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Effects of a new nitrification inhibitor 3,4-dimethylpyrazole phosphate (DMPP) on nitrate and potassium leaching in two soils

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Abstract

In this study, soil column was used to study the new nitrification inhibitor 3,4-dimethylpyrazole phosphate (DMPP) on nitrate (NO_3^--N) and potassium (K) leaching in the sandy loam soil and clay loam soil. The results showed that DMPP with ammonium sulphate nitrate (ASN) ($(NH_4)_2SO_4$ and NH_4NO_3) or urea could reduce NO_3^--N leaching significantly, whereas ammonium (NH_4^+-N) leaching increased slightly. In case of total N (NO_3^--N+NH_4^+-N), losses by leaching during the experimental period (40 d) were 37.93 mg (urea), 31.61 mg (urea+DMPP), 108.10 mg (ASN), 60.70 mg (ASN+DMPP) in the sandy loam soil, and 30.54 mg (urea), 21.05 mg (urea+DMPP), 37.86 mg (ASN), 31.09 mg (ASN+DMPP) in the clay loam soil, respectively. DMPP-amended soil led to the maintenance of relatively high levels of NH_4^+-N and low levels of NO_3^--N in soil, and nitrification was slower. DMPP supplementation also resulted in less potassium leached, but the difference was not significant except the treatment of ASN and ASN+DMPP in the sandy loam soil. Above results indicate that DMPP is a good nitrification inhibitor, the efficiency of DMPP seems better in the sandy loam soil than in the clay loam soil and lasts longer.

Key words: 3,4-dimethylpyrazole phosphate (DMPP); nitrate; potassium; leaching; sandy loam soil; clay loam soil

Introduction

Nitrate (NO_3^--N) leaching from agricultural land and the contamination of water resources is a major environment concern around the world (Di and Cameron, 2002, 2005; Liu *et al*., 2005). NO_3^--N is very mobile in the soil and, if not absorbed by the crop, it can be lost by leaching through water movement. Most of NO_3^--N in ground and surface waters is derived from leaching or running off from agricultural land (Liu *et al*., 2005). NO_3^--N, when presents at high concentration in drinking water, can be a health hazard for humans (Chen *et al*., 2003; Knobeloch *et al*., 2000). The concentration of NO_3^--N in drinking water recommended by the World Health Organization was $\leqslant$ 11.3 mg/L, by the USA was $\leqslant$ 10 mg/L, and by China was $\leqslant$ 20 mg/L (Chen *et al*., 2003; Wang *et al*., 2004; Zhu *et al*., 2005). The nitrate concentration has exceeded that level in China (Zhu *et al*., 2005).

Use of nitrification inhibitors can effectively suppress conversion of NH_4^+ to NO_3^-, and thus, its application could reduce NO_3^--N leaching, resulting in a reduction of water pollution (Di and Cameron, 2002, 2005; Hatch *et al*., 2005; Xu *et al*., 2005). There were few studies that showed the efficiency of nitrification inhibitor to retard NO_3^--N leaching depends on soil properties such as temperature, texture, organic matter (Ignacio *et al*., 2003; Zerulla *et al*., 2001), and it needs a further study.

The new nitrification inhibitor 3,4-dimethylpyrazole phosphate (DMPP, marketed by COMPO GmbH & Co. KG under the name ENTEC), which has been developed by BASF (BASF Agricultural Center, Limburgerhof, Germany). Field and laboratory studies showed that it decreased nitrous oxide (N_2O) and carbon dioxide (CO_2) emissions significantly and increased methane (CH_4) uptake (Hatch *et al*., 2005; Linzmeier *et al*., 2001; Weiske *et al*., 2001). Compared to dicyandiamide (DCD), it offers several advantages. It is effective at much smaller doses (Weiske *et al*., 2001; Zerulla *et al*., 2001) and unlikely to have deleterious effects on the crop growth (Macadam *et al*. 2003; Zerulla *et al*., 2001). So, it is may be a good nitrification inhibitor for China agriculture development. However, no study have reported on the effect of DMPP on NO_3^--N leaching under China climate.

Potassium (K) is less mobile and less prone to leaching

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than NO_3^--N, but K deficiencies may restrict crops to utilize N, resulting in increased potential for NO_3^--N leaching (Lægreid *et al*., 1999). Previous studies showed that nitrification inhibitors might reduce K leaching either, due to counter cations as NO_3^--N (Di and Cameron, 2002, 2005; Lægreid *et al*., 1999).

So far, the efficiency of DMPP on NO_3^--N and K leaching in different soils under China climate are little understood. In this study, filled-in soil polyvinylchloride (PVC) columns were used to investigate the effects of DMPP on NO_3^--N and K leaching in the sandy loam soil and clay loam soil.

1 Materials and methods

1.1 Soil

Two soil samples (0–20 cm layer) were used in this study. The sandy loam soil was collected in April 2005 from Hangzhou City, Zhejiang Province of China, with the following characteristics: organic matter 18.5 g/kg; pH 6.61; total N 1.06 g/kg; avail P 112.9 mg/kg; avail K 124.4 mg/kg; NO_3^--N 16.4 mg/kg; NH_4^+-N 2.0 mg/kg; and sand 16.4%, silt 61.1%, clay 22.5%. The clay loam soil was collected in April 2005 from Haining City, Zhejiang Province of China, with the following characteristics: organic matter 24.0 g/kg; pH 6.51; total N 1.49 g/kg; avail P 84.6 mg/kg; avail K 147.5 mg/kg; NO_3^--N 11.1 mg/kg; NH_4^+-N 1.8 mg/kg; and sand 3.6%, silt 44.5%, clay 51.9%. Each soil was air-dried, passed 2 mm sieve before packed into PVC columns.

1.2 Treatment

The characteristics of the tested nitrogen fertilizers are listed in Table 1, all of these fertilizers were obtained from BASF Company. Treatments using the nitrogen fertilizer included urea, urea+DMPP, ASN or ASN+DMPP per column for both soils. Each treatment received 0.6 g N of 1.0 kg soil, which equals to 180 kg N/hm^2. Soil columns without N application were for the control. Three replicate columns were used for each treatment.

1.3 Soil column step

Thirty PVC columns (48 mm inner diameter, 300 mm height) were vertical located on shelves in a glasshouse. The base of each PVC column was covered with two nylon meshes (< 1.0 mm) with elastic bands and quartzitic sands (about 25 g) to retain the soil, another 250 g soil without fertilizer was packed into each PVC column to achieve a bulk density of 1.3 g/cm^3 firstly, another 250 g soil mixed with fertilizer (0.6 g N/kg soil) was also packed into each PVC column, achieving the same bulk density as former. After that, the top of each column was covered with plastic membrane and quartzitic sands (about 25 g) to minimize soil disturbance when watering and prevented evaporation, details are described by the reference (Paramasivam and Alva, 1997). In the first day, distilled water was added into each column to make the soils saturated; in the day 2, 150 ml distilled water was added into each column and leachate was collected in tank until not existed in columns. The following incubation methods were the same as previously. Leachate was collected at 2, 5, 10, 21, 30 and 40 d of incubation.

At completion of the leaching, the plastic membranes were removed, and then soil in each column was pushed out, and was divided into 0–5 cm, 5–10 cm, 10–15 cm and 15–20 cm segments. All samples were stored at 4°C in fridge until analyzed.

1.4 Soil and leachate analysis

Soil samples were analyzed after extraction by 2 mol/L KCl. NH_4^+-N and (NO_3^-+NO_2^-)-N levels were determined by MgO-Devarda's ally method (Lu, 2000).

Leachate was collected, and its volume and NH_4^+-N, NO_3^--N, K concentration were determined after filtration. NO_3^--N was measured using ultraviolet spectrophotometry, using the wavelengths of 220 and 275 nm for nitrate and organic matter, respectively. NH_4^+-N was measured using Indophenol Blue Method (Lu, 2000). K was measured using a flame photometer (PFP7, Jenway Ltd, U.K.).

1.5 Calculation and statistical analysis

Leaching losses were calculated on the basis of their concentrations in the leachate collected from each PVC column and the volume of leachate. Data were analyzed using the one-way ANOVA of Statistical software for Windows (Statistica,Version 5.5). The Duncan's new multiple range difference tests were used to compare at 0.05 level.

2 Results

2.1 Leached NO_3^--N, NH_4^+-N and K

Application of DMPP reduced N leached in both soils. The loss of NO_3^--N in the leachate was lower from the soil in the treatment with DMPP than the soil that had received ASN or urea alone during the experiment period (Figs.1a1 and 1a2), and the opposite occurred with NH_4^+-N levels (Figs.1b1 and 1b2). It is interested to note that there was a peak level loss for NO_3^--N and NH_4^+-N, respectively. As for NO_3^--N, the peak occurred at 2 d from the treatment soil with ASN; as for NH_4^+-N, the peak occurred at 5 d.

Table 1 Characteristics of the tested nitrogen fertilizers

Type of fertilizer	Total N (%)	NH_4^+-N (%)	NO_3^--N (%)	Carbamide-N (%)
ASN	26.0	18.5	7.5	/
ASN+DMPP	26.0	18.5	7.5	/
Urea	46.0	/	/	46.0
Urea+DMPP	46.0	/	/	46.0

ASN: ammonium sulphate nitrate; DMPP: 3,4-dimethylpyrazole phosphate. The same indication is used for other tables below.

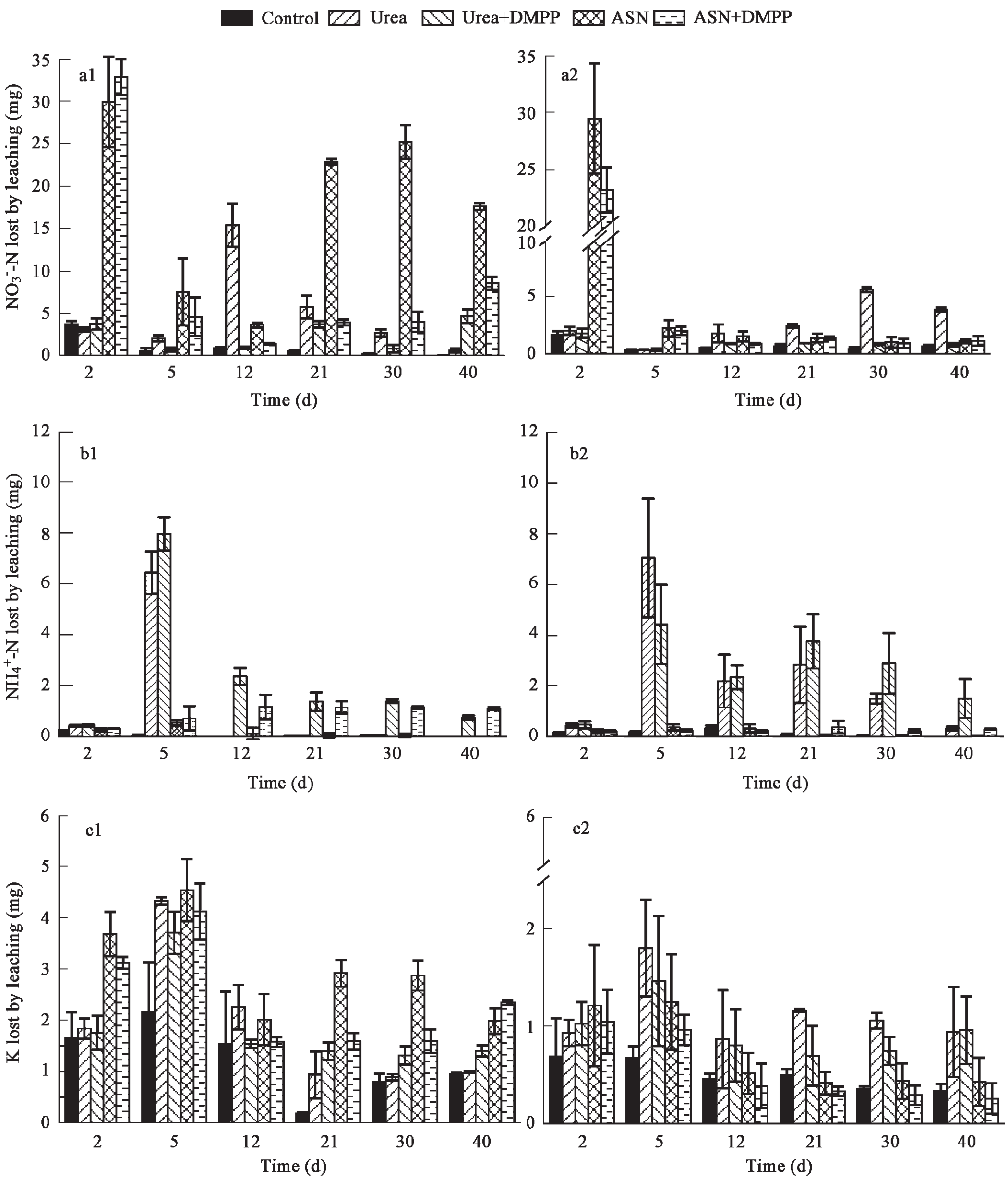

Fig. 1 Change of NO_3^--N (a), NH_4^+-N (b) and K (c) leached from sandy loam soil (a1, b1, c1) and clay loam soil (a2, b2, c2) in leachate. Values are means of three replicates and vertical bars represent SD.

The urea and ASN with DMPP can reduce the NO_3^--N loss in the leachate from the two soils, compared with urea and ASN, respectively. There were significant difference in the sandy loam soil after 12 d of incubation, however, for the clay loam soil it occurred later (21 d), the same trend happened for NH_4^+-N levels, this indicates DMPP is a good nitrification inhibitor, but there was a lag phase (about 12 d) before it took effect significantly (Figs.1a and 1b).

Total amount of various form N leached from the PVC columns are listed in Table 2. The leached NO_3^--N was predominantly and the NH_4^+-N was less. In the case of total N, losses by leaching during the experimental period (40 d) were 37.93 mg (urea), 31.61 mg (urea+DMPP),

Table 2 Total amount of various forms N and K leached from the PVC column (mg/column)

Treatment	Sandy loam soil				Clay loam soil			
	NH_4^+-N	NO_3^--N	$(NH_4^++NO_3^-)$-N	K	NH_4^+-N	NO_3^--N	$(NH_4^++NO_3^-)$-N	K
Control	0.28 d	5.97 e	6.25 e	7.31 d	0.75 b	4.27 d	5.02 d	3.02 c
Urea	8.35 b	29.58 c	37.93 c	11.24 c	14.28 a	16.26 c	30.54 b	6.77 a
Urea+DMPP	16.91 a	14.70 d	31.61 d	10.13 c	15.34 a	5.71 d	21.05 c	5.71 ab
ASN	1.36 d	106.74 a	108.10 a	18.03 a	1.00 b	36.86 a	37.86 a	4.27 bc
ASN+DMPP	5.38 c	55.32 b	60.70 b	14.37 b	1.49 b	29.60 b	31.09 ab	3.28 c

Values in each column followed by different letters meant a significant difference at 0.05 level.

108.10 mg (ASN), 60.70 mg (ASN+DMPP), 6.25 mg (control) in the sandy loam soil, and 30.54 mg (urea), 21.05 mg (urea+DMPP), 37.86 mg (ASN), 31.09 mg (ASN+DMPP), 5.02 mg (control) in the clay loam soil. The percentage of N losses by leaching with respect to N applied, discounting the leached N in the control, was 21.12% (urea), 16.91% (urea+DMPP), 67.90% (ASN), and 36.30% (ASN+DMPP) in the sandy loam soil and was 17.01% (urea), 10.69% (urea+DMPP), 21.89% (ASN), and 17.38% (ASN+DMPP) in the clay loam soil. The amount of NO_3^--N losses was different in the two soils, the fertilizer can be easily leached from sandy loam soil than from clay loam soil, especially the ASN and ASN+DMPP, and on average losses were about 2–3 times greater from the sandy loam soil than that from the clay loam soil. However, in both soils, the lowest NO_3^--N losses were found in the DMPP treatment, and the DMPP can reduce NO_3^--N significantly from the two soils. In contrast, the total amount of NH_4^+-N in leachate was higher in the treatment with DMPP, although the total N lost was greater in the treatment without DMPP. Therefore, the DMPP is efficient in reducing N leaching, but appears better in the sandy loam soil than applied in the clay loam soil.

DMPP added to ASN (ASN+DMPP) seemed better than added to urea (urea+DMPP). In the case of total N, with respect to ASN, ASN+DMPP reduced 47.40 mg N leaching from the sandy loam soil, while only 6.32 mg N was reduced in the treatment urea+DMPP when compared to urea. This phenomenon was also found for total K leached.

The result (Fig.1c) shows that DMPP can reduce K leached from both soils at least for 28 d, and the peak level occurred at 5 d, but there was no significant difference among the treatment with DMPP and without DMPP except the control during this time. Like the N (Table 2), the sandy loam soil leached more K than the clay loam soil. During the experiment period, in the sandy loam soil, the K level in the leachate was low in the treatment with urea+DMPP, ASN+DMPP than urea, ASN alone, respectively, but the difference was significant at 21–30 d, while in the case of clay loam soil the difference was significant at 21–30 d just between urea and urea+DMPP. In the first 12 d, the level decreased and increased slightly thereafter, the reason for this phenomenon may be the fixed K was replaced by the NH_4^+-N.

The total amount of K leached from the ASN+DMPP treatment was much lower than that found in the ASN treatment, and urea+DMPP has the same result as compared with urea alone, but the difference was not significant except the treatment ASN and ASN+DMPP in the sandy loam soil (Table 2).

2.2 Residual N in the soil

DMPP can maintain higher mineral N (NH_4^+-N+NO_3^--N), NH_4^+-N and lower (NO_3^-+NO_2^-)-N concentration in the two soils (Fig.2), and thus, the residual N was more in the treatment with DMPP than without DMPP. In the sandy loam soil, mineral N and NH_4^+-N concentration was significant greater in the ASN+DMPP than ASN alone in every soil depths, but there was no significant difference between urea and urea +DMPP except NH_4^+-N in the 10–20 cm. In the clay loam soil, mineral N and NH_4^+-N had the similar trend, but there was no significant difference. In the subsoil (15–20 cm layer) of sandy loam soil the concentration of (NO_3^-+NO_2^-)-N was 146.15, 68.14, 52.66, 43.20 mg/kg for ASN, ASN+DMPP, urea, urea+DMPP, respectively. Therefore, DMPP can reduce nitrate leaching into subsoil layer, and ASN+DMPP was better than urea+DMPP. However, the phenomenon was not obviously observed in the clay loam soil.

The NH_4^+-N/NO_3^--N ratio, which is considered a better and more sensitive indicator of effectiveness, as it is independent of the rate N, method of application, or spatial variability (Hauck, 1984), is also discussed (Fig.3). The NH_4^+-N/NO_3^--N ratios, in respect of control, urea, urea+DMPP, ASN, ASN+DMPP were 0.057–0.147, 0.116–0.122, 0.238–1.292, 0.091–0.127, 0.598–2.232 in the loam sandy soil and 0.944–1.156, 1.848–3.281, 1.488–2.885, 0.999–2.674, 1.150–4.382 in the clay loam soil. This indicates that DMPP was effective nitrification inhibitor, but it failed to be observed in the treatment of urea+DMPP in the clay loam soil.

3 Discussion

DMPP has already been identified by several authors as one of the most efficient nitrification inhibitors (Hatch *et al*., 2005; Linzmeier *et al*., 2001; Weiske *et al*., 2001), and better than the most used DCD and nitrapyrin (Zerulla *et al*., 2001). Our results showed that addition of DMPP maintained higher level NH_4^+-N and lower level NO_3^--N in the leachate (Figs.1a and 1b). Although the loss of NH_4^+-N was higher in the treatment with DMPP than without DMPP, which in contrast to NO_3^--N, the total N leached was less in the DMPP treatment. Above results consistent with previous studies (Serna *et al*., 2001; Xu *et al*., 2005). The amount of N lost by leaching was soil-derived N and fertilizer derived-N. Between soil N mineralization and immobilization turnover, nitrification inhibitors may cause a priming effect with a subsequent increase in the rate of soil organic matter mineralization and an extra release of soil organic N, the priming effect was real in the sandy loam soil where a net N release was observed, whereas in the clay loam soil the effect of the inhibitors was less pronounced (Gioacchini *et al*., 2002), so more mineral N (NO_3^--N, NH_4^+-N) will be leached from soil after fertilizer-derived N was leached almost over. During our experiment period, different N peak level occurred at different time. As for NO_3^--N, the peak occurred at 2 d from the treatment soil with ASN; as for NH_4^+-N, the peak occurred at 5 d. (Figs.1a and 1b), the loss of NO_3^--N and NH_4^+-N at 2 or 5 d might be derived from fertilizer N, while at 40 d should be derived from soil N. So, we need other experiments to prove this more detail.

Addition of nitrification inhibitors DMPP to fertilizers maintained soil N in NH_4^+-N form (Fig.2), which may increase N losses associated with NH_3 volatilization (Davies and Williams, 1995). However, the amount of NH_4^+-N

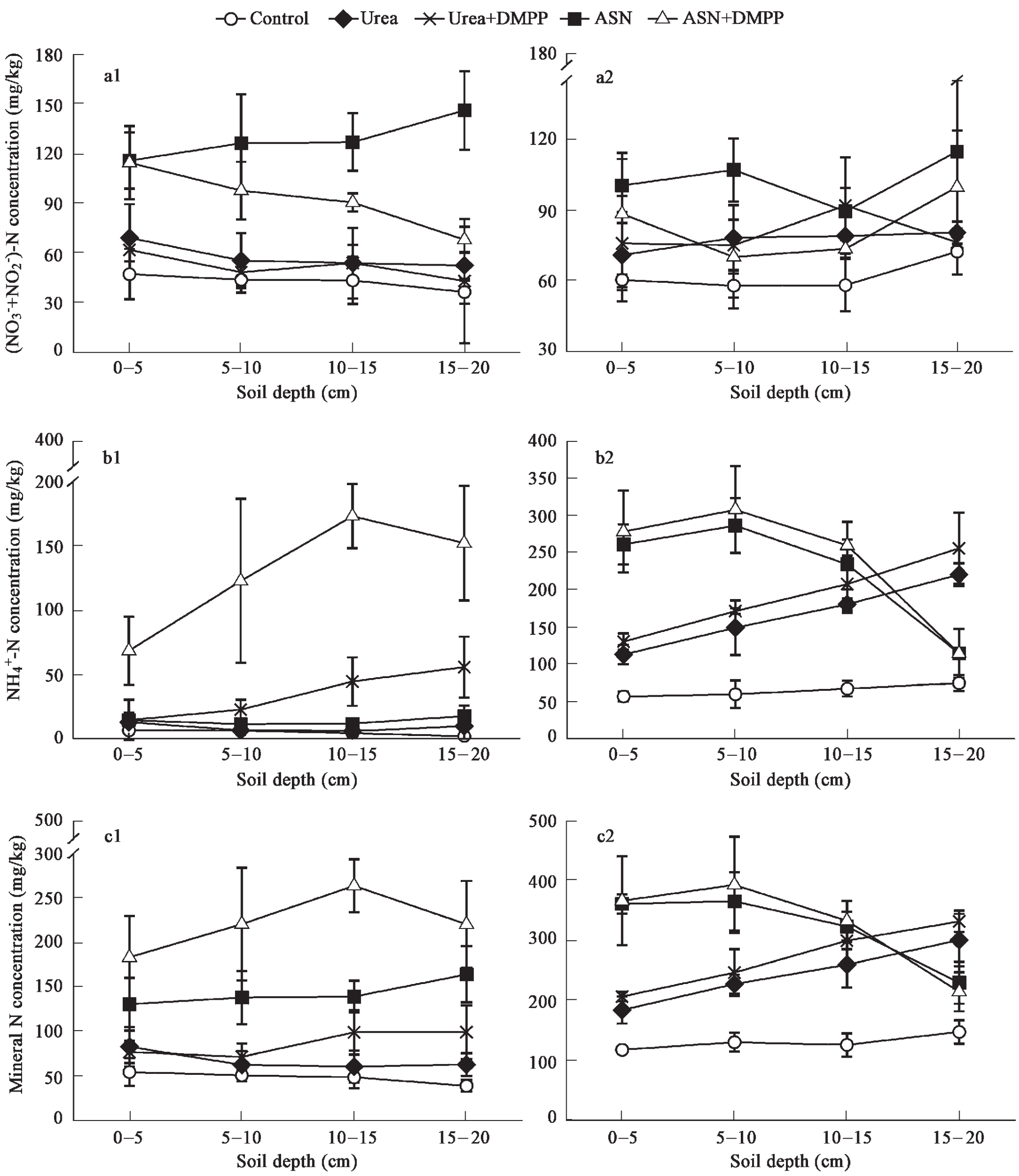

Fig. 2 Concentration of $(NO_3^-+NO_2^-)$-N (a), NH_4^+-N (b), mineral N (c) in different depths of sandy loam soil (a1, b1, c1) and clay loam soil (b1, b2, b3). Values are means of three replicates and vertical bars represent SD.

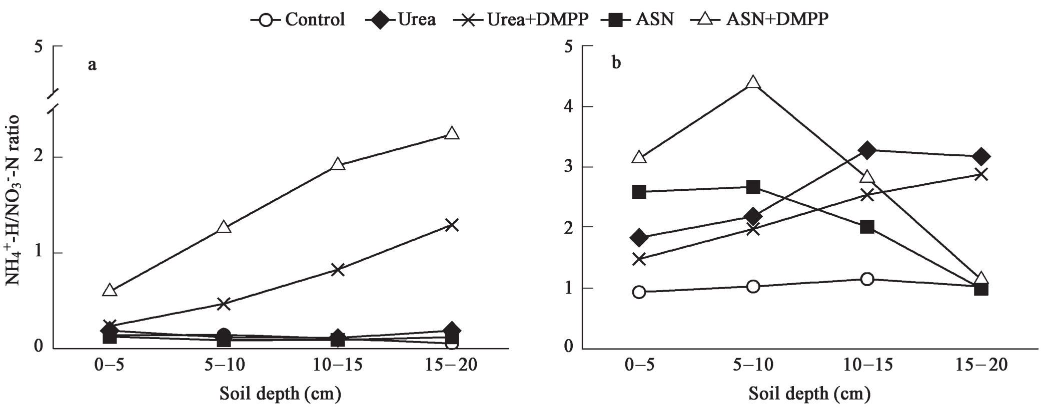

Fig. 3 Effects of DMPP on NH_4^+-N/NO_3^--N ratios in different depths of sandy loam soil (a) and clay loam soil (b).

lost in the form of NH_3 following slurry and slurry + DMPP applications was 7.8% and 11.0%, respectively, the increase induced by DMPP not being statistically significant (Menendez *et al*., 2006). These results may depend on soil properties and the soil pH in particular (Zerulla *et al.*, 2001).

Although K is less mobile and less prone to leaching than nitrate (Lægreid *et al.*, 1999), the nitrification inhibitor also reduce cations such as K^+, Ca^{2+}, Mg^{2+} leaching (Di and Cameron, 2004, 2005; Lægreid *et al.*, 1999). Di and Cameron (2005) research confirmed that the linear relationship was found between the NO_3^- concentration (x) and the sum of the three cations (y) in the leachate: $\hat{y}=3+1.22x$ ($R^2=0.93$ P <0.001). This is likely to have been due to the reduced requirement of counter cations in the leachate to maintain soil-solution charge balance as a result of reduced NO_3^- leaching (Di and Cameron, 2004, 2005; Lægreid *et al.*, 1999). However, due to the similar ionic radius of NH_4^+ (0.286 nm) and K^+ (0.266 nm), it shows that NH_4^+ may be displaced by K^+ (Kenan *et al.*, 1999). Some results found that NH_4^+ fertilizer applied into soil, it replaced Ca^{2+}, Mg^{2+} from the inerlayer, resulting in K^+ fixed in the no-exchange sites (Xie, 1981), but other research found NH_4^+ fertilizer may cause decreasing K fixed and increasing the K leached (Fan, 1993). Increase and decrease in NH_4^+ fixation may have resulted from the blocking of some exchanged sites by K. The fixation of NH_4^+ was reduced by K added before NH_4^+, and the reduction was proportional to the amount of K previously fixed (Kenan *et al.*, 1999). The mechanism for this phenomenon is not clear yet (Chen and Machenzie, 1992).

In addition, the relative inhibition of nitrification by inhibitors would be excepted to be lower with the alkaline-forming fertilizers such as urea and NH_3 than aciding-forming fertilizer such as $(NH_4)_2SO_4$, NH_4NO_3 or $(NH_4)_2HPO_4$. Recovery of N for nitrapyrin with the fertilizers applied as diammonium phosphate was much higher than applied as urea (Abbasi *et al.*, 2003). However, DCD was more effective in reducing N_2O emissions when applied with urea than $(NH_4)_2SO_4$ (McTaggart *et al.*, 1997). In our experiment, ASN+DMPP reduced more N leaching than urea+DMPP when compared to ASN, urea, respectively. But, this phenomenon was not happened in the clay loam soil.

The efficiency of nitrification inhibitor depended on soil properties such as temperature, texture, organic matter (Ignacio *et al.*, 2003; Zerulla *et al.*, 2001), this also applies to DMPP. Our results showed that the efficiency of DMPP seems better in the sandy loam soil than clay loam soil. However, only the simultaneous observation of several soil parameters can explain the intensity of inhibition of nitrification by DMPP (Zerulla *et al.*, 2001). Barth *et al.* (2001) showed with multiple regression that the sand content, proton concentration as well as microbiological parameters of soil, such as catalase activity, and the potential nitrification capacity, seem to have significant influences on the efficiency of DMPP in soils, however, other experiments are needed for further study.

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