

Application of microwave method for moisture determination of organic and organic-mineral soils

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Abstract. The problem of rapid drying arises when determining moisture, ash and organic matter content, as well as during many other soil tests. For highly-organic and organo-mineral peat soils the problem of advanced measurement of moisture content is of special importance, since after reweighing the dry sample increase in mass may be observed. The article examines the methods in determining the moisture content in peat and organic soils via microwave radiation, which will greatly speed up the process, simplify the complexity and cost of laboratory tests. The paper presents a detailed review of the methods determining moisture content in soils and characteristics, as well as application scope. The work contains the research results on moisture organic soils: drying in a microwave oven and the current domestic standards.

1. Introduction

The problem of rapid soil drying arises when determining moisture, ash and organic content as well as many other types of soil tests. For organic and organo-mineral peat soils, advanced moisture determination is of particular importance, since reweighing demonstrates the sample mass increase. Due to this fact, it is required to determine the smallest mass which is relevant to GOST 5180 [4].

The aim of this work is to study drying process in microwave ovens to determine the moisture content of organic and organo-mineral soils. The research objectives include: an overview of foreign and domestic scientific works on the testing techniques of moisture content in soils; sampling and laboratory tests to classify the indicators of composition and physical properties of peats and peat soils; measurement and comparison of their moisture by microwave and convection drying; measurement of organic matter loss after drying; development of brief instructions in using of microwave ovens for rapid drying of organic soils.

Literature and regulatory document reviews revealed variable methods for measuring soil moisture [1-14], and, accordingly, to current standards, a drying method in a convection oven is applied. In accordance with GOST 5180 [4] the soil sample weight of 15-50 g is dried to constant weight at a temperature of 105±2 °C for 5 hours, after which the drying continued to obtain the mass difference between two successive weighings (not more than 0.02 g), with a time interval of 2 hours of mass measurement. According to ASTM D2216 [10] drying dispersed and rock soils is conducted at a temperature of 110±5°C. To reduce gypsum dehydration in saline soils or decomposition of organic matter in peats, it is recommended to dry samples at 60° C or at room temperature in a desiccator. As defined in GOST 19723 [11] the peat portion of 15-20 g was dried within 4.5 hours at 105°C. The dried mass sample per hour is considered permanent if the difference in mass in two successive



weighings does not exceed 0.02 g. Based on GOST 11305 [12] the peat portion of 5 - 10 g is placed in a heated oven to a 105-110°C and dried within 2.5 - 4.0 hours, after weighing peat portions are dried for 30 minutes. If the mass loss does not exceed 0.01 g, the test is completed. In the accelerated method [12] peat portions weighing 5-6 g are placed in a drying oven heated to 165-170 °C and then dried at 145-150 °C within 30 min. If the peat portions contain moisture (moisture is the ratio of water mass to the initial mass of wet peat) more than 55 % – 45 min are required. Current measurements are performed in the case of determining a single peat portion. In accordance with ASTM D 2974-14 [13] the moisture peat content is determined by two methods: A) by drying at a temperature of 110±5° C and b) by dewatering in two stages: after evaporation at room temperature and after drying in an oven at 110±5° C (if peat is used as fuel).

According to ASTM D 4643 [14], the drying soil process in a microwave oven is also used to determine the moisture content. A peat portion of 200 g (for soils containing up to 10 % of the fraction greater than 2 mm) is placed in a microwave for 3 minute defrosting. Then the peat portion is weighed at 1 minute interval until re-weighing demonstrates mass loss of less than 0.1 %. Optimal power of the oven may be higher than in defrosting. The time of initial drying of clay soils can be increased to 12 minutes. Moisture is calculated as the ratio of the water mass to dry soil mass within the accuracy of 0.1 %.

2. Materials and methods

The authors studied the soil samples from the territory of Tomsk oblast. During the tests, following samples were analyzed: peat samples of varying decomposition, low-ash, wood, grass, moss, and grass-moss groups and peat clay and loam. In accordance with the procedures [2, 4, 5, 7]: moisture content (m), degree of peat decomposition (Ddp), organic content (Ir) and moisture at the boundary of liquid and fluidity (wL and wp) of organo-mineral soils were determined. During the laboratory investigations the following equipment was used: drying oven, moisture-testing oven SSP-0.25-100 and a dry-air oven SSVL-80-Kasimov, microwave ovens (LG of 900 Watts and Welton WMO-1700GW with variable regulation power, muffle oven MIN-10 UE, scale with accuracy of 0.01 and 0.001 g. To compare convection and microwave drying results each soil sample was divided into halves. Based on the methods [4, 11] from each half, 4 to 8 parallel 15-50 g peat portions were selected. Peatmoss samples were taken with a weight of 5-10 g [12], 15-20 g [11], as well as heavier samples of 100-200 g [14], in order to reduce the variation between parallel determinations of water-saturated samples. Drying was conducted before obtaining the mass difference of soils within containers in two weighings (not more than 0.02 g) [4]. The drying time of the samples in a convection oven complies with the standard requirements [4, 11, 12]. The testing time in the microwave was determined by the moisture content and sample weight and ranged from 10 to 30 minutes for a 50 g peat portion, and for peat portion of 100-200 g up to 1.5 hour (at radiation power ~200 W). The intervals between weighings were selected empirically in respect to the sample weight, moisture content, evaporation area, as well as the inertia of the microwave heating which resulted in significant drying slowdown (the intervals between weighings - less than 1-2 min). The first weighing was carried out at 1-2 minute interval for a sample weight up to 50 g and 3-7 minute interval for the samples of more than 50 g.

It is necessary to note the specific features of drying organic and organo-mineral soils (Table 1, Figure 1) such as, high moisture content in peat, process duration and possible organic matter loss at high temperatures. Maximum moisture content (1500-3000 % or more) was observed in slightly decomposed grass-peat and moss samples; average value of 400-600 % was revealed in highly - and medium -decomposed peat of wood and grass groups, minimum value of about 25-70 % - for soils with low peat content, which is typical for these varieties. Accordingly, increasing moisture content values are increasing too. If the sample weight will be increased from 5 to 20 g, then the dispersion of measurements results decreases slightly (Figure 1, b, d; PL. 1). Considerable variation of parallel definitions are observed, if a peat moss portion of 100-200 g (Figure 1, a). Increasing sample number to 8-10 peat portions did not improve the results (Figure 1).

Table 1. Results of the moisture content determination of organic and organo-mineral soils.

The name of the soil	Sample weight, g	The depth of sampling, m	Water content, % by drying in the oven						
			microwave			convection			
			Minimum	Maximum	Average	Minimum	Maximum	Average	
Organic soils	Least decomposed peat moss group	100–200	0,2	3125	3255	3188	2412	3327	2994
	Least decomposed peat moss group	15–20	1,0	2253	2794	2527	2390	3003	2761
	Medium decomposed peat herbal group	5–10	1,0	2200	2754	2513	2250	3063	2816
	Highly decomposed peat wood group	15–50	1,0	603	617	609	574	598	587
	Least decomposed peat grass-moss group	15–50	1,0	353	464	397	369	485	416
	Least decomposed peat grass-moss group	15–50	1,0	1925	2333	2097	2340	2506	2447
	Highly decomposed peat woody-herbal group	15–50	3,0	1443	1506	1464	1465	1966	1762
	Least decomposed peat moss group	15–50	1,0	394	402	398	416	438	428
Organic-mineral soils	Plastic peat clay	15–50	2,0	56	60	58	58	61	60
	Peat loam	15–50	4,5	29	31	30	27	30	29

The analysis of moisture vs the drying time graphs showed that all varieties can be divided into three main stages: the first stage (I) – initial, with a relatively upward slope, during which the heated moistured sample with a slight acceleration of evaporation is observed; second stage (II) – stage of evaporation, when main moisture volume evaporates from the sample; third stage (III) – final drying, characterized by a relatively slow evaporation of the remaining free water.

At the same time, investigated types of soils have particular characteristics of drying. Most water saturated least decomposed moss and grass-moss peats (Figure 1, b, C, d) are characterized by prolonged stage of moisture heat-up and rapid evaporation. Medium-decomposed peats of wood, grass (Figure 1, g), wood-grass and grass-moss groups are characterized by a longer second stage, due to the fact that the number of samples, has a fairly large area of evaporation and ensures good penetration of microwaves into the samples. Therefore, they are characterized by a short time of final stage of drying, during which the remaining moisture is easily removed. Separately, a group of soils with low peat content (Figure 1, g) and relatively low moisture content should be identified. These groups are unstable or have no initial stage (Figure 1, e) due to the rapid heating of the entire volume of the sample and intensive evaporation in the first minutes of the experiment.

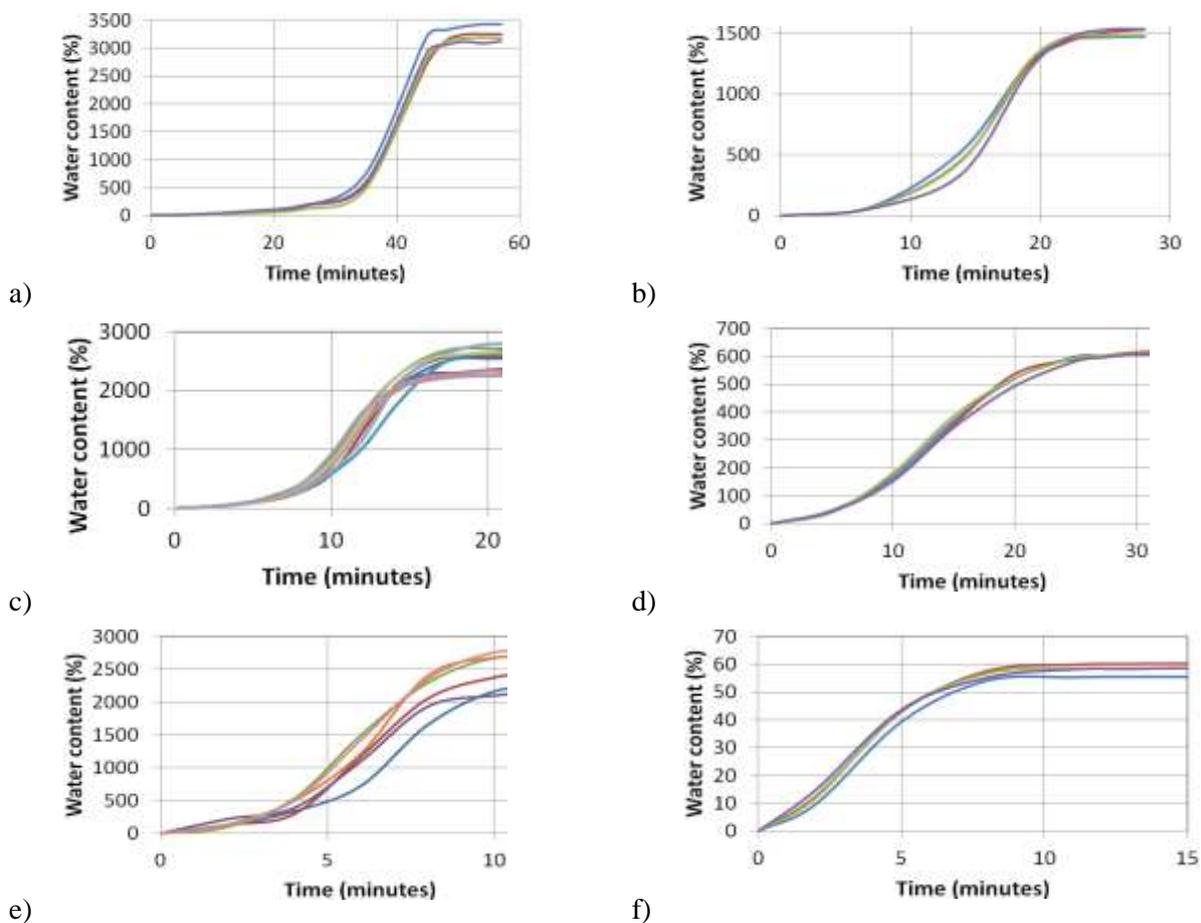


Figure 1. Dependence of soil moisture on drying time: a) least decomposed peat moss group (weight 200 g), b) least decomposed peat grass-moss group (15-50 g), c) least decomposed peat moss group (weight 15-20 g); d) medium decomposed peat grass group(15-50 g), e) least decomposed peat moss group (weight 5-10 g); f) clay with low content of peat (15-50 g).

This contributes to a relatively small sample surface area of evaporation and formation of a solid crust. As a consequence, high heating imposes restrictions on the material used for containers. In general, first two stages of drying take half of the testing time, during which up to 90% of the initial volume of moisture evaporates. The final stage of drying takes from 20 to 50 % of the experiment time.

To identify possible mass loss of soil due to the organic matter burning in convection and microwave ovens [10], methods of calcination were used in accordance with the applicable standards [13, 15-17]. Gravimetric method is based on determining organic matter in peat soils according to GOST 26213 and GOST 27784 [15, 16] after calcination at a temperature of $525 \pm 25^\circ\text{C}$. According to GOST 11306 [15] for the peat fuel production the ash content was determined by calcination at $800 \pm 25^\circ\text{C}$, and for agricultural and environmental at $525 \pm 25^\circ\text{C}$. In accordance with ASTM D 2974 [12] the ash content is determined by burning in a muffle oven: by “C” method at a temperature of $440 \pm 40^\circ\text{C}$ and “D” method (for peat, used as fuel) at a temperature of $750 \pm 38^\circ\text{C}$. The organic matter decomposition ranges between 229 and 579°C , with a maximum of $300\text{-}400^\circ\text{C}$ [18]. Finally, for the sample calcination the following temperatures were selected: 800, 525 and 350°C . The conducted tests (Table 2, for clarity, values are rounded to integers) showed a good correlation of the results of organic content determination after drying and other methods. Thus, it can be stated that organic

matter loss is the same both for the application of microwave and convection ovens (2-3 %). It should be noted that lower the temperature is, higher the ash content is.

Table 2. Organic matter content in organic and organic-mineral soils.

Name of the soil	Organic matter Content I_r , % after drying					
	Convection oven			Microwave oven		
	800°C	525°C	350°C	800°C	525°C	350°C
Least decomposed peat moss group	96	94	91	95	91	90
Highly decomposed peat wood group	90	92	85	90	92	87
Loam with low contents of peat	14	13	12	16	14	13

3. Results and discussion

The research results demonstrate that the variations in the moisture content of peats obtained at microwave drying are not higher than in convection drying in most cases (Table 1, Figure 1). In both cases variations fail to meet the standard requirements (allowable difference in moisture content over 100% is 5% [4], moisture – 1 % [11]). Mineral soils have little variation moisture content [14], as it has been proved. Variations of the obtained values are accounted for uniformity coefficient of the soil composition, irregularity in the distribution of organic remains and mineral inclusions, humus content of different fibering, and variable absorbing and water-holding capacity of plant remains. It should be noted that it is quite difficult to conduct testing in undrained peat areas, as well as to transport large naturally preserved samples. The moisture loss in these processes significantly exceed the difference of parallel measurements, therefore, it is rational to determine the physical properties of a single peat portion [12], by increasing the number of core samples (to 15-20 pieces) from each of the engineering-geological soil elements.

It was possible to make brief recommendations on the methodology for moisture content determination via microwave ovens for organic soils.

1. Appliance of a microwave oven excludes the use of metal containers, impenetrable to microwaves and resulting in a short circuit. Samples should also have a low content of electrically conductive minerals.

2. The optimal heating power of the microwave oven for drying organic soils is 200-500 watts (for small samples less heating power is recommended to strike a balance between optimal drying speed and heating). For drying highly-decomposed organic, organic with high ash content and organo-mineral cohesive soils, porcelain or ceramic containers withstanding high temperature drop are recommended. Plastic containers are not advised for highly decomposed organic samples as they melt in the final stages of drying. However, for least decomposed water-saturated peats, household plastic containers can be applied. Setting microwave power over 200W is recommended only for porcelain containers, as in the third (III) drying phase extensive heat occurs that can destroy most materials.

3. The optimal sample mass is determined by the initial water saturation of the soil and required weight range of 15-50 g for medium- and highly-decomposed peats and organic soils; peat portions up to 100-200g are recommended for least decomposed peat moss, peats with large plant remains, fibers or different fibering peats.

4. To reduce the moisture content variations, it is desirable to balance between the soil mass and the number of samples in the oven. To analyze medium- and highly-decomposed peats and peat soils, samples of 15-50 g. with the number from 3 to 6 pieces are recommended. For least decomposed water-saturated peats, peat portions of over 100 g are recommended for the number of samples not more than 2-3 pieces. For multiple moisture determination it is efficient to use an accelerated method according to [11].

5. The drying time for peat portions of more than 100 g is 30-40 minutes, for peat portions of 30-50 g (and for peat soils) is 10-15 minutes, for peat portions of 5-10 g is 3-5 minutes. The time intervals between reweighting are: 3-5 minutes for sample weighting more than 50 g; 1-2 minutes for peat portions of more than 5-10 grams and for soils with low peat content. Expanding the area of tested

sample evaporation intensifies drying process as well as reweighing via sample blending (e.g., for cohesive soils by rolling them into a thin layer and surfacing channels with a glass spreading rod).

4. Conclusions

Thus, the study confirmed that household microwave ovens are an efficient means for rapid moisture content determination of organic and organo-mineral soils, as the testing is carried out in short time, and the obtained data are as accurate as in applying the drying ovens. After being tested on moisture content, the soil calcination at different temperatures indicated that the organic matter loss is comparable as well. The positive aspect of this paper is that, reweighing a sample weight does not increase after microwave drying (Figure 1), which, in its turn, increases the accuracy of moisture content determination. The authors expect that the proposed recommendations could be used for low-budget equipment in engineering-geological surveys more extensively, as well as in the field and laboratory environment.

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