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Error caused by carbon dioxide in determination of ammonium by direct steam distillation of tropical wetland rice soils

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Summary A study was made with eight Philippine wetland rice soils to quantify the possible error caused by the CO_2 evolved during direct distillation of soil suspensions in aerobic and anaerobic conditions with MgO. The error caused by CO_2 was eliminated by absorbing the ammonia distilled in H_2SO_4, which was gently boiled to derive off the CO_2 absorbed. The possible error caused by CO_2 was not eliminated when boric acid was used for absorbing ammonia. The difference in NH_4^+ values determined by using sulfuric acid and boric acid methods gave an estimate of the error caused by CO_2. It was found that CO_2 evolved caused negative error in the NH_4^+ values obtained using the direct distillation of soil suspensions with MgO in presence of KCl. The magnitude of error was higher and significant with anaerobic soil samples but this error was negligible with aerobic soils.

Introduction

The nitrogen supplying capacity of soils play a dominant role in nitrogen nutrition of wetland rice because even in well fertilized paddies about half to two-thirds of nitrogen used by rice plant comes from the soil mineral N pool[2]. The potentially mineralizable N in wetland rice soils is usually measured by anaerobic incubation of soils for short periods varying from 6 to 14 days[3,5].

Waring and Bremner[8] proposed that the ammonium produced in soils under waterlogged conditions was a good measure of available N. These authors suggested direct distillation of the incubated soils with MgO in presence of 2 *M* KCl to determine NH_4^+ released. Our previous study[6] with Philippine wetland rice soils showed that this method gave inflated values for NH_4^+ released when direct distillation of soil samples with MgO as suggested by Bremner[1] and Keeney and Bremner[4] was followed as compared to when the filtered extracts of soils were distilled to determine NH_4^+. We also observed that during direct distillation of soil suspensions, CO_2 evolved caused negative error in NH_4^+–N values.

The present study examined the effects of CO_2 evolution on determination of NH_4^+ produced during direct distillation of the soil samples with MgO in the aerobic and anaerobic states. The results reported provide evidence to show that CO_2 evolved during direct distillation of anaerobic soils did cause negative error in exchangeable NH_4^+ values.

Materials and methods

The eight soil samples used (Table 1) had a wide range in pH, texture and organic matter and were collected from rice growing areas of the Philippines. For soil analysis reported in Table 1, pH was measured by a glass electrode using 1 : 1 soil to water ratio. Organic C was determined by the method of Walkley and Black[7].

To determine the effect of CO_2 on measurement of NH_4^+ in the anaerobic soils the following procedure was followed. The 10 g soil samples in triplicate were incubated under waterlogged conditions at 30°C with 25 ml water for 2 weeks. After incubation, one set of samples were distilled

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Table 1. Comparison of exchangeable NH_4^+ content (mg N/kg soil) of eight tropical rice soils in the aerobic and anaerobic states as measured by steam distillation of soil-KCl suspension using boric acid and sulphuric acid as absorbant for ammonia

Soil			Aerobic			Anaerobic		
Texture	pH	Organic matter (%)	H_3BO_3	H_2SO_4	Δ	H_3BO_3	H_3SO_4	Δ
Clay loam	6.3	1.1	34	35	−1	70	73	− 3*
Sandy loam	3.4	2.7	180	182	−2	231	238	− 7**
Clay loam	5.3	10.4	37	38	−1	434	442	− 8**
Clay	6.5	1.6	37	37	0	57	61	− 4**
Clay	7.5	3.9	35	35	0	95	101	− 6**
Sandy loam	6.5	2.2	37	38	−1	66	71	− 5**
Silty loam	5.3	26.8	68	70	−2	501	511	−10**
Silty loam	5.5	38.2	161	163	−2	503	515	−12**

*, ** Significant at 5% and 1% levels, respectively.

with MgO in presence of KCl and the ammonia distilled was absorbed in 25 ml of 2% H_3BO_3-indicator mixture; and titrated with 0.04 *M* H_2SO_4 to find the NH_4^+ produced. In the other case, the NH_4^+ distilled from soil samples with MgO in presence of KCl was absorbed in 25 ml of 0.04 *M* H_2SO_4. The CO_2 absorbed was driven off from the H_2SO_4 by gently boiling. The acid (with ammonia) was cooled and titrated with standard alkali and the amount of NH_4^+ released calculated. The differences between the two methods was ascribed to CO_2. Similar experiments were done to study the effects of CO_2 on NH_4^+ determination in the aerobic soil samples.

Results and discussion

For each of the 8 soils, distillation of the suspension gave higher values for exchangeable NH_4^+ when sulfuric acid was used for absorbing the ammonia distilled (Table 1). These difference were generally more in anaerobic soils with higher content of organic matter. Though the amounts of CO_2 produced during distillation of soil suspension with MgO were not measured, the error caused by CO_2 was indirectly eliminated. When boric acid was used for absorbing ammonium the CO_2 evolved was absorbed by the acid could not be expelled out by boiling because of possible loss of ammonia[1,9]. However, when sulfuric acid is used for absorbing ammonia, the interference due to CO_2 can be easily avoided by boiling H_2SO_4 for a short while to derive off CO_2[1]. Following this method it was found that the CO_2 introduced a negative error in the determination of NH_4^+ produced in anaerobic (waterlogged) soils with the result the values of NH_4^+ obtained by using H_3BO_3 for absorbing ammonia were lower than those obtained with H_2SO_4 as the absorbant. However, the error was negligible with aerobic soil samples (Table 1).

Based on these results it is suggested that CO_2 produced during distillation of the soil suspension with MgO following anaerobic incubation may cause negative error in determination of NH_4^+ if boric acid is used for absorbing ammonia. Our earlier studies showed that direct distillation of soils with MgO caused hydrolysis of organic matter at the high pH (9.9 to 10.7) obtained in aqueous suspensions resulting in inflated values for exchangeable NH_4^+ in aerobic and anaerobic soils[6]. Results of this study and our earlier observations suggest that the values of exchangeable NH_4^+ in waterlogged soils determined by the direct distillation technique are affected in two ways: (i) due to hydrolysis of organic matter, which inflates the NH_4^+ values and (ii) the CO_2 evolved causes negative error. The NH_4^+–N values thus obtained will be the resultant of interactive effects in the opposite directions due to hydrolysis of organic matter causing positive error and the effect of CO_2, which

causes negative error. Based on these findings, it is recommended that the direct distillation technique should not be followed for determining NH_4^+ in wetland tropical rice soils.

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