Measurement of Exchangeable NH_4^+ in Tropical Rice Soils[1]

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ABSTRACT

Measurement of exchangeable NH_4^+ by two methods—steam distillation of the filtrates of KCl extracts and of soil-KCl suspensions with MgO—was compared for 17 tropical soils covering a wide range in texture, pH, and organic matter. Distillation of soil suspensions gave significantly higher values for NH_4^+ both for aerobic and anaerobic soils, but the differences were much greater in the anaerobic soils. The higher values obtained by direct distillation of KCl-soil suspensions were probably due to hydrolysis of soil organic matter at the high pH values (9.9 to 10.7) brought about by the boiling MgO suspensions. We recommend the use of a filtered KCl extract instead of a soil suspension to measure exchangeable NH_4^+ in tropical rice soils.

Additional Index Words: **hydrolysis of organic N, soil-KCl suspensions, aerobic and anaerobic soils, pH of MgO-soil suspensions.**

THE HIGH COST of nitrogen fertilizer in South and Southeast Asia combined with the need for increased yields of rice has stimulated research on methods of using soil and fertilizer nitrogen efficiently. The measurement of exchangeable NH_4^+ in flooded soils is an important component of such research.

Exchangeable NH_4^+ in aerobic soils used to be measured by extracting soils with 1*N* KCl followed by steam distilling the filtrates with MgO into acids (Bengtsson, 1924; Harper, 1924; Gibbs et al., 1923; McLean and Robinson, 1924; Yuen and Pollard, 1953). Since Bremner (1965) and Keeney and Bremner (1966) showed that filtration could be omitted without sacrificing accuracy, direct distillation of the soil–KCl suspensions is now widely used (Simsiman et al., 1967; Racho and De Datta, 1968; Keeney and Bremner, 1969; Nelson and Bremner, 1972; Tabatabai and Bremner, 1972; Olsen et al., 1970; Manickam et al., 1974; Muthuswamy et al., 1975; Jolly and Pierre, 1977; Reddy and Patrick, 1977). However, apart from a report by Robinson (1967) that direct steam distillation of a humic ferrisol, incubated anaerobically, gave a much higher value for exchangeable NH_4^+ than a filtered KCl extract, there is little information on the reliability of the direct distillation of soil–KCl suspensions for the measurement of exchangeable NH_4^+ in tropical rice soils.

We compared the values for exchangeable NH_4^+ obtained by the two methods—steam distillation of filtered KCl extracts and of soil–KCl suspensions—for 17 soils of widely differing properties and found that distillation of soil suspensions gave higher values for aerobic and anaerobic soils but the differences were much greater in the anaerobic soils.

MATERIALS AND METHODS

The soil samples used had a wide range in pH, texture, and organic matter (Table 1) and were collected from important rice growing areas of the Philippines. The pH was measured with a glass electrode (soil: water ratio of 1:1) and organic carbon was determined by the method of Walkley and Black (1934).

To measure exchangeable NH_4^+ in aerobic soils, 10-g triplicate samples of the < 2-mm fraction of the air-dry soils were extracted with 100 ml of 2*M* KCl for 1 hour in a wrist action shaker; the suspension was filtered, and the entire filtrate steam distilled with 0.5 g dry, carbonate-free MgO for 6 to 7 min, at a rate of 5–6 ml/min. The time of distillation and amount of MgO used were those standardized in a preliminary study. The ammonia was absorbed in 25 ml of 2% boric acid and titrated with 0.02*N* H_2SO_4. The soil-suspension distillation method was identical with the filtered KCl-extract method except that the soil–KCl suspension was used in the distillation flask instead of the filtrate.

To measure the content of exchangeable NH_4^+ in anaerobic soils, 10-g soil samples were submerged in 25 ml of water and

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Table 1—Comparison of the exchangeable NH_4^+ contents of 17 tropical rice soils in the aerobic and anaerobic states, as measured by steam distillation of the filtrates of KCl extracts and soil–KCl suspensions.

Soil			Aerobic			Anaerobic		
Texture	pH	Organic matter	Filtrate	Soil suspension	Δ	Filtrate	Soil suspension	Δ
		%	NH_4^+-N, ppm			NH_4^+-N, ppm		
Sandy loam	7.4	1.0	21.8	22.5	0.7	26.7	35.3	8.6**
Clay loam	6.3	1.1	53.5	53.8	0.3	66.5	71.5	5.0**
Sandy loam	3.4	2.7	53.9	59.6	5.7**	65.6	76.4	10.8**
Clay loam	5.3	10.4	194.9	209.2	14.3**	428.9	485.0	56.1**
Clay	4.8	2.6	36.3	40.9	4.6**	68.0	74.0	6.0**
Clay	6.5	1.6	19.1	21.6	2.5	26.2	29.1	2.9*
Clay	7.5	3.9	35.2	40.1	4.9**	93.5	113.3	19.8**
Sandy loam	6.5	2.2	21.9	28.9	7.0**	71.2	77.5	6.3**
Loam	7.0	4.3	34.0	36.1	2.1	56.7	67.3	10.6**
Clay	6.1	2.4	7.5	11.1	3.6**	21.6	44.3	22.7**
Clay	3.5	3.8	375.8	444.3	68.5**	433.5	522.8	89.3**
Silt clay loam	8.7	1.7	14.7	14.9	0.2	28.9	30.3	1.4
Silt loam	7.4	2.9	51.6	60.9	9.3**	85.5	113.2	27.7**
Silty clay	5.3	26.8	51.7	73.2	21.5**	362.5	404.1	41.6**
Silty clay	5.5	38.2	73.6	125.8	52.2**	435.2	506.7	71.5**
Silt loam	7.7	2.0	7.4	10.9	3.5**	17.1	21.6	4.5**
Loam	7.1	3.9	1.6	7.0	5.4**	192.9	227.4	34.5**

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

Table 2—Comparison of the exchangeable NH_4^+ content of eight tropical rice soils in the aerobic and anaerobic states, as measured by steam distillation of the filtrates of KCl extracts and soil-KCl suspensions following exactly Bremner's procedure.

Soil			Aerobic			Anaerobic		
Texture	pH	Organic matter	Filtrate	Soil suspension	Δ	Filtrate	Soil suspension	Δ
		%	NH_4^+-N, ppm			NH_4^+-N, ppm		
Clay loam	6.3	1.1	24	32	8**	64	72	8**
Sandy loam	3.4	2.7	164	180	16**	212	236	24**
Clay loam	5.3	10.4	33	40	7**	386	440	54**
Clay	6.5	1.6	36	37	1	55	60	5**
Clay	7.5	3.9	30	33	3*	80	91	11**
Sandy loam	6.5	2.2	30	36	6**	69	75	6**
Silt loam	5.3	26.8	40	64	24**	428	506	78**
Silty clay	5.5	38.2	128	162	34**	433	511	78**

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

incubated at 30° C for 2 weeks. Exchangeable NH_4^+ was measured by distillation of the filtered KCl extracts and of soil-KCl suspensions of the incubated soils, as before.

RESULTS AND DISCUSSION

For each of the 17 soils, both aerobic and anaerobic, distillation of the suspensions gave higher values for exchangeable NH_4^+ than distillation of the filtered KCl extract (Table 1). The differences were greater in the anaerobic soils, in spite of a possible negative CO_2 error. There was a positive association between the organic matter content and Δ NH_4^+-N for both aerobic and anaerobic soils, but the correlation was poor ($r = +0.56$*).

The higher NH_4^+-N values obtained by distillation of the soil suspensions may be attributed to hydrolysis of soil organic matter at the high pH values brought about by the boiling MgO suspensions. Theoretically, as shown by Ponnamperuma (1967), MgO or $MgCO_3$ in equilibrium with CO_2 of air should have a pH of about 9.5. At the lower partial pressure of CO_2 in boiling aqueous suspensions of MgO, the pH should be higher. And indeed we observed pH values of 9.9 to 10.7 in such systems. Researchers are aware of the positive error caused by alkali hydrolysis. But Bremner (1965) stated that the error could be minimized by using a small amount of MgO and reducing the time of distillation. Our results show that inspite of these precautions, direct distillation of soil–KCl suspensions with MgO causes a positive error in the measurement of exchangeable NH_4^+ in tropical rice soils.

A later study of eight representative soils, both aerobic and anaerobic, by the exact procedure prescribed by Bremner (1965) and Keeney and Bremner (1966) confirmed the positive error caused by direct distillation of soil suspensions (Table 2).

We recommend the use of a filtered extract instead of a soil suspension to measure exchangeable NH_4^+ in tropical rice soils. Dr. F. E. Broadbent, Univ. of California, Davis, (personal communication) concurs.

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