Effect of Water Pretreatment on Total Nitrogen Analysis of Soils by the Kjeldahl Method[1]

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ABSTRACT

Effective methods are needed for determining total N when studying the fate of N fertilizer applied to soil. This study was concerned with the pretreatment of samples of a Vertisol (calcareous Typic Pellusterts) with water, 20 and 2 mL for macro- and semimicro-Kjeldahl methods, respectively, prior to digestion. The need for such a modification with certain clay soils was originally recommended in 1925, but its requirement in conjunction with modern Kjeldahl methods is rarely reported. The macro- and semimicro-Kjeldahl methods used involved digestion of soil with a H_2SO_4-K_2SO_4-$CuSO_4 \cdot 5H_2O$-Se mixture. Both techniques yielded digestion residues with dark-colored granules and low recoveries of N with soil samples ground to pass through a sieve with 0.8-mm openings (20 mesh), unless the soil was initially treated with water. For instance, N recovery from a surface Vertisol was increased by 29% as a result of a 30-min water soaking prior to a macro-Kjeldahl digestion. Extension of the digestion period up to 5 h after initial clearing did not eliminate the need for the water pretreatment. The requirement for the water pretreatment for semimicro-Kjeldahl methods was greatly reduced when the soil was ground to pass through a sieve with 0.16-mm openings (100 mesh). Increase in soil N due to water pretreatment was reduced from 35 to 6% by grinding the coarser soil to such fineness. A salicylic acid pretreatment, involving water addition after the nitration reaction, permitted satisfactory digestion. The water pretreatment effect appeared to be associated with incomplete digestion of organic N, not with the recovery of fixed NH_4^+, and was found with both surface and subsoils. Water pretreatment of samples from several horizons of an Alfisol (Udic Rhodustalfs) had little effect on recovery of soil N. A water pretreatment is probably not needed to obtain maximum recoveries of N if the nonmodified Kjeldahl method results in a postdigestion soil residue without dense, darkish granules. However, the water soaking was found to reduce bumping during digestion of many soils and is recommended for routine use.

Additional Index Words: **Bal pretreatment, Vertisol, Alfisol, soil particle size, fixed ammonium.**

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THE NEED TO SOAK certain heavy clay soils in water before determination of nitrogen by the Kjeldahl method was first reported by Bal (3). He found that the Kjeldahl nitrogen contents of an Indian "black cotton" soil, ground to pass through a sieve with 1-mm openings, were 0.030 and 0.042% N, respectively, without and with a prior soaking of 20 g of soil with 50 mL of distilled water for 30 min. The Kjeldahl method used involved the addition of $CuSO_4$, K_2SO_4, and 50 to 70 mL of strong H_2SO_4, but was otherwise undefined. Without the water pretreatment, henceforth called the Bal modification, the insoluble residue remaining after digestion was dark, coarse, and contained carbon. Treatment of the residue with water followed by a subsequent second Kjeldahl digestion resulted in additional recovery of nitrogen. The Bal modification was not as important for finely divided fractions of the problem soil.

The Bal modification effect was also demonstrated by Srinivasan (12, 13, 14), who concluded that the effect was associated with the presence of large amounts of clay, and the protection of the organic matter by a coating of silica. Kjeldahl nitrogen was determined by the Gunning and Hibbard method recommended by the Association of Official Agricultural Chemists (2), and involved digestion with a H_2SO_4-K_2SO_4-$FeSO_4$-$CuSO_4$ mixture until the digest was colorless.

Robinson et al. (9) developed a method to determine soil carbon, in addition to nitrogen, by a Kjeldahl digestion technique. The digestion mixture consisted of 25 mL of H_2SO_4, 15 g of K_2SO_4, and 0.3 to 0.4 g of $CuSO_4$ with an unspecified water of crystallization. The digestion was continued for 1 h after the contents of the flask assumed their final color. Recovery of soil carbon from air-dry samples ground to pass through a sieve with 1-mm openings was low, but the problem was eliminated by grinding the samples to pass through a sieve with 0.16-mm openings. Walkley (16), using the same digestion system, found that the Bal modification was not needed for all clay soils ground to pass through a sieve with 1-mm openings. However, water pretreatment of low organic matter, heavy clay soils from Southern Rhodesia and the Sudan resulted in Kjeldahl nitrogen values being increased by 23 and 25%, respectively. The Bal modification had a much smaller quantitative effect when finely ground soil was analyzed.

It is conceivable that the water pretreatment effect observed in the above reports may have been associated with short digestion periods and the type of acid–salt–catalyst mixture used. Nevertheless, Ashton (1) found that the Bal modification, although the effect was small (4.7% increase in nitrogen), was still needed for maximum recovery of nitrogen in a clay soil digested for 3 h after the flask contents had cleared in color. This worker also found that the water could be added after the completion of the nitration reaction in a salicylic acid modification of the Kjeldahl digestion procedure.

During the past 40 years changes in Kjeldahl methods for soils have included longer digestion times, use of superior acid–salt–catalyst mixtures, and greater use of semimicro methods with finely ground soils (6, 8). For instance, Bremner (5) found that a method recommended by the Association of Official Agricultural Chemists between 1925 and 1955, and used by Srinivasan (12, 13, 14) and possibly by Bal (3), gave results 10 to 37% lower than those by a method involving digestion of 10 g of soil with a mixture of 10 g of K_2SO_4, 1 g of $CuSO_4 \cdot 5H_2O$, and 0.1 g of Se and boiling for 5 h after clearing. Thus, there is a question whether the reported water pretreatment effects were associated with the type of Kjeldahl methods employed by the earlier researchers. Bremner (6) incorporated the Bal modification into several variations of his basic H_2SO_4–K_2SO_4–$CuSO_4 \cdot 5H_2O$–Se Kjeldahl technique.

The significance of fixed ammonium (10) in soils was

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not known when the Bal treatment effect was originally reported. Fine grinding of soils, which presumably reduces the magnitude of the Bal effect, would possibly increase the likelihood of fixed ammonium or of labile organic nitrogen trapped between unit layers of clays being released during a digestion. However, Bremner (4) found that the Bal modification had no effect on Kjeldahl nitrogen values of diverse soils, despite the fact that some of the soils contained large amounts of fixed ammonium. Stewart and Porter (15) indicated that certain soils rich in indigenous fixed ammonium gave low Kjeldahl nitrogen recoveries and dark residues after prolonged digestion unless a HF–HCl pretreatment was used. The Bal modification did not affect the recovery of this refractory nitrogen fraction.

During recent soil nitrogen balance studies at ICRISAT total nitrogen recovery from a Vertisol was incomplete without a water pretreatment. The primary objective of this study was to investigate the need of the Bal modification for a Kjeldahl nitrogen analysis with the H_2O–H_2SO_4–K_2SO_4–$CuSO_4 \cdot 5H_2O$–Se macro- and semimicro techniques (6) now widely used by soil scientists. Secondary objectives were to observe if the effect was associated with the fixed ammonium content of the soil, and to determine the influence of the water pretreatment on nitrogen recovery from selected horizons of an Alfisol, another soil widely distributed in the semiarid tropics.

MATERIALS AND METHODS

The soil used in most of these studies was a Vertisol (calcareous Typic Pellusterts) soil obtained from a field at ICRISAT, located on the Deccan Plateau of India. Selected properties of this surface, 0–15 cm, soil were: coarse sand = 15%; fine sand = 15%; silt = 19%; clay = 51%; organic carbon (Walkley–Black) = 0.60%; inorganic C = 0.52%; pH (soil-to-H_2O = 1:5) = 8.3; cation exchange capacity (CEC) = 56 meq/100 g; NO_3^--N = 13 ppm; NO_2^--N = < 0.1 ppm. The moisture content of the air-dried surface soil was 7.7%, and the 15-bar value was 22%. Unless otherwise stated, the soil was ground sufficiently fine to pass through a sieve (20 mesh) with 0.8-mm openings.

The Kjeldahl digestion methods were essentially based on those proposed by Bremner (6). Macro-Kjeldahl digestions were carried out in 800-mL Kjeldahl flasks and the basic procedure was as follows: 10 g of soil, 35 mL of concentrated H_2SO_4, and 11.1 g of K_2SO_4–$CuSO_4 \cdot 5H_2O$–Se (10:1:0.1), with digestion for 5 h, except where otherwise stated, after the flask's contents lost its intense dark color. Thirty-five rather than the recommended 30 mL of H_2SO_4 (6) were used to reduce the likelihood of Kjeldahl digests drying out during extended digestion periods with the particular macro-digestion unit used. The Bal-type modification normally consisted of adding 20 mL of distilled water to the soil in the Kjeldahl flask and allowing it to stand for 30 min, with occasional swirling, prior to addition of the acid–salt–catalyst mixture. Where the effect of the salicylic acid modification was studied, 40 mL of H_2SO_4 containing 1 g of salicylic acid were added to 10 g of soil and the mixture in a sealed flask was allowed to stand overnight. Five grams of $Na_2S_2O_3 \cdot 5H_2O$ were then added and the flask and its contents were gently heated until white fumes disappeared. The salt–catalyst mixture was then added and the regular digestion was then completed as previously described. The Ashton (1) water pretreatment for the salicylic acid-modified Kjeldahl procedure consisted of the addition of 50-mL of distilled water after the completion of the nitration reaction.

The semimicro-Kjeldahl procedure involved the addition of 1 g of soil to a 75-mL digestion tube. Four milliliters of H_2SO_4 and 1.1 g of K_2SO_4–$CuSO_4 \cdot 5H_2O$–Se (10:1:0.1) were added; the digestion for 5 h after clearing was completed on an aluminum block maintained at 360° C. The Bal modification consisted of the addition of 2 mL of distilled H_2O to the soil in the digestion tube followed by standing with occasional swirling of the suspension for 30 min prior to acid–salt–catalyst addition.

The contents of macro-Kjeldahl flasks after digestion were transferred to 250-mL volumetric flasks; 50- or 75-mL aliquots of the diluted digests were added to 300-mL distillation flasks and made alkaline with excess 10*N* NaOH. Ammonia released by steam distillation was collected in boric acid and titrated with standard acid. In the semimicro procedure usually the entire contents of the tube were steam distilled.

In the first experiment the influence of time of digestion after clearing and of time of soaking after water pretreatment on recovery of soil nitrogen by the macro-Kjeldahl method was studied. This was followed by a second experiment in which the recovery of soil nitrogen was studied when three pretreatments to include nitrate in a macro-Kjeldahl method were compared. The pretreatments, in addition to the control with no pretreatment for nitrate, were the usual salicylic acid modification, the Ashton–water addition version (1) of this technique, and the H_2O–$KMnO_4$–Fe method (6). The influence of a factorial combination of three treatments involving two soil particle sizes, < 0.8 and < 0.16 mm (passing 20- and 100-mesh sieves, respectively), two water pretreatments (0 and 2 mL of H_2O), and

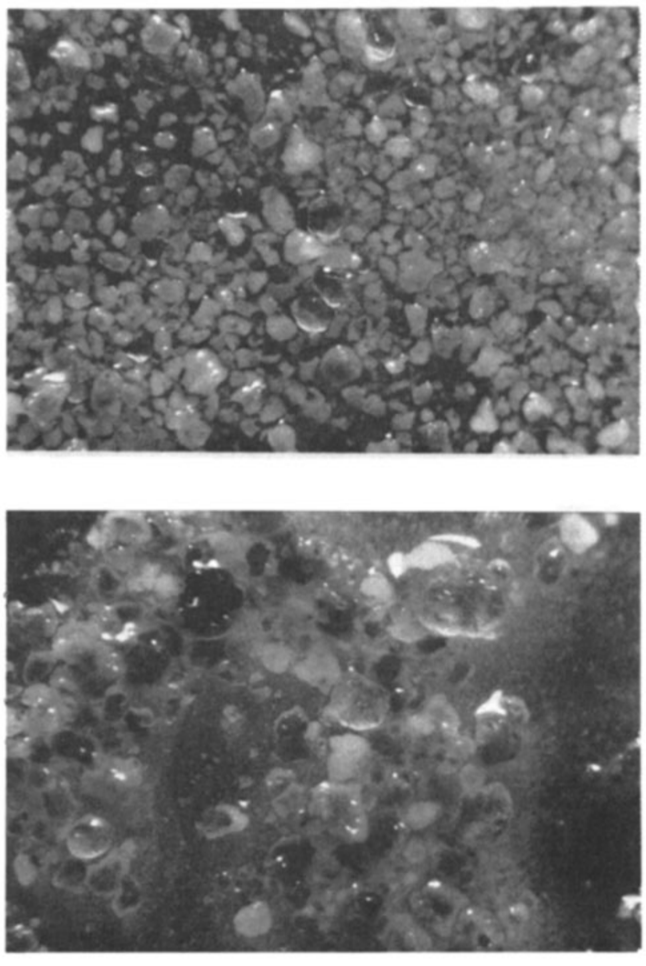

Fig. 1—Postdigestion residues of a Vertisol soil with (upper) and without (lower) initial water pretreatment treatments prior to addition of H_2SO_4 and the salt-catalyst mixture (approximate magnification = 40). Digestion time after initial clearing was 5 h.

two times of digestion after clearing (2 and 5 h) on recovery of nitrogen by the semimicro-Kjeldahl method was investigated in the third experiment.

The fourth experiment was designed to ascertain if the fixed ammonium fraction or incomplete digestion of organic N was contributing to the Bal effect. Several cores were taken to a depth of 170 cm from the general area where the original surface soil was obtained, subdivided into selected depth increments, and composited. The clay contents were 51, 51, 52, 55, 59, 61, and 61%, respectively, for the 0- to 15-, 15- to 30-, 30- to 60-, 60- to 90-, 90- to 120-, 120- to 150-, and 150- to 170-cm depth increments. Ammonium- and nitrate-extractable with 2*N* KCl (7), fixed ammonium (11), and total and organic nitrogen for each depth increment were determined. Total nitrogen in the seven soil samples was determined by the semimicro-Kjeldahl method, with and without a water pretreatment to allow quantification of the Bal effect.

In a final experiment, the effect of the Bal modification on the recovery of nitrogen by a semimicro-Kjeldahl method from selected horizons of an Alfisol (Udic Rhodustalfs) ground to pass through a sieve with 0.8-mm openings was studied. In all experiments each treatment was replicated three times.

RESULTS AND DISCUSSION

Water Pretreatment and the Macro-Kjeldahl Method

Although the black color of the solution phase disappeared approximately 30 min after the addition of the salt–catalyst mixture, darkish discrete granules remained, after an additional 5-h digestion, in the solid matrix within flasks in which there was no water pretreatment (Fig. 1). In contrast, the precipitate remaining after a water pretreatment was relatively white and less dense. The water pretreatment also reduced bumping during digestion.

Table 1—Influence of the Bal modification and period of digestion on the recovery of N by a macro-Kjeldahl method from the surface layer of a Vertisol ground to pass through a sieve with 0.8-mm openings.

Bal pretreatment	Digestion time†	N‡	Increase in recovery§
	h	ppm	%
None	2	437 (2)	--
None	5	454 (2)	3.9
20 mL of H_2O (30 min)	2	559 (4)	27.9
20 mL of H_2O (30 min)	5	564 (1)	29.1
20 mL of H_2O (16 h)	5	563 (5)	28.8

† Hours of digestion after initial clearing.
‡ Numbers in parentheses represent standard errors of mean values.
§ Percentage increase in recovery of N over the nonwater pretreatment with a 2-h digestion after clearing.

Table 2—Influence of procedures to include nitrate on the recovery of macro-Kjeldahl N from the surface layer of a Vertisol.

Kjeldahl modification	N§	Increase in recovery
	ppm	%
None	447 (1)	--
Salicylic acid	477 (8)	6.7
Salicylic acid + 50 mL of H_2O after nitration†	568 (3)	27.1
H_2O-$KMnO_4$-Fe‡	563 (3)	26.0

† Water addition procedure as recommended by Ashton (1); digestion proceeded for 5 h after initial clearing.
‡ Method as reported by Bremner (6).
§ Numbers in parentheses represent standard errors of mean values.

Increasing the period of digestion from 2 to 5 h after the initial clearing had only a small effect on recovery of soil nitrogen, especially if a Bal-type modification was employed (Table 1). In contrast, addition of water and soaking the soil suspension for either 30 min or 16 h before digestion caused large increases in soil nitrogen values. The data show that some form of water pretreatment was required for maximum nitrogen recovery from the surface Vertisol soil in question, even though the period of digestion after initial clearing was long, and a widely accepted acid–salt–catalyst digestion mixture was employed.

Modifications to Include Nitrate and the Water Pretreatment Effect

The H_2O–$KMnO_4$–Fe modification (6) recommended for quantitative inclusion of nitrate and nitrite in macro-Kjeldahl nitrogen determinations, and the Ashton salicylic acid modification (1) for recovery of nitrate, which involved a water pretreatment after the nitration reaction, resulted in the production of diffuse white precipitates, and increased recovery of nitrogen (Table 2). The salicylic acid treatment without a water pretreatment yielded a precipitate with darkish granules and low recovery of nitrogen. The increased efficacy of the Ashton modification, because of its magnitude, was associated almost certainly with increased recovery of reduced soil nitrogen, not of nitrate or nitrite.

Water Pretreatment and the Semimicro-Kjeldahl Method

Grinding soil to a fine state is generally recommended to reduce subsampling errors with semimicro-Kjeldahl methods (6, 8). The data in Table 3 demonstrate that reducing the particle size of the Vertisol soil from < 0.8 to < 0.16 mm increased greatly the recovery of nitrogen and reduced the magnitude of the Bal effect. The effect of the particle-size reduction in the present study was mainly associated with improved digestion of soil, rather than improvement in subsampling. One particularly noticeable feature was that finely divided soil samples yielded nearly white diffuse precipitates after digestion, irrespective of the imposition of the water pretreatment. However, the precision of the nitrogen values was also improved by use of finely divided soils. Extension of the

Table 3—Influence of a Bal modification, fineness of grinding, and time of digestion on the recovery of N from the surface layer of a Vertisol by a semimicro-Kjeldahl method.

Soil-particle size	Bal modification†	Digestion time‡	N§	Increased recovery due to modification
mm		h	ppm	%
<0.8	none	2	421 (4)	--
<0.8	+	2	570 (3)	35.4
<0.8	none	5	486 (10)	--
<0.8	+	5	571 (6)	17.5
<0.16	none	2	561 (2)	--
<0.16	+	2	595 (0)	6.1
<0.16	none	5	584 (0)	--
<0.16	+	5	595 (0)	1.9

† The soil suspension (1 g of soil + 2 mL of H_2O) was soaked for 30 min.
‡ Time of digestion after initial clearing.
§ Numbers in parentheses represent standard errors of mean values.

digestion period from 2 to 5 h in the absence of a water pretreatment had a substantially greater effect for the coarsely ground soil than that reported in Table 1 with the macro-Kjeldahl procedure. Nevertheless, even with the longer digestion period there was an appreciable increase resulting from the inclusion of a water pretreatment.

A slightly higher value of total nitrogen in the finely ground soil compared to that in the < 0.8-mm soil, 595 vs. 571 ppm N, was obtained with the treatment involving a 5-h postclearing digestion period and the Bal modification. A soil subsampling error or a direct grinding effect could have been responsible for such a result.

Soil Depth and the Magnitude of the Water Pretreatment

Data showing the values of certain nitrogen fractions, and the magnitude of the Bal effect for seven consecutive depth increments are given in Table 4. Levels of fixed ammonium were relatively similar throughout the profile, but the organic nitrogen content, like that for the magnitude of the Bal effect, fell with increasing depth. Regression of the magnitude of the Bal effect on levels of fixed ammonium, organic nitrogen, and on total nitrogen yielded correlation coefficients of 0.64(NS), 0.95 ($P < 0.001$, $n = 7$) and 0.94 ($P < 0.001$, $n = 7$), respectively. These associative relationships suggest that the water pretreatment effect was due to incomplete digestion of organic nitrogen, rather than to incomplete recovery of fixed ammonium.

Water Pretreatment and Nitrogen Determination in an Alfisol

The Bal modification, in marked contrast with the results obtained with the Vertisol soil, had little or no influence on the recovery of Kjeldahl nitrogen from selected horizons of the Alfisol (Table 5). Soil samples from the B21t and B22 t horizons were relatively high in clay, both containing 41%.

CONCLUSIONS

The results of this study confirm Bal's conclusion that certain clay soils need a water pretreatment in order to effect complete conversion of the nitrogen constituents into the ammonium form during a Kjeldahl digestion.

Table 4—Contents of 2*N* KCl-extractable inorganic N, fixed NH_4^+, and organic and total N, and the magnitude of the Bal effect in soils from selected depth increments of a Vertisol.

Soil depth	NH_4^+- + NO_3^--N	Fixed NH_4^+-N	Org. N†	Total N‡ *a*	Total N‡ *b*	Bal effect *b − a*	Fixed NH_4^+-N / Total N (*b*)
cm	ppm						
0-15	3	113	371	390	487	97	0.23
15-30	3	107	278	303	388	85	0.28
30-60	3	100	231	255	334	79	0.30
60-90	3	99	205	236	307	71	0.32
90-120	4	110	208	252	322	70	0.34
120-150	4	102	174	210	280	70	0.36
150-170	3	100	145	197	248	51	0.40

† Organic N = Total N (*b*) − (NH_4^+- + NO_3^--N) − fixed NH_4^+-N.
‡ Total N was determined on 1-g samples of soil ground to pass through a sieve with 0.8-mm openings; *a* and *b* indicate a semimicro-Kjeldahl N determination without and with a Bal modification, respectively.

The use of extended digestion periods and of the relatively effective H_2SO_4–K_2SO_4–$CuSO_4 \cdot 5H_2O$–Se digestion mixture (6) in our study did not eliminate the need for the water pretreatment with either surface or subsurface samples of the Vertisol. Thus, it would appear that the water pretreatment is still needed for nitrogen determination in some soils, especially if coarsely ground samples are used.

Many soils do not need a water pretreatment for quantitative recovery of soil nitrogen. The Vertisol soil is characterized by high levels of montmorillonite, but many soils such as a Fargo clay, a Vertic Haploboroll, in North Dakota have equally as high levels of montmorillonite, but do not require this water pretreatment.[3] The black cotton soil used by Bal would now be classified as a Vertisol. In studies where a water pretreatment has effected a large percentage increase in determined nitrogen, the soil normally had a low content of nitrogen (3, 13, 16), generally a high level of clay (3, 13, 16), and an alkaline reaction (16). The Vertisol used in this study meets these criteria.

Some analysts do not routinely soak soils in water prior to doing Kjeldahl nitrogen analyses. In many cases the pretreatment is not needed for maximum recovery of nitrogen, although less bumping may occur during digestion. However, if dense, darkish, granular-like particles are present in the flask after digestion incomplete recovery of nitrogen can be expected. A pretreatment with a mixture of HF–HCl, as reported by Stewart and Porter (15), may be required for complete nitrogen recovery in some soils rich in fixed ammonium. In other cases, the water pretreatment may be satisfactory. The causes for the anomalous data in the two cases are apparently not similar.

Because of considerations involving precision and subsampling, finely divided soil samples are recommended (6, 8) for use in semimicro-Kjeldahl methods. This fine grinding should also decrease the magnitude of any Bal-type effect.

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[3] J.T. Moraghan. Unpublished data. North Dakota State Univ.

Table 5—Influence of the Bal modification on the determination by a semimicro-Kjeldahl procedure of total N in selected horizons of an Alfisol.

Soil horizon	Clay	Total N† *a*	Total N† *b*
	%	ppm	
Ap	19	561 (10)	549 (15)
B1	30	647 (2)	642 (18)
B21t	41	823 (7)	846 (6)
B22t	41	482 (7)	498 (18)

† Numbers in parentheses represent standard errors of mean values; *a* and *b* indicate a semimicro-Kjeldahl N determination without and with a Bal modification, respectively.

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