GAU Res. J, 1999. 25 (1) : 9-14

Comparison of Kjeldahl's and Chromic Acid Methods of Nitrogen Determination

B. S. Trivedi, G. G. Patel, R. M. Desai and G. M. Padhiyar**

Soil Science and Agricultural Chemistry Research Project,
Gujarat Agricultural University, Navsari Campus, Navsari-396 450

ABSTRACT

The Kjeldahl's method of total N determination was compared with chromic acid method by analysing variable 30 soil and 29 plant samples. The chromic acid N agreed quite well with Kjeldahl's N in both the cases due to meagre variation in sample estimates and a nonsignificant 't' test. The Kjeldahl's and chromic acid N were significantly positively related with organic carbon of soils.

(Received on 13th Oct., 1997)

INTRODUCTION

Nitrogen, being one of the major nutrients, has significant importance in analysis of soil and plants. It is determined by routine Kjeldahl's method which is labourious and time consuming. Although, various steps have been included for hastening the process of digestion for soils (Bremner, 1965) and plant samples (Jackson, 1967) by modifying the catalysts addition, still however, the method has remained quite lengthy and time consuming. Looking to the principle involved in digestion, any strong oxidising agent can be used for hastening the process and the prolonged time could be reduced. This principle was used by Kempers (1977) for determining total-N through use of chromic acid. Sharma and Sud (1980, 1995) modified the chromic acid procedure for total-N determination and suggested its superiority over routine Kjeldahl's method. Keeping these facts in view, it was thought worth to compare the Kjeldahl's and the chromic acid method by determining total-N from variable soil and plant samples.

MATERIALS AND METHODS

Thirty surface soil samples (0.25 cm depth) were collected from cultivated fields belonging to Valsad, Navsari, Surat, Bharuch, Narmada, Banaskantha, Anand and Junagadh districts. The taxonomic details alongwith data on $CaCO_3$, clay pH, EC and OC (Organic Carbon) as determined by routine methods are given in Table 1. The ranges in $CaCO_3$, pH, EC and OC were 0.06 to 12.95%, 6.8 to 8.1, 0.11 to 0.81 dS m^{-1} and 0.30 to 2.27%. Although the clay content varied between 7

**Present address : Research Scientist (Soil Science), Gujarat Agricultural University, Navsari Campus, Navsari-396 450.

and 62%, most of the samples have clay texture. Twentynine plant samples (Table 2) representing grain and haulm/straw of different plant species were also collected. The total N was determined of these samples by Kjeldahl's method as per routine procedure (Jackson, 1967). In acid digestion method, the soil and plant samples were sieved through a sieve having 250 micron aperture (Sharma and Sud, 1980, 1995). The chromic acid was prepared by dissolving 50.0 g `AR' grade chromium trioxide in 100 ml of double distilled water. For digestion, 1.0 g soil was taken in 150 ml Erlenmeyer flask and 1.0 ml distilled water was added. After 30 minutes, 10 ml H_2SO_4 (1.84 sp. gr.) was added and the content was kept over night. On the next day, it was heated for 5 minutes on hot-plate and after cooling, 1.5 ml of 50% chromic acid was added and again it was heated for five minutes on a hot-plate. At this time, the entire content turns green. If black particles are seen, the content is reheated for 2-5 minutes after adding 1.5 ml of chromic acid. The acid digest then was transferred to one litre distillation flask and repeated washings were given to the flask till a volume of about 300 ml was obtained. The rest of the distillation procedure was same as is employed in Kjeldahl's method. The N was determined finally by titrating the distillate against 0.01 N H_2SO_4. In case of plants, the weight taken was 0.200 g and no water was added for wetting the samples. The rest of the procedure was same as indicated for soil. All the analysis were made in duplicate.

RESULTS AND DISCUSSION

The data on soil (Table 1) indicate very little variation in total N as determined by two methods. The total N as determined by Kjeldahl's method varied between 0.035 and 0.163 per cent whereas in case of chromic method, the range was from 0.034 to per cent. The experimental soils had loamy sand, silty clay loam, clay loam and clay textures with considerable variation in clay (7 to 62%) and organic carbon (0.30 to 2.27%) contents. In spite of this, the chromic acid N showed almost identical values to Kjeldahl's N. The overall mean were 0.089 and 0.0886 per cent, respectively for Kjeldahl's and chromic acid N. The CV percentages were only 2.25 and the `t' test was also nonsignificant indicating suitability of chromic acid method for total N determination from soil.

The C:N ratios determined under Kjeldahl's and chromic acid methods indicated quite meagre variation in individual samples. The overall mean for C:N was 10.47 in Kjeldahl's and 10.48 in chromic acid methods.

The correlation studies were made and the `r' values between clay and OC (+ 0.109) and clay and total N (+ 0.120 and + 0.118) as determined by these two methods were positive but nonsignificant indicating quite low association of OC and total N with clay in present cultivated soils. The findings are in agreement with those of Gupta *et al.* (1984) who observed a positive significant relationship between clay and total N only in continuous grassland and forest soils but not in cultivated mineral soils. The correlation studies were also made between OC and the total N as determined by these two methods. The relations were positive and highly significant with `r' values of + 0.996** for Kjeldahl's and + 0.995** for chromic acid N.

The regression equations developed for these two methods were :

(i) Kjeldahl's

$$N = 0.0015 + 0.0913\ OC \text{ and}$$

(ii) Chromic acid

$$N = 0.0016 + 0.0917\ OC.$$

In above equations, the OC stands for per cent organic carbon as determined by Walkley and Black's (Jackson, 1967) method. The values of the constant of both the equations

TABLE 1

Some of the chemical characteristics including details of taxonomy, total-N as determined by Kjeldahl's and chromic acid methods with C:N ratios in experimental soils.

Sr. No.	Villages	Soil-series	Great group	$CaCO_3$ (%)	Clay (%)	Texture	pH (1:2.5)	EC (1:2.5) dSm^{-1}	OC (%)	Kjeldahl's N (%)	Chromic acid N (%)	Variation over Kjeldahl's N (%)	C:N Kjeldahl method	C:N Chromic acid method
							VALSAD DISTRICT							
1.	Valvada	Sisodara	Vertic Ustochrept	0.80	42	c	6.8	0.11	0.93	0.092	0.093	+1.09	10.11	10.00
2.	Valvadi	Billimoda	Typic Ustochrept	1.0	45	c	7.0	0.32	1.68	0.160	0.157	– 1.88	10.50	10.70
3.	Tiskari	Baldha	Lithic Ustochrept	0.81	40	c	6.8	0.12	0.76	0.070	0.070	0.00	10.86	10.86
4.	Dungari	Billimoda	Typic Ustochrept	5.63	48	c	7.8	0.38	0.89	0.086	0.085	– 1.16	10.35	10.47
	Kumbharia	Gadat	Vertic Ustifluvent	1.10	50	c	7.6	0.25	1.74	0.158	0.156	– 1.27	11.01	11.15
							NAVSARI DISTRICT							
	Hanumanbari	Vadhwania	Vertic Ustochrept	0.85	45	c	7.4	0.31	1.21	0.117	0.119	+1.71	10.34	10.17
	Masa	Jalalpur	Vertic Ustochrept	1.35	48	c	7.1	0.23	0.98	0.103	0.101	– 1.94	9.51	9.70
	Navsari-1	Jalalpur	Vertic Ustochrept	2.52	60	c	7.8	0.34	0.70	0.062	0.061	– 1.61	11.29	11.48
	Navsari-2	Jalalpur	Vertic Ustochrept	2.00	62	c	7.9	0.47	0.89	0.084	0.087	+ 3.57	10.60	10.23
							SURAT DISTRICT							
.	Jamankuva	Vadhwania	Vertic Ustochrept	3.02	54	c	7.6	0.23	0.74	0.072	0.070	– 2.78	10.28	10.57
.	Nanicher	Gadat	Vertic Ustifluvent	1.30	51	c	7.2	0.20	0.62	0.061	0.060	– 1.64	10.16	10.33
12.	Dhata	Jalalpur	Vertic Ustochrept	1.10	55	c	7.5	0.19	0.81	0.078	0.079	+ 1.28	10.38	10.25
13.	Serula	Bedmal	Typic Ustochrept	1.02	46	c	7.1	0.19	0.68	0.063	0.064	+ 1.59	10.79	10.63
14.	Kharvada	Billimoda	Typic Ustochrept	1.12	42	c	6.9	0.22	1.32	0.128	0.125	– 2.34	10.31	10.56
15.	Kholvad	Gadat	Vertic Ustifluvent	2.30	34	sicl	7.6	0.31	0.68	0.058	0.058	0.00	11.72	11.72
16.	Vitan	Mulad	Udit Chromustert	2.00	46	c	7.5	0.37	0.70	0.062	0.062	0.00	11.29	11.29
17.	Velan	Sisodara	Vertic Ustochrept	1.16	44	c	7.6	0.25	0.42	0.043	0.045	+ 4.65	9.77	9.33
18.	Kuved	Onjal-I	Typic Halaquept	2.06	54	c	7.7	0.36	1.14	0.111	0.111	0.00	10.27	10.27

1. 2.	3.	4.	5.	6.	7.	8.	9.	10.	11.	12.	13.	14.	15.
BHARUCH DISTRICT													
19. Bharuch	Haldar	Typic Chromustert	1.00	54	c	7.7	0.27	0.30	0.035	0.034	– 2.86	8.57	8.82
20. Rahadpor	Haldar	Typic Chromustert	0.92	52	c	7.5	0.81	0.88	0.082	0.083	+ 1.22	10.73	10.60
21. Tanchha	Haldar	Typic Chromustert	3.10	54	c	7.9	0.42	0.39	0.039	0.037	– 5.13	10.00	10.54
22. Kosamba	Jalalpur	Vertic Ustochrept	1.60	43	c	7.7	0.31	0.37	0.042	0.043	+ 2.38	8.81	8.60
23. Navanagar	Tadkeshwar	Vertic Ustochrept	1.72	45	c	7.7	0.53	0.95	0.093	0.090	– 3.23	10.22	10.56
24. Vahiyan	Mulad	Udic Chromustert	2.52	45	c	7.7	0.68	1.76	0.163	0.167	+ 2.45	10.86	10.54
NARMADA DISTRICT													
25. Nikoli	Bedmal	Typic Ustochrept	2.02	36	cl	7.6	0.80	1.26	0.117	0.121	+ 3.42	10.77	10.41
26. Khupar	Baldha	Lithic Ustochrept	1.12	37	cl	6.9	0.20	0.80	0.073	0.073	00.00	10.96	10.96
27. Kakadamba	Vadhwania	Vertic Ustochrept	0.88	45	c	6.9	0.30	2.27	0.210	0.211	+ 0.48	10.81	10.76
BANASKANTHA DISTRICT													
28. Dantiwada	Dantiwada	Ustochrept	0.08	7	ls	7.9	0.30	0.50	0.044	0.042	– 4.55	11.36	11.90
ANAND DISTRICT													
29. Anand	Ankhi	Ustochrept	0.06	9	ls	7.6	0.18	0.60	0.059	0.061	+ 3.39	10.17	9.84
JUNAGADH DISTRICT													
30. Junagadh	Junagadh	Ustorthent	12.95	57	c	8.1	0.41	1.06	0.093	0.094	+ 1.08	11.39	11.28
Overall Mean								0.93	0.0890	0.0886	–0.45	10.47	10.48
									't' test	NS			
									(0.05)				
								CV %		2.25			

Note : The notations c, sicl, cl and ls, respectively indicate clay, silty clay loam, clayloam and loamy sand textural classes.

TABLE 2

Nitrogen content in grain and straw/haulm of different plant species as determined by Kjeldahl's and chromic acid methods.

Crop species	*Crop component*		*Kjeldahl's N (%)*	*Chromic acid N (%)*	*Variation over Kjeldahl's N (%)*
Legumes	Gram	(Grain)	3.31	3.33	+ 0.60
	Kideny bean	(Grain)	3.43	3.44	+ 0.30
	Cowpea	(Grain)	3.31	3.33	+ 0.60
	Black gram	(Grain)	3.04	3.06	+ 0.66
	Indian bean	(Grain)	3.89	3.92	+ 0.77
	Green gram	(Grain)	3.04	3.05	+ 0.33
	Green gram	(Haulm)	2.05	2.06	+ 0.49
	Pigeonpea	(Grain)	3.01	3.02	+ 0.33
Oilseeds	Safflower	(Grain)	2.67	2.72	+ 1.87
	Soybean	(Grain)	5.86	5.92	+ 1.02
	Castor	(Grain)	2.97	2.95	– 0.67
	Castor	(Haulm)	1.71	1.72	+0.58
	Sesamum	(Grain)	3.27	3.28	+ 0.31
	Sesamum	(Haulm)	0.84	0.83	– 1.19
	Groundnut	(Grain)	3.36	3.40	+ 1.19
	Groundnut	(Haulm)	1.86	1.84	– 1.08
	Rape-seed	(Grain)	4.09	4.12	+ 0.73
	Niger	(Grain)	3.63	3.65	+ 0.55
	Mustard	(Grain)	3.98	4.01	+ 0.75
	Mustard	(Haulm)	0.96	0.95	– 1.04
Cereals	Sorghum	(Grain)	2.48	2.46	– 0.81
	Wheat	(Grain)	2.51	2.46	– 1.99
	Wheat	(Straw)	0.52	0.52	0.00
	Fingermillet	(Grain)	0.87	0.86	– 1.15
	Kodomillet	(Grain)	0.88	0.90	+ 2.27
	Maize	(Grain)	1.56	1.53	– 1.92
	Pearl Millet	(Grain)	1.86	1.89	+ 1.61
	Paddy	(Grain)	0.98	0.97	– 1.02
	Paddy	(Straw)	0.59	0.58	– 1.69
Overall Mean			2.50	2.51	
		't' test (0.05)	NS		
		CV%	0.99		

are almost identical which further indicate suitability of rapid chromic acid method for total N determination from soil. Bremner (1965) has indicated inability of Kjeldahl's method for oxidising N-containing refractory heterocyclic compounds and azo compounds having N-N and N-O linkages and therefore suggested prolonged digestion with appropriate catalyst. Ordinarily, these compounds are present in high concentration in organic soils or soils containing quite high amount of organic matter but not in cultivated mineral soils which is the case in present study.

For judging the suitability of chromic acid method, the plant samples were selected in such a way that they give very wide variation in total N and hence legumes, oilseeds and cereals were used. The total N content in plant samples (Table 2) varied between 0.52 and 5.86% in Kjeldahl's method and 0.52 and 5.92% in chromic acid method indicating quite meagre variation between the values of N content as determined by these methods. This was supported by almost identical overall mean values in Kjeldahl's N (2.50%) and chromic acid (2.51%). The CV percentages were only 0.99 and the 't' test was also nonsignificant.

Since, the chromic acid method utilizes quite low weight of soil (1.00 g) and plant (0.200 g), the samples have to be passed through a sieve having 250 micron aperture. The H_2SO_4 used for titration should also be properly standardised exactly to 0.01 N strength. Barring these steps, the chromic acid method is quite rapid, economical and does not involve use of catalyst-salt mixture, special digestion apparatus and is free from toxic and corrosive SO_2 fumes. The present study indicated its suitability for determination of total N from variable soil and plant samples.

REFERENCES

Bremner, J. M. 1965. In methods of soil analysis Part-2, Am. Soc. Agron., Madison, Wisc., USA. pp. 1149-1178.

Gupta, R. D., Kaistha, B. P. and Kanwar, B. S. 1984. Native fixed Ammonium and other forms of Nitrogen in some soils under different Bio sequences of Himachal Pradesh. In Nitrogen in Soils, Crops and Fertilizers. *Bull. Indian Soc. Soil Sci.* **13** : 471-482.

Jackson, M. L. 1967. Soil Chemical Analysis, Prentice Hall of India Pvt. Ltd., New Delhi.

Kempers, A. J., 1977. Determination of the C/N ratio in a chromic acid soil digest. *Plant and Soil* **46** : 699-703.

Sharma. R. C. and Sud, K. C. 1980. An improved rapid chromic acid method for determining total nitrogen in surface soils. *J. Indian Soc. Soil Sci.* **28** : 232-235.

Sharma, R. C. and Sud, K. C. 1995. Economics of chromic acid method over Kjeldahl's method for estimation of total nitrogen in plants and soils. National Seminar on development in Soil Sci., 1995, 60th Annual Convention of Indian Soc. Soil Sci., Ludhiana. Abstr. C-8, pp.38.