



Use of electrical resistance measurement to assess the water saturation profile in porous limestones during capillary imbibition



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HIGHLIGHTS

- Development of a new method for estimation of water content in building stones.
- Relationship between electrical resistance and degree of saturation in stones.
- Profile of degree of saturation in porous stones during capillary imbibition.
- Imbibition curve of stones determined from electrical resistance measurement.

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ABSTRACT

Electrical resistance can be a relevant indicator for the assessment of water transfer in porous materials. The aim of this work was to establish a relationship between electrical resistance and water saturation in the case of two limestones used as building stones in the Château de Chambord in France: tuffeau and Richemont stone. For this purpose, firstly the electrical resistance matching different values of degree of saturation was investigated. Experimental measurements were performed to determine a relationship between the electrical resistance and the degree of water saturation, with high sensitivity. Secondly, using this relationship, the capillary imbibition curves and imbibition coefficients of the two stones were precisely determined by electrical resistance measurement.

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1. Introduction

The decay of built heritage can take different forms. In the case of degradation due to cycles of fatigue by wetting-drying, the building stones are mainly exposed to cracking, generated by mechanical stresses. The initiation and propagation of crack within the stone may result in other types of alteration such as spalling, flaking and scaling [1–4]. Water transfer is considered as the main cause of the stone's decay [5–9], as it can induce physical changes (e.g. changes in mechanical properties, hydraulic dilation [10]), and is a vector of degradation through the transport of pollutants and the dissolution/crystallization of soluble salts [11,12]. The stones the most exposed to degradations are those with a high porosity [13]. The porosity acts as a water tank due to the infiltration of water (capillary imbibition, rising damp, rain, etc.). Clay mineral

components in the stone also increase the water retention capacity and play an important role in the hydraulic behavior [14] and hydro mechanical behavior of the stone, as shown by Cherblanc et al. [15].

It is essential to determine the water content in order to correlate the action of the water in the stone and its degradation [16–18]. There are many methods for measuring water content, but all have drawbacks [19]. These techniques are generally used more in geophysics for the study of soils and less for the study of stone masonry. They can be grouped into direct and indirect methods:

Direct methods are based on determining water content by weight or by chemical analysis or volume measurements. Gravimetric methods consist in weighing the material in the wet (saturated samples) and dry states (samples oven-dried at 105 °C). This method is highly accurate and is used to calibrate indirect methods. Chemical analysis can determine water quantity by numerous standard analytical methods, among which the Karl Fischer method [20] is the most reliable. It consists in adding the

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Notations

ER	Electrical Resistance, in (Ω)	RH	Relative Humidity in (%)
Sr	degree of water saturation, in (%)	DMM	Digital MultiMeter

sample to an iodine or sulfur trioxide reagent in dry methanol and then determining the quantity of water by titration [21]. This method is less accurate than the previous one, however, and is generally applied on powders (usually used for brick), soils or food-stuffs. There are also other techniques such as nuclear magnetic resonance (NMR) and synchrotron measurements. The main disadvantage of these methods is the need for sampling, which is destructive for in-situ masonry studies, and this kind of measurement is available only in laboratory tests.

Indirect methods are based on variation in the physical properties of the material (conductance, resistance, capacitance, etc.) with changes in moisture content. This type of measurement requires a calibration test [22]. A powerful technique for measuring water content is neutron radiography [23,24] which makes a strong contrast between wet and dry areas, but it is complex to use for fieldwork. Gamma-ray and X-ray methods which determine water content by the attenuation of the ray beam can also be used [22,25]. They are very powerful techniques, which can map water content distributions in the sample, but are likewise difficult to use in the field. Capacitive methods are used in soil fieldwork based on the measurement of dielectric properties (complex permittivity) at a given frequency [26,27]. For example, in time domain reflectometry (TDR) an electromagnetic pulse propagates along a wave conductor, then the permittivity is determined by the measured propagation speed of this electromagnetic pulse [28]. While this method is less sensitive to soluble content and temperature, it is difficult to install and is affected by inaccurate installation [28]. Infrared thermography has also been applied to detect the presence of moisture in masonry but this technique can provide only qualitative results, and it is very dependent on the surrounding environment as the temperature and the humidity of the ambient air control the drying rate. Sensors that measure the humidity of the air in equilibrium with the wet building material stone can also be used to measure the water content in masonry. This method is limited by the correct installation of the probes, their measurement range (often limited to RH 20–98%) and their accuracy [29]. Another method called wooden dowels is based on capturing moisture by wooden dowels and then calculating its water content by sampling and weighing. This method is simple but it is invasive and it depends upon the exact location of the dowels and may affect the water movements within the masonry [19]. Electrical resistivity measurements have received more attention and have given valuable results on water movement in porous media such as rock [19,30–32] or concrete [33–38]. This method is based on the variation in electrical resistance (or resistivity) of the system ‘solid porous material + liquid water’ in which the solid porous material has a high electrical resistance and the liquid water has a low electrical resistance. The limit of this method is its sensitivity to the soluble salt content that may be present in the water [28].

To better understand the relationship between water transfer and the degradation of limestone, it is useful to determine the water transfer both in the laboratory and in situ. However, most techniques for assessing water content are destructive, invasive for historic masonry, difficult to install in situ or restricted to the laboratory. The reasons are related to both the material and to the practical manipulation of the techniques themselves, which may be destructive (e.g. sampling) or invasive, their sensitivity,

their accuracy, their complexity for fieldwork, and the negative effect of implanting probes which may influence the measurement environment and can capture more moisture [30]. Thus, only electrical resistance measurement seems to be the most sensitive and the simplest method to use both in the laboratory and on site.

Our objective was to develop an easy to use method to measure the water saturation profile of limestones simultaneously in order to evaluate the water content and the water penetration height in the stone. This study therefore proposes an indirect and non-destructive local measurement of water saturation of porous stones along a range of heights in a sample during capillary imbibition. Two types of limestones, both used for the construction and the restoration of the ‘Château de Chambord’, were investigated: tuffeau and Richemont stone [39].

The method chosen to assess water saturation was the electrical resistance method because of its above-mentioned advantages. Firstly, a relationship between the electrical resistance (ER) and the degree of water saturation (Sr) of the considered material was developed for the calibration curve. Secondly, electrical resistance measurements were carried out during a total imbibition test on the two limestones. The relationship determined by calibration measurement was applied to determine the hydraulic behavior during capillary imbibition by deducing the local degree of saturation of the stones, and hence the position of the water capillary front, using the recorded electrical measurements.

2. Materials

Tuffeau is a siliceous limestone, used as a building stone in the ‘Centre-Val-de-Loire’ region of France. It was widely used for the construction of cultural heritage buildings in the Loire Valley such as the Château de Chambord. This stone has been studied in many previous research investigations [8,29,40]. It is impacted by various weathering processes such as spalling, moss and lichen development, scaling and exfoliation [39]. Tuffeau is composed of a major calcite phase (50%), a high siliceous fraction (40%: opal CT and quartz) and a significant clay content (10%: glauconites, smectites, illite). Fig. 1a presents the Scanning Electron Microscope images (SEM) for the tuffeau. The tuffeau texture is multi-scale and it is possible to differentiate the size of the different minerals and pores. Tuffeau is composed of grains of calcite, quartz, opal, clays (glauconite) and mica. Their sizes are wide-ranging from 10 μm for opal spherules to 100 μm for quartz. Due to these differences in particle types and sizes, tuffeau fabric is heterogenous, with a large pore size distribution. Fig. 2a presents the pore size distribution determined by Mercury Intrusion Porosimetry (MIP). This limestone is very porous with a porosity close to 45% and a bimodal pore distribution. Tuffeau has a wide range of pore diameter sizes, from 0.002 μm to 20 μm , with a mean pore diameter of 1.8 μm and a bi-modal porous network (first peak at 8 μm ; the second at 0.01 μm) [8,29]. The main peak is around the pore diameter of 8 μm , and represents the capillary pores. These large pores with a diameter ranging from 1 μm to 10 μm account for more than half of the investigated pores. The second peak, about 0.01 μm , is due to the rough surface of spherules of opal, and to the clayey minerals. It should be noted that the larger pores observed by SEM (about 100 μm in diameter) were poorly detected in the MIP test, mainly due to the effect of the ‘‘ink bottle’’ phenomenon.

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