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Crystallization Cycles in Masonry Walls: Experimental Technique to Develop Accelerated Aging on a Real Scale

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Abstract

Moisture in historic built heritage is one of the main degenerative agents, because it supports or manifests itself through multiple pathologies. Current knowledge allows for the diagnosis and assessment of the problem, but there are deficiencies in the evaluation of corrective systems due to the time it takes for moisture to become significant. In response, this study proposes an experimental methodology that aims to reproduce the accelerated aging of real scale specimens under laboratory conditions. Thereby improving the understanding of the impact of moisture related deterioration on masonry structures. To achive this, eight masonry walls were constructed and subjected to eight cycles of sulfate crystallization. They were saturated with different sulfate concentrations (5% or 10%) and exposed to different drying conditions (outdoor or solar dehydrator) in order to identify the factors favoring sulfate crystallization and the resulting deterioration. The progress of the experiments was monitored using a hygrometer, a thermographic camera and photogrammetry. The results indicate that it is possible to induce efflorescence in real scale specimens. Temperature and moisture monitoring helped identify the solar dehydrator as a more effective drying treatment. While digital photogrammetry was considered inefficient for quantifying volumetric damage, since this technique can present errors greater than 2%, a value exceeding the observed wear. Reason why the weight of material detached at the end of the experiment was recorded and a positive correlation between the increase in sulfate concentration and the use of the dehydrator was observed. Finally, pertinent considerations are made to improve the experimental conditions.

Keywords

crystallization, masonry walls, moisture, temperature, photogrammetry, degradation

1 Introduction

In the context of historic heritage buildings, moisture is considered to be an important factor associated with sick building syndrome. This is due to the fact that moisture alters the feeling of comfort, changes the environmental conditions inside the buildings, promotes the development of fungi, mites, and dust, affecting the health of users (Casas Figueroa, 2018). Furthermore, it can affect the image and structural integrity of the materials that make up the built heritage (Silva et al., 2003; Šťastný et al., 2021), since moisture is a factor that can cause any of the following classified pathologies:

1. cracking;
2. delamination;
3. induction of failure;
4. discoloration;
5. biological colonization (ICOMOS, 2008).

For example, differences in moisture and temperature lead to the expansion and contraction of materials, favoring appearance of cracks (López-Doncel et al., 2018). Water also dissolves and transports minerals, causing erosion, staining and the development of efflorescence or surface (Balog et al., 2016). These are cumulative degenerative pathologies, they can become significant issues and even compromise the structural integrity of elements (Navarro Hernández, 2013; Villegas et al., 2014).

Regarding the diagnosis and characterization of moisture as a pathology in buildings, it is widely studied

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(Pipiraite, 2018), with techniques ranging from simple observation to the use of specialized instruments, for instance:

1. Mapping involves using a subjective and contextual scale based on colors, fills, or shading (Arriaga de la Cerda, 2017; Marín Chaves et al., 1994).
2. Volumetric surveys rely on digital systems like photogrammetry or laser scanning for accurate digitization of elements' current state (Agüera-Vega et al., 2018; Almac et al., 2016; Cipriani et al., 2015).
3. The X-ray diffraction studies material composition and structure, identifying saturation states beneath the surface (López-Arce, 2012; Pérez García, 2001).
4. Instruments like thermographic cameras and solid hygrometers, record temperature and moisture, aiding in identifying the origin or causes of the pathology (Cañola et al., 2020; Coletti et al., 2023; Erazo-Aux et al., 2022; Serna Moreno, 2016).

However, some authors point out that in practice there are weaknesses in the implementation of corrective interventions in historic buildings. This is due to the difficulty of having comprehensive knowledge due to the peculiarities of the buildings, such as the composition of the walls, the origin of moisture, possible interventions, etc. (Franzoni et al., 2023; Lubelli et al., 2018; Šťastný et al., 2021). This can have repercussions as moisture can take time to manifest or become visible (Alkhateeb, 2011; da Costa Ribeiro et al., 2018); for example, in the Ögryte New Church, Sweden, previous interventions were recorded, consisting of sacrificial plaster, were recorded without favorable results (Balksten and Strandberg-de Bruijn, 2021).

In pursuit of this, an accelerated aging technique based on sodium sulfate crystallization cycles is employed (Cultrone and Pardo, 2008; Rivas Brea et al., 2008); where utilizes the efflorescence capacity of sodium sulfate, which crystallizes within the porous network of the materials and induces significant degradation. This test involves cyclic saturation of material samples in a sodium sulfate solution, followed by drying in an oven and standing at room temperature to record data (Liu et al., 2020; Morales Tassinari et al., 2020). This crystallization simulates accelerated wear due to moisture and facilitates the evaluation of corrective systems (Speri et al., 2017).

The objective of this investigation is to propose, develop, and evaluate an experimental approach based on sodium sulfate crystallization cycles as a test method for simulating accelerated aging on real scale. To achieve this experiment involves constructing and degrading eight combined masonry walls, applying and evaluating different treatments: saturation with varying concentrations of brine and two drying alternatives. The obtained results are periodically recorded for temperature, moisture, and volume in each cycle.

2 Materials and methods

The experimental process followed the same concept as Sodium Sulfate Crystallization Cycles (SSCC): which involved the stages of saturation, drying, and recording. For this research, it was proposed to extrapolate this technique to apply on a real scale. This is because standard conventional dimensions encompass cubes measuring 5 to 7.5 cm on each side. Instead, the plan is to test mixed brick and stone masonry walls.

2.1 Placement and auxiliaries

The experimentation has been conducted outdoors, requiring the preparation of the ground with an impermeable membrane and brick borders to prevent leakage. Separating the eight walls in two groups, on each platform necessitates a space measuring 4.00 m in length and 1.50 m in width, accommodating four walls with a separation of 1.0 m between them, a distance sufficient for capturing necessary photographs (Fig. 1).

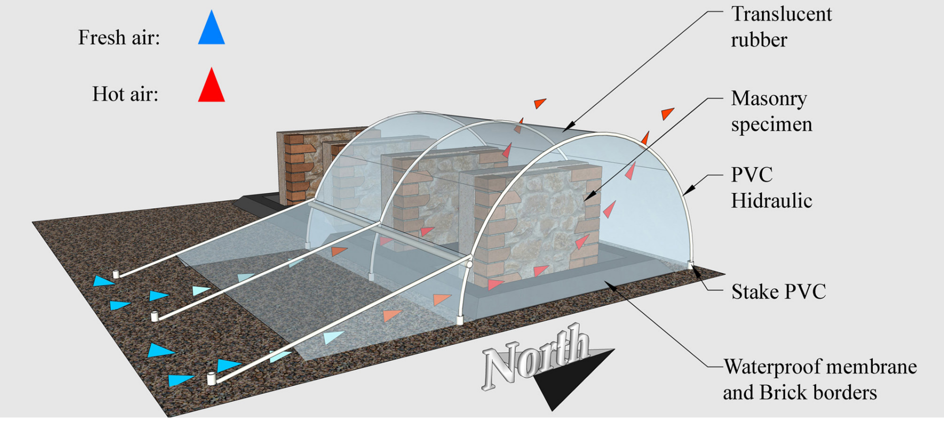

Fig. 1 Disposition and operation of the solar dehydrator

To enhance ventilation conditions and thermal gains provided by the environment, two solar dehydrators were needed (UNAM, 2023); the construction of these dehydrators was based on a framework of hydraulic PVC pipes and an enveloping translucent rubber cover.

To optimize the dehydrator's performance, understanding the geographical location was essential: GMT −6.00 time zone area, latitude of 19.8547 and a longitude of −104.2320. In this site, during the autumn-winter period when the experimentation was conducted, the sun's inclination was towards the south (SunEarthTools, 2023). The designated orientation for the walls also had to ensure wind circulation between them. As such, the walls arranged in a North-South longitudinal orientation (Fig. 1).

Similarly, the specimens were placed in an open area where they were not affected by the shadows of nearby buildings or surrounding vegetation, thus ensuring solar incidence.

2.2 Study materials

Representing the construction system that makes up the walls and buttresses of the temple of Nuestra Señora de la Candelaria, a 16th-century historic monument located in the town of Villa de Purificación, in the province of Jalisco, Mexico (Ascencio, 2004; INAH, 2022; Regalado, 2013). Eight walls (specimens) were constructed with the following dimensions: 30 cm thick, 100 cm long, and 75 cm high, for an estimated volume of 225,000 cm^3. The materials used in their construction include fired red brick at the ends, basalt stone in the center, and mortar to join the pieces and plaster one side of the specimen (Fig. 2). The mortar used is composed of the following proportions: one part cement, two parts lime, and eight parts sand. The physical properties of these materials are described in Table 1.

2.3 Instruments

A thermographic camera attached to a smartphone is used for temperature monitoring; its characteristics are: IR Resolution: 120 × 90, measurement range: −20 ~ 120 °C, accuracy: ±3.

A hygrometer for solids is used to record moisture data; its characteristics are: recordable moisture range: 0% to 70%, accuracy: ±0.5%, probe length: 1.10 cm.

2.4 Experiments and records

The eight specimens were subjected to eight SSCC each. Differentiating the different treatments by groups as described in Table 2. Subsequently, the adaptation and application of each of the processes necessary to develop this methodology is described:

1. Saturation: Saturation process, knowing that moisture infiltrates the walls due to factors such as porosity, absorption, water availability, and evaporation (Franzoni et al., 2023). Therefore, the specimens were wetted by capillary action by flooding their base. This was done by estimating the amount of water required to saturate each wall, considering an extrapolation of the absorption of the different materials and the estimated volume of the specimen. A total of 20 liters of water was obtained, of which 5 liters were applied every 30 minutes for two hours. The specimens are left to rest for the remainder of the day.

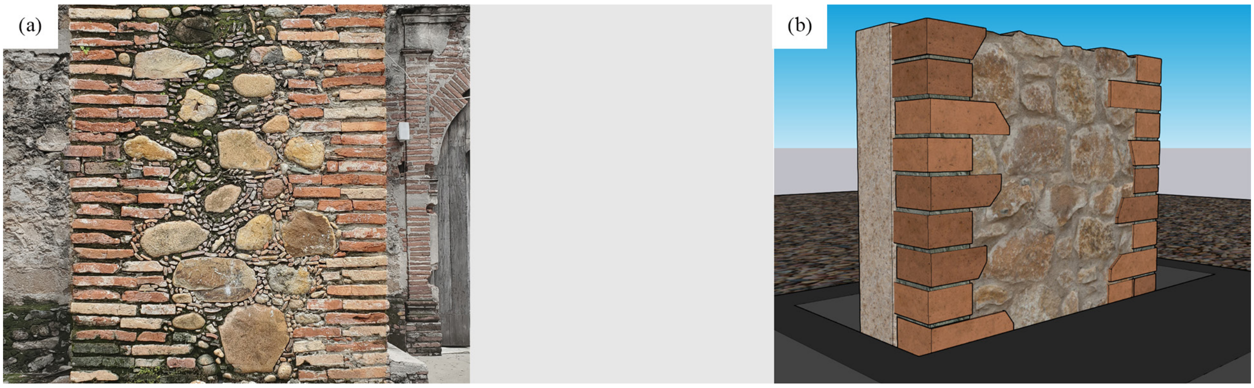

Fig. 2 (a) Typology of masonry walls of the historic temple, (b) Conceptual segment for the elaboration of the wall

Table 1 Physical properties of material

Material	Porosity (%)	Absorption (%)	Bulk density (g/cm^3)	Matrix density (g/cm^3)
Red brick	43.93	23.69	1.50	2.69
Mortar	25.92	11.22	2.31	3.11
Stone fathom	12.54	4.84	2.58	2.96

Table 2 Layout and experimental treatments

Input or treatments	Brine with 5%	Brine with 10%
Dehydrator drying	Walls 1 and 2	Walls 5 and 6
Weather drying	Walls 3 and 4	Walls 7 and 8

2. Drying: Drying process, two ideas are rescued from other research: the use of a ventilation system favors the drying (Mayo Corrochano and Lasheras Merino, 2012; UNAM, 2023); increases in temperature favor the development of efflorescence (Angeli et al., 2010; Lezzerini et al., 2022). Therefore, in this experiment, the use of the solar dehydrator has been implemented. Likewise, a contrast treatment is needed, which consists in drying outdoors. For both drying treatments, a period of six days was considered sufficient. The duration of one week per cycle: it was conceived after observing a significant decrease in moisture recorded by a hygrometer and the disappearance of stains in a pilot test with conventional water.
3. Data recording: To record the volume, it should be noted that these are objects with a free surface, making it impossible to use conventional methods, for this reason the use of digital photogrammetry has been incorporated as a reliable technique to represent the volume of complex shapes (Janvier et al., 2016; Pepe et al., 2023). The degradation is expected to be significant, since photogrammetry is known to have an error of 6% in unfavorable conditions (regular shapes and poor lighting) (Castro Figueroa, 2016); or a precision of 2% in favorable situations (irregular and opaque shapes) (Vizcaino et al., 2022).

For the development of the photogrammetry technique, each specimen was processed separately, requiring the capture of 42 images to create each model. The digitization process was carried out using Metashape (Agisoft, 2020) software (Fig. 3). Models generated through this process often need repairs to become virtual solid elements, and this was performed Meshmixer (Autodesk, 2021). To conclude the volumetric quantification, SketchUp (Trimble, 2023) was employed, using cubic centimeters as the measurement unit. Lastly, for a comparative value against the volume, the final weight of the material precipitated at the base of each specimen was quantified.

In addition, to evaluate the relevance of the use of the dehydrator, the moisture and temperature values were monitored, recording the average values for each face of the specimen. To obtain the average moisture, 12 points distributed on each face were recorded, while the temperature was obtained through dedicated markers from the MobIR application (Guide Sensmart, 2022), the thermographic camera's own software (Fig. 3).

3 Discussion of results

The experimental process resulted in the application of eight SSCC, one per week for a period of two months. This sequential process resulted in a gradual deterioration of the masonry walls. It is evident that the efflorescence mainly affected the first 22 cm from the base of the walls. The progression began with the appearance of white patches or localized wet areas and culminated in detachment of a significant amount of material from the mortar. In particular, the bricks showed a moderate degree of damage, while the stones showed only minor discoloration.

As a subjective visual result, different results can be observed in each group of specimens at the end of the experiment with their respective treatments (Fig. 4). Greater degradation can be seen in those treated with a higher concentration of brine (specimens 5, 6, 7 and 8). In addition, it is evident that the dehydrator favored degradation in both groups (specimens 1, 2 and 5, 6).

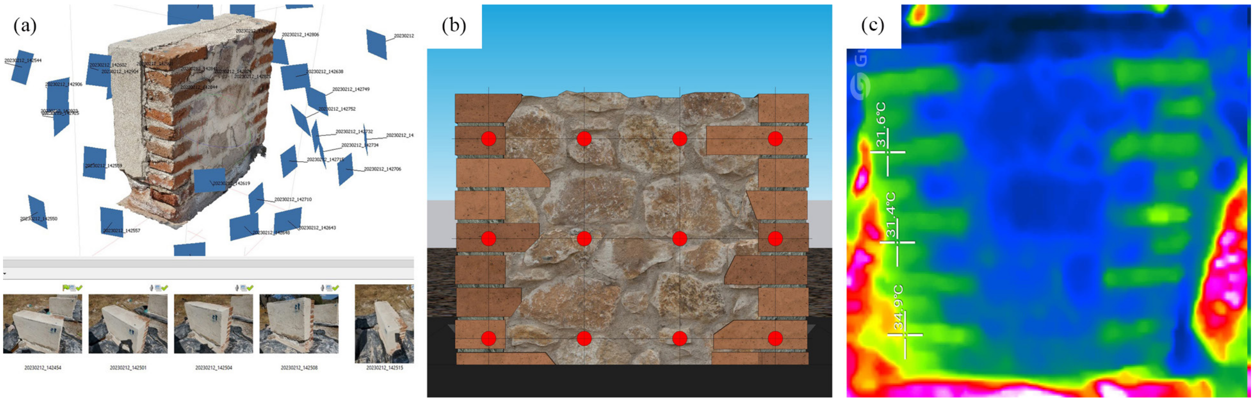

Fig. 3 (a) Acquisition by photography from Metashape, (b) Measurement of moisture by hygrometer, (c) Record of temperature by MobIR application.

Input or treatments	Brine with 5%		Brine with 10%	
Dehydrator drying	1 	2 	5 	6 
Weather drying	3 	4 	7 	8 

Fig. 4 Final state of the specimens according to their treatment group, side view

3.1 Volumetric monitoring by photogrammetry

As a result of the volumetric monitoring by photogrammetry, there is a graphic representation of the volume expressed in cubic centimeters for each specimen, recording the changes in each crystallization cycle.

It is worth noting that the volume can be altered due to the expansion and spalling of some materials. Similarly, it's important to mention that there is a 2% margin of error inherent to the photogrammetry technique used for volume registration. Nonetheless, there is a slight tendency to record the deterioration of the specimens (Fig. 5).

By replacing these volume values with percentage changes, where the origin (0) is considered as the initial real value and the degradation is considered as a positive value (Fig. 6), it is possible to better interpret and explain the changes recorded by photogrammetry.

In the case of specimens treated with a 5% brine solution, it is evident that specimens 1 and 2 exhibit initial volume variations, possibly due to photogrammetry registration errors. It is from cycle 5 that an increase in volume is reflected, which corresponds to the real state of the walls that have expanded due to spalling. For cases 3 and 4, the volumetric description does not seem to correspond to their real state, since they showed only slight chipping, and such changes are not justified.

On the other hand, the walls treated with a 10% brine show more compatible graphs among their pairs. In cases 5 and 6, initial changes are observed that could possibly be an adjustment of the precision of the volume, since the real deterioration started from cycle 4, where the graph shows a similarity with the real state. For cases 7 and 8, similar changes and a possible increase in volume due to spalling

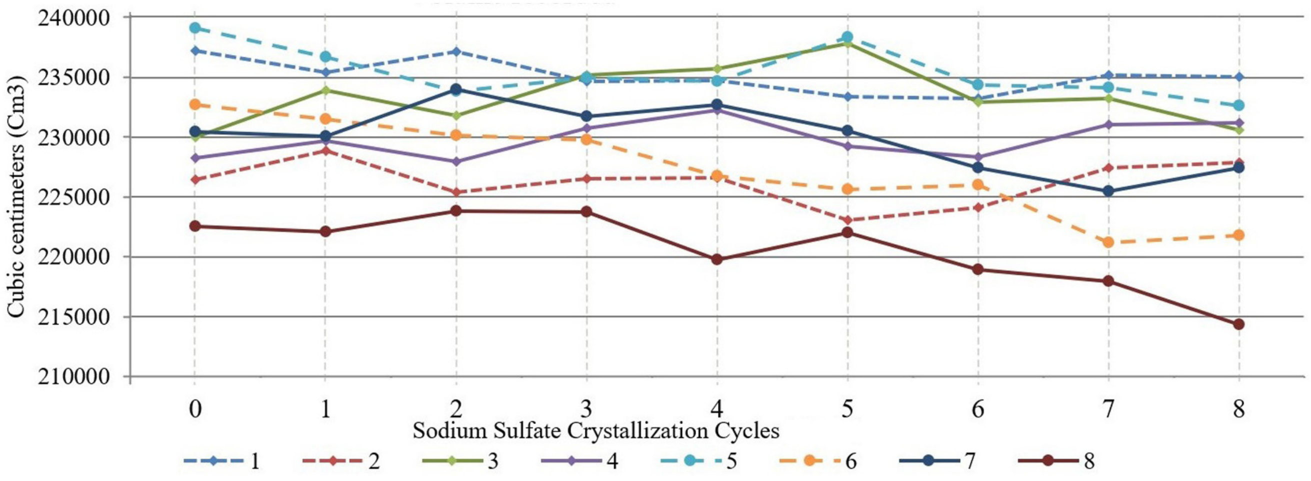

Fig. 5 Volume registered after each SSCC

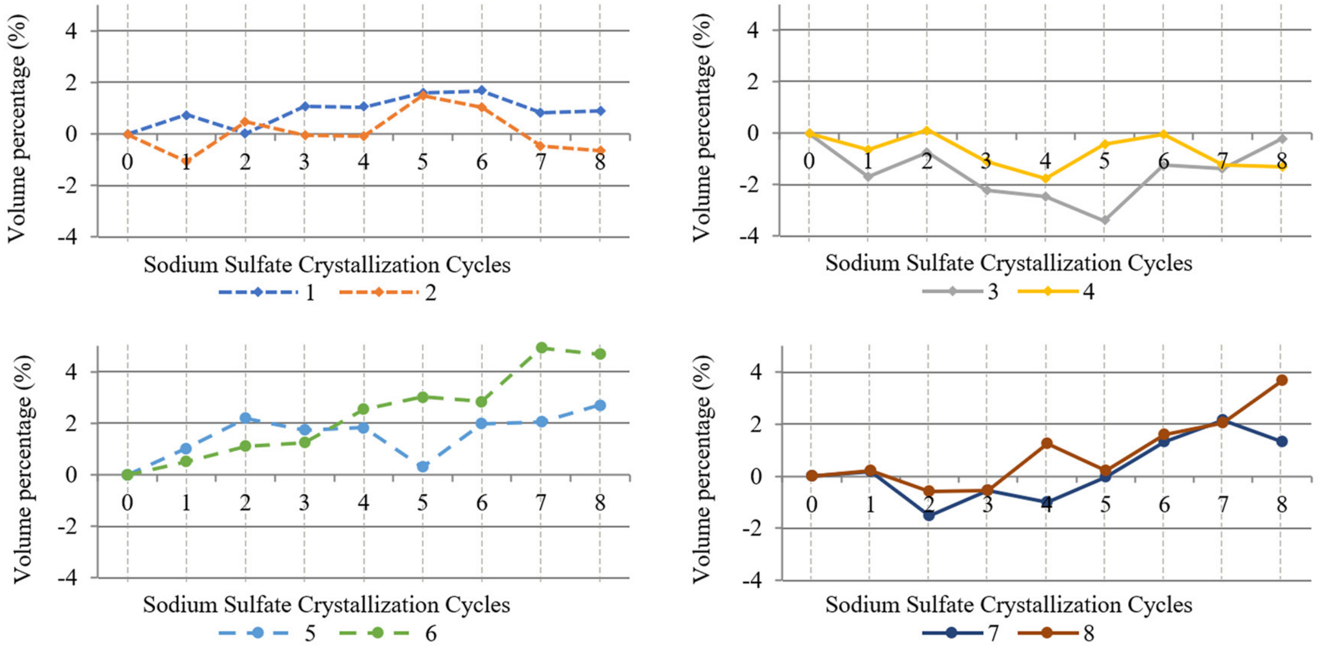

Fig. 6 Percentage change in volume recorded by photogrammetry

are observed between cycles 2 and 5. From there, the registered changes correspond to the visually observed degradation, from cycle 5 to 8.

3.2 Moisture and temperature monitoring

Moisture is a relevant factor for the accelerated aging technique, since sodium sulfate crystallization is achieved by drying (López-Arce et al., 2008). Therefore, the moisture behavior is recorded in the dry stages (indicated by a number only, for example 0, 1, 2, etc.) and the saturated stage (whit letter S plus number, for example S.1, S.2, S.3, etc.). The data are represented by the average moisture of the two groups of specimens: one for those dried with a solar dehydrator (orange line) and another for those treated outdoors (blue line), see Fig. 7.

It is important to note that moisture is affected by external environmental issues (Fig. 7), this can generate inconsistent data, such as higher moisture in the dry stage than in the saturated stage, such as the point of origin 0 and 3. In general, there is a clear difference between the saturated and dry stages, with the solar dehydration treatment being the one that achieves the greatest contrast. In addition, a lower percentage of moisture is always registered in the dry conditions compared to those treated outdoors.

Temperature is also a relevant factor contributing to the development of efflorescence (Angeli et al., 2010), so tracking was also carried out. The behavior of temperature was recorded during the dry stage (indicated by a number only, for example 0, 1, 2, etc.) and the saturated stage (whit letter S plus number, for example S.1, S.2, S.3, etc.). The data represents the average temperature in both groups of specimens: one for those dried using a solar dehydrator (orange line) and the other for those treated outdoors (blue line), see Fig. 8.

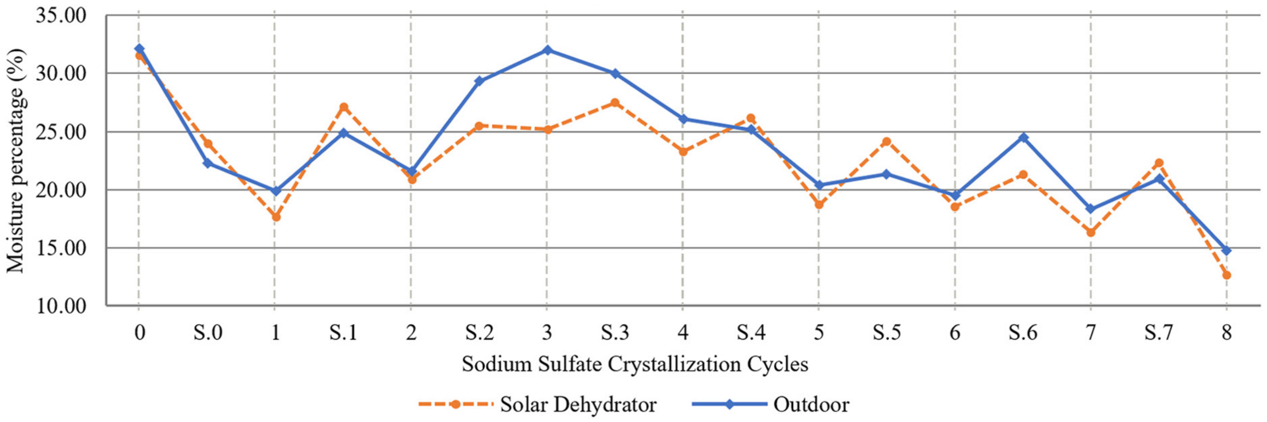

Fig. 7 Average registered moisture; dry and saturated state

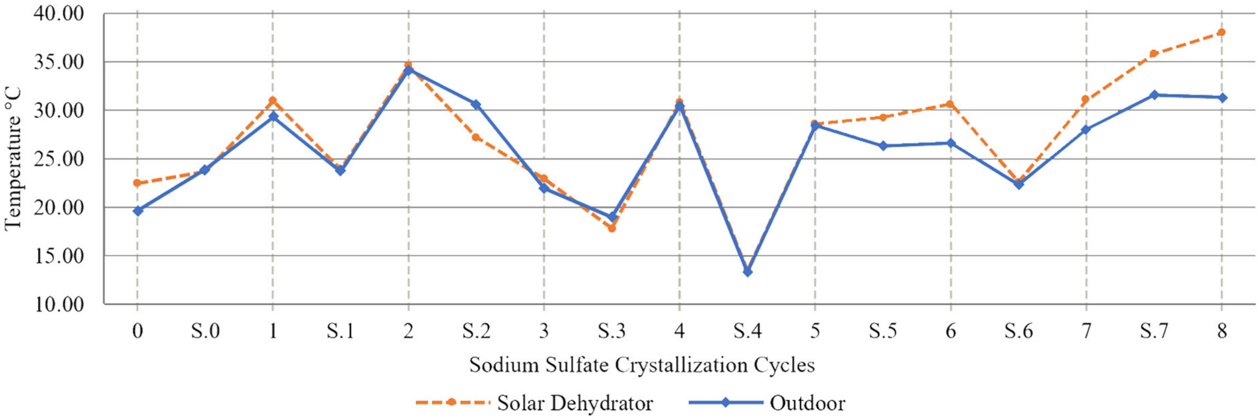

Fig. 8 Average registered temperature; dry and saturated state

The temperature is influenced by external environmental factors, making it challenging to discern differences between the saturated and dry stages. However, in specific cases, the expected temperature trends can be observed, such as during transitions from point S.0 to S.2 and from S.3 to S.5, where temperature gains are recorded during the dry stage and decreases during saturation. Notably, the solar dehydrator treatment consistently results in higher temperatures at the conclusion of the drying process (indicated by whole numbers: 0, 1, 3, 6, 7, and 8) in comparison to specimens treated under outdoor conditions.

3.3 Correlation between treatments

In order to assess the possible correlation between the increase in sodium sulfate concentration in the brine and the different drying processes applied to the specimens, a two-level, two-factor factorial design (Melo Martínez et al., 2020). Since the volume values obtained by photogrammetry may not be representatives of degradation, the analytical procedure was adapted to study it by quantifying the material detached from each sample. For this purpose, the dry weight of the material precipitated at the base of each specimen was recorded at the end of experiment, as detailed in Table 3.

In order to study how the independent variables (treatments) interact with the dependent variable (degradation of the specimens), it is necessary to arrange the treatments according to the factor analysis procedure. In this arrangement, 1 corresponds to the quadrant where the treatments are at their lowest level, while AB corresponds to the quadrant where the treatments are at their highest level, as shown in Table 4.

Table 3 Final degradation of the walls, recorded in kilograms (Kg)

Treatments levels	Brine with 5% (B−)	Brine with 10% (B+)
Solar dehydrator drying (A+)	2.08 Kg	6.51 Kg
	1.77 Kg	8.52 Kg
Weather drying (A−)	1.37 Kg	4.23 Kg
	1.29 Kg	5.18 Kg

The cumulative effects can be plotted visually to illustrate the relationship between events and their outcomes. The lines have a slope that progresses from lower to higher values, indicating a positive correlation between treatments (Fig. 9).

4 Conclusions

The incorporation of alternative knowledge into construction, exemplified in this case by the use of sulfate crystallization and elements such as the solar dehydrator, has facilitated the development of the experimental methodology presented here. This methodology has effectively induced accelerated aging in real scale masonry walls, highlighting the potential of implementing sodium sulfate

Table 4 Calculation to evaluate the correlation

(A) Drying	(B) Brine	Treatment	Replicas		Add.	U.
Weather (−)	5% (−)	1	1.37	1.29	2.66	Kg
Dehydrator (+)	5% (−)	A	2.08	1.77	3.84	Kg
Weather (−)	10% (+)	B	4.23	5.18	9.40	Kg
Dehydrator (+)	10% (+)	AB	6.51	8.52	15.03	Kg

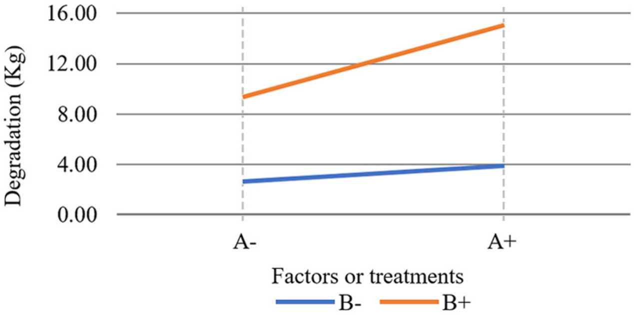

Fig. 9 Correlation graph of sulfate saturation and drying process

crystallization cycles as a laboratory test for large specimens. This methodology experimental can be applied to the simulation of construction elements of historical heritage, considering that these elements may be composed of different materials that interact differently when modified by moisture or efflorescence (Szemiot et al., 2023).

Regarding the volumetric recording by photogrammetry, its high capacity to digitize objects in a real environment is recognized, but its precision is considered inefficient in measuring the degradation produced by eight SSCC. This may be due to three factors:

1. Natural lighting produces high contrasts in photographs that end up being unsuitable for the Metashape (Agisoft, 2020) software interpretations.
2. Degradation caused by efflorescence is associated with an increase in volume due to expansion, spalling and crystallization (Balog et al., 2016) resulting in changes in the volume records.
3. It is estimated that the degradation caused by eight SSCC represents less than 2% volume degradation, which is below the accuracy of the technique (Castro Figueroa, 2016; Vizcaino et al.,2022).

The monitoring of moisture and temperature shows that the incorporation of a solar dehydrator favors the development of efflorescences, since it manages to reduce the percentage of moisture and increase the temperature in the specimens, two important factors for the development of SSCC in the laboratory (Angeli et al., 2010). This can be visually confirmed by comparing the degradation between the different treatments carried out. It is estimated that these results could be improved by integrating ventilation systems or mechanical conditioning.

Finally, it has been shown that there is a positive correlation between the concentration of sodium sulfate and the use of the solar dehydrator, where the use of the dehydrator (factor A) and a higher concentration of sodium sulfate (factor B) contribute to the degradation of the specimens, with the concentration of sodium sulfate having a more significant effect; in the same way, the interaction of both is also important.

Therefore, it is estimated that the experimental methodology proposed to generate accelerated aging can be useful for simulated evaluations of the built historical heritage. It can become a complementary tool to evaluate the efficiency of corrective systems aimed at solving moisture problems that attack the base of construction elements through filtration, capillarity, or absorption. In the same way, it implies an optimization or an alternative in the study processes for those investigations interested in understanding the behavior of the moisture phenomenon or, more specifically, efflorescence. This methodology can reduce testing times, as some authors, such as Balksten and Strandberg-de Bruijn (2021), reported that it took three and a half years to ensure that certain lime hemp specimens no longer transmitted efflorescent minerals.

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