

Nitrogen Contents and Ammonium Fixing Capacities of Soil Separates

DARRELL W. NELSON

Department of Agronomy, Purdue University, West Lafayette, Indiana 47907

Introduction

Nelson *et al.* (1977) have recently found significant positive correlations between the clay content and the levels of organic C, total N, total P, total S, and fixed ammonium N and ammonium fixing capacities of 48 Indiana surface soils. These data suggested that organic matter, fixed ammonium, and ammonium fixing capacities of soils were primarily associated with the clay fraction. However, little information is available on the relative amounts of organic C, total N, and fixed ammonium or ammonium fixing capacities of soil separates. Nommik (1965) in reviewing data from several studies reported that the ammonium fixing capacities of soils tend to increase with increasing clay content, but some studies suggested that the sand and silt fractions also fix added ammonium N. A few studies have shown that the clay fraction has higher contents of fixed ammonium and potassium than silt or sand fractions (Hinman, 1964; McLean and Brydon, 1963), however, Genrich (1972) found that the silt fraction of Iowa soils contained much higher levels of fixed potassium than the clay fraction. The clay fraction of Iowa soils has been shown to contain most of the organic C, total N, and total S (Genrich, 1972).

The lack of definitive information on the distribution of organic matter, fixed ammonium, and ammonium fixing capacities in soil separates prompted this investigation. The objective of the work was to determine the relative amounts of organic C, total N, exchangeable ammonium, fixed ammonium, and ammonium fixing capacity present in sand, silt, and clay fractions of diverse agricultural soils.

Materials and Methods

The soils selected for the study (Table 1) represented a wide range in texture, organic matter content, clay mineralogy, and fixed ammonium N concentration. The soils were collected from throughout North America: Clarence and Marshall from Iowa; Yolo from California, Nicollet from Minnesota, and Whatcom from British Columbia. The Clarence and Yolo samples were subsoils, whereas the other samples were surface soils. The clay fraction of Marshall, Nicollet, and Yolo soils was composed largely of montmorillonite with lesser amounts of illite and kaolinite. The principal clay mineral in Clarence soil was illite, although significant amounts

TABLE 1. *Characteristics of soils used in the study.*

Soil ⁺	Depth	Clay	Silt	Sand	Organic C	Total N	Fixed NH ₄ ⁺ - N	pH
	cm	% by weight			μg/g			
Clarence sic	38-58	43	44	14	0.87	920	217	6.0
Yolo cl	38-58	33	47	21	0.79	810	273	7.6
Marshall sic1	0-15	33	65	2	2.20	2090	268	4.8
Nicollet 1	0-15	24	37	39	1.56	1340	85	6.9
Whatcom sil	0-15	11	75	14	1.27	810	96	6.4

⁺ sic, silty clay; cl, clay loam; sic1, silty clay loam; 1, loam; sil, silt loam.

of montmorillonite, vermiculite, and kaolinite were present. Chloritized vermiculite was the predominate clay mineral in the Whatcom soil.

Soil sample (< 2-mm) were separated into sand, silt, and clay fractions by an ultrasonic dispersion and sieving-sedimentation technique (Genrich and Bremner, 1974) designed to preclude alteration of the chemical composition of fractions during separation. To verify the efficacy of the separation procedure, the mechanical composition of soils was also determined by the pipette method following dispersion of solids with sodium metaphosphate and hydrogen peroxide (Kilmer and Alexander, 1949). Organic C, total N, fixed ammonium N, and exchangeable ammonium N in whole soils and soil separates were determined by procedures of Mebius (1960), Bremner (1965), Silva and Bremner (1966), and Keeney and Bremner (1966), respectively. The pH of whole soils was measured by glass electrode in a 1:1 soil:water mixture.

Ammonium fixing capacity was determined by three methods. In all methods the whole soils or separates (0.5 g) were amended with 1 ml of 1N NH_4Cl , incubated or dried, and analyzed for fixed ammonium N by the procedure of Silva and Bremner (1966). The difference in fixed ammonium N before and after amendment of the sample with 1N NH_4Cl was defined as ammonium N fixing capacity. After soils or soil separates were amended with 1N NH_4Cl a portion of the samples were allowed to stand at 25°C for 24 hours (moist ammonium N fixing capacity), a portion of the samples were dried in a dessicator at 25°C (air-dry ammonium N fixing capacity), and the remainder of the samples were dried at 105°C (oven-dry ammonium N fixing capacity).

All concentration data are expressed on a moisture-free basis and represent averages for three replicate determinations. Data presented in Tables 2 and 4 represent average values for the 5 soils. Data for all soils followed the trends exhibited by the average values for the 5 soils. Linear correlation analyses were performed by procedures outlined by Steele and Torrie (1960).

Results and Discussion

On the average the concentrations of organic C, total N, exchangeable ammonium N, and fixed ammonium N in the clay fraction were higher than those in the whole soils or silt and sand fractions (Table 2). Likewise, the ammonium N fixing capacities of soils and separates followed the order: clay > whole soil > silt > sand. The concentrations of organic C, total N, and exchangeable ammonium N in the clay fraction were two and three times the levels in the whole soils and the silt fractions, respectively. However, the fixed ammonium N and moist and air-dry ammonium N fixing capacities were only about 1.5 times higher in the clay fraction than in the whole soil. Oven-drying NH_4Cl -treated samples markedly increased the

TABLE 2. *Average organic C, total N, and fixed ammonium concentrations and ammonium fixing capacities of whole soils and soil separates.*

Soil component	Organic C	Total N	Organic C/N	Exch. $\text{NH}_4^+ - \text{N}$	Fixed $\text{NH}_4^+ - \text{N}$	Ammonium Moist	fixing Air-dry	capacity Oven-dry
	%			$\mu\text{g/g}$		μg	N/g	
Whole	1.34	0.119	13.6	15	188	97	185	201
Clay	2.92	0.278	12.0	28	311	127	276	414
Silt	0.98	0.066	18.8	8	130	47	69	87
Sand	0.66	0.021	44.6	2	60	17	33	33

ammonium N fixing capacity of the clay fraction as compared to the increases obtained from oven-drying whole soils.

The organic C/organic N ratio in whole soils and soil separates followed the order: clay < whole soil < silt < sand (Table 2). This finding suggests that organic matter associated with the clay fraction was well humified (low C/N) whereas organic matter in the sand fraction was largely undecomposed residues (high C/N). Air-drying markedly increased the ammonium N fixing capacity of soils and soil separates, but oven-drying had little effect above that of air-drying in all sample except the clay fractions. This finding suggests that the mechanism for ammonium fixation in clay differs from that of the other soil separates.

It is significant that the silt fraction of soils contained relatively high concentrations of organic C, total N, and fixed ammonium N and had substantial ammonium N fixing capacity because silt is the predominate separate in many Indiana soils. The sand fraction of soils contained higher levels of organic matter and fixed ammonium N than would be expected because many soil scientists consider sand to be essentially inert. It was especially surprising to find that sand had a modest ammonium N fixing capacity.

Calculations were made to determine if the method used to obtain the soil separates altered the chemical composition of fractions. The organic C, total N, and fixed ammonium N concentrations of Nicollet and Whatcom soils were determined by chemical analysis and estimated by summing the amounts present in the three size fractions (Table 3). In all cases the values obtained by summation agreed with

TABLE 3. *Comparison of organic C, total N, and fixed ammonium N concentrations determined directly on whole soils and calculated from analysis of soil separates.*

Soil	Method of estimation	Organic C	Total N	Fixed NH_4^+ -N
		----- % by weight -----		$\mu\text{g/g}$
Nicollet 1	Direct	1.56	0.134	85
	Sum of separates	1.46	0.133	95
Whatcom sil	Direct	1.27	0.081	96
	Sum of separates	1.29	0.083	87

those from direct analysis. This finding indicates that the method used to fractionate soils did not alter the characteristics of soil separates.

Although the clay fraction represented an average of only 29% of the soil mass, 58% of the organic C, 65% of the total N, and 52% of the fixed ammonium N in soil was present in this fraction (Table 4). In addition, the clay fraction was

TABLE 4. *Percentage of the total amounts of organic C, total N, fixed ammonium N, and ammonium-fixing capacities of soils present in soil separates.*

Soil component	Mechanical composition	Organic C	Total N	Fixed NH_4^+ -N	NH_4^+ -N fixing capacity		
					Moist	Air-dry	Oven-dry
				----- % of total amount -----			
Clay	29	58	65	53	53	62	67
Silt	53	40	34	42	43	33	30
Sand	18	3	2	5	4	5	4

responsible for 53 to 67% of the ammonium N fixing capacity of soils. The silt fraction representing 53% of total soil mass contained 40, 34, and 42% of the organic C, total N, and fixed ammonium N in soils, respectively. From 30 to 43% of the ammonium N fixing capacity of soils was associated with the silt fraction. The sand fraction (18% of soil mass) accounted for < 5% of the organic C, total N, and fixed ammonium N in soils and was responsible for < 5% of the ammonium N fixing capacity of soils. These findings point out the importance of the clay fraction as a 'storehouse' for nutrients, but also suggest that the silt fraction is of significance in regards to nutrient cycling in soils.

Linear correlation analyses were performed to determine statistical relationship between the amounts of soil separates (clay and silt) and the fixed ammonium N concentration and the ammonium N fixing capacities of soils (Table 5). The fixed ammonium N levels were correlated with the % clay in soils, but were not correlated with % silt. However, a higher correlation coefficient was obtained between fixed ammonium N and the sum of % clay and a fraction (.33 to .5) of % silt than between fixed ammonium N and % clay. This finding suggests that the silt fraction is important in contributing to the fixed ammonium N level in soils, but the significance of the silt fraction is much less than that of the clay fraction. The moist and air-dry fixing capacities of soils were correlated with the % clay, but not with % silt. Combining % clay with some portion of % silt did not result in improved correlations. This finding suggests that silt plays a small role in influencing the magnitude of ammonium N fixation in soils although the silt fraction does contribute to the fixation capacity.

Literature Cited

1. BREMNER, J. M. 1965. Total nitrogen. In C. A. Black (ed.) *Methods of Soil Analysis*. Part 2. American Society of Agronomy, Madison, Wis. pp. 1149-1178.
2. GENRICH, D. A. 1972. Isolation and characterization of sand-, silt-, and clay-size fractions of soils. Ph.D. Thesis. Iowa State Univ., Ames, Ia., Diss. Abstr. 33:1342B. Univ. Microfilms, Ann Arbor, Mich.

TABLE 5. *Relationships between the proportion of soil separates and the fixed ammonium N concentrations and ammonium fixing capacities of soils.*

Independent variable	Dependent variable	Correlation + coeff. (r ²)
% clay	Native fixed NH_4^+ -N, $\mu\text{g/g}$	+ .54*
% silt	Native fixed NH_4^+ -N, $\mu\text{g/g}$	+ .00
% clay + 0.5 (% silt)	Native fixed NH_4^+ -N, $\mu\text{g/g}$	+ .73 **
% clay + .33 (% silt)	Native fixed NH_4^+ -N, $\mu\text{g/g}$	+ .74**
% clay	Moist NH_4^+ -N fixing cap., $\mu\text{g/g}$	+ .63*
% silt	Moist NH_4^+ -N fixing cap., $\mu\text{g/g}$	+ .50
% clay + 0.5 (% silt)	Moist NH_4^+ -N fixing cap., $\mu\text{g/g}$	+ .14
% clay + 0.33 (% silt)	Moist NH_4^+ -N fixing cap., $\mu\text{g/g}$	+ .32
% clay	Air-dry NH_4^+ -N fixing cap., $\mu\text{g/g}$	+ .71 *
% silt	Air-dry NH_4^+ -N fixing cap., $\mu\text{g/g}$	+ .38
% clay + 0.5 (% silt)	Air-dry NH_4^+ -N fixing cap., $\mu\text{g/g}$	+ .26
% clay + 0.33 (% silt)	Air-dry NH_4^+ -N fixing cap., $\mu\text{g/g}$	+ .46

+ *, significant at the 95% confidence level; **, significant at the 99% confidence level.

3. GENRICH, D. A. and J. M. BREMNER. 1974. Isolation of soil particle-size fractions. *Soil Sci. Soc. Amer. Proc.* 38:222-225.
4. HINMAN, W. C. 1964. Fixed ammonium in some Saskatchewan soils. *Can. J. Soil Sci.* 44:151-157.
5. KENNEY, D. R. and J. M. BREMNER. 1966. Determination and isotope ratio analysis of different forms of nitrogen in soils. 4. Exchangeable ammonium, nitrate, and nitrite by direct distillation methods. *Soil Sci. Soc. Amer. Proc.* 30:583-587.
6. KILMER, V. J. and L. T. ALEXANDER. 1949. Methods of making mechanical analysis of soils. *Soil Sci.* 68:15-24.
7. MCLEAN, A. J. and J. E. BRYDON. 1963. Release and fixation of potassium in different size fractions of some Canadian soils as related to their mineralogy. *Can. J. Soil Sci.* 43:123-134.
8. MEBIUS, L. J. 1960. A rapid method for determination of organic carbon in soils. *Anal. Chim. Acta* 22:120-124.
9. NELSON, D. W., C. B. ROTH, and L. E. SOMMERS. 1977. Chemical and mineralogical characteristics of selected Indiana soils. *Proc. Indiana Acad. Sci.* 86:435-444.
10. NOMMIK, H. 1965. Ammonium fixation and other reactions involving a nonenzymatic immobilization of mineral nitrogen in soil. *In* W. V. Bartholomew and F. E. Clark (ed.) *Soil Nitrogen*. American Society of Agronomy. Madison, Wis. pp. 198-258.
11. SILVA, J. A. and J. M. BREMNER. 1966. Determination and isotope ratio analysis of different forms of nitrogen in soils. 5. Fixed ammonium. *Soil Sci. Soc. Amer. Proc.* 30:587-594.
12. STEELE, R. G. D. and J. H. TORRIE. 1960. *Principles and Procedures of Statistics*. McGraw-Hill Book Co., New York, N.Y.