3.9 to +0.5‰),  $+6.3 \pm 0.4\text{‰}$  (from +5.3 to +7.2‰), and  $+16.0 \pm 0.4\text{‰}$  (from +9.3 to +20.9‰), respectively. Therefore, the  $\delta^{15}\text{N}$  of three sources were clearly separated each other without overlapping; synthetic fertilizers lower than +1.0‰, manures between +5.0 and +8.0‰, and composts over +9.0‰ (Fig. 1). Around 50% of the 37 compost samples had  $\delta^{15}\text{N}$  between +15.0 and +18.0‰ (Fig. 1).

The  $\delta^{15}\text{N}$  values of synthetic fertilizers (-3.9 ~ +0.5‰) observed in this study were in good agreement with other studies. Overall, the  $\delta^{15}\text{N}$  values for synthetic fertilizer are

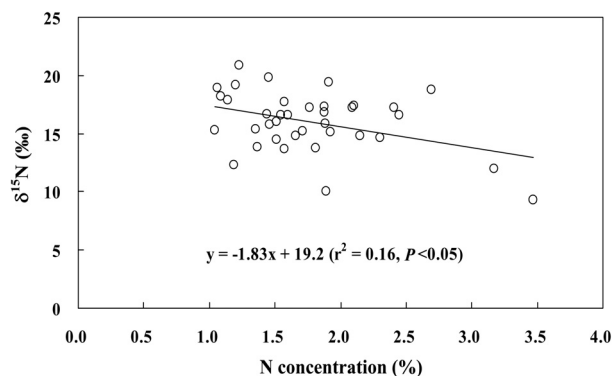


**Fig. 1.** Histograms of  $\delta^{15}\text{N}$  values of synthetic fertilizer (n=8), manure (n=4), and compost N (n=37). Arrows show the observed range of each N source.

between -8.0 and +6.2‰ with 90% ranging from -3.0‰ to +2.0‰ (Freyer and Aly, 1974; Kreitler, 1977; Mariotti and Letolle, 1977; Kreitler et al., 1978; Bateman and Kelly, 2007). Such a relatively low  $\delta^{15}\text{N}$  of synthetic fertilizer reflects their N source; atmospheric  $\text{N}_2$  which has a  $\delta^{15}\text{N}$  value of 0‰, and the differences in  $\delta^{15}\text{N}$  among fertilizers are generally considered a result of fractionation during subsequent processing of the N fixed through the Haber-Bosch process (Flipse and Bonner, 1985).

The  $\delta^{15}\text{N}$  of cattle and pig manure slurry ranging from +5.3 to +7.2‰ suggests that the manure samples have been experienced with N loss (particularly via  $\text{NH}_3$  volatilization) to some degree that causes N isotopic fractionation as original N excreted from animal is depleted in  $^{15}\text{N}$ , while N incorporated into protein retaining in animal body is enriched in  $^{15}\text{N}$  (Kreitler, 1975; Gormly and Spalding 1979). For examples, Kreitler (1975) reported that  $\delta^{15}\text{N}$  of the original urea in the excreted N was -3.5‰. Gormly and Spalding (1979) have also shown that  $\delta^{15}\text{N}$  of fresh animal manure was not different from  $\delta^{15}\text{N}$  of soil- or fertilizer-derived N (<+5.0‰). Because of high urea-N concentration (accounts for up to 50% of total N) in livestock manure, volatilization of  $\text{NH}_3$  following urea hydrolysis is the predominant pathway of N loss from manure after excretion (Kerley and Jarvis, 1996). It has been shown that the ratio of  $^{14}\text{NH}_3$  to  $^{15}\text{NH}_3$  volatilization rate is between 1.020 and 1.029, indicating that volatilization of  $^{14}\text{NH}_3$  is faster than  $^{15}\text{NH}_3$  by 2.0 to 2.9% (adopted from Chang and Choi, 2009). Choi (2002) demonstrated  $^{15}\text{N}$  enrichment of manure slurry by  $\text{NH}_3$  volatilization through a laboratory incubation study with human, pig, and cattle manure;  $\delta^{15}\text{N}$  of manure N increased from +11.8 to +16.7‰ for human manure, +10.3 to +51.4‰ for pig manure, and +7.2 to +33.1‰ for cattle manure with decreasing  $\text{NH}_4^+$  concentration in the manure samples.

Enrichment of  $^{15}\text{N}$  in N derived from manure can further progressed during composting as around 50% of N in the manure can be lost mainly via  $\text{NH}_3$  volatilization (Hao et al., 2004; Larney et al., 2006). In our study, the higher  $\delta^{15}\text{N}$  of compost than raw manure could be an indicative of the  $^{15}\text{N}$  enrichment resulting from N isotopic fractionation associated with N loss (Fig. 1). Meanwhile, variation in  $\delta^{15}\text{N}$  compost might be due to the different magnitude and extent of  $\text{NH}_3$  volatilization among composts being affected by composting conditions and materials used. Kim et al. (2008) found that  $\delta^{15}\text{N}$  of N in cattle manure composting mixture increased from +7.8‰



**Fig. 2. Relationship between N concentration and corresponding  $\delta^{15}\text{N}$  of livestock manure composts (n=37).**

at the initiation of composting to +9.9‰ when rice hull was used as bedding material and from +11.4 to +14.3‰ when saw dust was mixed with the manure. They attributed such different pattern to contrasting characteristics of rice hull and saw dust in terms of their  $\delta^{15}\text{N}$  and decomposability. The negative correlation between N concentration and the corresponding  $\delta^{15}\text{N}$  of compost N is also notable (Fig. 2). This relationship suggests that  $\delta^{15}\text{N}$  of compost increases with decreasing N concentration due to  $^{15}\text{N}$  enrichment of compost via N loss.

## Conclusions

The fact that  $\delta^{15}\text{N}$  values of compost were higher than those of synthetic fertilizer suggests that application of compost to fields may result in different  $\delta^{15}\text{N}$  of nitrogenous compounds in soil-plant-groundwater system, compared with synthetic fertilizer application. Therefore, this study shows the potential use of  $\delta^{15}\text{N}$  technique in discriminating compost-derived N and fertilizer-derived N in agricultural system.

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## 화학비료, 가축분뇨 및 퇴비의 질소동위원소비

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화학비료, 가축분뇨 및 퇴비 등 주요 질소원의 질소동위원소비 ( $\delta^{15}\text{N}$ ) 차이를 조사하기 위해 각각 8, 4, 37점의 시료를 채취하여  $\delta^{15}\text{N}$ 을 분석하였다. 평균  $\delta^{15}\text{N}$  값은 화학비료가  $-1.5 \pm 0.5\%$  (범위:  $-3.9 \sim +0.5\%$ ), 가축분뇨가  $+6.3 \pm 0.4\%$  ( $+5.3 \sim +7.2\%$ ), 가축분퇴비가  $+16.0 \pm 0.4\%$  ( $+9.3 \sim +20.9\%$ )였다. 화학비료가 타 질소원에 비해  $\delta^{15}\text{N}$  값이 낮은 것은 화학비료 제조시 이용하는 질소원인 대기  $\text{N}_2$ 의  $\delta^{15}\text{N}$  값 (0%)을 반영하기 때문이다. 반면, 가축분에 비해 퇴비의  $\delta^{15}\text{N}$  값이 높은 것은 퇴비화 과정 중 일어나는 질소손실 (특히, 암모니아 휘산)과 관련된 질소동위원소분할효과 ( $^{14}\text{N}$ 의 손실속도)  $^{15}\text{N}$ 의 손실속도)에 의한 퇴비 중  $^{15}\text{N}$  농축에 의한 결과로 판단된다. 따라서, 본 연구는  $\delta^{15}\text{N}$  분석을 통해 현재 우리나라 농업 시스템에서 가장 널리 이용되고 있는 두 가지 질소원 (화학비료와 퇴비)을 구분할 수 있음을 보여준다.