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# **ANALYSIS OF SPALLING IN TUFFEAU: CASE STUDY OF THE CASTLES OF CHAMBORD AND CHAUMONT-SUR-LOIRE IN FRANCE**

**Sarah Janvier-Badosa<sup>1\*</sup>, Kévin Beck<sup>2</sup>, Malek Balawi<sup>2</sup>, Marie Ternoy<sup>2</sup>, Romain Janvier<sup>2,3,4</sup>, and Xavier Brunetaud<sup>2</sup>**

## **KEYWORDS**

Spalling, gypsum, limestone, tuffeau, case study

## **ABSTRACT**

The main historic monuments of the Loire Valley in France are built with tuffeau. This very porous and soft siliceous limestone presents many technical and aesthetic advantages justifying its wide use. Among the most damaging degradations of tuffeau, spalling is the most destructive one.

This study aimed at verifying the profiles of gypsum content observed in spalling of tuffeau at the castle of Chambord during a previous study. Here, samples from both the Castle of Chambord and from the Castle of Chaumont-sur-Loire were collected to provide more extensive and thus representative data for monuments made out of tuffeau in the Loire Valley.

For all samples, in both castles, a systematic presence of gypsum was observed in areas subjected to spalling. The gypsum content is always very low at the surface and progressively increases (always around 1%) up to the depth of the zone where the crack is present, and then decreases along depth. No other salt was detected. This study also proved that preexistent cracking is not necessary for gypsum to crystallize. All these statements are totally in accordance with the previous study, hence allowing to confirm its representativeness.

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## 1 INTRODUCTION

In the Loire Valley, in France, most of the historical monuments are built with a locally-extracted stone, tuffeau. It is a soft white fine-grained sedimentary limestone, with high porosity (about 45%). The composition and properties of tuffeau vary slightly from one quarry to another. It contains calcite, quartz, opal-CT, micas and clay minerals [1] [2] [3].

Tuffeau is subject to many forms of degradation. However, among the most common ones in the Loire Valley, the most damaging is spalling (Figure 1). Spalling is the progressive development of a crack parallel to the surface exposed to the environment, generating the detachment of a surface layer [1] [4]. This plate eventually falls, leaving a powdery surface. The morphology of the spalling observed in the constructions in tuffeau in the Loire Valley is generally the same, even if the thickness of the plate can vary from a few millimeters to a few centimeters, with an average value around 1 cm. A study of the distribution of spalling has shown that spalling develops preferentially on smooth walls, near reliefs, in areas exposed to run-off and wind, with moisture supply and quick drying [5].

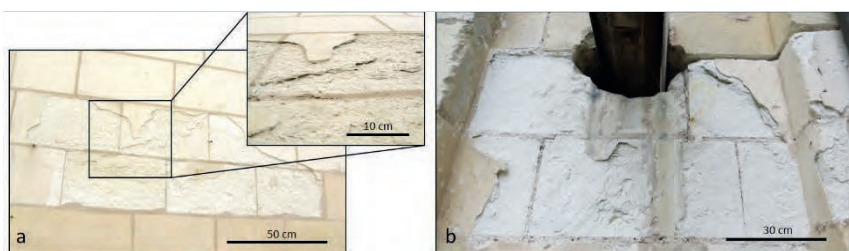


Figure 1: Examples of spalling in tuffeau: (a) castle of Chambord (2012), (b) castle of Chaumont-sur-Loire (2020).

Several studies concerning the spalling of tuffeau were conducted since the 1960s (mainly French case studies, for diagnosis) and they highlight the presence of gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) in the cracking zone. Black crusts and flaking, degradation that contains gypsum, are widely studied, however the phenomenon of spalling has still not been fully explained. A study of the location and the concentration of the gypsum according to the depth was carried out in detail on a spalling zone of the castle of Chambord, presenting different stages of evolution of spalling [6]. This study showed a particular distribution of the gypsum with small amounts of gypsum on the surface and a maximum around 1 cm of depth, most often corresponding to the cracked zone (thickness of the plate); deeper in the stone, the gypsum content decreases with depth. However, this study was only conducted on a single area of stone presenting spalling. In order to progress in the understanding of this degradation phenomenon, this case study proposes to answer several questions: Is this particular profile systematic in spalling, and therefore is it found for different tuffeau on different monuments? Are the profiles of gypsum contents measured in the different areas similar?

## 2 SITES AND MATERIALS

### 2.1 Presentation of the castles of Chambord and Chaumont-sur-Loire, France

Among the historical constructions in tuffeau in the Loire Valley, two prestigious monuments were chosen for this study: the castle of Chambord and the castle of Chaumont-sur-Loire (Figure 2).

The construction of the castle of Chambord started in 1519, on the ruins of a medieval building, by the order of Francis I<sup>st</sup>. Castle of Chambord is the largest castles in the Loire Valley. Different changes were made to the configuration of the monument, mainly during the reign of Louis XIV. These main changes were applied during a relatively short period, even if restorations and maintenances operations were also regularly performed.

The castle of Chaumont-sur-Loire was built in several steps of construction, destruction and improvements. It started during the end of the 15<sup>th</sup> century on the ruins of a medieval fortress. Then, it becomes a pleasure castle, with a typical Renaissance style. Diane de Poitiers, favorite of the King of France Henry II, gave its overall present configuration. During the 18<sup>th</sup> century, the north wing is demolished to open the inner courtyard on the Loire river. Several others buildings can be found in the domain, including the prestigious stables, built during the late 19<sup>th</sup> century.

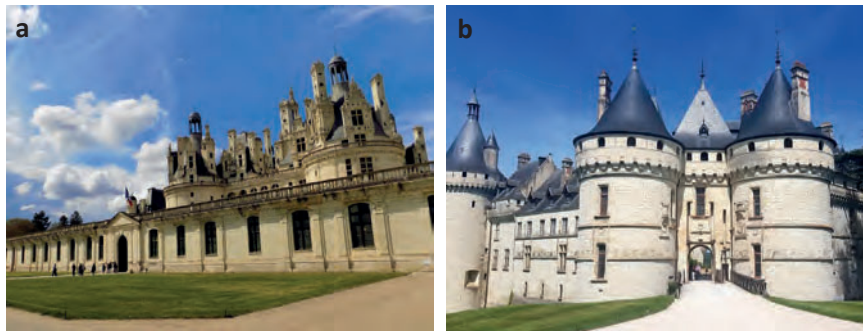


Figure 2: (a) Castle of Chambord (2019); (b) Castle of Chaumont-sur-Loire (2019) (b).

Even if their style is different, both castles were built during the same period with very similar stones and techniques. They also present the same degradations. However, they are located in distinct environments. While the castle of Chambord is isolated in the middle of a forest park of 5000 ha, the castle of Chaumont-sur-Loire, even if it is surrounded by parks and gardens, is next to a village and many roads, in a semi-rural area. The choice of both monuments stem from the objective of avoiding strictly urban environments, generating specific and thus distinct degradations forms.

### 3 MATERIALS AND METHODS

#### 3.1 Sampling

In both castles, samples were collected so as to represent different environmental configurations (accessibility or not to wind, rain, runoff), though the sampling methodology was specific for each monument. On the castle of Chambord, samples were collected by scratching the outer surface of the plate, on the back-side of the plate, and on the powdery zone where the plate has already fallen. Different situations were studied and presented in Figure 3: near recent restorations (1; 2; 3); in sheltered areas (4; 5), and in elevated areas with different directions (6; 7; 8; 9).

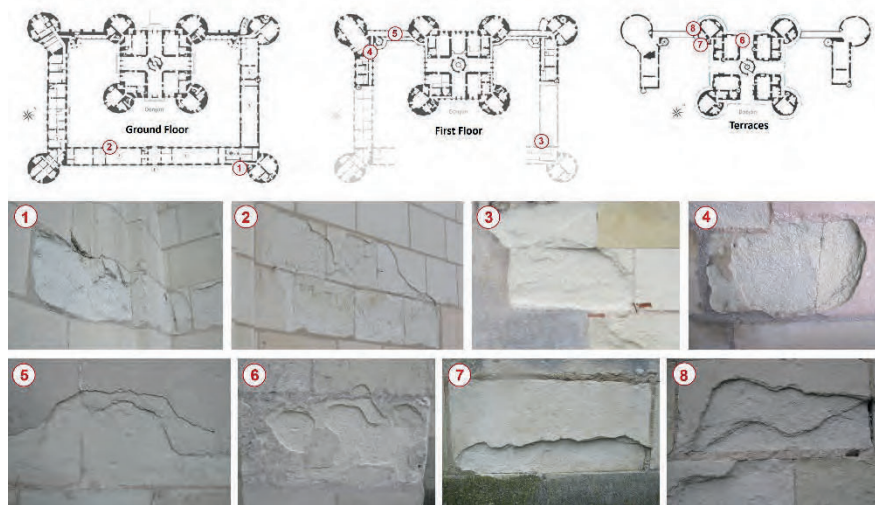


Figure 3: Localization of the samples collected on the castle of Chambord.

On the castle of Chaumont-sur-Loire, samples were collected by progressive drilling so as to obtain profiles along depth (Figure 4). Drilling was performed with a 10 mm diameter drill. The resulting powder was collected every centimeter up to 5 cm. Two zones were selected for this study:

- The "B" zone, situated on the Amboise tower, built in the 16<sup>th</sup> century, at the South-West of the castle, and exposed to South-East. Two samples were drilled: one in a reference stone (Bp1) which is visually healthy, another in a stone subjected to spalling (Bp2).
- The "G" zone, situated in the North-West of the main building of the stables, built in the 19<sup>th</sup> century. Spalling affects the whole height of the selected pilaster while occupying around 33% of its surface.

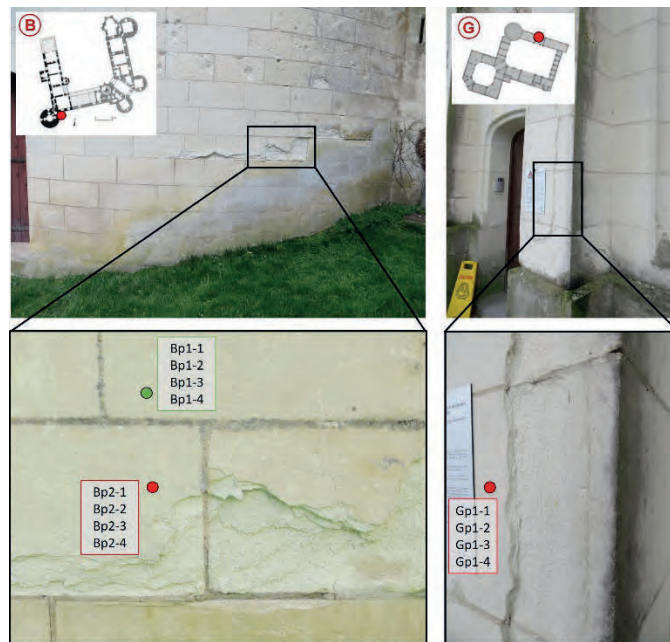


Figure 4: Localization of the samples collected on the castle of Chaumont-sur-Loire.

### 3.2 XRD and ionic chromatography

Samples were characterized by complementary physical and chemical techniques. The mineralogical characterization of samples was performed by X-ray powder diffraction (XRD) in order to identify their constituent minerals and detecting the possible presence of exogenous minerals (angle  $2\theta$  from  $5$  to  $60^\circ$  using  $\text{Cu-K}\alpha$  wavelength).

The soluble compounds were quantified via aqueous phase ion chromatography, both cations ( $\text{Na}^+$ ,  $\text{NH}_4^+$ ,  $\text{Mg}^{2+}$ ,  $\text{K}^+$ ,  $\text{Ca}^{2+}$ ) and anions ( $\text{Cl}^-$ ,  $\text{NO}_2^-$ ,  $\text{NO}_3^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{PO}_4^{3-}$ ) were analyzed. The ionic chromatography sample preparation protocol is that used in the analysis of stone samples for heritage conservation-restoration [7].

## 4 RESULTS AND DISCUSSION

### 4.1 Castle of Chambord

Powder samples were characterized by powder XRD analysis so as to detect exogenous minerals, especially gypsum. The main XRD peak attributed to gypsum is for  $2\theta=11,6^\circ$ . As shown in Figure 5, all samples present the same pattern: a systematic presence of the peak related to gypsum in the cracked zone, sheltered from the rain, whereas this peak is insignificant on the surfaces directly in contact with potential rain, i.e. external surface of the plate or of the powdery surface where the plate has already fallen. No other exogenous minerals were found.

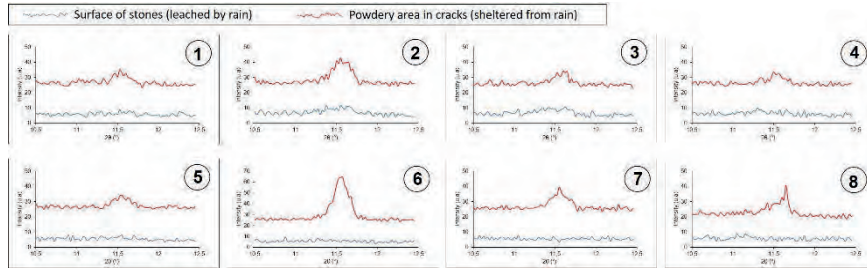


Figure 5: XRD pattern of the samples collected in the castle of Chambord (zoom around  $2\theta=11,6^\circ$  for the main peak of gypsum).

These samples were then prepared for ion chromatography analysis to quantify the gypsum content. Figure 6 presents the result of the quantification of gypsum assuming all sulphates dosed by ion chromatography are related to gypsum.

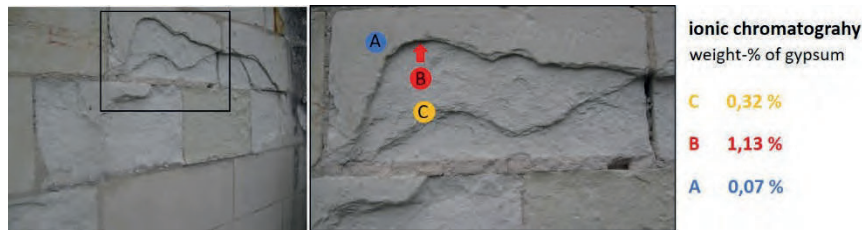


Figure 6: Gypsum quantification by Ion Chromatography for Chambord sample #8.

Figure 6 presents results for sample #6 from the castle of Chambord as it is representative of all the samples tested. The powdery surface (C), already subjected to spalling and thus now directly exposed to the environment, presents a lower gypsum content compared to the cracked zone of the plate (B). No gypsum can be found at the external surface of the plate (A). Most of the gypsum is thus located in the cracked zone as long as the plate has not fallen yet. Once the plate falls, this area is then subjected to loss of matter, mostly through leaching by rain runoff. This pattern of gypsum localization is totally in accord with the previous study conducted on samples cored in stones subjected to spalling in the castle of Chambord [6].

#### 4.2 Castle of Chaumont-sur-Loire

Samples collected in the castle of Chaumont-sur-Loire also contain gypsum according to XRD analysis (Figure 7) on zone "G", with no other observed exogenous salts. Quantification of gypsum profile by ion chromatography shows the same pattern as for the previous study [6]: low quantify at the surface of the plate, a maximum value at the cracked zone, and a decreasing profile deeper after the crack. Maximum values of gypsum content are still around 1%.

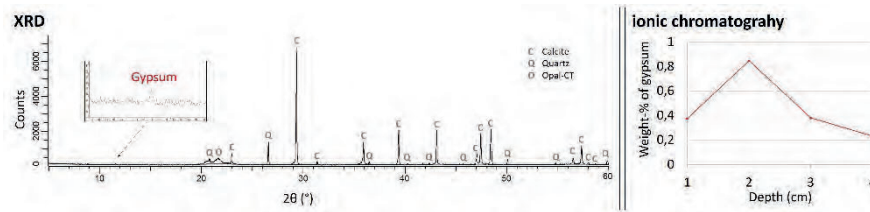


Figure 7: Gypsum identification by XRD and quantification by ion chromatography, on "G" zone at the castle of Chaumont-sur-Loire.

In the "B" zone, the same profile is observed (see Figure 8). An interesting point is that a similar profile can be found in the reference stone, which is visually not affected by spalling, though in a stone directly in contact with a stone subjected to spalling. This fact was also observed in the previous study on the castle of Chambord [6]. The confirmation of this observation tends to prove a maximal quantity of gypsum can be found in the depth corresponding to the future crack before the cracking happens.

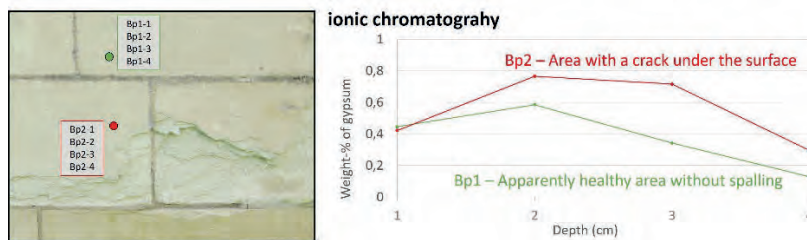


Figure 8: Quantification of gypsum in the "B" zone at the castle of Chaumont-sur-Loire

### 4.3 Questioning about sulphates

All tested samples showed the occurrence of gypsum in spalling, with a maximum value (around 1%) at the depth corresponding to the cracking zone. This statement is valid irrespective to the site, the position in the site, the exposition, the height, and the environment. Indeed, the literature reports that other stone types are affected by spalling. In many cases, authors observed the occurrence of gypsum in the degraded stone. Wendler et al. [8] made a similar characterization of salt profile within sandstone and limestone, with maximum content where damage to the stone matrix is also maximal. This zone also presents the maximal water content [9] [10], enhancing the crystallization of salts during drying. Other similar observations were made on the molasses of the cathedral of Lausanne, in Switzerland, where gypsum content could reach from 1 to 3 % in the cracking zone.

The localization of the maximum amount of gypsum is correlated with the depth corresponding to the cracking and so, the spalling. Hence, is it legitimate to consider the different potential roles of gypsum in spalling:

- It is a triggering factor of the cracking? (initiator)
- Does it propagate a preexisting crack? (enhancer)



- It is only a consequence of spalling with no link with its origin? (no role)

The analyses conducted in this study showed there can be gypsum at the typical depth of cracking before any crack occurs. The opposite, i.e. the occurrence of a crack with no gypsum, has never been observed. Hence, the assumption that a pre-existent crack is required for gypsum to crystallize can be rejected. So, gypsum is most probably supposed to crystallize before cracking occurs. The mechanisms of gypsum ingress in the stone and its potential role in cracking were studied [11]. However, the possibility of crack initiation due to the accumulation of gypsum has not been proven yet, whether for tuffeau or other types of stone subjected to spalling.

## 5 CONCLUSIONS

The phenomenon of spalling has questioned the scientific community for a long time. Though, no study has been able to identify and prove the existence of triggering factors yet.

This study aimed at verifying the profiles of gypsum content observed in spalling of tuffeau at the castle of Chambord during a specific study [6]. Other samples were collected at the castle of Chambord added to the castle of Chaumont-sur-Loire to provide more extensive and thus representative data for monuments built in tuffeau in the Loire Valley, in France.

In this study, for all samples, in both castles built in tuffeau, a systematic presence of gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) was observed in areas subjected to spalling. The gypsum content is always very low at the surface and progressively increases (always around 1%) up to the depth corresponding to the crack zone, and then decreases with depth. No other salt was detected. This study also proved pre-existent cracking is not necessary for gypsum to crystallize. All these statements are totally in accordance with the previous study [6], hence allowing to confirm its representativeness.

However, the role of gypsum in spalling, whether initiator, enhancer or purely spectator, has not been solved yet.

## ACKNOWLEDGEMENTS

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