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Determination of Organic Matter by Using Titrimetric and Loss on Ignition Methods for Northern Iraqi Governorates Soils

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Abstract

The study was carried out to determine soil organic matter content (SOM) using loss on ignition (LOI) and titrant methods. Eighty-four composite soil samples were collected from surface soil of randomly different locations in four governorates of northern parts of Iraq. The percent mean values of SOM recorded were 2.34, 2.399 and 1.821, respectively for Erbil, Sulaymaniyah, Kirkuk, and Duhok soil samples for the LOI method, while lower values were obtained 1.39, 1.595 and 0.98% respectively by using the titrant method for the same soil samples. A low relationship between two SOM procedures was obtained (r^2 = 0.462, 0.585 and 0.241) respectively for the same governorates soil samples. Current results revealed that the titrant method is more accurate and convenient to be used for SOM determination of soils in our area, and further studies must be done by using other methods to find the more suitable procedure for this purpose.

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1. Introduction

Soil organic matter is important for nutrient cycling and the maintenance of soil structure and fertility [1]. Decomposition is a biological process in which complex organic molecules in decaying matter are physically breakdown and biochemically transformed into simpler organic and inorganic molecules [2]. Organic matter improves the physical, chemical, and biological properties of the soil. It's a key factor in soil fertility and productivity [3].

Soil organic matter (SOM) is defined as all organic material found in the soils irrespective of origin or decomposition [4]. It is represented by soil humus which consists essentially of a series of products, ranged from decayed plants, animals, organisms, microbial synthesized compounds produced as a result of microbial decay [5]. Soil organic matters play an important role in soil quality, productivity and nutrient supply, reducing soil erosion and increasing water holding capacity [6,7]. Measurement of SOM has become a common procedure in soil science. To determine soil organic carbon, several methods have been used, each with its advantages and disadvantages in terms of accuracy, expense and convenience [8].

Loss on ignition (LOI) is one of the widely used methods for estimation of soil organic matter, soil organic carbon in agriculture, forest soils and sediments. It is characterized by simple, quick, inexpensive, safe and less labor-intensive procedures compared with Walkley and Black (Titrimetric method) [9]. The loss of soil mass at high temperatures (500-575 ºC) as a result of oxidation is proportionally related to soil organic matter content (10).

The titrimetric method is one of the common soil analysis procedures for soil organic matter determination in most laboratories. This procedure involves the reduction of potassium dichromate ($K_2Cr_2O_7$) by organic carbon compounds, in which unreduced dichromate titrant against ferrous ammonium sulfate to the measurement of oxidizable organic matter, the data converted to percent organic matter using a constant factor [11].

This study aimed to evaluating and comparing two procedures employed to determine soil organic matter in four governorates in the northern part of Iraq.

2. Materials and Methods

Study area:

The Governorates Erbil, Dohuk, Kirkuk and Sulaymaniyah are located in the northern part of Iraq. The region lies between latitudes 34°42' N and 37°22' N and between longitudes 42°25' and 46°15' East.

The climate of the area is characterized as an arid and semi-arid climate. It is hot and dry in summer and cold and wet in winter, with short spring and autumn seasons compared to summer and winter. The maximum daily temperature reaches as high as 50 °C in hot summer periods, while the minimum daily temperature can drop to −10°C in cold winter. Annual rainfall for Erbil, Kirkuk and Duhok ranged from 500-600 mm, while for Sulaymaniyah it ranged from 600-700 mm and it exceeds 1100 mm in mountain areas [12].

Sample collection:

Eighty-four (84) soil samples were collected from surface soils in four governorates during the summer months at four northern Iraqi Governorates (Figures 1-3). Soil samples were collected from each location at the surface horizons (depth of 30 cm) at random sampling points to form a composite sample. Air-dried soil samples were sieved (< 0.2 mm) and used for the determination of soil organic matter using two different methods.

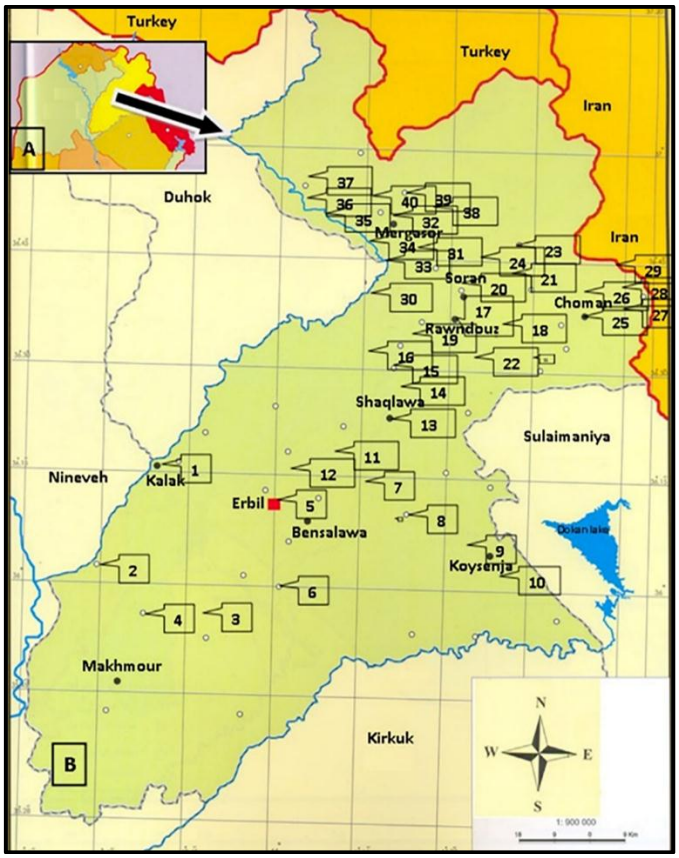

Figure 1. (a) Northern governorate of Iraq, (b) Erbil governorate, the location of sampling sites.

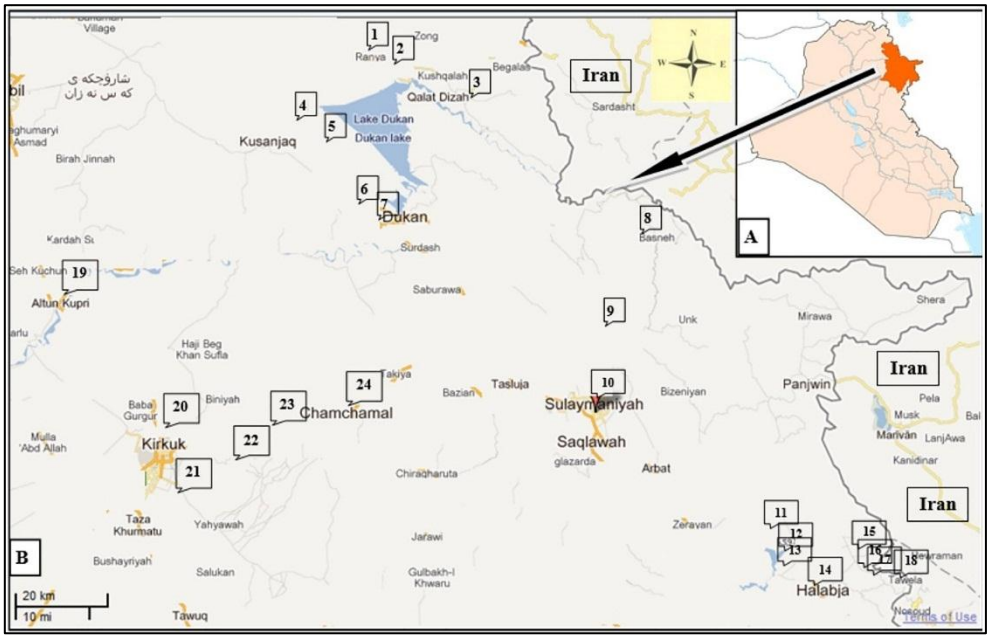

Figure 2. (a) Iraq, (b) Sulaimaniyah- Kirkuk governorates, the location of sampling sites.

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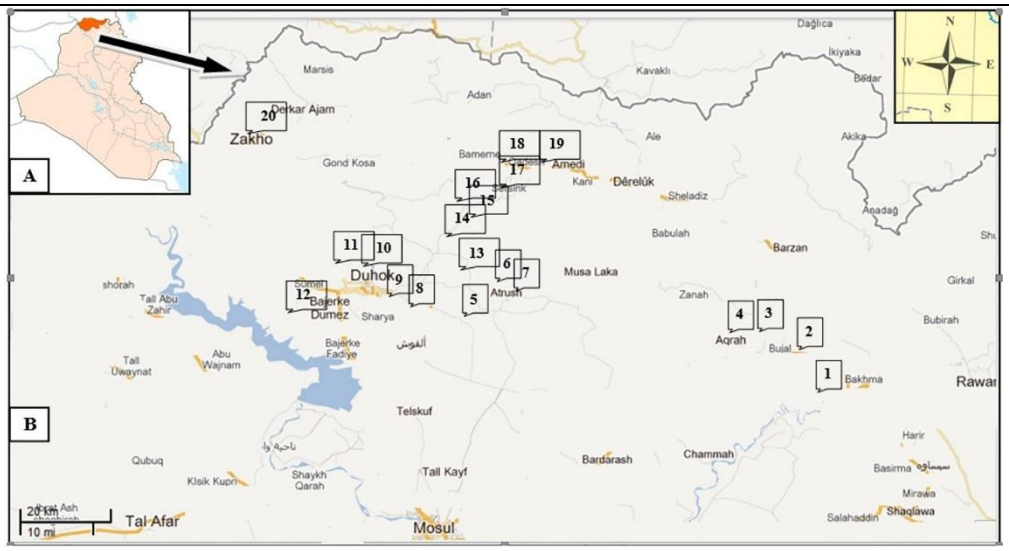

Figure 3. (a) Iraq, (b) Duhok governorate, the location of sampling sites.

One gram of oven-dried soil samples was exposed to combustion at 550 ºC for 4 hours in a muffle furnace. After cooling to room temperature in desiccators, and weighting [13]. Soil organic matter was calculated as follows:

Organic matter% = [(*Weight of soil oven dried* 105 ºC – *Weight of soil after combustion at* 550 ºC)/ (*Weight of soil oven dried* 105 ºC)]×100

The titrant method (Walkley and Black 1934) [14], is a procedure for determining soil organic matter by using $K_2Cr_2O_7$ and concentrated H_2SO_4 [15]. One gram of soil sample was placed in a 500 ml conical flask. Ten milliliters of 1N potassium dichromate ($K_2Cr_2O_7$) and 20 ml of concentrated sulfuric acid (H_2SO_4) were added to the soil while shaking it to ensure well mixing of the soil with the reagents. Soil sample reagent mixture was boiled at the hot plate for 30 minutes, after cooling 200 ml of distilled water, 10 ml of concentrated H_3PO_4 (85%) and 1 ml of diphenyl-amine indicator were added. The excess dichromate that was not reduced in the reaction was determined by volumetric titration using ammonium ferrous sulfate, when the color flashes from violet-blue changed to bright green, then the organic matter was calculated as follows:

$$Oxidizable\ organic\ carbon\ \% = \frac{(B-S)\times M\ of\ Fe\times 12\times 100}{gram\ of\ soil\times 4000}$$

where:

B = ml for blank titration

S = ml for the sample titration

12/4000 = milliequivalent weight of carbon in gram.

Total organic carbon multiply by 1.724 to the obtained percentage of oxidizable organic carbon

$$Organic\ matter\ \% = total\ organic\ carbon\ \times 1.724$$

Descriptive statistical analysis and Pearson correlation between mean values of soil samples were used to compare between two soil organic matter procedures by using (SPSS 20) program.

3. Results and Discussion

As shown in (Tables 1-3) the SOM content of studied sites was ranged between (0.65 to 4.77%) for the LOI method, while it was ranged between (0.025 to 4.08%) for the titrant method. High values of SOM were recorded by both methods, regardless of the soil type or form of the land use. This may be attributed to the soil samples were collected at surface and uncultivated soils that contain a high percentage of organic matter, from litters of trees, shrubs and herbs. Decomposition and decay of these litter by microbial activities produced humus that mixed with other soil mineral matters [5]. The mountain soil content high organic matter than in plains and foothills; this may be due to higher rainfall and moisture content which encourage more biomass production. In addition, the lower temperature also decreases the rate of mineralization in this area [16]. Maulood and Darwesh in 2020 [17] reported 0.22 to 2.15% SOM for upper parts of Erbil Governorates. Higher SOM was recorded by [1] for both methods in the same studied area. However, higher SOM of 7.82% for Erbil soil samples was recorded by [18]. They mentioned that in the northern governorates soils, with some cases where organic content exceeds 5%, particularly in natural vegetation and it may be due to the effects of variations in local conditions on the amount of plant residues added to soils, the rate of decomposition. These SOM content was higher than that of other Iraqi soils [3].

The results of LOI were higher than the titrant method, the mean value of SOM in the soil of Erbil governorate was 2.34, 1.39% respectively with a differences value of 0.95 between both methods (Table 4). Similar results were recorded for soil samples of other studied governorates. The correlation coefficient of studied soil samples of governorates between two different procedures was (r^2 = 0.680, 0.765) at $P \leq 0.01$ and (0.491) at $P \leq 0.05$, respectively (Table 1-3).

The linear regression for SOM by LOI and titrant methods, in Erbil soil samples, have a low value (r^2 =

0.462), while for Sulaimaniyah- Kirkuk and Duhok governorates have an (r^2 = 0.585, 0.241) respectively (Figures 4 and 5). These low regression values between LOI and titrant method is attributed to that LOI has a large variation in a mass loss, due to weight losses from structural water, and carbonate at high temperature [19]. Chatterjee et. al in 2009 [20] reported that LOI is occasionally used as a guide for carbon and organic matter content in soils. Many workers consider it too unreliable, due to potential losses of volatile salts, structural water and ammonia during combustion. While Sepahvand in 2020 [21] commented that the LOI procedure gives a rough indication for SOM. Bakr and El-Ashry in 2018 [6] reported to high linear regression obtained between both methods (0.93) but they recommended that the lower combustion temperatures of 300 to 375 °C for 2 hours produced the best matches between the measuring methods.

Aregahegn in 2020 [8] suggested that the titrant method, essentially estimating only organic carbon, with heating or without, the oxidation will not be complete. Using $K_2Cr_2O_7$ as the oxidant, that promotes only partial oxidation of the carbon. The carbon in the organic fractions that are surrounded and protected by the mineral fraction, the refractory carbon and inorganic carbon well not be fully attacked during the acid digestion. Therefore, the external heating for wet oxidation method could be used as a reliable and efficient for determining SOC without overestimating due to the oxidation of the inorganic carbon [19].

Table 1. Soil organic matter (%) determination by LOI and titrimetric methods in the soil of different locations from Erbil governorate.

Sites No.	Locations	Ignition method	Walkley and Black method	Sites No.	Locations	Ignition method	Walkley and Black method
1	Kalak	3.40	3.31	21	Halgort Mountain	4.11	4.09
2	Guwer	1.94	0.34	22	Akoyan	2.66	1.50
3	Yarmcha	2.66	1.50	23	Rosty	3.27	1.76
4	Mala-Qara	1.04	0.67	24	Hasarot	1.09	0.72
5	Erbil	1.73	0.98	25	Choman	1.90	0.72
6	Qushtapa	1.72	1.29	26	Rayat	1.42	1.03
7	Derband	4.46	3.54	27	Azadi	1.18	0.59
8	Degala	0.78	0.59	28	Haji- Omran	3.37	3.32
9	Koysenjaq	0.65	1.50	29	Haji- Omran	1.85	1.45
10	Koysenjaq	1.60	1.11	30	Chame Beaje	1.42	1.03
11	Qarasenj	2.03	1.09	31	Khalan	1.9	0.31
12	Bastora	1.19	0.39	32	Mergasor	2.73	0.52
13	Shaqlawa	2.57	0.03	33	Malman	2.37	2.79
14	Mama-Jelka	4.19	1.07	34	Soran	3.76	2.64
15	Hareer	1.84	0.72	35	Ashkafta	2.66	0.54
16	Spelek	2.92	0.98	36	Rezan	2.21	0.39
17	Jundeyan	2.22	1.50	37	Barzan	1.19	0.72
18	Zar- Gali	2.91	0.88	38	Maran	1.67	1.76
19	Rawndouz	2.75	1.50	39	Peran	3.8	2.46
20	Bradost	1.9	0.65	40	Mandan	4.51	4.09
		LOI method				Titrant method	
Mean		2.34				1.39	
Minimum		0.65				0.025	
Maximum		4.51				4.08	
Range		3.86				4.06	
Standard Error		0.163				0.162	
Correlation		0.689**					

Finally, it was concluded from the above results that LOI is less accurate than the wet oxidation method for SOM determination, and the last method is more convenient to be used for estimation of SOM in soils.

Table 2. Soil organic matter (%) determination by LOI and titrimetric methods in the soil of different locations from Sulaymaniyah and Kirkuk governorates.

Sites No.	Locations	Ignition method	Walkley and Black method	Sites No.	Locations	Ignition method	Walkley and Black method
1	Ranya	3.37	3.26	13	Serwan	4.77	4.03
2	Ranya	3.33	2.12	14	Halabja	3.27	1.29
3	Qalat Dizah	1.6	0.91	15	Khormal	3.68	2.07
4	Haybat Sultan	1.16	1.24	16	Bayara	2.39	0.57
5	Kani Watman	2.98	1.94	17	Balkha	3.07	1.55
6	Kelka smaqa	1.95	1.50	18	Tawela	2.36	2.59
7	Dukan	1.28	1.58	19	Altun Kupri	2.44	1.19
8	Basneh	2.4	0.65	20	Kirkuk	0.83	0.26
9	Qalat Cholan	2.46	1.68	21	Kirkuk	1.16	0.98
10	Sulaymaniyah	2.01	1.76	22	Chemano	1.73	1.06
11	Said Sadek	4.51	3.47	23	Qara Hanjer	1.16	1.47
12	Derbandikhan	1.88	0.54	24	Chamchamal	1.8	0.57

	LOI method	Titrant method
Mean	2.39	1.59
Minimum	0.83	0.26
Maximum	4.77	4.03
Range	3.94	3.77
Standard Error	0.242	0.196
Correlation	0.765**	

Table 3. Soil organic matter (%) determination by LOI and titrimetric methods in the soil of different locations from Duhok governorates.

Sites No.	Locations	Ignition method	Walkley and Black method	Sites No.	Locations	Ignition method	Walkley and Black method
1	Qandil bridge	1.22	0.54	11	Duhok	1.67	0.57
2	Gawelan	1.5	1.45	12	Zaweta	2.36	0.47
3	Aqrah	0.75	0.78	13	Mam- Yazden	3.15	0.98
4	Gali- Zanta	2.63	2.06	14	Swaratuka	1.68	1.50
5	Lalesh mountain	2.78	2.09	15	Solaf	3.17	1.63
6	Atrush	1.18	0.72	16	Serseng	0.65	0.47
7	Atrush mountain	2.45	1.01	17	Serseng	1.42	1.03
8	Baadrae	2.02	1.01	18	Kani chnarkae	0.9	0.98
9	Gali- Derkae	1.4	0.85	19	Qadash	1.72	0.72
10	Duhok	1.85	0.49	20	Zakho	1.92	0.26

	LOI method	Titrant method
Mean	1.82	0.98
Minimum	0.65	0.26
Maximum	3.17	2.09
Range	2.52	1.83
Standard Error	0.166	0.116
Correlation	0.492*	

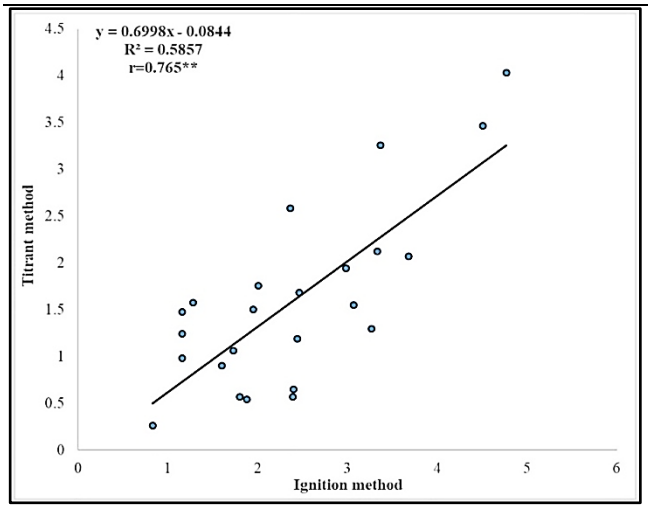

Figure 4. Relationship between LOI and titrant methods of Sulaymaniyah and Kirkuk governorates soil samples.

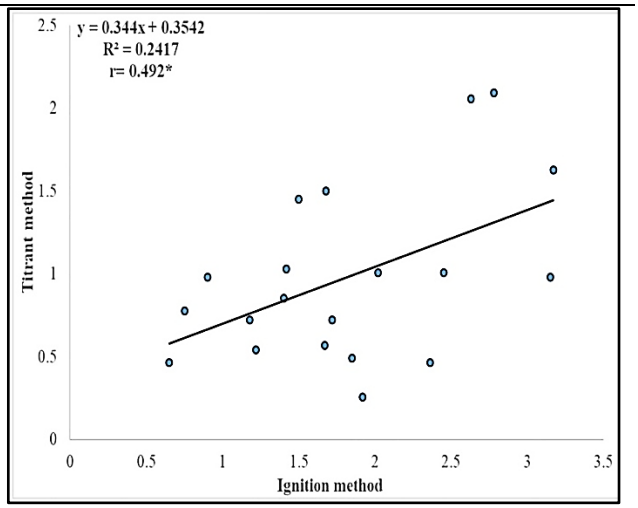

Figure 5. Relationship between LOI and titrant methods of Duhok governorate soil samples.

Table 4. Mean values of soil organic matter (%) determination by LOI and titrant methods from governorates with their differences values between methods.

Governorates	LOI method	Titrant method	Differences
Erbil	2.34	1.39	0.95
Sulaymaniyah and Kirkuk	2.39	1.59	0.80
Duhok	1.82	0.98	0.84

4. Conclusion

The results of the study demonstrated that the values obtained by the LOI method differed significantly from those produced by the titrant method. The two techniques were found to be reliable in this study, but the titrant method was more suitable. The soil in the northern parts of the studied area belongs to the Mollisols and Vertisols, which are rich in organic matter, due to the effect of soil-forming factors and the type and density of vegetation at each location.

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