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INTERNATIONAL JOURNAL OF CURRENT RESEARCH

*International Journal of Current Research*
*Vol. 7, Issue, 11, pp.22989-22993, November, 2015*

ISSN: 0975-833X

## RESEARCH ARTICLE

# DETERMINATION OF CARBON TO NITROGEN RATIO IN SOILS OF A SEMI-DESERT DRY CLIMATE

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**ARTICLE INFO**

***Article History:***
Received 14th August, 2015
Received in revised form
22nd September, 2015
Accepted 07th October, 2015
Published online 30th November, 2015

***Key words:***

Walkley-Black method, Tinsley method, Wet Combustion method, arid regions, soil C/N ratio.

**ABSTRACT**

The carbon to nitrogen ratio (C/N) determination is one of the most important parameters of soil characterization. The validity of the methods used for determining organic carbon and total nitrogen in some Aridisols soils collected from Khartoum State, Sudan was studied. Organic carbon (O.C) was determined by wet combustion method which was considered as a reference or standard method against Walkley-Black and Tinsley methods that are widely used in the Sudan. In the latter two methods, the organic material was found to be poorly oxidized, and the use of their conventional conversion factors will thus lead to underestimation of O.C. Using the wet combustion method as standard, new conversion factors were suggested, which were about 30% and 20% higher than those used in Walkley-Black and Tinsley methods, respectively. The contribution of non-exchangeable nitrogen to total nitrogen was determined, and was found that it significantly affected the C/N especially in the subsoil.



***Citation***: Gaffer, M.O., Sulieman, H. A., Elmahi, Y. E. and Elhag, A. Z. 2015. "Determination of carbon to nitrogen ratio in soils of a semi-desert dry climate", *International Journal of Current Research*, 7, (11), 22989-22993.

## INTRODUCTION

The ratio of carbon to nitrogen (C/N) in arable soils usually ranges between 8:1 and 15:1, with the median being between 10:1 and 12:1 (Brady & Weil, 2008; Persson & Kirchmann, 1994). However, a ratio of about 20:1 is generally considered as the approximate threshold between net mineralization and net immobilization of soil nitrogen (Killham, 1994). Brady and Weil (2008) pointed out that, generally the C/N ratio tended to be lower in soils of arid regions than in those humid regions having the same average annual temperature. In many soils of Northern Sudan, however, especially the Aridisols, the C/N ratio was found much lower than this range. For example, Khodary, (1979), reported, in eight soil profiles, an average C/N of 2.75 for the top soil (0-30 cm), and 2.5 for the sub-soil in the Nile State. Moreover, Fadil (1979) reported in another area at the north eastern part of the White Nile State average values of 5.0 in the top soil (0-30 cm) and 3.2 in the sub-soil (30-60 cm) in 5 soil profiles. This phenomenon is widespread and these are only few examples. Globally, organic carbon is measured by oxidation to $CO_2$ and is directly measured as $CO_2$ or by sample weight loss or by back-titration of the excess of the added oxidant. Wet combustion recovers 100% of organic carbon values (Vogel, 1994), while Tinsley-indirect combustion method- can recover most of it.

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However, the Walkley-Black method gives variable recovery of soil organic C (Batjes, 1996). Nevertheless, a standard conversion factor of 1.33 for incomplete oxidation of carbon is commonly used to convert Walkley-Black carbon to the total organic-C content in organic matter.However, the factors adopted by various workers vary greatly between and within soils because of differences in the nature of organic matter with soil depth and vegetation type (Grewal *et al*., 1991). Therefore, the Walkley-Black method gives only an approximation of soil organic carbon content (Batjes, 1996).

Organic carbon is usually determined in the Sudan by the Walkley-Black (1947) method and or Tinsley method (1950) and the total nitrogen by the Kjeldahl method described by Bremner (1996). Walkley-Black and Tinsely methods involve many assumptions whose validity is not well proven in arid zone soils as the methods were developed and tested in temperate soils. Therefore, the objectives of this experiment were to investigate: (a) the validity of the Walkley-Black and Tinsely methods for organic C determination in arid-zone soils and (b) the contribution of non-exchangeable ammonium to the total nitrogen in determination of the actual C/N. Three soils lying in the semi-desert dry climate, Sudan (Adam, 2008) were used for this purpose.

### Materials

Present study was carried out on three Sudanese soil profiles around Khartoum State, namely Soba, Shambat and Kuku having a semi-desert dry climate (Adam, 2008). These soils are utilized by co-operative and private growers for the production of irrigated forage, vegetables and fruit crops. The experimental site is an area of old Nile terraces, almost flat, with alluvial soils derived from the weathering of basic-igneous rocks of the Ethiopian high lands (Whiteman, 1971). Based on the differences in texture, structure, color and consistency, Shambat and Kuku profiles were divided into four layers while Soba profile was divided into five layers. Soba soil is classified as Typic Haplocambid, while both Shambat and Kuku soils are classified as Sodic Haplocambid (Soil Survey Staff, 2014). Soil samples were collected from the observed specified layers. The basic physical and chemical characteristics of samples from the three different profiles are presented in Table 1.

A standard multiplication factor of 1.33 was used to compensate for incomplete oxidation.

The second method used is described by Tinsely (1950). Expecting that the oxidation was not complete, Tinsley's conversion factor of 1.2 was used in this experiment. Thirdly the wet combustion method of Allison (1960) was used as a standard method. The soil was treated and heated with the recommended oxidation agent and strong acids to convert all forms of organic carbon into $CO_2$ which was then absorbed in alkali and determined by titration. Total soil nitrogen was determined by macro-Kjeldahl method described by Bremner (1996). The soil samples were gently crushed to pass a 0.5 mm sieve, and the reaction was enhanced by adding salts to raise the temperature of the digestion mixture and by adding selenium as a catalyst to promote oxidation. Two grams of 0.5 mm soil samples were used for determination of non-exchangeable nitrogen according to the method described by Bremner (1960).

**Table 1. The basic physical and chemical characteristics of samples from the three different profiles**

| Profile | Depth (cm) | Sand % | Silt % | Clay % | pH (paste) | ECe dS/m | Salt % | $CaCO_3$ % | M.C % | Gypsum meq/100 g | ESP | CEC meq/100 g |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0-20 | 34.5 | 17.5 | 48.0 | 8.4 | 1.5 | 47.0 | 7.9 | 4.2 | 0.0616 | 22.6 | 35.0 |
| | 20-44 | 59.5 | 10.0 | 30.5 | 8.61 | 6.2 | 58.0 | 7.5 | 6.4 | 0.07 | 38.38 | 39.0 |
| | 44-74 | 49.5 | 15.0 | 35.5 | 8.75 | 11.8 | 53.0 | 5.43 | 4.2 | 4.275 | 45.38 | 37.0 |
| Soba | 74-112 | 42.0 | 12.5 | 45.5 | 8.79 | 7.6 | 60.0 | 4.2 | 5.3 | 0.256 | 39.92 | 40.0 |
| | 112-180 | 59.5 | 10.0 | 30.5 | 8.82 | 5.0 | 47.0 | 3.83 | 6.4 | | 33.58 | 38.0 |
| | 0-23 | 44.5 | 17.5 | 38.0 | 8.3 | 5.8 | 52.0 | 7.4 | 5.3 | 0.16 | 33.88 | 36.0 |
| | 23-66 | 44.5 | 17.5 | 38.0 | 8.23 | 7.4 | 52.0 | 8.8 | 4.4 | 2.76 | 45.62 | 35.0 |
| | 66-112 | 39.5 | 12.5 | 48.0 | 8.64 | 2.4 | 89.0 | 7.0 | 4.6 | 0.975 | 34.42 | 38.0 |
| Kuku | 112-191 | 27.0 | 15.0 | 58.0 | 8.89 | 2.1 | 119.0 | 2.1 | 6.4 | 0.975 | 37.87 | 41.0 |
| | 0-25 | 34.2 | 26.3 | 39.5 | 7.91 | 7.5 | 60.0 | 6.8 | 6.5 | 0.95 | 21.5 | 40.0 |
| | 25-50 | 21.1 | 23.6 | 55.3 | 8.04 | 10.0 | 75.0 | 6.2 | 5.4 | 6.8 | 31.8 | 48.0 |
| | 50-90 | 21.1 | 23.6 | 55.3 | 8.3 | 9.6 | 81.0 | 7.4 | 6.0 | 0.32 | 35.6 | 51.0 |
| Shambat | 90-140 | 15.5 | 26.6 | 57.9 | 8.76 | 8.8 | 106.0 | 6.2 | 6.6 | 0.05 | 34.0 | 55.0 |

| Profile | Depth (cm) | $Ca^{2+}$ meq/l | $Mg^{2+}$ meq/l | $Na^{+}$ meq/l | $K^{+}$ meq/l | $CO_3^{2-}$ meq/l | $HCO_3^{2-}$ meq/l | $Cl^{-}$ meq/l | $SO_4^{2-}$ meq/l |
|---|---|---|---|---|---|---|---|---|---|
| | 0-20 | 1.0 | 0.8 | 12.6 | 0.07 | 0.6 | 1.5 | 12.25 | 0.12 |
| | 20-44 | 18.0 | 5.0 | 52.2 | 0.13 | 0.75 | 1.8 | 7.25 | 65.42 |
| | 44-74 | 20.2 | 4.8 | 130.4 | 0.27 | 0.8 | 2.45 | 10.0 | 142.42 |
| Soba | 74-112 | 3.4 | 1.4 | 73.9 | 0.095 | 0.78 | 2.6 | 7.25 | 88.16 |
| | 112-180 | 4.0 | 1.2 | 47.8 | 0.08 | 0.78 | 1.85 | 14.2 | 36.25 |
| | 0-23 | 5.6 | 1.0 | 50.0 | 0.09 | 0.8 | 1.3 | 9.0 | 45.59 |
| | 23-66 | 17.2 | 4.6 | 67.4 | 0.1 | 0.9 | 1.6 | 7.0 | 79.7 |
| | 66-112 | 1.0 | 0.4 | 23.9 | 0.05 | 1.14 | 2.2 | 6.25 | 15.76 |
| Kuku | 112-191 | 0.8 | 0.4 | 17.4 | 0.03 | 1.1 | 2.0 | 6.25 | 9.28 |
| | 0-25 | 22.66 | 7.34 | 68.4 | 0.1 | 0.2 | 1.2 | 14.3 | 87.7 |
| | 25-50 | 13.33 | 6.0 | 91.5 | 0.31 | 0.25 | 1.5 | 16.6 | 92.79 |
| | 50-90 | 5.3 | 2.7 | 94.6 | 0.38 | 0.28 | 1.3 | 13.8 | 88.59 |
| Shambat | 90-140 | 1.3 | 2.0 | 81.8 | 0.21 | 0.22 | 1.2 | 14.5 | 69.39 |

### Methods

#### Determination of organic carbon by different methods

First organic C was determined by Walkley-Black method (1947). Chlorides which cause an underestimation of carbon were eliminated by the precipitation by silver sulphate. To reduce the effect of iron, the soil samples were air dried for more than two days before analysis. A modification of Walkley-Black used by the Sudan Soil Survey was followed. This includedgrinding the soil to pass a 0.5 mm sieve and digesting the mixture over a steam bath for one hour.

## RESULTS AND DISCUSSION

Walkley-Black and Tinsely methods for the determination of the organic carbon were developed for temperate zone soils and they received wide application in tropical regions. Yet very little effort was spent in order to examine the validity of these methods under arid tropical conditions (Bornemisza *et al*., 1979; Gang *et al*., 2012; Jia-Ping *et al*., 2013; Nelson & Sommers, 1996). Data in Tables 2, 3 and 4 show that the organic carbon content using these two methodswas low as compared to wet combustion method. Moreover, these tables show the contribution of non-exchangeable nitrogen towards total N and the C/N.

**Table 2. The carbon to nitrogen ratio using wet combustion method for organic carbon in soil before and after the removal of non-exchangeable nitrogen**

| Profile | Depth (cm) | O.C % | Total N % | Non-exch. N % | Corrected Total N % a | C/N ratio (B.C.N)* | C/N ratio (A.C.N)** |
|---|---|---|---|---|---|---|---|
| | 0-20 | 0.6 | 0.0546 | 0.007 | 0.0476 | 11.0 | 13.0 |
| | 20-44 | 0.36 | 0.049 | 0.0084 | 0.0406 | 7.0 | 9.0 |
| | 44-74 | 0.24 | 0.0434 | 0.0112 | 0.0322 | 6.0 | 8.0 |
| Soba | 74-112 | 0.18 | 0.029 | 0.014 | 0.015 | 6.0 | 12.0 |
| | 112-180 | 0.12 | 0.029 | 0.014 | 0.015 | 4.0 | 8.0 |
| | 0-35 | 0.76 | 0.091 | 0.0126 | 0.0784 | 8.0 | 10.0 |
| | 35-77 | 0.48 | 0.056 | 0.014 | 0.042 | 9.0 | 11.0 |
| | 77-136 | 0.6 | 0.0728 | 0.015 | 0.0578 | 8.0 | 10.0 |
| Shambat | 136-200 | 0.36 | 0.0434 | 0.0168 | 0.0266 | 8.0 | 14.0 |
| | 0-23 | 0.36 | 0.0518 | 0.0084 | 0.0434 | 7.0 | 8.0 |
| | 23-66 | 0.29 | 0.035 | 0.0098 | 0.0252 | 8.0 | 11.0 |
| | 66-112 | 0.19 | 0.028 | 0.0112 | 0.0168 | 7.0 | 11.0 |
| Kuku | 112-191 | 0.15 | 0.021 | 0.0112 | 0.0098 | 7.0 | 15.0 |
| | | | | | Average | 7.39 | 10.77 |

∗ (B.C.N) = Before correction of total nitrogen.
** (A.C.N) = After correction of total nitrogen.
a= Corrected total nitrogen % = (total nitrogen – fixed nitrogen).

**Table 3. The C/N ratio using Walkley-Black method for organic carbon in soil before and after the removal of non-exchangeable nitrogen**

| Profile | Depth (cm) | O.C % | Total N % | Non-exch. N % | Corrected Total N % a | C/N ratio (B.C.N)* | C/N ratio (A.C.N)** |
|---|---|---|---|---|---|---|---|
| | 0-20 | 0.38 | 0.0546 | 0.007 | 0.0476 | 7.0 | 8.0 |
| | 20-44 | 0.3 | 0.049 | 0.0084 | 0.0406 | 6.0 | 7.0 |
| | 44-74 | 0.21 | 0.0434 | 0.0112 | 0.0322 | 5.0 | 7.0 |
| Soba | 74-112 | 0.16 | 0.029 | 0.014 | 0.015 | 6.0 | 11.0 |
| | 112-180 | 0.11 | 0.029 | 0.014 | 0.015 | 4.0 | 7.0 |
| | 0-35 | 0.5 | 0.091 | 0.0126 | 0.0784 | 5.0 | 6.0 |
| | 35-77 | 0.33 | 0.056 | 0.014 | 0.042 | 6.0 | 8.0 |
| | 77-136 | 0.4 | 0.0728 | 0.015 | 0.0578 | 5.0 | 7.0 |
| Shambat | 136-200 | 0.27 | 0.0434 | 0.0168 | 0.0266 | 6.0 | 10.0 |
| | 0-23 | 0.3 | 0.0578 | 0.0084 | 0.0434 | 6.0 | 7.0 |
| | 23-66 | 0.25 | 0.035 | 0.0098 | 0.0252 | 7.0 | 10.0 |
| | 66-112 | 0.15 | 0.028 | 0.0112 | 0.0168 | 5.06 | 9.0 |
| Kuku | 112-191 | 0.13 | 0.021 | 0.0112 | 0.0098 | 6.0 | 13.0 |
| | | | | | Average | 5.69 | 8.46 |

∗ (B.C.N) = Before correction of total nitrogen.
** (A.C.N) = After correction of total nitrogen.
a= Corrected total nitrogen % = (total nitrogen – fixed nitrogen).

**Table 4. The C/N ratio using Tinsley method for organic carbon in soil before and after the removal of non-exchangeable nitrogen**

| Profile | Depth (cm) | O.C % | Total N % | Non-exch. N % | Corrected Total N % a | C/N ratio (B.C.N)* | C/N ratio (A.C.N)** |
|---|---|---|---|---|---|---|---|
| | 0-20 | 0.42 | 0.0546 | 0.007 | 0.0476 | 8.0 | 9.0 |
| | 20-44 | 0.288 | 0.049 | 0.0084 | 0.0406 | 6.0 | 7.0 |
| | 44-74 | 0.216 | 0.0434 | 0.0112 | 0.0322 | 4.0 | 7.0 |
| Soba | 74-112 | 0.16 | 0.029 | 0.014 | 0.015 | 6.0 | 11.0 |
| | 112-180 | 0.11 | 0.029 | 0.014 | 0.015 | 4.0 | 7.0 |
| | 0-35 | 0.528 | 0.091 | 0.0126 | 0.0784 | 6.0 | 7.0 |
| | 35-77 | 0.3 | 0.056 | 0.014 | 0.042 | 5.0 | 7.0 |
| | 77-136 | 0.432 | 0.0728 | 0.015 | 0.0578 | 6.0 | 7.0 |
| Shambat | 136-200 | 0.288 | 0.0434 | 0.0168 | 0.0266 | 7.0 | 11.0 |
| | 0-23 | 0.3 | 0.0518 | 0.0084 | 0.0434 | 6.0 | 7.0 |
| | 23-66 | 0.24 | 0.035 | 0.0098 | 0.0252 | 7.0 | 10.0 |
| | 66-112 | 0.186 | 0.028 | 0.0112 | 0.0168 | 7.0 | 11.0 |
| Kuku | 112-191 | 0.144 | 0.021 | 0.0112 | 0.0098 | 7.0 | 15.0 |
| | | | | | Average | 6.00 | 8.92 |

∗ (B.C.N) = Before correction of total nitrogen.
** (A.C.N) = After correction of total nitrogen.
a= Corrected total nitrogen % = (total nitrogen – fixed nitrogen).

Generally, the variation between replicates of the same sample was very little and did not exceed 5% for C and 4% for non-exchangeable N.

The determination of actual C/N ratio is very important because this ratio affects both mineralization and immobilization of soil nitrogen and hence its availability to plants (Bengtsson *et al*., 2003; Brady, 1974; Broadbent, 1953; Frankenberger & Abdelmagid, 1985; Killham, 1994). A narrow C/N ratio may give false indication of a good fertility status, if much of the nitrogen was coming from the non-exchangeable $NH_4$, which may not be readily available for the involved microbes during the short wet spells in arid zones. The results show that the removal of fixed N from the total N substantially increased C/N ratios in the three profiles. The organic N, being naturally small in these soils, will be substantially overestimated by contribution of non-exchangeable $NH_4$ held by clays. During the short wet episodes, cyanobacteria and N-fixing blue green algae may grow on wet soil crusts (Aranibar *et al*, 2003; Tiet *et al*., 2005; West, 1991). High temperatures may promote subsequent rapid mineralization of this newly formed, easily oxidisable, microbial organic matter, into $CO_2$,lost in atmosphere, and $NH_4$ fixed by soil clays. This will result, time and time again, in increase of fixed N (albit small) in the ammonium form, affecting the fixed total N ratio. Another source of error seems to be the determination organic carbon. There is a significant increase in C ($P = 0.01$) in the top soil for the three profiles as determined by wet combustion compared to Walkley-Black and Tinsley methods. This shows that the oxidation conditionsin case of the Walkley-Black and Tinsley methods were not strong enough to attack all organic matter. Consequently, the conversion factors of 1.33 and 1.2 used in these methods, respectively, may not be valid in these arid zone soils, especially in the top soils. In this experiment, the Walkley-Black conversion factor for the top soil approaches 2.0 for the first two soils as can be seen in Table 5 if the organic carbon is to become equivalent to total C as determined by wet combustion.

It is evident that, especially in the top horizons of these soils, the high temperature in semi-desert climates may promote rapid mineralization during the short wet spells and only the toughest forms of C remain un-mineralized (Gregory & Nortcliff, 2013). The remaining tough C will accumulate year after year and its proportion will gradually increase relative to easily oxidisable C. Therefore, the tough C accumulating in the top soil in this way may remain relatively inert to oxidation by the conditions furnished by the Walkley-Black or Tinsley methods. To compensate for incomplete oxidation of organic materials by Walkley-Black and Tinsley methods, average conversion factors were established which were 1.66 for Walkley-Black method and 1.49 for Tinsley method instead of the traditional 1.33 and 1.2 factors, respectively (Table 5 and 6). Even these suggested factors may cause a gross underestimation of organic carbon in the top surface soil.

It is clear that the organic matter in the deeper horizons of the three soils is more easily oxidizable by the two milder methods. This may be due to the fact that plant residues in the sub-soil, being older, were subjected for longer times to the action of such organisms as termites and earthworms which make them more oxidizable. The portion of tough organic carbon oxidized by wet combustion may not be practically important. It may not be oxidisable (in the short wet spells) by microbes during mineralization and immobilization, so its active contribution to soil fertility is rather doubtful. Further investigations are recommended in this aspect.

**Table 5. Estimation of a conversion factor for Walkley-Black method of determination of organic carbon using wet combustion as standard method**

| Profile | Depth (cm) | Organic C % Wet combustion | Organic C % Walkley-Black | Conversion factor |
|---|---|---|---|---|
| | 0-20 | 0.6 | 0.29 | 2.06 |
| | 20-44 | 0.36 | 0.23 | 1.56 |
| | 44-74 | 0.24 | 0.16 | 1.5 |
| Soba | 74-112 | 0.18 | 0.123 | 1.46 |
| | 112-180 | 0.12 | 0.09 | 1.33 |
| | 0-35 | 0.76 | 0.39 | 1.94 |
| | 35-77 | 0.48 | 0.254 | 1.88 |
| Shambat | 77-136 | 0.6 | 0.31 | 1.93 |
| | 136-200 | 0.36 | 0.21 | 1.71 |
| | 0-23 | 0.36 | 0.23 | 1.56 |
| | 23-66 | 0.29 | 0.19 | 1.52 |
| Kuku | 66-112 | 0.19 | 0.115 | 1.65 |
| | 112-191 | 0.15 | 0.1 | 1.5 |
| | | | Average | 1.66 |

**Table 6. Establishment of a conversion factor for Tinsley's method of determination of organic carbon using wet combustion as standard method**

| Profile | Depth (cm) | Organic C % Wet combustion | Organic C % Tinsley | Conversion factor |
|---|---|---|---|---|
| | 0-20 | 0.6 | 0.35 | 1.71 |
| | 20-44 | 0.36 | 0.24 | 1.5 |
| | 44-74 | 0.24 | 0.18 | 1.33 |
| Soba | 74-112 | 0.18 | 0.133 | 1.35 |
| | 112-180 | 0.12 | 0.092 | 1.3 |
| | 0-35 | 0.76 | 0.44 | 1.72 |
| | 35-77 | 0.48 | 0.25 | 1.92 |
| | 77-136 | 0.6 | 0.36 | 1.66 |
| Shambat | 136-200 | 0.36 | 0.24 | 1.5 |
| | 0-23 | 0.36 | 0.25 | 1.44 |
| | 23-66 | 0.29 | 0.2 | 1.45 |
| | 66-112 | 0.19 | 0.155 | 1.22 |
| Kuku | 112-191 | 0.15 | 0.12 | 1.25 |
| | | | Average | 1.49 |

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