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TECHNICAL NOTE

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New Method to Determine The True Water Content of Organic Soils

ABSTRACT: The true water content of organic soils is overestimated when the standard oven drying temperature of $110 \pm 5^\circ\text{C}$ is used due to some oxidation of the solids, and underestimated when the recommended oven drying temperature of 60°C is used due to some residual pore water remaining in the voids. A new method is presented to identify the precise oven drying temperature for which the mass of the residual pore water exactly balances the loss in the mass of solids due to oxidation. The true water content is calculated on the basis of the specimen dry mass recorded for this oven drying temperature.

A larger error arose when the water content of the test soil (70 % organic content) was determined using the specimen dry mass recorded at 60°C rather than the standard drying temperature of $110 \pm 5^\circ\text{C}$. Longer drying times were also required for the specimen dry mass to equilibrate, making it more inconvenient from a practical standpoint. Some oxidation of the organic solids occurred for oven drying above 80°C . The true value of the water content corresponded to an oven drying temperature of 86°C .

KEYWORDS: oven drying, organic soil, water content determination, drying temperature

The oven drying method, ASTM Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass (D2216), is the preferred method for determining the water content, defined as the ratio of the mass of pore water in the void space to the mass of solid particles, expressed as a percentage. The water content of the wet specimen is calculated as the reduction in the specimen mass, expressed as a percentage of its equilibrium dry mass, after oven drying at the standard temperature of $110 \pm 5^\circ\text{C}$. The method assumes that the reduction in the specimen mass is entirely due to the evaporation of the pore water in the voids. Other forms of water, namely absorption water (the thin layer of tightly bound water molecules surrounding

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the solid particles), and chemically combined water of hydration, are not taken into account in determining the soil water content.

However, oven drying of organic soils at 110 ± 5 °C may cause a reduction in the mass of the solid particles due to some slight oxidation of the organic fraction. Ensuing water content calculations will slightly overestimate the true value of the water content. Lower oven drying temperatures of typically 60–80 °C (Jarrett 1983) are routinely used to reduce the level of oxidation. However, small quantities of pore water that would otherwise evaporate at 100 °C remain in the dried specimen when lower oven temperatures are used. The remaining unbound water in the soil comprises pore water, and for the organic fraction, internal capillary water and intracellular water (Möller 1983). Water content calculations will slightly underestimate the true water content value in this case. Research in the literature on the oven drying characteristics of organic soils, particularly in relation to determining the water content, is limited. Previous work includes that of Skempton and Petley (1970) who studied the oven drying behavior of a range of peats and other organic soils over the oven temperature range of 60–110 °C.

A laboratory test program was undertaken to assess the sensitivity of the dry mass of organic soil to increasing oven-drying temperatures. A new method that uses the same apparatus of the standard oven drying method (ASTM D2216) is presented for organic soil to identify the precise oven drying temperature for which the mass of the solid particles, and hence the water content, are accurately measured.

Experimental method

The test soil was organic sludge from a wastewater treatment plant. Some index properties of the sludge material are given in Table 1. The high plasticity of the test soil can be explained by the high organic content of approximately 70 % dry mass, which was measured indirectly using ignition tests (ASTM Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils, D2974). X-ray diffraction analysis indicated that the mineralogy of the inorganic clay fraction comprised quartz, calcite and kaolinite.

TABLE 1—Some index properties of the test soil (O’Kelly 2005).

Property	Value
Liquid limit	315 %
Plastic limit	55 %
Plasticity index	260
Shrinkage limit	14 %
Specific gravity of solids	1.55
Particle size distribution	90 % passing 425 µm sieve
Loss in dry mass on ignition	70 %

Drying tests were conducted on wafer-thin specimens of the sludge material. The wet masses of the specimens were such that each had an oven dry mass of at least 5 g. The equilibrium dry masses of the specimens were determined for different oven drying temperatures, commencing at 60 °C, and increasing in seven temperature steps, up to a maximum value of 150 °C. The drying

oven was thermostatically controlled such that the uniformity of the oven-chamber temperature remained within 1.5 °C of the set temperature value, which was measured using an alcohol thermometer. The dry masses of the specimens were generally recorded at four-hour intervals to determine when the specimen masses had equilibrated for a given drying temperature. Hence, the organic soil fraction experienced the full effects of potential oxidation. The specimens were allowed to cool in a desiccator container to ambient laboratory temperature before the specimens were weighed. The specimen masses were measured to 0.01 g, the same precision as used for the standard oven-drying method (ASTM D2216).

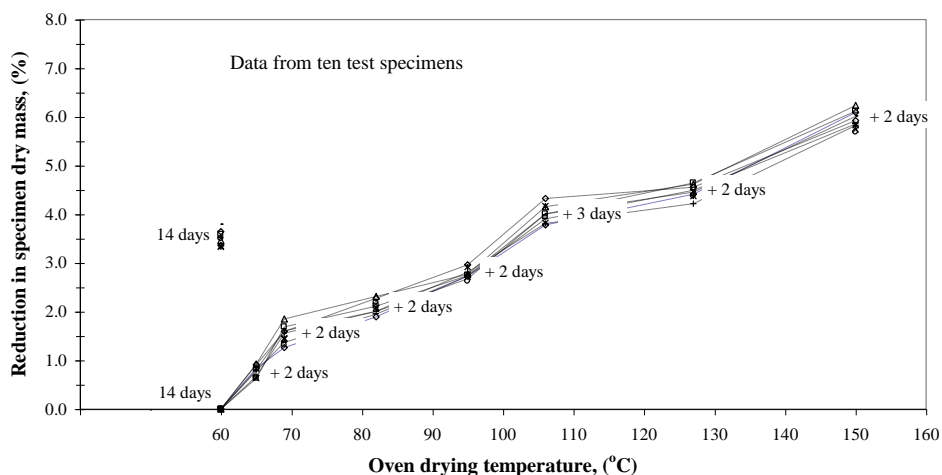
Distilled water was added to the sample drying-containers to partially submerge the specimens after recording the dry masses of the specimens for an oven temperature of 150 °C. The wet specimens were allowed to stand for seven days. Full re-saturation was possible using wafer-thin specimens. The dry masses of the specimens were determined for a second time using an oven drying temperature of 60 °C. The drying tests commenced using an oven temperature of 60 °C as this is the lower end of the drying temperature range reported in the literature for water content determinations on organic soils. ASTM D2216 also recommends using this oven temperature for water content determinations on organic soils.

The test results are presented as the reduction in the specimen dry masses versus oven drying temperature, (Fig. 1). The reductions in the specimen dry masses were expressed as a percentage of the initial dry masses recorded for 60 °C ($m_{60^{\circ}C}$) using Eq. 1. The wet specimens required 14 days oven drying at 60°C to fully equilibrate. Between two and three days oven drying were sufficient for the specimens to equilibrate at higher oven temperatures. Specimens dried above 95 °C produced a pungent odor.

$$\left(\frac{m_{60^{\circ}C} - m_{t^{\circ}C}}{m_{60^{\circ}C}} \right) \times 100 \quad (\%) \quad (1)$$

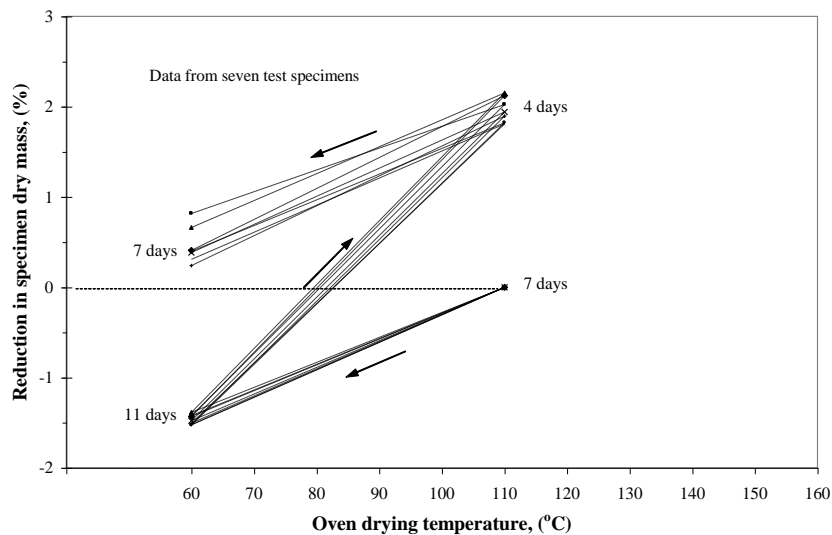
where $m_{t^{\circ}C}$ = the specimen dry mass recorded for oven drying at t °C.

FIG. 1—Reduction in specimen dry mass versus oven drying temperature.



Further tests that involved cycles of oven drying and re-saturation were conducted to show that the removal of pore water from the void space was reversible. The test soil was organic sludge material from the same source but which had been biodegraded to a higher degree. The mineralogy of the inorganic clay fraction was the same but the organic content of approximately 50 % dry mass (ASTM D2974) was significantly lower. Seven specimens of the wet sludge material were initially oven dried at 110 °C for seven days, and the equilibrium dry masses were measured to 0.01 g. Then distilled water was added to the sample drying containers and, over the next ten days, the specimens were allowed to fully re-saturate. The wet specimens were reheated again and the equilibrium oven dry masses were determined, first after oven drying at 60 °C over eleven days, and then after oven drying at 110 °C over another four days. The re-saturation and drying cycle was repeated on the specimens at the end of which the equilibrium dry masses of the specimens were recorded after seven days oven drying at 60 °C. The results of the drying and re-saturation cycles, presented as the reductions in the specimen dry masses (Eq. 1) versus oven drying temperature are shown in Fig. 2.

FIG. 2—Cycles of oven drying and re-saturation.



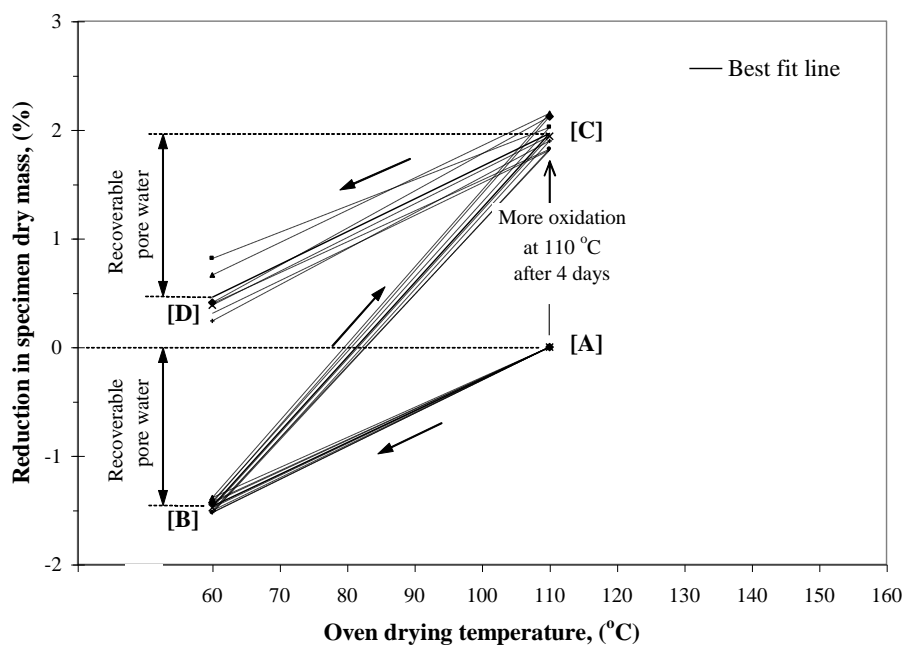
Analysis and Discussion

Given that complete evaporation of the pore water occurs at 100 °C, the following are concluded from a closer inspection of Fig. 3, in which [A] to [D] refer to data labels:

- i) The reduction between the initial [A] and final [C] specimen dry masses at 110 °C occurred due to some further oxidation only.
- ii) Equal recoveries in the specimen dry masses for oven drying at 110 °C and then at 60 °C, over the drying cycles [A] to [B], and [C] to [D], indicate that the removal of pore water was fully recoverable.

- iii) Residual pore water accounted for 1.5 % of the specimen dry masses for oven drying at 60 °C.
- iv) Equal differences in the specimen dry masses recorded at 60 °C and at 110 °C for the first and second drying cycles indicate that no oxidation occurred for oven drying at 60 °C.

FIG. 3—Removal of pore water is reversible.

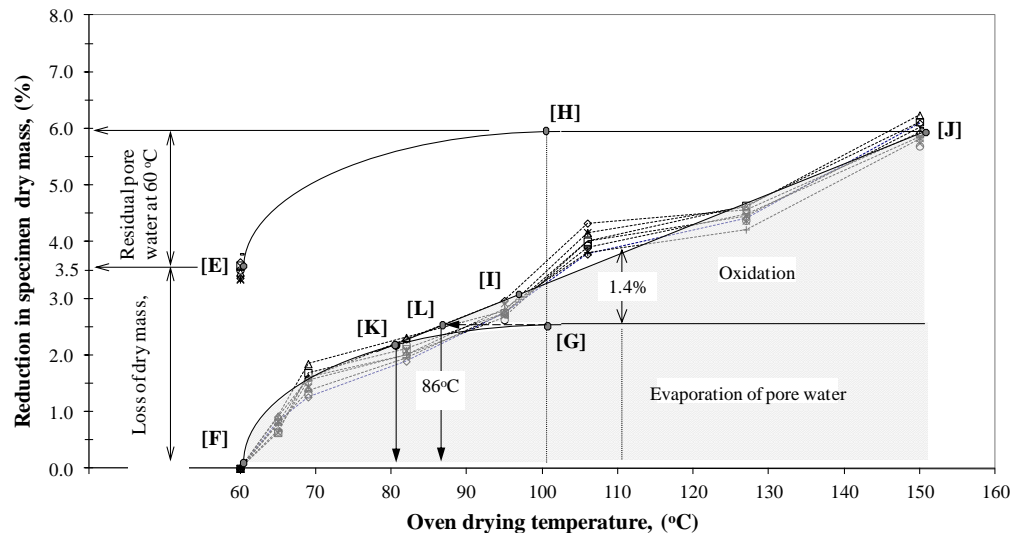


Under certain circumstances, full re-saturation may not be possible for other test soils, with the degree of resaturation achievable depending on the clay mineralogy. Other phenomenon, such as the permanent removal of inter-sheet water in for example halloysite 10Å (Mitchell, 1993), or the water of hydration in for example gypsum, can occur.

Given that the removal of pore water was fully reversible for the test soil, the following are concluded from a closer inspection of Fig. 4, in which [E] to [L] refer to data labels:

- v) A 6.0 % reduction in the specimen dry masses occurred overall on drying the specimens at an oven temperature of 150 °C. An apparent 2.5 % dry mass was recovered on reheating the specimens for the second time at the lower oven temperature of 60°C [E].
- vi) The dry mass recovery was due to small amounts of pore water, which had previously evaporated at 150 °C [J], remaining in the specimens on reheating at 60 °C.
- vii) The difference between the initial [F] and final [E] specimen dry masses of 3.5 % was due to some oxidation of the organic fraction that occurred at higher oven temperatures.

FIG. 4—Construction of empirical drying curves.



Empirical drying curves were fitted to the data. Drying curves for the initial and re-saturated specimens were fitted over the oven temperature range of 60–100 °C. [E] and [F] were known start, and [H] and [G] were known end points for the hyperbolic drying curves. A best-fit oxidation line, constructed tangential to the lower drying curve, was drawn through the data for oven temperatures of 95–150 °C, corresponding to [I] and [J], respectively. The oven temperature corresponding to the tangent point [K] was the temperature at which oxidation of the susceptible organic fraction largely began. This occurred for an oven temperature of approximately 80 °C was supported by the fact that oven drying of the test soil only produced a pungent odor for higher oven temperatures. Other researchers, for example MacFarlane and Allen (1963), also reported that some oxidation of other organic soils began for oven drying temperatures between 80–85 °C. The data recorded for oven drying at 105 °C deviated slightly above the best-fit oxidation line since the specimens were first subjected to the latent heat of vaporization of water at this test temperature.

The hatched regions in Fig. 4 indicate the relative significance of the evaporation and oxidation processes, which were responsible for the reductions in the specimen dry masses with increasing oven temperature. The oven drying temperature [L] at which the mass of the pore water remaining in the void space exactly balances the mass loss of the solid particles corresponds to the intersection of the oxidation line and the horizontal line representing complete evaporation of the pore water. The total mass of the solid particles prior to heating equals the specimen dry mass recorded for this oven temperature of approximately 86 °C. The true value of the water content is calculated on the basis of the specimen dry mass measured for this oven temperature. A marginal loss of only 1.4 % in the dry mass of the solid particles occurred due to oxidation at the standard drying temperature of 110 ± 5 °C.

Note that residual pore water accounted for 1.5 and 2.5 % of the specimen dry masses when the test soils of 50 and 70 % organic contents, respectively, were oven dried at 60 °C. Skempton and Petley (1970) reported that pore water, which remained in a peat specimen after oven drying

at 60 °C for 24 hours, had accounted for 4.2 % of the specimen dry mass. Hence, this would suggest that the amount of residual pore water remaining in the test specimens was a function of the organic content.

Determination of water content of test soil

The reduction in the dry mass due to oxidation of the solid particles at the standard oven drying temperature of 110 ± 5 °C was only 1.4 % for the test soil. A larger error would arise if the water content calculations were based on the specimen dry mass recorded for oven drying at 60 °C, as recommended for organic soils. For example, consider the wet test soil with a water content value of 85.0 %, calculated on the basis of a specimen dry mass ($m_{60^\circ C}$) of 5.00 g, recorded for an oven drying temperature of 60 °C. The apparent mass of the pore water of 4.25 g in the wet soil is calculated as the specimen dry mass times the water content value. A reduction of 3.9 % (Fig. 4), equivalent to 0.20 g, in the specimen dry mass would occur due to oxidation at the standard oven drying temperature of 110 °C. A higher water content value of 92.7 % would arise from the relative reduction in the specimen dry mass to 4.80 g ($m_{110^\circ C}$), and apparent mass of pore water in the wet soil of 4.45 g. The true value for the water content of 89.9 % is determined on the basis of the 2.5 % (Fig. 4), equivalent to 0.13 g, relative reduction in the specimen dry mass ($m_{86^\circ C}$) for oven drying at 86 °C. Skempton and Petley (1970) reported similar findings for oven drying tests on peat and other organic soils. In addition, longer drying times were required for the specimen dry mass to equilibrate when lower oven drying temperatures were used, making it more inconvenient from a practical standpoint.

Conclusions

A simple and practical method that uses the same apparatus as the standard oven drying method was presented to identify the precise oven drying temperature for which the mass of the dry solids, and hence the true water content, are accurately measured for organic soils. The new method uses a curve fitting technique to assess the contributions of evaporation and oxidation in reducing the equilibrium dry mass of the test specimen with increasing oven temperature. Cycles of oven drying and re-saturation on organic soil (50 % organic content) indicated that the removal of pore water was fully reversible, which is key to the accuracy of the method. Some oxidation occurred for oven drying above 80 °C. A 1.4 % loss in the mass of the dry solids occurred at the standard drying temperature of 110 ± 5 °C. The small amounts of residual pore water at lower drying temperatures were a function of the soil organic content.

The true value of the water content was calculated on the basis of the specimen dry mass recorded for an oven drying temperature of 86 °C, at which the mass of residual pore water exactly balanced the loss in the mass of the solids due to oxidation. There was no real advantage gained in determining the water content of the test soil on the basis of the specimen dry mass recorded for oven temperatures of 60–80 °C, rather than the standard drying temperature of 110 ± 5 °C. In fact a larger error arose when the water content of the test soil was calculated on the basis of the specimen dry mass recorded for oven drying at 60 °C. Longer drying times were also required for the specimen mass to equilibrate when lower oven drying temperatures were used, making it more inconvenient from a practical standpoint.

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