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A method for retrospectively calculating the water content for silt-dominated desiccated core samples

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Introduction

When dating sediments by luminescence the water content is one of the most important values needed for age estimation and often places the largest uncertainty on dose rate calculation. If sediments are sampled in situ it is a straightforward process to measure their present day water content directly, although more consideration is required to assess what this figure would have been over geological time. For example, hydrological conditions may have experienced important changes in the past, as is often the case of fluvial sediments (Tanaka et al., 1997). This paper addresses the problem of retrospectively estimating the water content of core samples, and describes the procedure applied to a sedimentary core that was completely dry prior to sampling undertaken for luminescence dating. Following desiccation, the coarser, more sandy sediments are prone to disaggregation and so are unsuitable for luminescence dating as it is likely that many of the grains will have been exposed to light. Sampling was therefore restricted to the consolidated silty sediments where it was clear that the inner parts of the core had not been exposed to light subsequent to extraction. As the samples remained below the water table throughout burial it was assumed that all pore space in the sediment was originally filled with water. As the water evaporated during storage the sediment contracted and calculating the amount of this shrinkage is a relatively simple procedure. The amount of shrinkage can then be converted to pore space available for filling by water (Aitken, 1998, p. 82), but this does not take account of the pore space still remaining in the sample and which is also required to make an accurate assessment of original water content. This can be obtained by measuring the amount of water required to saturate a known volume of sample, but as the sediment under consideration very quickly disaggregated in water, it made a reliable measurement impossible. Instead, an alternative method to identify pore space volume, and subsequently water content, was used and is described here. The results were then evaluated by applying to fresh core samples and comparing the results with the conventional method for calculating water content.

Experimental procedure

A sedimentary core, drilled four years earlier in Azzano, northeastern Italy, was sampled for luminescence dating in 2006. The core was 260 m long, with the top lying only 9 m above sea level (a.s.l.), and composed of sand, silt and clay sediments, of which only the consolidated silty sediments were sampled (Fig. 1). As it had been exposed to air during storage almost all the water originally present had evaporated.

Figure 1: *Azzano Decimo core, samples ADC18 and 19. Samples were between 8 and 20 cm in length, and only taken from those parts that remained completely consolidated.*

When measuring the water content of fresh samples, they are usually weighed before and after oven drying, and water content (w) is normally expressed as % by weight using the equation

$$w = \frac{M_w - M_d}{M_d} \text{ x } 100, \qquad (1)$$

where M_w and M_d are the weights of the wet sediment and dry sediment respectively (terms used in this paper are listed in the Appendix). In order to identify an upper limit for water content, the fraction of the total volume that can be occupied by water, the pore space volume (V_p), is required (Aitken, 1998, p 63); this is a simple task when the original bulk volume (V) of the sample is known, and can be calculated using the equation

$$V_p = V - \frac{M_d}{\rho_d} \qquad (2)$$

where ρ_d is the density of the particles within the sediment. Water content can then be calculated by modifying Equation 1 to read

$$w = \frac{\left(\frac{V_p}{\rho_w}\right)}{M_d} \times 100, \qquad (3)$$

where ρ_w is the density of water. As the core samples had remained saturated during burial it was the original pore space volume of the sediment that was required to identify water content, and this required a little more effort to establish their original bulk volume; the samples were taken from one half of the core and it could not be assumed they had been cut exactly in half. In order to identify the original water content, two unknown values were required; the pore space volume of the completely dry samples ($V_p(d)$), together with the amount of shrinkage that the samples had undergone. To determine $V_p(d)$, pieces between 5-18 cm^3 of each sample were cut and, to ensure that only sediment grains and pore space were present, samples were first air dried, and then oven-dried overnight at 105°C. The samples were then weighed (M_d) and their bulk volume (V_d) determined using mercury porosimetry (Rootare, 1970). As a non-wetting liquid, mercury will not penetrate the sediment under ambient pressure and instead, forms an envelope around the material to be measured. Samples were submerged in mercury, and measurement of the displaced liquid was used to calculate V_d. The density of the grains (ρ_d) making up individual samples was measured on additional portions of disaggregated oven-dried sediment, using a gas pycnometer, after which present pore space ($V_p(d)$) of the sample was calculated using the equation

$$V_p(d) = V_d - \left(\frac{M_d}{\rho_d}\right). \qquad (4)$$

Determination of the amount of shrinkage that had occurred was conducted on the bulk samples (Fig. 1) following oven drying. The entire length of the core had been split in half and samples taken from one half. The present diameter of the samples (d_d) and of the coring equipment (d_o) was identified, and used to estimate the original bulk volume (V) using the equation

$$V = V_d\left(\frac{d_o^{\,3}}{d_d^{\,3}}\right). \qquad (5)$$

The linear values for diameter were cubed ($d_d^3$and d_o^3) to ensure that the ratio between them reflected a change in volume, rather than just length which would result in an underestimation. This assumes that shrinkage occurs to the same magnitude in all dimensions which may not exactly be the case, but was considered the most appropriate approach when the sample length was often not too much longer than its diameter, as illustrated in Fig. 1. To identify the original pore space volume (V_p), a similar correction was also made to present pore space ($V_p(d)$) of the sample using the equation

$$V_p = V_p(d)\left(\frac{d_o^{\,3}}{d_d^{\,3}}\right), \qquad (6)$$

after which Equation 3 could then be solved to identify the original water content (as % by weight).

Testing the method

In order to check the validity of the pore space volume approach in evaluating water content, samples were taken from three further cores. One was 41 m long and drilled one year earlier from Lake Fimon (FM1-12), northeastern Italy, and had subsequently been stored in a plastic liner. This sediment was higher in both organic and clay components than the Azzano samples. Two more, 12 and 40 m long cores, were taken from Niederweningen, northern Switzerland (NWG1/1-11 and NWG2/1-12) and were sampled shortly after coring. While most of these were from silty sediments, they also included some more sandy samples as well. Once again only the consolidated sediments were sampled. While it is possible that each of the cores may have lost some moisture following storage, they were considered to be in a similar condition to those samples taken normally for luminescence from fresh exposures. Two sets of samples were taken from each of the cores. Water content was determined on one set using the conventional method where samples were weighed

before and after drying at 105°C (w_o). The second set were air dried and then oven-dried overnight at 105°C to simulate the Azzano samples; water content was determined using mercury porosimetry (w_p) and shrinkage of the samples was also measured. Particle density was only measured for all samples in NWG1 (n = 12) and averaged 2.62 g cm^{-3}. This value was applied to the samples that were more sandy at the top of NWG2, but for the lower silt/clay samples a value of 2.75 g cm^{-3} was applied; this was the average value for the original Azzano samples to which the fine sediments of NWG2 were far more similar. For the Fimon samples, a value of 2.65 g cm^{-3} was applied as, although these were very silty, they were known to also contain an organic component.

Water content (w_{LOI}) of the Fimon core, at 25-50 cm intervals, had already been measured prior to loss on ignition (LOI) measurements. While this is done in exactly the same way as the conventional oven dry method, samples for LOI are approximately 1 cm^3, and so between 5 and 18 times smaller than those used for the former method.

As core samples may often remain exposed to air for several hours when first opened and during cataloguing of the samples, the effect of this on measured water content was investigated. Three different sizes of fresh sample (NWG2/6) were cut and weighed, with the smallest sample being similar in size to those taken for w_{LOI}. They were then weighed intermittently as they stood exposed in the luminescence laboratory at room temperature over the space of 3 hours; these measurements were used to identify water content as a function of time.

Results

Fig. 2(a) compares the water content calculated using both the oven dry and pore space volume methods for NWG1, 2 and Fimon. NWG1 and 2 record water contents of up to 30 % using the w_o, and values calculated using w_p are within 20 % of these, which would result in a variation in age of ± 4%. The Fimon samples were all higher in water content than both NWG1 and 2, and w_p underestimates the oven dry method by more than 40 % in some cases, which would lead to much larger age underestimations. For those more sandy samples that disaggregated on drying it was not possible to derive water content values using w_p. Fig. 2(b) compares the water content calculated for the Fimon samples using w_{LOI} and w_o values. These show a systematic underestimation using the w_{LOI} measurements although for almost all samples, this is no more than 10% below that derived by w_o.

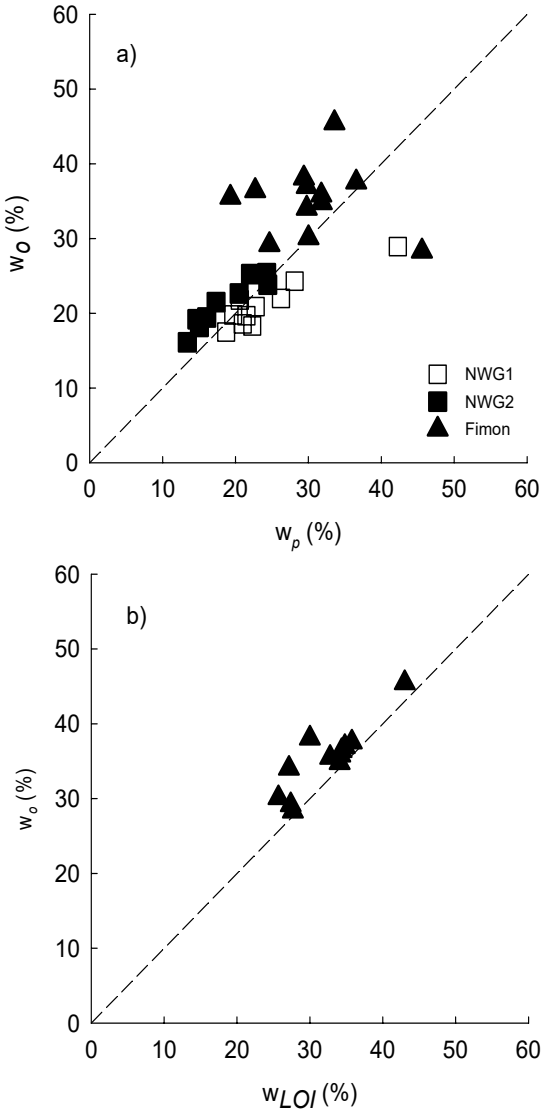

Figure 2: *(a)Comparison of pore space volume (w_p) and conventional oven dry(w_o) methods used to identify water content (expressed as % by weight). (b) Comparison of oven dry (w_o) and preliminary LOI (w_{LOI}) measurements made to determine water content for the Fimon samples. Dashed lines signify unity.*

The measured water content of samples as a function of time while exposed in the laboratory is plotted in Fig. 3; this shows that the smallest samples, which have the largest surface area relative to volume, exhibit the greatest loss of water. The largest piece, with a volume of approximately 32 cm^3 and a surface area of around 60 cm^2, lost 19% of its water content after 3 hours. The smallest piece with a volume of approximately 1 cm^3 and 6 cm^2 surface area, had lost 80% of its water content in the same time.

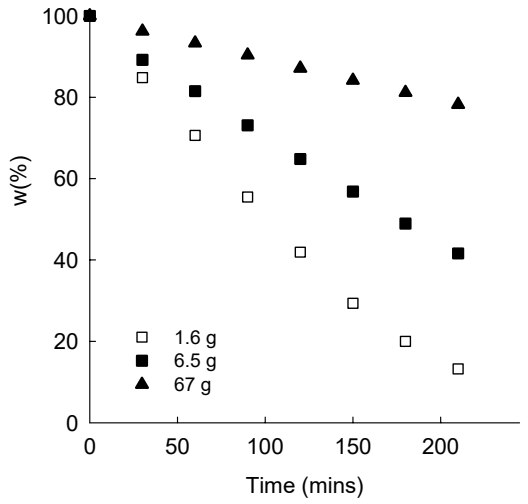

Figure 3: *Water content as a function of time during exposure at room temperature in the luminescence laboratory. This shows a correlation between relative surface area and moisture loss, with the greatest loss occurring in the smallest sample.*

Discussion

NWG1 and 2 samples show a good correlation between the two methods and suggest that the pore space volume method of calculating water content works well for these samples, while the Fimon samples on the other hand, show far more spread in the values. This is believed to stem primarily from the difficulty in measuring shrinkage of those samples that were less 'blocky' and subsequently distorted on drying, and also highlights that it is the reliable measurement of shrinkage on which estimating original water content relies. For example, NWG1/4 shrank from 4.0 cm to 3.7 cm, and a water content of 26% was subsequently calculated. Shrinkage of 0.1 cm less would produce a dry water content value of 21 %, and go on to produce a ~5 % lower age. The Azzano samples appear to have retained their original shape well and shrunk in a uniform manner. These samples remained undisturbed while they dried slowly and without heat, and their much larger volume (some pieces up to 20 cm long) led to a much more regular contraction. The possibility that cubing a linear measurement of shrinkage to convert it to volume may overestimate the amount of shrinkage, has already been mentioned, although it appears to have been appropriate in this study. It may be unrealistic to assume cores are always cut exactly in half, and if this were the case then it is not possible to estimate the true diameter of dry samples. For this reason a more accurate method of measuring the shrinkage was to lay each piece on circular templates of varying diameters, which provided a more reliable measurement and also confirmed that they had contracted uniformly. The original core was 8.89 cm in diameter and all samples were found to have shrunk to between 8.2-8.4 cm; an indication of the very similar sediments that were sampled throughout the core.

The need to determine individual particle densities for all samples was considered. The Azzano samples had particle densities between 2.65 and 2.85 g cm^{-3}, and applying a mean value of 2.65 g cm^{-3} only altered age calculations by ± 2 %. Although this figure is small, the value increases as sediment becomes finer and particle density increases suggesting that its actual determination may be preferable. It should also be mentioned that the comparison of the texture and appearance of unmeasured samples with a reference set of core sediments with a range of known particle densities, proved successful in the estimation of appropriate values.

It is important to be aware of possible moisture loss that may have occurred before water content measurements are made. Although the influence of compaction during coring needs to be considered for modern soft sediments (Zheng et al., 2002), the effect on the samples in this study, should be no more than that applicable to any sediments taken using a sampling cylinder. It has also been suggested that a small amount of water may be lost during core retrieval and extraction (Forman et al., 2007). A new core may also be exposed for several hours on first opening and cataloguing, and this can result in further water loss. While neither of these losses would influence water content estimation obtained using the pore space volume method, the oven dry method would be unable to identify the original water content.

Conclusions

While immediate measurement of fresh samples remains preferable, measurement of porosity offers a useful alternative in retrospectively estimating the water content of core sediment. Determining the present day pore space volume is simple, and quick to achieve, but using this to calculate the in situ water content is only possible when the subsequent shrinkage to samples can be accurately assessed. This is most likely where samples have remained undisturbed and dried slowly, resulting in a uniform shrinkage. It is highly unlikely that a core has been cut exactly in half, and so matching the curvature of cores to templates, rather than measuring the diameter, will provide more reliable results and also identify any distortion that may have occurred during drying.

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Appendix

d_d	Diameter of dried core sample (cm)
d_o	Diameter of coring equipment (cm)
M_d	Weight of dry sediment (g)
M_w	Weight of wet sediment (g)
ρ_d	Density of sediment grains (g cm^{-3})
ρ_w	Density of water (g cm^{-3})
V	Original bulk volume (cm^3)
V_d	Bulk volume of dried sample (cm^3)
$V_p(d)$	Pore space volume of dried sample (cm^3)
V_p	Original pore space volume (cm^3)
w	Water content (% by weight)
w_{LOI}	Water content determined prior to LOI measurements (samples ~ 1 cm^3)
w_o	Water content determined using weight of samples before and after drying oven drying at 105°C (% by weight)
w_p	Water content determined using mercury porosimetry following air drying and oven drying at 105°C (% by weight)

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Reviewer

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