

Evaluation of a Suitable Extractant for the Extraction of Inorganic Nitrogen from Coal Mine Soils

M.Q. KHAN, S.H. SHAH¹ AND MUSHK-I-ALAM

Faculty of Agriculture, Gomal University, D.I. Khan, Pakistan.

¹*Soil Biochemistry, Agricultural Research Institute, Temab, Peshawar, Pakistan.*

(Received 8th May, 1990, revised on 4th August, 1990)

Summary: Various Potassium salts (0.5 M K₂SO₄, 1 M KCl and 1 M CH₃COOK) were tested for the extraction of inorganic nitrogen (NH₄, NO₃ & NO₂-N) from coal mine soils. Although the amount of NH₄-N extracted by various extractants was significantly different, there was no regular increase/decrease in the levels of NH₄-N extracted by the various salt solutions. The amount of NO₃-N extracted by KCl was significantly more compared to K₂SO₄ or CH₃COOK which was attributed to the Cl ions interference in the colorimetric determination of NO₃-N. The K₂SO₄ salt solution did not interfere and was, therefore, recommended to be used as an extractant for the colorimetric determination of soil inorganic nitrogen.

Introduction

Plant available nitrogen is usually a major plant growth limiting factor in coal mine soils. Measurement of inorganic nitrogen (exchangeable ammonium, nitrite and nitrate) in soil is an accepted and straight forward procedure. The methods which have been adopted for determination of exchangeable ammonium, nitrite and nitrate in soils involve extraction of these forms of nitrogen and analysis of the extract by colorimetric or distillation techniques. Colorimetric methods have been extensively employed for determination of inorganic forms of nitrogen in soil extracts.

Various chemical salt solutions, such as potassium chloride, potassium sulphate, potassium acetate, sodium chloride, calcium chloride and Morgan's reagent (10% sodium acetate, 3% acetic acid pH 4.8) varying both in concentration and pH have been used for extracting inorganic forms of nitrogen [1]. Jackson [2] recommended the use of 10% sodium chloride solution acidified to pH 2.5 for extracting ammonium-N in soils. Sahrawat and Prasad [4] proposed the use of Morgan's reagent for simultaneous extraction of ammonium, nitrite and nitrate nitrogen from soil. Sahrawat [5] compared potassium chloride, sodium chloride, sodium acetate and Morgan's reagent solutions for extracting ammonium nitrogen from rice soils. He reported the efficiency of these chemicals in the following descending order by using the recovery of the known amount of ammonium-N applied to soils as a criterion.

KCl > NaCl > CH₃COONa > Morgan's reagent.

Extraction is one of the most important steps for the colorimetric determination of ammonium and other inorganic forms of nitrogen in the coal mine soil. In order to avoid any problem in the preparation of extracts for determination of ammonium, nitrite and nitrate-N in soils, it is necessary to obtain clear, colourless extracts which do not contain substances that interfere with the colorimetric method of determination of these forms of nitrogen. It has been reported by Bremner [1] that procedures involving extraction with potassium chloride or sodium chloride solutions yielded turbid or coloured extracts, and chloride interfered with the methods generally favoured for colorimetric determination of nitrate. Moreover, it is also important to use an extractant that contains very low levels of inorganic nitrogen as impurities or that can be purified very easily. The aim of this experiment was to select a suitable extractant for coal mine soils amongst the various potassium salts commonly used for the extraction of inorganic nitrogen from soils.

Materials and Methods

Reagents

Analar grade reagents and deionized water was used throughout.

1. Potassium sulphate solution (0.5 M)
2. Potassium chloride solution (1 M)
3. Potassium acetate solution (1 M)

The potassium sulphate and potassium chloride solutions were prepared from their respective salts using deionized water. The solutions were boiled and stirred at pH 11.0 for 15 minutes and then readjusted to pH 5.5 - 6.0 after cooling.

The potassium acetate solution was not boiled at pH 11.0 keeping in view the trace amount of ammonium contamination and buffering nature of the salt. Khan [3] reported negligible amounts of ammonium (0.1 - 0.2 mg/kg salt) in the batch number of potassium acetate used in this experiment.

Soil samples

Eight soil samples collected from the following experimental plots at Baads were used as test samples.

1. Naturally vegetated area

No treatment had been applied to this area of the bing and the spoil had become naturally vegetated mainly with trees and associated undergrowth. Two soil samples one each from organic and mineral horizons from this area were collected. The sample from the organic horizon was further subdivided horizontally into three portions, indicated as natural organic 1,2 and 3.

- 2 - Chicken manure treated plot.
- 3- NPK fertilizer treated plot.
- 4- Birdsfoot trefoil species plot
- 5- White clover species plot.

The above mentioned four plots had been specifically reclaimed and sown with a grass/clover mixture or as otherwise mentioned.

Procedure

An amount of fresh soil (partially air-dried to allow sieving through 4 mm sieve) equivalent to 2.5 g on oven dry basis was shaken for 2 hours at 2°C with 50 cm³ of extracting solution. The suspensions were filtered through previously washed Whatman filter paper No. 40 and the filtrates obtained were analysed for ammonium, nitrite and nitrate-N with a Technicon Auto-Analyzer. All the analytical standards used contained equal concentrations of the corresponding extractant as the soil extracts.

Results and Discussion

The criteria used for the selection of the extractant were [1] amount of inorganic nitrogen extracted from soil samples [2] level of inorganic nitrogen in the extractants [3] interference with colorimetric method of nitrate-N determination.

Some of the results of the experiment, as shown in Table-1 indicate that significantly different amounts of ammonium nitrogen were extracted by the different extractants from half of the coal mine soil samples tested. There was, however, no trend whereby one extractant consistently removed more ammonium nitrogen than another.

Table-1 Ammonium-N extracted from 8 soil samples using 0.5 M potassium sulphate, 1M potassium chloride and 1 M potassium acetate

Sample	Ammonium-N (mg/kg oven dry soil)		
	Potassium sulphate	Potassium chloride	Potassium acetate
Natural mineral	1.3	1.4	1.4
Natural organic 1	19.4	18.7	20.2
Natural organic 2	9.0	9.2	10.5*
Natural organic 3	3.0	3.0	2.8
Chicken manure plot	4.5	2.6	2.8*
Fertilizer plot	3.3	3.0	2.6
Birdsfoot trefoil plot	0.1	0.1	1.2*
White clover plot	2.5	2.2	3.3*

*indicates a significant difference between extraction solution (P < 0.001)

Table-2. Nitrate-N extracted from 8 soil samples using 0.5 M potassium sulphate, 1 M potassium chloride and 1 M potassium acetate.

Sample	Nitrate-N (mg/kg oven dry soil)		
	Potassium sulphate	Potassium chloride	Potassium acetate
Natural mineral	0.2	0.3	0.0 *
Natural organic 1	0.2	0.3	0.3 *
Natural organic 2	0.6	1.9	0.2 *
Natural organic 3	0.9	1.8	0.0 *
Chicken manure plot	0.6	1.0	0.5 *
Fertilizer plot	0.3	0.7	0.1 *
Birdsfoot trefoil plot	0.6	0.9	0.5 *
White clover plot	0.2	0.5	0.2 *

*indicates a significant difference between extraction solution (P < 0.001)

The results of the experiment documented in Table-2 show that in spite of the very small amounts of nitrate nitrogen extracted by different reagents

from various soil samples, potassium chloride (1 M) extracted significantly more nitrate nitrogen from all the coal mine soil samples. The use of potassium chloride (1 M) cannot be recommended because it sometimes yielded colored extracts and also due to the chloride ion interference in the colorimetric determination of the nitrate nitrogen. The comparatively higher levels of nitrate nitrogen extracted by this salt solution may be due to the chloride ion interference.

As for as the level of contamination is concerned, Khan [3] studied the nitrogen contamination in several batches of potassium sulphate, potassium chloride and potassium acetate. He reported.

1 - Very low levels of nitrite in the various batches of potassium salts.

2- Higher and variable levels of ammonium in potassium sulphate compared with Pot:chloride and Pot:acetate salts.

3 - Comparatively higher levels of nitrate in potassium acetate salts.

Keeping in view the higher and variable levels of nitrate-N contamination in potassium acetate salts, its use will be a bit risky because it cannot be purified easily as there is no simple method for the removal of nitrate-N.

Potassium sulphate solution does not interfere in the nitrate nitrogen analysis and contains low levels of nitrate nitrogen contamination. It does contain ammonium nitrogen contamination but it can be easily purified by boiling and stirring for fifteen minutes at pH 11.0 (Khan [3]). Ammonium is converted into ammonia by increasing the pH of the solution to 11.0 with potassium hydroxide solution. Boiling and stirring for a period of 15 minutes will expel all the ammonia in the solution.

Coal mine soil extracted with potassium sulphate produced precipitation in some cases when the extract was stored at 2°C for 2 days but this drawback can be ignored as it does not affect the amount of nitrogen extracted from the soil. It was therefore, recommended to use 0.5 M potassium sulphate as an extractant for the extraction of ammonium, nitrate and nitrite nitrogen from the coal mine soils.

References

1. J.M. Bremner, *Methods of Soil Analysis Part 2, Agronomy*, 9, 1179 (1965).
2. M.L. Jackson, *Soil Chemical analysis*, Prentice Hall, New Delhi, 18 (1967).
3. M.O. Khan, Ph.D. Thesis, Glasgow Univ. (1987).
4. K.L. Sahrawat and R. Prasad, *Plant and Soil*, 42, 305 (1975).
5. K.L. Sahrawat, *Commun. In Soil Science, Plant Anal*, 10, 1005 (1979).