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Standardization activity in the evaluation of moisture content

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ABSTRACT

This review paper comments the international standards to measure the moisture content in building materials, i.e. EN 772-10:1999; EN 13183-1:2002; EN 13183-2:2002; EN 13183-3:2005; EN 1428:2012; EN-ISO 11461: 2014; EN-ISO 15512:2014; ISO 11465:1993; ISO 12570: 2013; ISO 16979:2003 and ISO 760:1978. The above standards do apply to new building materials, with standardized composition and shape, in satisfactory state of conservation, without sampling restrictions. If they are applied to aged and deteriorated materials, as in the field of cultural heritage, the results may be misleading. The paper discusses the difference between 'moisture content' and 'water content' and the various problems met with cultural heritage materials, e.g. ethical problem when test specimens are needed; the biased response when wood was attacked by moulds or insect tunnelling, or was impregnated with oil, wax or preservatives; or when masonry contains soluble salts or subsurface discontinuities. The most recent, comprehensive standard is presented, i.e. EN16682 (2017) '*Conservation of cultural heritage – Methods of measurement of moisture content, or water content, in materials constituting immovable cultural heritage*' that considers all existing methods and discusses pros and cons of each of them in relation with the real world of aged and deteriorated materials.

1. Introduction

This review paper will introduce the topic of Modhima and comment the European and international standards to measure the moisture content in building materials. They have been produced by the European Committee for Standardization (CEN), that is an association that brings together the National Standardization Bodies of 34 European countries and by the International Organization for Standardization (ISO), that is a worldwide federation of national standards bodies (ISO member bodies). CEN standards are abbreviated EN owing for European Norms. The work of preparing ISO or EN Standards is normally carried out through Technical Committees with specific competence on the topic. A number of standards produced by ISO have been adopted by CEN and appear under the two headings EN-ISO. Some specific standards have been produced in association between ISO and CEN.

The EN standards [1–5], the ISO standards [6–10], and the EN-ISO standards [11,12] provide a precise operative protocol and describe the instrumentation needed for some selected methods to measure the moisture content. Each standard deals with only one method that is specified in the title e.g. gravimetric determination, chemical titration, azeotropic distillation, electrical resistance measurement, capacitance measurement, etc. The standardization by single subject may be confusing because all standards, and therefore all methods, are apparently at the same level, and the user lacks a comprehensive framework to assist him in the choice of the most convenient one for every particular case study. In addition, not all the existing methods have been standardized.

In this review, the standards [1–12] for normal materials will be considered and commented for uncertainties or inappropriate use, i.e. when the test material is deteriorated. The difficulties, and the misleading results, that the user may have if a standard for normal materials [1–12] is applied to cultural heritage materials will be also considered.

In 2017, CEN, Technical Committee 346, has produced a comprehensive standard, EN 16682 [13] specific for cultural heritage, but usable for most materials, which constitutes an authoritative guideline to choose and use all existing methods and advices about misuse and misinterpretation of readings. This paper will outline the benefits that the user may gain from this holistic standard as well as some outstanding problems.

In the following, for the sake of clarity, when standards are introduced, they will be indicated with their number, full title and bibliographic reference.

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2. Materials and methods, with discussion

The various methods are here shortly presented, followed by a comment about potential flaws in the case of misuse, e.g. application to non-recommended materials, or materials that have been altered over time, or are in bad condition of conservation.

2.1. Gravimetric method

A number of EN and ISO standards concern the so-called gravimetric method. It consists in determining the mass of a moist test specimen of the material, drying it to constant mass, determining the new mass, calculating the ratio of the loss in mass during drying to the mass after it has been dried . The result should be expressed in % of the dry test specimen.

The following standards refer to it:

- EN 13183-1. *Moisture content of a piece of sawn timber – Part 1: Determination by oven dry method* [1].
- EN 772-10. *Methods of test for masonry units – Part 10: Determination of moisture content of calcium silicate and autoclaved aerated concrete units* [5].
- ISO 11465. *Soil quality – Determination of dry matter and water content on a mass basis – Gravimetric method* [6].
- ISO 12570. *Hygrothermal performance of building materials and products – Determination of moisture content by drying at elevated temperature* [7].
- ISO 12571. *Hygrothermal performance of building materials and products – Determination of hygroscopic sorption properties* [8].
- ISO 16979. *Wood-based panels – Determination of moisture content* [9].
- EN-ISO 11461. *Soil quality – Determination of soil water content as a volume fraction using coring sleeves – Gravimetric method* [11].
- EN 16682. *Conservation of cultural heritage – Methods of measurement of moisture content, or water content, in materials constituting immovable cultural heritage* [13].

Discussion. The gravimetric method is very popular, accurate and absolute. 'Absolute' means a method whose readings can be expressed in terms of the International System of units (SI), i.e. grams, corresponding to the weight of the moist and dry specimen. When the reading is expressed in %, the SI formally disappear, but they have been essential in determining the final reading.

The standards [1,5–9,11] describe how to proceed, but they do not establish whether this method should be preferable to others. As opposed, the American standard ASTM D4442 '*Standard Test Methods for Direct Moisture Content Measurement of Wood and Wood-Based Materials*' [14] specifies that the gravimetric method should be considered "*primary*" method, and others should be considered "*secondary*" ones. Similarly, EN 16682 [13] establishes priorities in methods, and establishes that the gravimetric method and the Karl Fisher Titration should be used as primary reference.

In the gravimetric method, sampling is made on the site, but weighing and drying are performed in the laboratory. In the field of cultural heritage, sampling constitutes an ethical problem because it is a destructive methodology that should not be applied, except for exceptional, well justified cases, that should be under the supervision and responsibility of the competent authority. A practical problem with cultural heritage materials is the lack of homogeneity in the material, and specimens taken in different parts may provide different results and it is not possible to take benefit of the average of several specimens to avoid sampling damage. In particular, in order to minimize this damage, specimens are generally extracted from less representative parts, e.g. near the borders, in already damaged areas, or their depth is limited to a surface layer. The risk for scarce representativeness cannot be avoided increasing the number and dimension of the test specimens, as discussed.

This method is simple and objective. It may provide reliable results, except when the material includes components not resistant to oven temperature. For instance, if the material has been treated with, or includes oils, waxes, resins, or other substances that at oven temperature become volatile organic compounds (VOC) and are outgassed, the mass difference of the specimen after drying is due to the sum of the two contributions, i.e. water and VOC. Therefore, the gravimetric oven drying method overestimates the moisture content when the test specimen includes substances that may be outgassed. In any case, the official definition of "*moisture content*" is based on this method and is potentially affected by this flaw.

In order to avoid this bias, ISO 12571 [8] specifies two alternative methods for determining hygroscopic sorption properties of porous building materials: (i) using desiccators and weighing cups (desiccator method), considered the reference method; (ii) using a climatic chamber (climatic chamber method). The latest standard EN 16682 [13], produced in 2017, offers a wider choice of drying alternatives: (i) ventilated oven for heat resistant materials, thermostable up to $(103 \pm 2)^{\circ}$C; (ii) vacuum drying for materials not resistant to oven temperature, but resistant to low pressure; (iii) desiccant drying for materials neither resistant to oven temperatures nor resistant to low pressures; (iv) compressed air drying for materials neither resistant to oven temperatures nor resistant to low pressures; (v) adsorption drying for materials neither resistant to oven temperatures nor resistant to low pressures.

In wood, the gravimetric method is unaffected by pest attack, including insect tunnelling; in masonry, by the presence of salts.

2.2. Karl Fischer Titration

A chemical method, i.e. the Karl Fischer Titration (KFT) is specific to detect water in material test specimens that should be crushed to a fine powder. KFT can be performed in two basic modes. (i) Volumetric mode, where the water content of the material sample is measured with an iodine solution. This mode determines the volume of water extracted from the specimen, considering that its mass expressed in g equals its volume in cm^3. (ii) Coulometric mode, where the water content is measured with electrolysis. This mode determines the mass of water extracted from the specimen from the number of electric charges used in the electrolysis. The former mode is more robust, the latter requires smaller specimens. In both cases, the mass of the moist test specimen should be previously determined with a precision balance.

Reference should be made to the following standards

- ISO 760. *Determination of water – Karl Fischer method (General method)* [10].
- EN-ISO 15512. *Plastics – Determination of water content* [12].
- EN 16682. *Conservation of cultural heritage – Methods of measurement of moisture content, or water content, in materials constituting immovable cultural heritage* [13].

Discussion. KFT is an accurate and absolute method. EN 16682 [13] establishes that it can be used as a primary reference in addition to the gravimetric method. In the case of cultural heritage materials, KFT is limited by the ethical problem of sampling, like the gravimetric method.

In the standards [10,12], the KFT is expressed as water content of the test specimen, divided by the mass of the moist specimen. However, it is possible to convert from the moist specimen mode to the dry mode, as for the gravimetric oven drying method, and EN 16682 [13] establishes that the readings should be converted to dry, and provides the conversion formula.

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