Bull. Environ. Contam. Toxicol. (1987) 38:377–380

Environmental Contamination and Toxicology

Determination of Exchangeable Inorganic Nitrogen Species in Wetland Soils

S. Brodrick, P. Cullen, and W. Maher

Water Research Centre, Canberra College of Advanced Education, P.O. Box 1, Belconnen, ACT, 2616 Australia

Thredbo Village, an Australian Alpine resort, discharges secondary treated sewage effluent through an adjacent wetland area to trap suspended and dissolved material before water is discharged into the Crackenback river (Brodrick 1985). We are currently evaluating the effectiveness of this wetland in removing nitrogen species from the sewage effluent by denitrification and required a means of determining the exchangeable nitrogen species in wetland soils.

A review of the literature revealed that potassium chloride is commonly used to extract exchangeable nitrogen species from soils (Bengtsson 1924; Bremner 1965; Sahrawat 1979). However a great variation occurs with the use of extraction techniques with regard to the method of agitation (Terry and Nelson 1975; Haines et al. 1977; Henriksen 1980), the molarity of potassium chloride solution (Sorensen 1978; Blackburn and Kenriksen 1983; Smith and Patrick 1983), the extraction period (Klingensmith and Alexander 1983; Jenkins and Kemp 1984) and the solvent to soil ratio (Henriksen 1980; Burford and Bremner 1975) used.

It is also noted that most workers when extracting nitrogen species do not detail their efficiencies in quantifying exchangeable inorganic nitrogen species. This communication reports our attempt to use potassium chloride to extract exchangeable inorganic nitrogen species from wetland soils.

MATERIALS AND METHODS

Soil was collected from an Alpine wetland area subject to treated effluent from a sewage treatment works (Brodrick 1985). Soil contained 45-50% clay and a large amount of partially decomposed organic matter.

A Technicon Autoanalyser II was used to measure nitrate + nitrite N and ammonia N in extracts. Both autoanalyser manifolds were operated in accordance to A.P.H.A. standard methods (1980).

Send reprint requests to P. Cullen at the above address.

RESULTS AND DISCUSSION

The extraction technique initially employed was based on that of Henriksen (1980). One gram soil samples were placed into centrifuge tubes with 25 mL of 2M potassium chloride. After capping and inverting the tubes three times, the samples were centrifuged (4500 rpm) for 10 min, and the supernatants were removed and analysed for nitrogen species. Four consecutive extractions were performed. Yields of nitrate + nitrite N were similar for the first three extractions (Table 1) and started to decline in the fourth. Clearly this method was not an effective way to extract exchangeable nitrate + nitrite.

Table 1. Extraction of nitrate + nitrite N from soil using Henriksen's (1980) method

Extraction(1)	1	2	3	4
Nitrate + nitrite N (µg)	12.5±2.2	12.5±0.4	12.4±1.0	10.06±0.34

(1) Four replicate samples

To increase the interaction between the extractant solution and soil, agitation with a wrist-action shaker was introduced, and the extraction period extended to two hours. The yield of nitrate and nitrite N was increased to 20.8±3.9 µg N; consequently, agitation was used in all further experiments. To determine the optimum extraction period, subsamples were extracted with 25 mL of the potassium chloride solution, and the nitrate-nitrite N content was measured in the centrifuged supernatants at different time intervals (Figure 1). There was no increase in the nitrate + nitrite N yield after two hours. Longer extraction periods resulted in a decrease of nitrate + nitrite N and suggested that readsorption occurred. There was little difference in the yield of nitrate + nitrite N when the volume of extractant was varied (Table 2), and a volume of 25 mL was selected for convenience. Re-extraction with successive 25 mL volumes of extractant solution (Table 3) continued to yield nitrate + nitrite N and ammonium N even after three extractions. The apparent failure of the extraction procedure to quantitatively extract exchangeable nitrogen species may have been the result of other nitrogen forms as well as the exchangeable nitrogen species being extracted. The efficiency of the extraction procedure to extract known amounts of exchangeable nitrogen species was investigated by equilibration of soil with nitrogen species for two hours followed by extraction of soils with 25 mL of 2M potassium chloride for two hours. The recoveries of added nitrate N (1.6 µg/g) ranged from 78-98% (average 84.6±1.2%; n = 4), and ammonia N (24 µg/g) were 70-100% (average 81.3+3.4%; n = 4)

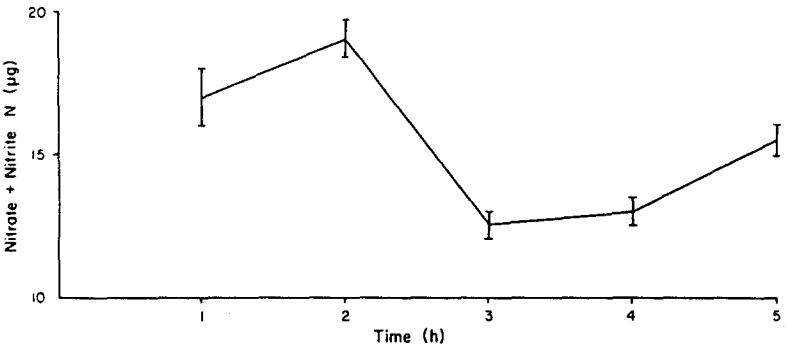

Figure 1. Effect of extraction time on the nitrate-nitrite N content of the potassium chloride extractant. (n=4)

Table 2. Effect of volume of extractant on yield of nitrate + nitrite N.

Extractant Volume (mL)	20	25	40	50
Nitrate + Nitrite N (µg)	20.8±1.0	19.3±0.9	17.8±0.6	19.7±0.4

Extraction time 2 hours, four replicates.

Table 3. Effect of successive extractions on the yield of nitrate + nitrite N and ammonium N from soil

Extraction No.	1	2	3
Nitrate + Nitrite N (µg)	5.3±3.2	7.9±1.6	9.2±1.4
Ammonium N (µg)	369±17	283±10	260±12

25 mL 2M KCL; 2 hour extraction period. Four replicates.

Our results show that the techniques usually employed to extract exchangeable nitrogen species from soils could not be used to quantify the apparent exchangeable inorganic nitrogen species in the Thredbo wetland soil. It is unclear whether the extraction reagent is unable to quantitatively extract exchangeable nitrogen species, or if other nitrogen forms as well as the exchangeable nitrogen species are being extracted. Further investigations of the use of potassium chloride as an extractant

solution needs to be conducted before the use of this solution is accepted as the standard technique for extraction of exchangable nitrogen species from wetland species.

REFERENCES

A.P.H.A. Standard Methods (1980).

Bengtsson N (1924) The determination of ammonia in soil. Soil Sci 18:255-278.

Blackburn TH and Henrisken K (1983) Nitrogen cycling in different types of sediment from Danish Waters. Limnol and Oceanog 28:477-493.

Bremner JM (1965) Inorganic forms of Nitrogen. In "Methods of Soil Analysis Vol. 2" (CA Black ed). Chpt 84 Am Soc Agron Madison Wis.

Brodrick SJ (1985) The influence of secondary treatment effluent on denitrification in a natural wetland. M App Sci thesis. Canberra College of Advanced Education.

Burford JR and Bremner JM (1975) Relationships between the denitrification capacities of soils and total water soluble and readily decomposable soil organic matter. Soil Biol Biochem 7:389-394.

Haines E Chalmers A Hanson R and Sherr B (1977) Nitrogen pools and fluxes in a Georgia salt marsh. Estuarine Interactions (ML Wiley ed) p241-253.

Henriksen K (1980) Measurement of in situ rates of nitrification in sediment. Microb Ecol 6:329-337.

Jenkins MC and Kemp WM (1984) The coupling of nitrification and denitrification in two estuarine sediments. Limnol and Oceanog 29:609-619.

Klingensmith KM and Alexander V (1983) Sediment nitrification, denitrification and nitrous oxide production in a deep Arctic Lake. Appl and Environ Microbiol 46:1084-1092.

Sahrawat KL (1979) Evaluation of some chemical extractants for the determination of exchangeable ammonium in tropical rice soils. Comm in Soil Sci and Plant Anal 10:1005-1013.

Smith CJ and Patrick WH (1983) Nitrous oxide emission as affected by alternate anaerobic and aerobic conditions from soil suspensions enriched with ammonium sulphate. Soil Biol Biochem 15:693-697.

Sorensen J (1978) Denitrification rates in marine sediment as measured by the acetylene inhibition technique. App and Env Microbiol 36:139-143.

Terry RE and Nelson DW (1975) Factors influencing nitrate transformations in sediments. J. Environ Qual 4:549-553.

Received May 19, 1986; accepted November 1, 1986