

LOSS OF NITRATE NITROGEN IN SOIL COLUMNS*

M.M. IQBAL** and B.P. WARKENTIN

McGill University, Montreal, Canada

(Received September 29, 1976; revised September 18, 1977)

Abstract. Extremely high losses of $\text{NO}_3\text{-N}$ were observed in the lab columns of sandy loam and clay soils amended with or without sucrose and incubated at room temperature for one week at two moisture levels; field capacity and saturation in the lower half of columns. The sucrose addition had no significant influence on N losses, because the soils did not seem to be lacking in the energy source. Also, there was no marked difference in the pattern of losses between the two moisture levels showing that conditions are such that N loss will also occur at field capacity.

During a field study on nitrogen balance following application of animal manure to soil,⁵ it was found that 77-100% of the applied N was not accounted for in leaching, uptake by corn or residual N in soil. The authors suggested that such losses could be due either to denitrification or to conversion of available N into unavailable forms. A follow up study to estimate the losses of added N from soils used in the field study was initiated in the lab to determine what mechanisms led to the N loss in the field and to what extent these mechanisms could contribute to the losses of applied N from soil. The experiments was designed to study the influence of water content and readily oxidizable energy sources on N losses.

Materials and Methods

The soils used in the study were Ste. Rosalie clay and Ste. Demase sandy loam surface soils. They were air-dried, passed through a 2 mm sieve and stored. The physicochemical analysis of the soils is as under:

		Ste. Rosalie clay	Ste. Demase sandy loam
Carbon	(%)	1.2	2.4
Total N	(%)	0.114	0.143
pH(1:1 soil water suspension)		5.3	5.3
Clay	(%)	50	14
Sand	(%)	20	75

Clear lucite cylinders 6.94 cm inside diameter and 30 cm long were packed as uniformly as possible with 1362 g each of the soils to a bulk density of 1.2 g cm^{-3} . The following moisture treatments were established in the soil columns. The calculated amounts of water were added to each soil.

(a) Field capacity moisture content throughout the length of soil column. The field capacity moisture content, as determined in the lab, was 25% for sandy loam and 28% for clay.

(b) Saturation in the lower half of soil columns. The saturation moisture content was 52% and 56% for sandy loam and clay soils, respectively.

The leachate from the field capacity columns, after incubation, was collected by an opening at the bottom. The leachate from the half-way saturated columns was obtained by a horizontal 'U' shaped glass tube, one end of which was attached to the bottom of the column and the other was perpendicular to the length of soil columns in the middle. A readily oxidizable energy source in the form of sucrose was added in solution to half of the columns at the rate of 0.1% (1.3 g/column).

Incubation Procedure

The behaviour of nitrate in soil can accurately be traced with reference to chloride. Since chloride is not changed through microbial activity, it can provide a reasonable estimate of mineral N transformations in soil. In the present study too, chloride was used as a tracer for nitrate behaviour. The columns were wetted with the calculated amounts of water containing 0.005 M CaSO_4 . The Ca^{++} was used to prevent dispersion of soil particles. Then 25 ml of solution containing 25 mg $\text{NO}_3\text{-N}$ and 5 mg Cl was added to each column at the top. The nitrate plus chloride solution was mixed with the CaSO_4 solution used for wetting the soil. The solution was prepared by dissolving 6.52 g of $(\text{CaNO}_3)_2 \cdot \text{H}_2\text{O}$ and 0.416 g of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ litre of distilled water. Columns were allowed to stand at room temperature for one week. At the end of this period, the soil columns were displaced with 0.005 M CaSO_4 solution. The displacing solution was applied until the leachate contained essentially no NO_3 or Cl.

Treatment of Leachate

The leachates obtained from the sandy loam columns were of yellowish red colour which interfered in the NO_3

*This is a part of Ph.D. Thesis submitted by the senior author to McGill University.

**Present address: Atomic Energy Agricultural Research Centre, Tandojam, Pakistan.

analysis on autoanalyser. The colour, which was due to iron, was removed by treating the leachate with NaOH solution at pH 10-12. The solution was filtered and the clear filtrate after adjusting the pH to 7-9 range was used for NO₃ and Cl determinations. The colour was more intense in saturated than in field capacity treatments. The leachates were analysed for NO₃ and Cl on the Technicon Autoanalyser. Excess Cl, which interferes in the NO₃ analysis, was removed by

adding 2 ml of 0.02 N Ag₂SO₄ solution to about 25 ml of the leachate. A turbid precipitate appeared. It was filtered and the clear filtrate was used for the NO₃ analyses. Nitrate was determined by the colorimetric method of Kamphake *et al.*⁶ using a heating bath to promote colour development. Chloride was determined by Method No. 12-68 of Technicon Autoanalyser Methodology.

After collecting 400 ml of leachate, the columns

TABLE 1. AMOUNTS OF NO₃-N AND Cl RECOVERED IN LEACHATE FROM SANDY LOAM AND CLAY COLUMNS.

Moisture treatment	Leachate fractions (ml)	NO ₃ - N(mg)*			Cl (mg)*		
		Control	NO ₃	NO ₃ +Sucrose	Control	NO ₃	NO ₃ +Sucrose
<i>Sandy loam</i>							
	50	0.04	0.30**	0.23	19.8	22.3	21.8
Field capacity	50	0.01	0.11	0.06	8.0	9.6	11.3
	100	0.02	0.14	0.12	4.8	4.5	5.0
	100	0.10	0.10	0.06	1.1	2.2	2.4
	100	0.07	0.05	0.06	2.6	2.2	2.1
	<u>400</u>	<u>0.24</u>	<u>0.70</u>	<u>0.53</u>	<u>34.3</u>	<u>40.8</u>	<u>42.6</u>
	Net recovery	—	<u>0.46</u>	<u>0.29</u>	—	<u>6.5</u>	<u>8.3</u>
Half-way saturated	50	0.24	0.64	0.58	19.3	18.1	16.5
	50	0.01	0.04	0.03	7.0	8.5	6.9
	100	0.02	0.05	0.03	4.6	5.8	5.0
	100	0.02	0.06	0.02	2.0	3.0	2.6
	100	0.02	0.06	0.06	1.6	2.7	1.8
		<u>400</u>	<u>0.31</u>	<u>0.85</u>	<u>0.72</u>	<u>34.5</u>	<u>38.1</u>
	Net recovery	—	<u>0.54</u>	<u>0.41</u>	—	<u>3.6</u>	<u>1.7</u>
<i>Clay</i>							
Field capacity	50	0.44	0.38	0.52	25.0	25.0	25.8
	50	0.15	0.38	0.04	12.8	14.1	14.0
	100	0.26	0.95	0.03	5.4	7.9	9.1
	100	Trace	0.25	0.01	1.6	2.5	2.4
	100	0.23	0.59	0.15	0.6	1.2	1.3
	<u>400</u>	<u>1.08</u>	<u>2.55</u>	<u>0.75</u>	<u>45.4</u>	<u>50.7</u>	<u>52.6</u>
	Net recovery	—	<u>1.47</u>	<u>0.3</u>	—	<u>5.3</u>	<u>7.2</u>
Half-way saturated	50	1.68	1.35	1.20	24.5	24.4	19.5
	50	0.01	0.01	0.10	12.5	12.9	9.3
	100	0.01	Trace	Trace	4.8	9.1	7.5
	100	0.01	0.06	0.01	0.9	1.0	5.7
	100	0.01	0.05	0.01	0.6	0.3	0.6
		<u>400</u>	<u>1.72</u>	<u>1.46</u>	<u>1.32</u>	<u>43.3</u>	<u>47.7</u>
	Net recovery	—	<u>0.26</u>	<u>0.40</u>	—	<u>4.4</u>	<u>1.3</u>

*NO₃-N added: 25 mg/column. Chloride added: 5 mg/column.

**Each figure is an average of 2 determinations except for control columns where only one determination was made.

were cut into two halves and the soil was analyzed for NO_3 to find out the amount remaining in the soil.

Results and Discussion

Nitrate Recovered in Leachate. The amounts of nitrate and chloride recovered in 4 successive fractions of leachate totaling 400 ml are given in Table 1. Fairly high amount of chloride was present in the soil originally as is indicated by the chloride recovery in the control columns. Recovery of added Cl was variable. In NO_3 columns of clay soil almost all of the added Cl (5 mg) appeared in 400 ml of the leachate whereas in NO_3 columns amended with sucrose, the recovery was erratic. It resulted from the low permeability of these columns which, in some of the columns, was so low that the displacing solution remained ponded on the soil surface for a long time. Attempts to improve the leaching by inserting a wire into the columns to remove air bubbles apparently resulted in the solution passing through the column as such.

Amount of NO_3 recovered in the leachate was very little. This low recovery of nitrate compared to chloride indicates that NO_3 is either retained in the soil or has been lost from the system.

Nitrate Left in Soil. The amount of nitrate left in the soil columns after 400 ml of leachate had been collected, is given in Table 2. More nitrate was left in sandy soil than in clay. The reverse is true for nitrate recovered in leachate. The nitrate content of the lower half of the soil columns was always lower than that of the upper half. High variability is a characteristic of such measurements, but the lower portions of columns were comparatively less variable with respect to NO_3 -N values. The average coefficient of variability was 19% in the upper half of soil columns and 5% in the lower half showing that more uniform and higher losses occurred at the lower depths. It is not uncommon to expect such situations in the field. Low oxygen tension and suitable amounts of readily oxidizable carbon at lower depths may lead to considerable loss of applied nitrogen.¹¹

Effect of Sucrose. Sucrose addition at the rate of 0.1% of soil weight basis does not seem to have any pronounced effect on N losses (Table 1). The only pronounced effect of sucrose was observed in clay columns wetted to field capacity where about one third less nitrate was recovered compared to no sucrose columns. This shows that probably enough carbon is present in these soils naturally (sandy loam= 2.4%, clay= 1.2%) and the added sucrose (1.3 g/column) is over and above the need of soil organisms or retention of NO_3 in the soil.

Similar observations were made by McGarity⁷ who found that addition of 0.1% glucose at various moisture

TABLE 2. NO_3 -N PRESENT IN THE SOIL COLUMNS (mg) AT THE END OF THE EXPERIMENT

Column portion	Control		NO_3 alone		NO_3 + sucrose	
	F.C.	Sat'd.	F.C.	Sat'd.	F.C.	Sat'd.
<i>Sandy loam</i>						
Upper half	11*	12	14	10	14	12
Lower half	11	6	7	6	7	7
	<u>22</u>	<u>18</u>	<u>21</u>	<u>16</u>	<u>21</u>	<u>19</u>
<i>Clay</i>						
Upper half	10	4	8	2	6	2
Lower half	6	1	6	2	3	1
	<u>16</u>	<u>5</u>	<u>14</u>	<u>4</u>	<u>9</u>	<u>3</u>

*Each figure in the treated columns is an average of 2 determinations; in control column of one determination.

levels to a 0-2.5 cm sample of permanent pasture soil with a carbon content of 2.6% did not stimulate greatly the rate of gas production. The amount of gas produced was actually less with glucose than without. He suggested that more NO_3 -N was immobilized as microbial protein in the presence of glucose. Perhaps this was also a factor in the present study.

Effect of Moisture Content. It is seen from Table 1 that less NO_3 was recovered in columns at field capacity than those saturated in the lower half. This is contrary to expectations.^{7,8,10} The lower losses in the half way saturated columns may be attributed to the lack of a sufficient induction period. Table 1 shows that the amount of nitrate collected in the first 50 ml fraction of the leachate from half way saturated columns of both soils was higher than the corresponding field capacity columns. The nitrate recovered in the subsequent aliquots of leachate from the half saturated columns was lower. This indicates that an induction period, greater than one week, is needed to have the comparable losses in half way saturated columns.

In general, more nitrate was left in the soil at field capacity than in half way saturated columns (Table 2). The differences were more marked in clay soil.

Loss of Added Nitrate. The percentage of added NO_3 lost in the various treatments is given in Table 3. It is observed that essentially all of the added N has been lost from the system. In addition, some NO_3 -N mineralized from the soil organic matter (upto 28% in clay and upto 5% in sandy loam) has also disappeared.

TABLE 3. LOSS OF ADDED NITRATE FROM THE SOIL COLUMNS.

Moisture content	Energy source	NO ₃ -N added (mg)	NO ₃ -N recovered in*			NO ₃ -N lost (mg)	% of added N lost
			Leachate (mg)	Soil (mg)	Total (mg)		
<i>Sandy loam</i>							
Field capacity	No sucrose	25	0.5	-0.8	-0.3	25.3	101
	Sucrose	25	0.3	-0.9	-0.6	25.6	102
Half-way saturated	No sucrose	25	0.5	-1.7	-1.2	26.2	105
	Sucrose	25	0.4	0.6	1.0	24.0	96
<i>Clay</i>							
Field capacity	No sucrose	25	1.5	-2.2	-0.7	25.7	103
	Sucrose	25	-0.3	-6.7	-7.0	32.0	128
Half-way saturated	No sucrose	25	-0.2	-1.1	-1.3	26.3	105
	Sucrose	25	-0.4	-1.1	-1.5	26.5	106

*After subtracting the values from control columns.

As the NO₃ recovered in leachate and the NO₃-N remaining in the soil have been determined the estimated N loss is due either to tie-up of N into microbial tissue or to denitrification. The net loss is probably the result of a combination of several reactions.

The major loss of N probably has occurred through immobilization. In this process, soil microorganisms use inorganic N for the synthesis of their cell tissue resulting in the tie-up of N in organic form which is somewhat resistant to further biological degradation. Removal by this mechanism is limited by the availability of energy source. An easily decomposable energy source when added to soil stimulates the activity of microorganisms greatly. They utilize C in respiration as energy source and N for their body building. They multiply in number considerably until the C content of the material they are decomposing decreases to the extent when C to N ratio of the material approaches to about 10:1. If the energy source is low in N, as sucrose is, the microorganisms will utilize the available NH₄ and NO₃ from soil around them. This disappearance of available N from soil will be the results of immobilization. In our soils, there is enough C present naturally. In addition half of the soil columns received additional sucrose. The low content of inorganic N compared to the easily available C suggests that a considerable amount of N from added NO₃ as well as from soil has been incorporated into microbial cells. The organic N content of soil, which is a good indicator of immobilization, was not determined, hence the

exact amounts of N immobilized are not known.

In denitrification process, nitrogen is lost in the form of molecular N or nitrous oxide gas. Direct measurement of these gases was not made in the present study hence it is difficult to assess the losses due to this process. Losses due to denitrification are generally considered to be of little economic importance in well aerated soils.^{1,4,12} A number of workers,^{2,9} however, suggest that denitrification occurs in the anaerobic zones within a generally aerobic soil matrix. Soil moisture in such cases interacts with soil to influence aeration and thence denitrification. The N loss in field capacity columns in the present study may, in part, be due to this mechanism.

Some N may have disappeared as a result of accumulation of some intermediates which lie between nitrite and molecular N. Nommik⁸ found the accumulation of such compounds in certain stages of his denitrification experiments. These compounds were not trapped by the analytical methods applied. The disappearance of NO₃ in this way was not accompanied by an evolution of corresponding amounts of N₂O or N₂ gases. He termed such losses as 'apparent N losses' and observed extremely high losses of this kind from Ultuna soil. He cited other workers who had obtained similar 'apparent N losses'.

Conclusions

Extremely high losses (almost 100%) of the applied

N from sandy loam and clay soils as influenced by water content and energy source were observed in the lab. A major portion of these losses was considered to be due to immobilization. It is doubtful whether the conditions in the field are as ideal as those prevailing in the laboratory, but still the data show the possibility and magnitude of this process when the conditions become near ideal. It is a common practice to add to soil the organic material which is low in N for maintaining the soil organic matter content. For decomposition of such material, the soil population will remove all the available NH_4 and NO_3 present in the soil for their building and proliferation. On the other hand, if the added organic material is high in N, its decomposition will release NH_4 or NO_3 into the soil. This implies that organic material high in C and low in N should not be applied to a growing crop otherwise the crop will show signs of N deficiency.

References

1. F.E. Broadbent, Proc. Symp. on Nitrogen in Soil and Water (Univ. of Guelph, 1971), p. 56.
2. F.E. Broadbent and F.E. Clark, Soil Nitrogen (American Society of Agronomy, Madison, Wisconsin, 1965), p. 344.
3. J. C. Corey, D. Kirkham and D.R. Nielsen, Iowa Academy of Sci., 47 (1967).
4. E.T. Croswell and A.E. Martin, Soil Biol. and Biochem., 6 (1974).
5. M.M. Iqbal, Nitrogen Movement and Losses from Application of Animal Wastes to Soil, Ph.D Thesis (McGill Univ., 1973).
6. L.J. Kamphake, S.A. Hannah and J.M. Cohen, Water Res., 1, 205 (1967).
7. J.W. McGarity, Plant and Soil, 14, 1 (1961).
8. H. Nommik, Acta Agric. Scand., 6, 195 (1956).
9. V.B.D. Skerman and I.C. Macrae, Can.J. Microbiol., 3, 215 (1957).
10. M.E. Tusneem and W.H. Patrick, Jr., Bulletin No.657 (Louisiana State Univ. and Agri. Exptl. Stn., 1971).
11. M.G. Volz, H.S. Ardakani, R.K. Schulz, H.L. Stolzy and A.D. McLaren, Agron. J., 68, 621 (1976).
12. S.A. Waksman, *Principles of Soil Microbiology* (Bailliere, Tindall and Cox, London, 1927).