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A comparative study to determine soil organic carbon content in soils of Haryana

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Abstract

A field survey and laboratory experiment were carried out for comparative study to determine soil organic carbon content in soils of Haryana during 2018-19 in four soil orders Entisol, Inceptisol, Aridisol and Alfisol of Haryana. The surface and subsurface samples were collected using GPS.

Higher organic carbon content measured by dry combustion method compared to wet oxidation. Between wet oxidation methods Colorimetric method gives higher recovery than WB method. There is high correlation between dry combustion-WB method and dry combustion-Colorimetric method. Organic carbon recovery estimated higher in surface soil of Aridisol. A higher correction factor reported for Alfisol in wet oxidation methods. Subsurface soil has high correction factor than subsurface soil.

Keywords: WB method, colorimetric method, dry combustion method

Introduction

Soil carbon storage is the third largest carbon pool in the earth system that plays an important role in the global carbon cycle and climate change. Soil may contain carbon in organic and inorganic forms. The organic form comprises soluble organic compounds (carbohydrate and protein), amorphous organic compounds (humic acid, fat, wax, lignin and polyuronide) and organo-mineral complexes (Schnitzer, 1991) [15] and the inorganic form is present as calcium carbonate. The term total carbon (TC) is the sum of both forms.

The soil carbon pool is 3.3 times the size of the atmospheric carbon pool (760 Pg) and 4.5 times the size of biotic carbon pool (560 Pg) (Lal, 2004) [11]. In India, the SOC, SIC and total soil carbon pool has been estimated as 22.72 ± 0.93 Pg, 12.83 ± 1.35 Pg and 35.55 ± 1.87 Pg, respectively (Sreenivas *et al.*, 2016) [18].

Soil organic matter (SOM) is the organic fraction of soil. It is a complex mixture of plant and animal products in different stages of decomposition, soil microorganisms and substances produced by them (Chan *et al.*, 2001) [2]. SOC is essential for the improvement of soil quality, food production, maintaining clean water and reduction of increased CO₂ in the atmosphere. While its inorganic counterpart is responsible for organic carbon decomposition, development of soil salinity, restriction of root proliferation and immobilization of soil plasma (Singh *et al.*, 2007) [16].

Universal and harmonized quantification of SOC content is essential. A major challenge in SOC determination is the absence of a single method that can be applied in all situations due to the fact that SOC is not evenly distributed over different areas, depths, soil types and topography (FAO 2017) [8]. There are several methods for the determination of organic carbon (OC) and TC, each of which has its own advantages and disadvantages.

Wet chemistry method includes the digestion and extraction technique that is based on destruction of all the organic matter in the samples by oxidation. Dry chemistry method includes combustion that is performed by burning the sample at high temperature (Dias *et al.*, 2012) [7]. Among these analytical methods, the Walkley and Black (WB) and Dry Combustion (DC) methods are most commonly used for the quantification of SOC.

WB method (Walkley and Black, 1934) [19] is most widely recognized as simple, inexpensive and rapid method for determination of OC, which requires minimum equipments compared to other methods (Nelson and Sommers, 1982) [12]. Therefore, this method is preferred for routine purpose over other methods. Although this method is helpful but it has certain drawbacks, for example, accuracy problems due to manual titration, use of correction factor, environmental concerns because of the formation of acid wastes containing toxic form of chromium (Cr⁺⁶), potential safety problems associated with use of concentrated sulfuric acid (Kerven *et al.*,

2000)^[8] and labour intensive in nature. Therefore, this method is less suitable for large area analysis.

Colorimetric method is used to quantify SOC through the determination of absorbance. This method is advantageous because it is rapid, economical, more oxidation of organic carbon compared to WB method, not affected by presence of carbonates and any laboratory capable of soil testing can perform this method. So, use of colorimetric method rather than titration can increase the precision of wet oxidation method (Soon and Abboud, 1991)^[17].

An automated technique based on DC of OC by CHNS analyzer is adopted recently. The advantages of this method are accuracy, safety, reliability, repeatability, automated analysis procedure, simultaneous determination of C and nitrogen, destruction of all forms of C, avoid the use of potentially dangerous chemicals, can be connected to mass spectrometer for analysis of stable isotope, minimum sample preparation and sample analysis time is short. The main disadvantage of this method is high purchasing cost of equipment (Nelson and Sommers, 1996)^[13] and considerable space needed for the analyser.

Materials and Methods

General description of study area

Haryana lies in the North Western region of India between the latitudes from 27°39' N to 30°55' N and longitudes from 74°27' E to 77°36' E comprising twenty-two districts covering an area of 44,212 square kilometers with 1.4% of India's land area. Climatologically Haryana lies in sub-tropical belt.

Distribution and characteristics of different Soil Orders of Haryana

According to soil taxonomy, the Haryana soils are divided into four orders. The area and location are presented in the following table:

Table 1: Location and extent of study area

Sr. No.	Order	Area (%)	Districts
1	Inceptisol	58	All districts
2	Entisol	29	All districts
3	Aridisol	9	Sirsa, Fatehabad, Hisar and Bhiwani
4	Alfisol	2	Karnal and Kurukshetra

- Inceptisol:** The samples were collected from Rohtak, Sonapat, Sirsa, Fatehabad and Panipat districts where the annual rainfall varies from 500 to 1000 mm. Texture from coarse loam to fine loam.
- Entisol:** These samples were collected from Yamuna belt of Haryana dominated by recent alluvium soil.
- Aridisol:** These samples were collected from parts of Sirsa, Fatehabad, Hisar and Bhiwani districts having average rainfall of 300 mm. Wind erosion is a serious problem in these soils.
- Alfisol:** The samples were collected from Karnal and Kurukshetra districts. The problem of salinity and alkalinity appear in irrigated areas.

Collection and analysis of soil samples

A total of 160 samples comprising 20 samples of surface (0-15cm) and 20 sub-surface samples (15-30cm) were taken from each order. Soil samples were air dried, ground and sieved from 0.2mm sieve for analysis of organic carbon.

Soil organic carbon: Soil organic carbon content from oven dried fine sieved samples were determined by following three methods:

Wet oxidation by Walkley and Black method

Organic carbon in soil oxidized by mixture of potassium dichromate and sulphuric acid, utilizing the heat of dilution of sulphuric acid. Samples were left on asbestos sheet for 30 min for the completion of the reaction. The highest temperature attained by heat of dilution of sulphuric acid is 120°C that is capable of oxidizing all the active forms of C but not the recalcitrant form of C. Now water is added to quench reaction. The excess of potassium dichromate left unreduced by organic carbon was titrated against standard ferrous ammonium sulphate in presence of sodium fluoride or phosphoric acid and diphenylamine indicator. Sodium fluoride or phosphoric acid is used for sharp end point due to their flocculating effect. At the end point color changes from violet to green (Walkley and Black, 1934)^[19].

Wet oxidation by Colorimetric method

A known weight of soil sample is treated with mixture of potassium dichromate and sulphuric acid. The mixture was left on asbestos sheet for 30 min for the reaction to be completed. Contents were cooled and 100 ml volume was made up after the centrifugation. The intensity of chromium sulphate formed is measured at 600 nm in colorimeter. A standard solution of sucrose is used for calibration of results (Datta *et al.*, 1962)^[5].

Dry combustion by CHNS analyzer

Total carbon in soil samples were analyzed by CHNS analyzer. Combustion was carried out in a source of pure oxygen and helium as carrier gas. Sulfadiazine was used as a standard compound for the calibration of results. A 20 mg of soil sample was weighted on an inert tin capsule. Sample combustion started at 1150 °C in pure oxygen. Carrier gas was turned on later. After the clean swept of first chamber gases were carried to the post chamber and reduction chamber. These chambers were maintained at 800-850 °C. At last gases were carried to detector. In first chamber tungsten oxide was used. In second chamber copper oxide and platinum as a catalyst were used. Copper was used in third chamber. Detection in a sequence of N₂, CO₂ and SO₂ were made with the help of thermo conductivity cell. Carbon content obtained by using the software provided with analyzer (Dias *et al.*, 2013)^[7].

TOC analysis after dry heat combustion

TOC content can be determined indirectly from the difference between TC and soil inorganic carbon (SIC) that are measured separately. TC measured by dry combustion using CHNS analyser and inorganic carbon by volumetric method.

$$\text{TOC} = \text{TC} - \text{SIC}$$

Calculation of recovery and correction factor

The % recovery obtained by WB method compared to CHNS analyser is calculated as-

$$R_{\text{WB}} = \text{SOC}_{\text{WB}} \times 100 / \text{TOC}$$

Where,

R_{WB} = % recovery by WB method

SOC_{WB} = SOC % by WB method

TOC = Total organic carbon or SOC% by CHNS analyser

The correction factor for WB method compared to CHNS analyser is calculated as-

$$CF_{WB} = 100 / R_{WB}$$

Where,

CF_{WB} = Correction factor for WB method

The % recovery obtained by Colorimetric method compared to CHNS analyser is calculated as-

$$R_C = SOC_C \times 100 / TOC$$

Where

R_C = % recovery by Colorimetric method

SOC_C = SOC % by Colorimetric method

The correction factor for Colorimetric method compared to CHNS analyser is calculated as-

$$CF_C = 100 / R_C$$

Where,

CF_C = Correction factor for Colorimetric method

Results and Discussion

Soil Organic Carbon Content:

1. Walkley and Black method

OC content determined by WB method (Table 2) in Entisol varied from 0.33 to 0.54 with an average value of 0.43% for the surface samples and 0.20 to 0.43 with an average value of 0.32% for subsurface samples. In this 80% of samples fall in low category and 20% in medium category in surface and all the soil samples fall in low category in subsurface. In Inceptisol, OC ranged from 0.23 to 0.52 with an average value of 0.40% in surface samples and 0.22 to 0.46 with a mean value of 0.35% in subsurface samples. 15% sample of surface falls in medium category and 85% in low category. While all the samples in subsurface reported in low category. Surface samples of Aridisol show OC variation from 0.15 to 0.40 with mean value of 0.29%. OC in Subsurface samples varied from 0.08 to 0.34 with an average value 0.23%. Soil samples of this order present in low category. In Alfisol for surface samples OC ranges from 0.36 to 0.76 with mean value of 0.54%. While for subsurface it ranged from 0.34 to 0.57 with a mean value of 0.44%. In surface 5% samples falls in high category, 60% in medium category and 35% in low category. While in subsurface 15% samples represent medium

category and 85% low category.

Colorimetric method

Organic carbon determined by Colorimetric method (Table 2) in Entisol for surface samples ranged from 0.42 to 0.78 with a mean value of 0.57%. While in subsurface from 0.22 to 0.56 with an average value of 0.41%. In surface 30% samples fall in low category, 65% in medium category and 30% in high category. While in subsurface samples 25% in medium category and 75% in low category. Inceptisol order represents a range of OC from 0.33 to 0.77 with a mean value of 0.53% for surface soil and an average value of 0.47% and range of 0.29 to 0.61 in subsurface samples. 30% samples of surface are present in low category, 65% in medium category and 5% in high category. While in subsurface 30% samples fall in low category and 70% in medium category. Aridisol ranged OC from 0.16 to 0.46 with a mean value of 0.33% in surface. Subsurface samples of this order range OC from 0.10 to 0.38 with a mean value of 0.27% OC. All samples of Aridisol are present in low category irrespective of soil depth. Alfisol OC ranged from 0.51 to 0.89 with mean OC content of 0.69% in surface samples and 0.44 to 0.73 with a mean of 0.57% in subsurface samples. 30% of surface samples fall in low and 70% in medium category. While in subsurface 65% samples fall in medium category and rest in low category.

Dry combustion method

Dry combustion reference method represents (Table 2) a range of OC in Entisol from 0.46 to 0.87 with a mean value of 0.66% in surface and subsurface samples of this order contains mean carbon content of 0.50 which ranges from 0.28 to 0.70. In this 5% samples represent low category, 75% medium and 5% high in surface soil while for subsurface 55% samples belong to medium and 45% low category. In Inceptisol surface samples presents TOC range from 0.40 to 0.86 with mean of 0.63 and subsurface samples contains average 0.56% TOC and range from 0.35 to 0.71. 10% samples of surface falls in low, 80% in medium and 10% in high category while in subsurface 15% of OC belong to low category and 85% to medium. Aridisol represents a range of OC from 0.17 to 0.48 with a mean value of 0.35 in surface samples and from 0.10 to 0.40 with an average value of 0.29 in subsurface samples. 100% samples of this order belong to low category. OC content in surface soil of Alfisol ranges from 0.62 to 1.21 with an average value of 0.90%. Subsurface soils of the same order present OC range from 0.54 to 1.02 with a mean value of 0.76. 20% samples of surface belong to medium category and rest from high category. While in subsurface samples 45% belong to low and 55% to high category.

Table 2: Soil organic carbon content determined by Walkley and Black method, Colorimetric method and Dry combustion method

Order	Depth (cm)	Walkley and Black method (%)			Colorimetric method (%)			Dry combustion method (%)		
		Maximum	Minimum	Mean	Maximum	Minimum	Mean	Maximum	Minimum	Mean
Entisol	0-15	0.54	0.33	0.43	0.78	0.42	0.57	0.87	0.46	0.66
	15-30	0.43	0.20	0.32	0.56	0.22	0.41	0.70	0.28	0.50
Inceptisol	0-15	0.52	0.23	0.40	0.77	0.33	0.53	0.86	0.40	0.63
	15-30	0.46	0.22	0.35	0.61	0.29	0.47	0.71	0.35	0.56
Aridisol	0-15	0.40	0.15	0.29	0.46	0.16	0.33	0.48	0.17	0.35
	15-30	0.34	0.08	0.23	0.38	0.10	0.27	0.40	0.10	0.29
Alfisol	0-15	0.76	0.36	0.54	0.89	0.51	0.69	1.21	0.62	0.90
	15-30	0.57	0.34	0.44	0.73	0.44	0.57	1.02	0.54	0.76

Comparison of Methods of Organic Carbon Determination

Comparison of Walkley and Black method and Dry combustion method

The data represent that (Table 3) in Entisol OC recovery varies from 59.88 to 71.94 with an average value of 65.79% in surface soil while in subsurface it varies from 58.48 to 69.93 with a mean value of 64.93%. The correction factor (CF) varies from 1.39 to 1.63 with an average of 1.52 for surface while for subsurface CF ranges from 1.38 to 1.70 with a mean of 1.54. OC recovery in Inceptisol for surface ranges from 57.47 to 68.49 with a mean value of 63.01%, for subsurface varies from 55.87 to 67.56 with a mean value of 61.49%. Here CF varies from 1.46 to 1.74 with an average value of 1.59 for surface and from 1.48 to 1.79 giving an

average value of 1.63 for subsurface. In Aridisol for surface samples OC recovery varies from 75.76 to 89.29 with an average value of 82.61% and from 72.46 to 88.50 with an average value of 80.23% for subsurface. The CF in this order varies from 1.12 to 1.32 with a mean value of 1.22 for surface samples and from 1.13 to 1.38 with an average value of 1.25 for subsurface samples. In Alfisol, surface samples have OC recovery from 57.14 to 67.57 with a mean value of 60.16% and from 55.87 to 64.52 with a mean value of 59.46%. The correction factor varies from 1.48 to 1.75 with a mean of 1.67 and from 1.55 to 1.79 with a mean of 1.69 for surface and subsurface samples. Similar results of Walkley and Black recovery were reported: 72-86% by Charles and Simmons, 1986; 71% by Fernandes *et al.*, 2015 ^[9]; 75% by De Leenheer and Van Hove, 1958 ^[6]; 76.6% by Byers *et al.*, 1977 ^[1].

Table 3: Comparison of recovery and correction factor for Walkley and Black method with Dry combustion method

Order	Depth (cm)	Recovery (%)			Correction factor		
		Maximum	Minimum	Mean	Maximum	Minimum	Mean
Entisol	0-15	71.94	59.88	65.79	1.63	1.39	1.52
	15-30	69.93	58.48	64.93	1.70	1.38	1.54
Inceptisol	0-15	68.49	57.47	63.01	1.74	1.47	1.59
	15-30	67.56	55.87	61.49	1.79	1.48	1.63
Aridisol	0-15	89.29	75.76	82.61	1.32	1.12	1.22
	15-30	88.50	72.46	80.23	1.38	1.13	1.25
Alfisol	0-15	67.57	57.14	60.16	1.75	1.48	1.67
	15-30	64.52	55.87	59.46	1.79	1.55	1.69

Comparison of Colorimetric method and Dry combustion method

The OC content determined by CHNS analyser differs from colorimetric method. According to (Table 4) Entisol shows OC recovery from 79.37 to 92.59 with an average value of 86.2% in surface and from 76.92 to 90.91 with an average value of 84.75% in subsurface. CF varies from 1.08 to 1.26 with mean value of 1.16 and from 1.10 to 1.30 with mean value of 1.18 for surface and subsurface samples respectively. In Inceptisol, recovery varies from 78.13 to 90.91 with an average value of 84.53% for surface samples and from 77.52 to 87.72 with a mean value of 81.77% for subsurface samples. CF in this ranged from 1.10 to 1.28 with mean value of 1.19 in surface samples and from 1.14 to 1.29 with an average value of 1.22 in subsurface samples. Aridisol represent OC

recovery from 90.09 to 100 with an average value of 94.51% and from 86.21 to 100 with mean value of 93.13% in surface and subsurface samples respectively. CF in this order varies from 1.00 to 1.11 with mean value of 1.06 for surface samples and from 1.00 to 1.16 with mean value of 1.08 for subsurface samples. Alfisol shows recovery from 73.53 to 81.97 with mean 76.26% for surface samples and from 70.92 to 76.92 with mean 74.58% for subsurface samples. The correction factor varies from 1.22 to 1.36 with an average value of 1.31 for surface samples and from 1.30 to 1.41 with an average value of 1.34 for subsurface samples. Similar results by Sato *et al.*, 2014 ^[14] and Conyers *et al.*, 2011 ^[4] also suggested that combining wet digestion with colorimetric method provides a simple way of determination of OC than titration.

Table 4: Comparison of recovery and correction factor for Colorimetric method with Dry combustion method

Order	Depth (cm)	Recovery (%)			Correction factor		
		Maximum	Minimum	Mean	Maximum	Minimum	Mean
Entisol	0-15	92.59	79.37	86.20	1.26	1.08	1.16
	15-30	90.91	76.92	84.75	1.30	1.10	1.18
Inceptisol	0-15	90.91	78.13	84.53	1.28	1.10	1.19
	15-30	87.72	77.52	81.77	1.29	1.14	1.22
Aridisol	0-15	100	90.09	94.51	1.11	1.00	1.06
	15-30	100	86.21	93.13	1.16	1.00	1.08
Alfisol	0-15	81.97	73.53	76.26	1.36	1.22	1.31
	15-30	76.92	70.92	74.58	1.41	1.30	1.34

Conclusion

It can be concluded from this study that OC content differ in different methods of chemical analysis. Recovery of WB method was not similar in all soils against standard 77% recovery. Considering an average recovery for different soil orders, the correction factors of this study can be used for determining the SOC content in soils of Haryana. Therefore,

in different soil testing laboratories in Haryana state, the above correction factor should be used for making precise recommendations for nitrogenous fertilizers.

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References

1. Byers SC, Mills EL, Stewart PL. A comparison of methods of determining organic carbon in marine sediments with suggestions for a standard method. *Hydrobiologia*. 1977;58:43-47.
2. Chan KY, Bowman A, Oates A. Oxidizable organic carbon fractions and soil quality changes in an oxic paleustalf under different pasture leys. *Soil Science*. 2001;166(1):61-67.
3. Charles MJ, Simmons MS. Methods for the determination of carbon in soils and sediments. *Analyst*. 1986;3.
4. Conyers MK, Poile CGJ, Oates AA, Waters DD, Chan KY. Comparison of three carbon determination methods on naturally occurring substrates and the implication for the quantification of soil carbon. *Soil Research*. 2011;49:27-33.
5. Datta NP, Khera MS, Saini TR. A rapid colorimetric procedure for the determination of the organic carbon in soils. *Journal of Indian society of soil science*. 1962;10:67-74.
6. De leenheer L, Van Hove J. Determination of the soil organic carbon content: Critical study of titrimetric methods. *Pedologie*. 1958;8:39-77.
7. Dias RDS, Abreu CAD, Abreu MFD, Ferreiro JP, Matsura EE, Gonzalez AP. Comparison of methods to quantify organic carbon in soil samples from Sao Paulo State, Brazil. *Communication in Soil Science and Plant Analysis*. 2013;44(1-4):429-439.
8. FAO. Soil Organic Carbon: The hidden potential. Food and agriculture organization of the United Nations. Rome; c2017.
9. Fernandes RBA, Junior IAC, Junior ESR, Mendonça ES. Comparison of different methods for the determination of total organic carbon and humic substances in Brazilian soils. *Revista Ceres*. 2015;62:496-501.
10. Kerven GL, Menzies NW, Geyer MD. Soil carbon determination by high temperature condition, a comparison with dichromate oxidation procedures and the influence of charcoal and carbonate carbon on the measured value. *Communications in Soil Science and Plant Analysis*. 2000;31:1935-1939.
11. Lal R. Soil carbon sequestration impacts on global climate change and food security. *Science*. 2004;304:1623-1627.
12. Nelson DW, Sommers LE. Total carbon, organic carbon, and organic matter. pp. 539-579. In: A.L. Page *et al.* (eds.), *Methods of Soil Analysis. Part 2: Chemical and Microbiological Properties*. Agron. Monogr. No. 9. 2nd ed. American Society of Agronomy, Madison, WI, 1982.
13. Nelson DW, Sommers LE. Total carbon, organic carbon, and organic matter. D. L. Sparks, (ed.) In *Methods of Soil Analysis part 3 ed.* SSSA and ASA, Madison, WI; c1996. p. 961-1010.
14. Sato JM, Figueiredo CC, Marchão RL, Madari BE, Benedito IEC, Busato JG, *et al.* Methods of soil organic carbon determination in Brazilian savannah soils. *Scientia Agricola*. 2014;71:302-308.
15. Schnitzer M. Soil organic matter: The next 75 years. *Soil science*. 1991;151:41-58.
16. Singh SK, Singh AK, Sharma BK, Tarafdar JC. Carbon stock and organic carbon dynamics in soils of Rajasthan, India. *Journal of Arid Environments*. 2007;68:208-221.
17. Soon YK, Abboud S. A comparison of some methods for soil organic carbon determination. *Communication in Soil Science and Plant Analysis*. 1991;22:943-954.
18. Sreenivas K, Dadhwal VK, Kumar S, Harsha GS, Mitran T, Sujatha G, *et al.* Digital mapping of soil organic and inorganic carbon status in India. *Geoderma*. 2016;269:160-173.
19. Walkley A, Black IA. An examination of the Degtjareff method for determining soil organic matter, and a proposed modification of the chromic acid titration method. *Soil Science*. 1934;37:29-38.