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Influence of soil specimen preparation and test methods on soil organic matter content

Keywords:

soil organic matter content, oxidation method, loss of weight on ignition, specimen preparation, organic soil

Wpływ przygotowania próbek gruntów i metodyki oznaczania na zawartość części organicznych w gruncie

Słowa kluczowe:

zawartość części organicznych w gruncie, metoda utleniania, strata masy przy prażeniu, przygotowanie próbek, grunt organiczny

Abstract

Soil organic matter content (SOM) is used e.g. in soil classification and erosion evaluation. However, its value depends on soil specimen preparation and test method so it is useful to deal with these issues. Totally 150 specimens of soils CI, MS (from Bielsko-Biała, Poland) and different 3 soils, all classified as GC (from Ujsoly, Poland) were tested. To obtain values of SOM, the oxidation method using 30% hydrogen peroxide solution (H₂O₂) and loss of weight on ignition method (LOI) at 800°C (LOI-800) according to Polish Standard PN-88/B-04481 and LOI at 440°C according to the ASTM D 2974-87 (LOI-440) were applied. The tests were carried out for particles smaller than 0.5 mm. For every soil, 30 specimens (every with a mass about 10 g of dry soil) were prepared from 2400 g of dried soil for 3 methods, so for every method, 10 specimens, taken from various soil amount in frame of total 800 g were prepared. The results showed that values of SOM of tested soils varied from 0.33% to 6.09%. The largest relative difference in values of SOM, caused by soil specimen preparation, was 97.63% (H₂O₂, soil GC) and the smallest relative difference was 1.65% (LOI-800, soil CI). The largest relative difference in values of SOM, caused by test method was 1126.53% (LOI-800 and H₂O₂, soil GC) and the smallest relative difference was 15.17% (LOI-800 and H₂O₂, soil CI). The various values of SOM caused by different test method differently classify soils from organic point of view by the ISO14688-2:2004.

Abstrakt

Przeanalizowano wpływ przygotowania próbki gruntu i metody oznaczania na zawartość materii organicznej (SOM). W sumie przebadano 150 próbek gruntów (przygotowanych z różnej ilości gruntu) z Bielska-Białej (CI, MS) oraz z Ujsół (GC). Aby uzyskać wartości SOM zastosowano metodę utleniania 30% roztworem nadtlenku wodoru (H_2O_2) oraz metodę straty masy przy prażeniu (LOI) w temperaturze 800°C (LOI-800) i 440°C (LOI-440). Badania przeprowadzono dla cząstek gruntu mniejszych niż 0,5 mm. Wartości SOM wahały się od 0,33 do 6,09%. Największa względna różnica wartości SOM spowodowana przygotowaniem próbek wyniosła 97,63% (H_2O_2 , grunt GC), a najmniejsza 1,65% (LOI-800, grunt CI). Największa względna różnica wartości SOM spowodowana metodą badania wyniosła 1126,53% (LOI-800 i H_2O_2 , grunt GC), a najmniejsza 15,17% (LOI-800 i H_2O_2 , grunt CI). Uzyskiwane różne wartości SOM mogą skutkować odmiennym klasyfikowaniem gruntu pod kątem organicznym.

1. INTRODUCTION

A soil organic matter content (SOM) is an important soil property and is used e.g. in soil classification and erosion evaluation. To determine SOM, there are many methods which can be divided to major groups: direct methods and indirect methods. In the direct methods, the SOM is directly measured. Loss on ignition (LOI) method and H_2O_2 method are the two primary methods belonging to this category. In the indirect methods, the concentration of organic carbon is measured by chemical methods. The quantity of SOM is then determined by multiplying the concentration of organic carbon by a factor which varies with soil type and depth. This factor has a typical range of 1.7~2.1 [Huang et al. 2009].

Since various methods give various results, many authors deal with influence of test method on SOM. The temperature in LOI method is a key factor in determination of SOM and various standards prescribes various temperature. So e.g. according to the PN-88/B-04481 (1988), temperature should be 600–800°C; according to the ASTM D2974 (1987), temperature should be 440°C.

Concerning soils in Poland, Łądkiewicz et al. [2017] introduced values of SOM, obtained for 14 specimens from various locations in Poland. Authors applied Tiurin's method and also LOI method at 3 various temperatures: 440, 600 and 800°C. Results show that values of SOM obtained by LOI method are larger than that one obtained by Tiurin's method: 19% more at temperature 440°C, 48% more at temperature 600°C and 64% more at temperature 800°C. Based on values of SOM obtained by LOI method at 2 as high organic. Based on values of SOM obtained by LOI method at 2 as high organic. Based on values of SOM obtained by COI method at 2 as high organic. Some of SOM obtained by Tiurin's method, 7 soils are classified as low organic, 5 as medium organic and 2 as high organic. Authors state that applying LOI at higher temperature than 440°C causing risk of overestimation of SOM thus soils can be wrongly classified.

Further comparison of values of SOM of Mio-Pliocene clay in Warsaw, Poland, obtained by H_2O_2 method, Tiurin's method, LOI method (440°C) and thermal method (DTA) was carried out by Kaczmarek and Gawriuczenkow [2016]. Results show that SOM obtained by LOI method were ~7 times higher than those of the other methods. The H_2O_2 method revealed a negative result ~ -0.89%, due to oxidation of iron and hydration of its compounds. Based on these results, authors concluded that the Tiurin's method is the recommended technique for the assessment of organic matter content in cohesive soils.

Germaine and Germaine [2009] state that criteria for judging the acceptability of test results obtained by LOI method according to ASTM D2974 (1987) have not been determined. However, based on a brief study of performing twelve tests on a potting soil specimens with SOM of approximately 21%, two SOM tests performed properly by a single operator in the same laboratory in the same time period should not differ by more than about 4%. If the range of one set of measurements exceeds 4%, individual techniques should be evaluated. The likely problems are: incomplete ignition of organics, poor massing technique, insufficient initial specimen mass, insufficient cooling time or heterogeneity of specimens.

Taking into account the fact, that mass of a specimen is much smaller in comparison with collected amount of soil i.e. soil sample, heterogeneity of specimens could be a key factor influencing the values of SOM. Nguyen [2018] carried out totally 54 specimens of soils CG, CL, CI and CH from Lipnik, Międzyrzecze and Nieboczowy, Poland. To obtain values of SOM, the H_2O_2 method according to the PN-88/B-04481 was applied. The tests were carried out for particles smaller than 2 mm. For every sample, 3 specimens (every with a mass about 10 g of dry soil) were prepared by such a way that the first one is taken from whole sample after drying about 200 g of wet soil (TestNo1). The second and third one was taken from 50 g of remaining sample mass after taking the first one (TestNo2 and TestNo3). As an oxidizing agent, the 30%

hydrogen peroxide solution was used. The results showed that values of organic matter content of soils ranged from 0.03% to 2.74%.

The largest difference in values of SOM, caused by soil specimen preparation, was 0.10% (absolute difference between 0.13% and 0.03%, the values of SOM from TestNo2 and TestNo3, soil CI, Międzyrzecze), corresponding to relative difference 333.33%. Such high relative difference is caused by the fact that there are very small values of SOM so small absolute difference gives large relative difference. For soil CG (Lipnik) with the highest value of SOM (2.52% from TestNo3), the difference in values of SOM, caused by soil specimen preparation (TestNo2 and TestNo3) was 0.31% (corresponding to relative difference 14.03%).

Further comparison was made between value of SOM from TestNo1 and average values of SOM from TestNo2 and TestNo3. The largest difference in values of SOM, caused by soil specimen preparation in this case was 0.10% (absolute difference between 0.22% and 0.12%, the values of SOM from TestNo1 and average value of SOM from TestNo2 and TestNo3, soil CI, Międzyrzecze), corresponding to relative difference 83.33%. Again, such high relative difference is caused by the fact that there are very small values of SOM so small absolute difference gives large relative difference. For soil CG (Lipnik) with the highest value of SOM (2.74% from TestNo1), the difference in values of SOM, caused by soil specimen preparation was 0.38% (absolute difference between 2.74% and 2.36%, the values of SOM from TestNo1 and average value of SOM from TestNo3), corresponding to relative difference 15.86%.

From above mentioned literature review we can see that soil specimen preparation as so as test method have large influence on the value of SOM, further research on these topics will be useful. So we deal with these issues in this paper.

2. METHODOLOGY OF RESEARCH

To determine values of SOM, 2 samples of soils from Bielsko-Biała (Poland), marked as B1 and B2 were taken. These soils will be used in cover layer of an embankment so values of SOM will be used for their classification and erosion evaluation (Figure 1). In Figure 2 we can see 3 specimens of soils from Ujsoły (Poland), marked as U1, U2 and U3. These soils are original slope soils so values of SOM will be also used for their classification and erosion evaluation.



Fig. 1. Soil sample No. B1 and B2 from Bielsko-Biała, Poland (photo by G. Nguyen).

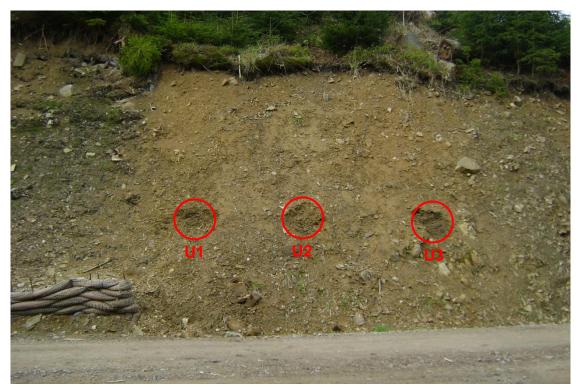


Fig. 2. Soil sample No. U1, U2 and U3 from Ujsoły, Poland (photo by G. Nguyen).

Determination of soil particles size distribution was carried out in accordance with the BS 1377:1990. Part 2 (wet sieving method and sedimentation by the hydrometer method). Soil basic parameters such as water content (w), liquid limits (w_L) and plastic limits (w_P) were also determined in accordance with mentioned standard. Based on obtained values, soils classifications were carried out in accordance with the British Standard BS 5930:2015. Soils properties are posted in Table 1.

Soil characteristics	Bielsko-Bia	ała (Poland)	Ujsoły (Poland)				
Specimen number	B1	B2	U1	U2	U3		
Soil classification by BS 5930:2015	CI	MS	GC	GC	GC		
Clayey fraction amount (%)	7.9	8.6	7.1	12.2	8.9		
Silty fraction amount (%)	63.5	52.7	20.2	20.3	17.5		
Sandy fraction amount (%)	22.8	31.7	35.1	27.0	35.5		
Gravelly fraction amount (%)	5.8	7.0	37.6	40.5	38.1		
Amount of fraction under 0.5 mm	85.9	78.8	47.2	48.5	43.3		
Water content (w) (%)	17.2	16.1	25.2	27.9	16.8		
Plastic limit (w _P) (%)	25.4	20.6	27.5	27.7	25.6		
Liquid limit (w∟) (%)	36.6	32.7	46.6	45.2	49.1		
Plasticity index I _P (%)	11.2	12.1	19.1	17.5	23.5		

Tab. 1. Research soil properties.

There are many methods to determine SOM but taken into account our laboratory equipment, purpose of the test (obtained value of SOM will be applied in soil classification and determination of soil erodibility factor K), geographical locations of soils (Poland) as so as previous research, it will be suitable to continue carrying out tests for determination of SOM

in accordance with the articles 4.4.4.1 and 4.4.4.2 of Polish Standard PN-88/B-04481 (determination of SOM by H_2O_2 and LOI-800 methods) and additionally also in accordance with the ASTM D2974 (determination of SOM by LOI-440 method).

To analyse influence of soil specimen preparation on values of SOM, for every above mentioned soil sample, about 2400 g of dried soil containing soil particles smaller than 0.5 mm was prepared. Applying the quartering method, mentioned amount of soil was divided to 3 equal parts, every from which has amount about 800 g and was used for one method. In frame of 1 method, about 10 g of soil from the first half (400 g) was taken and subjected to TestNo1. Further 10 g of soil from the second half (400 g) was taken prior TestNo2. The remained part about 780 g was divided by the quartering method to 4 equal parts (about 195 g), from which 4 specimens of amount about 10 g were taken and subjected to TestNo3, TestNo4, TestNo5 and TestNo6. After that the remained amount was about 185 g of 1 quarter was divided by the quartering method to 4 specimens of amount about 10 g were taken and subjected to TestNo10. So for 1 soil, 30 soil specimens were prepared and were tested by 3 various methods (H₂O₂, LOI-440 and LOI-800). Totally, 150 specimens of 5 soils were tested.

According to H_2O_2 method, soil specimen of mass about 10 g in a beaker (mass of the beaker will be m_t) was dried to a constant mass at 105–110°C (mass of the beaker and soil will be m_{st}). Then about 30 cm³ of 30% hydrogen peroxide solution is poured into the beaker, cover the beaker and gradually warm up to approximately 60°C, making sure that the content of the beaker does not foam too strong and does not spray. Heating was carried out during about 3 hours to the moment when after adding another 10 cm³ of 30% hydrogen peroxide solution, there are no gas bubbles. After the heating is finished, the contents of the beaker should be boiled, after that we will obtain slurry of a dense consistency; content of the beaker should not splatter. Then the beaker is placed in a dryer and dries to constant mass at 105–110°C. After cooling in the desiccator to room temperature, a beaker together with content is weighed with accuracy not less than 0.01 g (mass of the beaker and soil after drying will be m_u). SOM is calculated applying formula:

$$SOM = \frac{(m_{st} - m_u)}{(m_{st} - m_t)}$$
(1)

where:

 m_t – mass of the beaker, (g) m_{st} – mass of the beaker and soil, (g) m_u – mass of the beaker and soil after drying (g).

By the Standard, it is not necessary to carry out more specimens and calculate their average, so practically value of SOM obtained from one specimen could be sufficient.

According to LOI-800 method, specimen of mass about 10 g in a crucible is placed in a muffle furnace and temperature in the furnace is gradually increased to 600–800°C (in the tests, temperature 800°C was applied and this temperature was reach after 1 hour) and hold for at least 4 hours (in the tests, temperature 800°C was hold for 4 hours). After cooling in a desiccator, mass of soil with crucible was determined. After that the procedure is repeated until there is no change in mass from the previous measurement. The formula (1) is applied for calculation of SOM value. The same procedure is applied for LOI-440 method but temperature is 440°C instead of 800°C.

In Fig. 3 we can see burning temperature 440°C in muffle furnace L 9/11/B180 (upper) and ten specimens (TestNo1 to TestNo10) of soil sample U1 from Ujsoły after burning (lower).



Fig. 3. Burning temperature 440°C in muffle furnace L 9/11/B180 (upper) and ten specimens (TestNo1 to TestNo10) of soil sample U1 taken from Ujsoły after burning (lower).

3. RESULTS

The values of SOM of 150 specimens are posted in Table 2 and graphical presentation of values of SOM as so as different values of SOM obtained from various methods can be seen in Figure 4. As we can see, specimen preparation has the largest influence when applying H_2O_2 method and the smallest influence when applying LOI-800 method. This rule is valid for all 5 soil samples (see values in line "Relative difference (Max-Min) (%)" in Table 2 and columns for Max. and Min. values of SOM in Figure 4). It is necessary to note, that it is applied for relative differences (the largest difference between max. and min. value of SOM in frame of 1 method is 0.74% (soil GC, U1, LOI-400 method).

The largest relative difference between max. and min. value of SOM in frame of 1 method (showing the largest influence of specimen preparation) is 97.63% (soil GC, U3, H_2O_2 method, between TestNo5 and TestNo6). The smallest relative difference between max. and min. value of SOM in frame of 1 method (showing the smallest influence of specimen preparation) is 1.65% (soil CI, B1, LOI-800 method, between TestNo5 and TestNo9).

By comparison of position (placement) of max. and min. value of SOM in the Table 1 we can conclude that there is no rule applied for influence of specimen preparation on SOM value. It seems that specimen in TestNo10 (taken from about 46 g of dried soil, not 400 g or 195 g) could have the largest SOM value (5 from 15 cases), however, in case of soil GC (U1, LOI-800 method), TestNo10 specimen has the lowest SOM value.

Concerning influence of test method on SOM value, we can see that H_2O_2 method gives the smallest SOM value and LOI-800 method give the largest SOM value. The largest absolute difference in SOM values, obtained by various methods, is 5.52%, corresponding to relative difference 1126.53% (soil GC, U3, LOI-800 and H_2O_2 method). The smallest absolute difference in SOM values, obtained by various methods, is 0.71%, corresponding to relative difference 15.7% (soil CI, B1, LOI-800 and LOI-440 method).

Generally, H_2O_2 method gives small SOM values in comparison with LOI-440 and LOI-800 methods. Even the smallest difference in SOM values, obtained by H_2O_2 and LOI-800

method, reaches value 3.08%, corresponding to relative difference 133.33% (soil CI, B1). Difference in SOM values, obtained by LOI-440 and LOI-800 methods are not so large but it is not negligible. The largest absolute difference reaches value 2.29%, corresponding to relative difference 61.51% (soil GC, U3).

		SOM (%)														
Locations, specimen number, test method		Bielsko-Biała (Poland)						Ujsoły (Poland)								
		B1 (Cl)			B2 (MS)			U1 (GC)			U2 (GC)			U3 (GC)		
		H ₂ O ₂	LOI-400	LOI-800	H ₂ O ₂	LOI-400	LOI-800	H ₂ O ₂	LOI-400	LOI-800	H ₂ O ₂	LOI-400	LOI-800	H ₂ O ₂	LOI-400	LOI-800
Taken from about 400 g of soil	TestNo1	2.50	4.59	5.37	1.05	3.26	4.03	0.50	3.95	5.76	1.02	4.00	5.99	0.51	3.75	6.01
	TestNo2	2.26	4.67	5.36	1.17	3.07	4.04	0.39	3.73	5.74	0.98	4.27	5.89	0.44	3.75	6.02
Taken from about 195 g of soil	TestNo3	2.25	4.74	5.39	1.17	3.06	4.10	0.38	3.85	5.78	1.08	4.23	5.97	0.47	3.67	6.08
	TestNo4	2.30	4.70	5.38	1.23	3.15	4.00	0.42	3.40	5.76	1.01	4.08	5.95	0.38	3.83	6.07
	TestNo5	2.27	4.73	5.44	1.24	3.28	3.95	0.59	3.97	5.85	1.08	4.19	5.91	0.33	3.42	6.06
	TestNo6	2.15	4.49	5.40	1.16	3.18	3.96	0.59	4.09	5.87	1.28	4.12	5.94	0.64	3.75	6.09
Taken from about 46 g of soil	TestNo7	2.39	4.64	5.39	1.26	3.23	4.02	0.62	4.15	5.78	1.21	4.18	5.98	0.57	3.72	5.65
	TestNo8	2.14	4.71	5.39	1.23	3.23	4.00	0.52	3.54	5.82	1.12	4.18	5.92	0.41	3.69	6.00
	TestNo9	2.47	4.73	5.35	1.23	3.16	3.99	0.34	3.61	5.83	1.09	4.18	5.94	0.56	3.82	6.03
	TestNo10	2.40	4.76	5.40	1.26	3.25	4.02	0.47	3.97	5.74	1.33	4.31	6.01	0.58	3.82	6.08
Average		2.31	4.68	5.39	1.20	3.19	4.01	0.48	3.83	5.79	1.12	4.17	5.95	0.49	3.72	6.01
Max.		2.50	4.76	5.44	1.26	3.28	4.10	0.62	4.15	5.87	1.33	4.31	6.01	0.64	3.83	6.09
Min.		2.14	4.49	5.35	1.05	3.06	3.95	0.34	3.40	5.74	0.98	4.00	5.89	0.33	3.42	5.65
Absolute difference (Max-Min)		0.36	0.26	0.09	0.21	0.22	0.14	0.28	0.74	0.13	0.35	0.32	0.12	0.32	0.41	0.44
Relative difference (Max-Min) (%)		16.78	5.83	1.65	19.67	7.25	3.67	80.08	21.78	2.34	35.05	7.95	1.99	97.63	1 2.0 8	7.73
Classification of organic soils according to ISO14688-2:2004 (based on average value of SOM)		low organic content	low organic content	low organic content	-	low organic content	low organic content		low organic content	low organic content		low organic content	low organic content		low organic content	medium org. content
Abs. and relat. diff. between LOI-400 and H_2O_2		2.37 (102.60%)			1.99 (165.83%)		3.35 (697.92%)			3.05 (272.32%)			3.23 (659.18%)			
Abs. and relat. diff. between LOI-800 and H_2O_2		3.08 (133.33%)			2.81 (234.17%)		5.31 (1106.25%)			4.83 (431.25%)			5.52 (1126.53%)			
Abs. and relat. diff. between LOI-800 and LOI-400		0.71 (15.17%)			0.82	0.82 (25.71%)		1.96 (51.17%)			1.78 (42.69%)			2.29 (61.51%)		

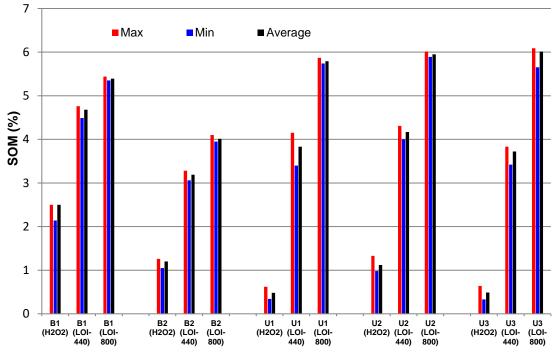


Fig. 4. Graphical presentation of SOM values of differently prepared specimens, obtained by various test methods.

Various SOM values, obtained by various test method cause different soil classification from organic point of view. The ISO 14688-2:2004 differs soils from organic point of view: low organic (SOM from 2–6%), medium organic (SOM from 6–20%) and high organic (SOM is larger than 20%). Classification of organic soils according to the ISO14688-2:2004 (based on average value of SOM) is posted in Table 2. As we can see, based on SOM values obtained by H_2O_2 method, 4 soils are classified as without organic content, only 1 is classified as low organic but based on SOM values obtained by LOI-800 method, 4 from 5 soils are classified as low organic and 1 soil is classified as medium organic. Extreme case can be seen for soil GC (U3), where 3 methods give 3 different SOM values providing 3 different soil classifications from organic point of view.

4. DISCUSSION

Research results confirm some conclusions of various researchers on the fact that SOM values obtained by LOI-800 are larger than these ones obtained by LOI-440 and difference between SOM can cause difference in soil classification from organic point of view [Łąd-kiewicz et al. 2017]. It seems that Kaczmarek's and Gawriuczenkow's [2016] result showing that SOM obtained by LOI method were ~7 times higher than those of the other methods could be extreme but in this case, SOM value obtained by LOI-800 is up to 11.2 times larger than H_2O_2 method. In comparison with Germaine's and Germaine's [2009] result, where based on a brief study of performing twelve tests on a potting soil specimens with SOM of approximately 21%, two SOM tests performed properly by a single operator in the same laboratory in the same time period should not differ by more than about 4%, in this case range of one set of measurements exceeds 4% (min. 5.83% and max. 21.78%, see these values in Table 2 only for LOI-440 method, since only these can be compared with Germaine's and Germaine's [2009] method). The reason is that SOM values of soils in this case are not so high (only 4.76% and 4.12%) so relative differences are large.

As results show, SOM values obtained by H_2O_2 method are very small in comparison with LOI-440 and LOI-800 method. It is believed that H_2O_2 did not remove all organic matter in soil. It can be seen also from the fact, that just heterogeneity of specimens, caused by sample preparation, has the largest influence in just this H_2O_2 method.

4. CONCLUSION

- Soil specimen preparation has the largest influence on SOM value when applying H₂O₂ method and the smallest influence when applying LOI-800 method. It is suggested that H₂O₂ did not remove all organic matter in soil. Therefore it is recommended carrying out more specimens even the Standard does not prescribe.
- 2. Heterogeneity of soil specimens, caused by specimen preparation, has the smallest influence on SOM value when applying LOI-800 method. This fact can confirm that SOM are burned well at this temperature.
- No rule applied for influence of specimen preparation on SOM value. Maximal and minimal SOM value from 10 specimens (TestNo1 to TestNo10) is randomly assigned to various specimens, not depending on the fact that specimens were taken from about 46 g of dried soil, or 400 g or 195 g.
- 4. SOM values obtained by H₂O₂ method are the lowest and by LOI-800 are the highest. Difference in SOM values, obtained by LOI-440 and LOI-800 methods are not negligible.
- 5. Various SOM values, obtained by various test methods cause different soil classification from organic point of view.

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