



# Coupled thermal–hygric characterisation of elastic behaviour for soft and porous limestone

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## HIGHLIGHTS

- Soft limestone was exposed to  $-8\text{ }^{\circ}\text{C}$  to  $40\text{ }^{\circ}\text{C}$  and 0–100% relative humidity.
- The measurements include strain, elastic modulus and Poisson's ratio.
- The DOE methodology was successfully applied to obtain mathematical relationships.
- Determined elastic properties were compared with elastic properties at dry state.
- Dry state elastic properties are inaccurate to simulate field conditions.

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## ABSTRACT

The experimental work presented in this study was focused on examining the effect of the variation of two parameters: temperature and water saturation, on the elastic modulus, Poisson's ratio, and dilation properties of a limestone used widely in French construction called tuffeau. Experimental results were treated using a design of experiment to assess the coupling effects of the studied parameters, and to create mathematical models used to estimate the measured elastic properties in the range of  $-8\text{ }^{\circ}\text{C}$  to  $40\text{ }^{\circ}\text{C}$  for temperature and 0–100% for water saturation. The elastic properties, generally characterised in the dry state or without real coupling with water saturation, proved to be inaccurate for most environmental conditions, suggesting that the proposed models represent an essential contribution to any further research involving the simulation of outdoor conditions in soft limestone construction.

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## 1. Introduction

Soft and porous limestones have been used in the construction of individual houses, official buildings, churches, cathedrals and castles in France. This stone continues to be used in the replacement and repair of stone construction. Moreover, the aesthetic aspect and low density of porous limestone promotes its use today in wall cladding in order to provide a prestigious exterior wall area for buildings [1]. However, these interesting properties make soft limestone very sensitive to weathering when exposed to outdoor environmental conditions. Different weathering processes can occur such as thermal stress, condensation, freezing, salt crystallisation, and biological colonisation. Accelerated ageing laboratory tests, like salt crystallisation tests and freeze–thaw tests, can be

used as quality standards to enable selection of the appropriate stone as a building material [2]. Moreover, a recent study managed to estimate some stone mechanical properties based on non-destructive testing, thanks to the use of artificial neural networks [3].

Even if the accumulation of temperature and humidity cycles is frequently quoted as a major source of stone degradation in the literature, there is still a need for improving the assessment of the consequences of this weathering process. Recent studies [4–7] focused on investigating the role of the daily variation of temperature in the development of thermal stresses within the stones, considering that the thermal stress depends only on the thermal dilation. Furthermore, the calculation of these stresses was carried out on the assumption that the linear thermal expansion coefficient ( $\alpha$ ) and the mechanical properties of the stone, elastic modulus ( $E$ ) and Poisson's ratio ( $\nu$ ) were considered constant. However, these mechanical properties depend on both the temperature and the degree of water saturation of the stone. Moreover, the constant

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values used in the simplified approach stem from standard characterisation, usually in the dry state, whereas the actual state of soft stones in the field may be very different. Therefore, the accurate calculation of the thermal stresses of stones requires the knowledge of the effect of temperature and degree of water saturation on the stone's mechanical properties ( $E$  and  $\nu$ ).

Stone used in buildings suffers stress for restraining thermal dilation, but there is another significant source of dilation: hygric dilation, due to variation in water content. Hence, the calculation of the stress induced by the daily variation in environmental conditions must take into account not only the effect of the restrained thermal dilation, but also the dilation induced by the coupled effects of the daily variation in temperature and degree of water saturation. The resulting thermal–hygric stress depends on the mechanical properties,  $E$  and  $\nu$  as a function of the temperature and relative humidity of the stone, while the strain results from the free thermal–hygric dilation (liner thermal–hygric expansion coefficient).

Most stones show thermal dilation or/and hygric expansion when subjected to variation in temperature and humidity. These two behaviours strongly depend on the origin of the stone (sedimentary, metamorphic, magmatic), its texture (grain shape, grain size distribution, degree of grain interlocking), and its mineral composition [8,9]. Degradation by thermal dilation is not restricted to the dry stones; on the contrary this degradation can be more severe for wet stones. For example, Koch and Siegesmund [10] pointed out that saturated marbles are more sensitive to thermal dilation degradation than the dry ones. Moreover, the presence of clay minerals is another factor that increase the degradation by thermal dilation or/and hygric expansion.

This research stems from a more global approach concerning the conservation and monitoring of the construction stones at the Chateau of Chambord, France, built in tuffeau, a soft and porous limestone. To date, there is no experimental study concerning the mechanical properties of stone under the coupled effect of temperature and humidity variation. Therefore, to estimate the stresses within the stone there is a need to obtain a mathematical relationship to link the mechanical properties of the stone to both temperature and water saturation.

The objective of this paper is to experimentally examine the effect of variations in both temperature and water saturation on the mechanical and dilation properties of a soft limestone. An experimental plan was designed, and the results were analyzed, statistically tested and finally modelled thanks to the use of a design of experiments (DOE). DOE is a methodology that ensures the quantification of interactions between parameters (here: temperature and water saturation) and allows statistical analysis of the relevance of the resulting model. The methodology of the design of experiments (DOE) was used to rigorously assess the coupling effects and to develop mathematical relationships that can be used to estimate any value of elastic modulus, Poisson's ratio and strain based on the knowledge of temperature and water saturation variations in the stone.

## 2. Material and methods

### 2.1. Stone sample properties

Tuffeau is a soft, porous, clayey, and fine-grained limestone used in the construction of most of the castles in the Loire Valley in France [11]. It is from the Turoonian age, the upper Cretaceous period, approximately 88–92 million years ago. It is a light-weight stone showing a white colour.

The main mineral phases of tuffeau are 50% calcite (sparite, micrite, and marine fossils as coccoliths), 10% quartz, 30% opal (cristobalite–tridymite) and about 10% of clayey minerals and mica, resulting in a micro-porous fabric [11,12]. Previous results showed that tuffeau has negligible close porosity [12]. Similar observations were found for other sedimentary stones [2]. Thus, in this study, the vacuum saturation method was adopted to measure the stone's physical properties; total

porosity, apparent density and skeletal density. The unconfined compressive strength and the tensile strength of the stone in both the dry and saturated states were measured using an Instron 4485 press machine. Table 1 summarises the characterisation of the studied stone for physical and mechanical properties. Fig. 1 presents the water retention curve of tuffeau [11,12]. The variations in the degrees of water saturation of tuffeau resulting from exposure to different conditions of relative humidity are presented. Measurements for water retention curves used three complementary techniques in order to apply different suctions linked to relative humidity thanks to Kelvin's law: saturated salt solutions (RH < 98%), osmotic solutions and tensiometric plates (RH > 98%) [11–13]. The variations of water saturation are very limited for relative humidity up to 76%, while significant increases in water saturation are observed when the relative humidity is close to 100%. In a fully saturated state, tuffeau exhibits high water-holding capacity, since it is characterised by high-fine porosity and contains a significant amount of clayey minerals.

### 2.2. Sample preparation

Tuffeau samples, 40 mm in diameter and 80 mm in height, were cored out perpendicularly to the bedding plane from a larger block. Attention was paid to selecting samples free from visible cracks and flaws.

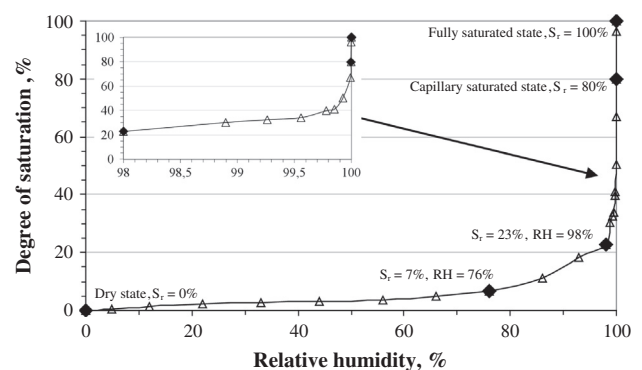
Prior to testing, tuffeau samples were prepared in different conditions of relative humidity (0%, 76%, 98%, 99.99%, and 100%) corresponding to different degrees of water saturation (0%, 7%, 23%, 80% or 100%). These degrees of saturation were chosen to be representative of the water retention curve of tuffeau: dry and fully saturated states, and three other points required to describe the water retention curve (see Fig. 1). The different degrees of water saturation imposed on the tuffeau samples are based on the water retention curve which links the relative humidity (or suction) to the water content (and the degree of saturation). So, three consecutive techniques were used:

#### 2.2.1. Preparing the sample with 7% and 23% of water saturation

The water saturation of 7% and 23% results from exposure at a relative humidity of 76% and 98%, respectively. The preparation of the sample was done in a climatic chamber with controlled temperature and relative humidity. A dry sample was exposed to a temperature of 20 °C and a relative humidity of 76%. The sample was left inside the chamber long enough to reach equilibrium (constant weight). At that time, the dilation of the sample was monitored using the strain meter (see Section 2.3). In fact, the dilation of the sample increased significantly in the first 24 h, and thereafter increased only slightly. Finally, after 96 h the sample showed no further increases in dilation, i.e. the measured strain data were always stabilized, indicating that the sample had already reached equilibrium with the environment inside the climatic chamber. At 76% RH, when the weight is stabilized, the sample

**Table 1**  
Characterisation of the studied stone.

Stone property	Property value
Skeletal density, g/cm <sup>3</sup>	2.57 ± 0.01
Apparent density, g/cm <sup>3</sup>	1.30 ± 0.02
Total porosity, %	45.0 ± 0.52
<i>Unconfined compressive strength, MPa</i>	
Dry state	11.67 ± 0.33
Saturated state	4.83 ± 0.33
<i>Brazilian tensile strength, MPa</i>	
Dry state	1.30 ± 0.11
Saturated state	0.38 ± 0.08



**Fig. 1.** Water retention curve of tuffeau [11,12].

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