Focus on soluble salts transport phenomena: The study cases of Leonardo mural paintings at Sala delle Asse (Milan)

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The program of investigations on "Sala delle Asse", which hosts a monochrome landscape attributed to Leonardo, in the Sforza Castle in Milan recently concluded the first step. Results of the analytical tests for the characterization of materials and their damages showed the high diffusion and concentration of nitrates and sulphates on the surface of the monochrome at the edge with the restoration mortars, on the right side of the north-western wall. On the base of the scientific literature and laboratory tests, the researchers identified a threshold of RH above which deliquescence of salts could easily occur. Microclimatic monitoring results informed that during the most humid days in spring, summer and fall, RH trespasses this threshold, with a frequency of about 30 events/year. After an accurate analysis of air temperature (T °C) and relative humidity (RH) resulted that the exterior changes especially affect the interior climate at some summer conditions as middle-high speed of wind and, especially, its direction due to some cracks and holes in the north western exterior wall of the hall.

Keywords: Microclimate
Air mixing ratio Psychrometry
Nitratine Magnesium
sulphates Wind
Restoration Leonardo da Vinci
Monochrome

1. Introduction

The historical documentation regarding Sala delle Asse, located in one of the corner towers in Sforza Castle (Milan; Fig. 1), reports that Leonardo had the task of decorating the hall in 1498.

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Fig. 1. Sforza Castle. Milan; credits of the website of the protection of CH, Region Lombardy http://www.lombardiabeniculturali.it/blog/articoli/1815/.

Fig. 2. Sforza Castle. Milan. Sala delle Asse, view of the north eastern side, with the monochrome on the left and with part of the vault, before restoration (December 2012).

Fig. 3. Sample 1 C. Monochrome wall: white efflorescences are mainly located alongside the border of a patch composed of a different cement mix compared to the surrounding matrix.

Currently, it is not known if the artist accomplished the task alone or if somebody else completed the decoration, nevertheless Sala delle Asse was his second huge job, as a mural painter in Milan, overshadowed by The Last Supper alone. One year later, after the defeat of Milan's Duke, Leonardo left the town and, probably, his unfinished painting in Sala delle Asse. Under the French domination, from 1499 the castle went under a wide-ranging refurbishment, to host the army headquarter. Sala delle Asse became a horse stable and several layers of whitewash coated the paintings.

Fig. 4. Polychrome vault: white efflorescences are unevenly distributed forming a whitish covering layer.

Fig. 5. Polychrome vault: soluble salt crystal aggregates morphology.

Fig. 6. Sample 1. White efflorescence analysed by X Ray diffraction (see Fig. 7).

Luca Beltrami, in charge of restoration works, as the castle was turned into a museum, discovered the ornamental paintings in 1893, based on Paul Müller-Walde plaster removal trials; Beltrami removed the whitewash, unveiling the poor condition of the paintings. Although he did not document the remains of the decoration that appeared underneath the whitewash, his decision to intervene

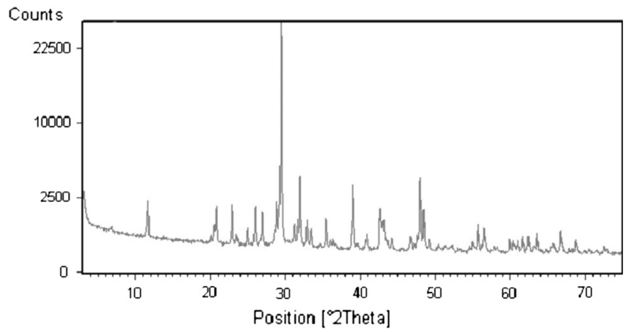


Fig. 7. Sample 1, X Ray diffractogram of powder coming from efflorescence. The pattern shows the presence of nitratine and gypsum as soluble salts. Other insoluble mineral components are present, coming from the plaster (barite and clay).

on the paintings took place quite soon, and eight years after, the painter Ernesto Rusca had the task of integrating and repainting the remains, completing the works in 1902. Beltrami and Rusca did not recognize Leonardo's monochrome painting remnants on the two walls (Fig. 2). In 1954–56, Ottemi Della Rotta removed most of the Rusca's over-paintings with the aim of showing better the layers underneath; he used very invasive mechanical cleaning and, furthermore, he did not document the used methods.

At present, the poor condition of the paintings require a thorough intervention, especially, the monochrome decoration on the northwestern and northeastern walls, certainly painted by Leonardo himself: the assessment results are that soluble salts efflorescences whitened the surface, altering the pictures and endangering the plaster cohesion. Whitish formations are mainly located on the right part of the wall with the monochrome by Leonardo, even if their distribution is also on several other parts such as the vault, their micro-distribution is quite peculiar; in fact, they are concentrated in the ancient plaster close to the modern plaster patches (Fig. 3). Soluble salts whiten the painting with a thin powdery layer not present on the ribbons' decorative elements (Fig. 4); at closer observation, it is possible to see crystals aggregates in slight relief compared to the painting surface (Fig. 5).

Documentation regarding the assessment of materials and their state of conservation [5] permit to hypothesize that the paintings require a complex, urgent, articulated intervention above all for the safeguard of the remaining Leonardo monochrome fragments (see Fig. 6).

The verification of the initial hypothesis of the damage causes, exposed in the preliminary phase of the analysis in 2013 [9], indicated further studies were necessary on the most critical areas.

On the basis of the damage map, the first analytical campaign had the aim of ascertaining and localizing whether anomalous water content is present in the masonry.

Despite the masonry thickness and the elevation of the hall (first floor), the damage at the bottom of the masonry showed clear signs of water phase transition (liquid/vapor and vice versa). Moreover, due to the extension of the areas where diffused salt crystallization is on both the monochrome and the vault, in May 2012, the authors started the first step of a microclimatic monitoring to detect dangerous variations of air temperature (T °C) and relative humidity (RH) that could accelerate the soluble salt solution transport phenomena and increase the damage pattern. The present paper refers to the results obtained through monitoring and investigating during the last four years. The authors presented a preview of these results in previous papers [12].

Table 2
Ionic chromatography results of some samples coming from efflorescence formations.

Sample	Nitrates (%)	Sulphates (%)	Sodium (%)	Magnesium (%)	Calcium (%)
SA1	38,65	6,48	18,27	0,00	2,39
SA2	44,72	5,12	20,50	0,00	2,12
SA3	17,01	18,79	7,57	1,34	7,45
SA4	12,10	22,69	5,27	1,58	8,37

2. Materials and methods

2.1. Preliminary tests on the study cases

The authors applied an IR Thermography (IRT) scanning and gravimetric tests [10,7] as preliminary assessment for mapping the presence of water in the structure. NDT method showed that structures and surfaces are dry [3,8,9].

In the areas where the thermal gradient could show the presence of evaporative flux as on going, the authors applied the gravimetric tests on a set of few samples composed of very small fragments of restoration mortar [11] and they evaluated the presence of soluble salts.

The application of Nuclear Magnetic Resonance (NMR) and Evanescent Dielectric Field (EDF) on the masonry, confirmed the results of the IRT and gravimetric data regarding the presence and quantity of water.

2.2. Analysis for the restoration

The following results come from the analytical plan supporting the intervention. The soluble salts formed whisker-like arrangements, mostly localized in the outer plaster surface, close to the recent cement plaster patches. The authors sampled the whitish formations and analysed the samples by means of X Ray Diffraction (X PanalyticalX'Pert PRO). Nitratine (sodium nitrate $NaNO_3$) came out as the main component of efflorescences (Fig. 7 shows the diffractogram). Gypsum ($CaSO_4{\cdot}2H_2O$) and magnesium sulphates (both hexahydrite $MgSO_4$ $6H_2O$ and epsomite $MgSO_4{\cdot}7H_2O$) are also present as minor components. Nitratine efflorescences are mostly located in the proximity of the north-eastern window, on the monochrome surface. On the contrary, the sulphate salts are

Table 1
X Ray Diffraction results of some samples coming from efflorescence formations.

Sample	Location	Material description	Results
SA 1	Monochrome wall. Surface close to the window	Efflorescence. Pale grey powder	Nitratine = xxx; gypsum = xx; baryte = x; montmorillonite = tr
SA 2	Monochrome wall. Surface close to the window	Efflorescence. Pale grey powder	Nitratine = xxx; gypsum = xx; baryte = x; montmorillonite = tr; hornblende = tr
SA 3	Monochrome wall	Efflorescence. Dark grey powder	Nitratine = xxx; gypsum = xxx; quartz = x; plagioclase = tr
SA 4	Monochrome wall	Efflorescence. Dark grey powder	Nitratine = xxx; gypsum = xx; quartz = xx; plagioclase = tr.; kornblende = tr
SA 6	Monochrome wall. North Angle. Close to probe 1 (see Fig. 10)	Deposit and efflorescence. Pale grey powder	Gypsum = xxx; hexaydrite = xx; calcite = x; epsomite = x; quartz = x
Sa 7	Close to probe 12 (see Fig. 10)	Deposit and efflorescence. Pale grey powder	Calcite = xxx; quartz = x; gypsum = x; hexaydrite = xx

diffused in a lower amount, nevertheless their presence in every sample shows a widespread sulphation of the plaster; soluble salt analyses are summarised in Tables 1 and 2. Fragments of the original plaster were analysed by means of Ionic Chromatography (Dionex DX 100). The results obtained corroborated XRD outcomes, quantifying the concentration of ions in the plaster: nitrates reach 1% w/w, and their amount increases with the depth of the plaster. On the contrary, sulphates reach their maximum around 0.3% w/w on the surface of the plaster.

3. Results

The results of the first part of the investigation excluded that water infiltration is the cause of the monochrome damage; on the contrary, the main causes are probably microclimatic unbal-ance and the diffusion of soluble salts [1,4]. The salts provenance could be identified in the cement mix design of the mortar over several restoration projects, composing both the patches and vast integrations surrounding the monochrome surface; inappropriate cleaning products, supposed to be based on alkali and applied in previous interventions, could explain the large amount of sodium present. The location of the most damaged areas (close to the north-eastern window) shows that further tests should investigate the microclimate variation, and the rate of air mixing close to the windows, because of the lack of tightness of the window frames. Up to now, monitoring results partially explained the dynamic of the natural ventilation inside the hall, therefore the new investigation areas included the rooms nearby as well, together with an increase in the number of sensors applied in the main hall and the repetition of the psychrometric survey in the second and third years of the analytical campaign. The aim of the psychrometric investigation is to evaluate the temperature and humidity unbalances of the microclimate and to define the thresholds for the optimal conservation of the precious surface in the room [15].

3.1. Psychrometry

The verification of the existing unbalance by means of the repetition of seasonal and daily psychrometric measurements allowed the authors to locate the most significant places to install the additional probes. As an example, we can analyze the maps of December 2013 (in Figs. 8 and 9).

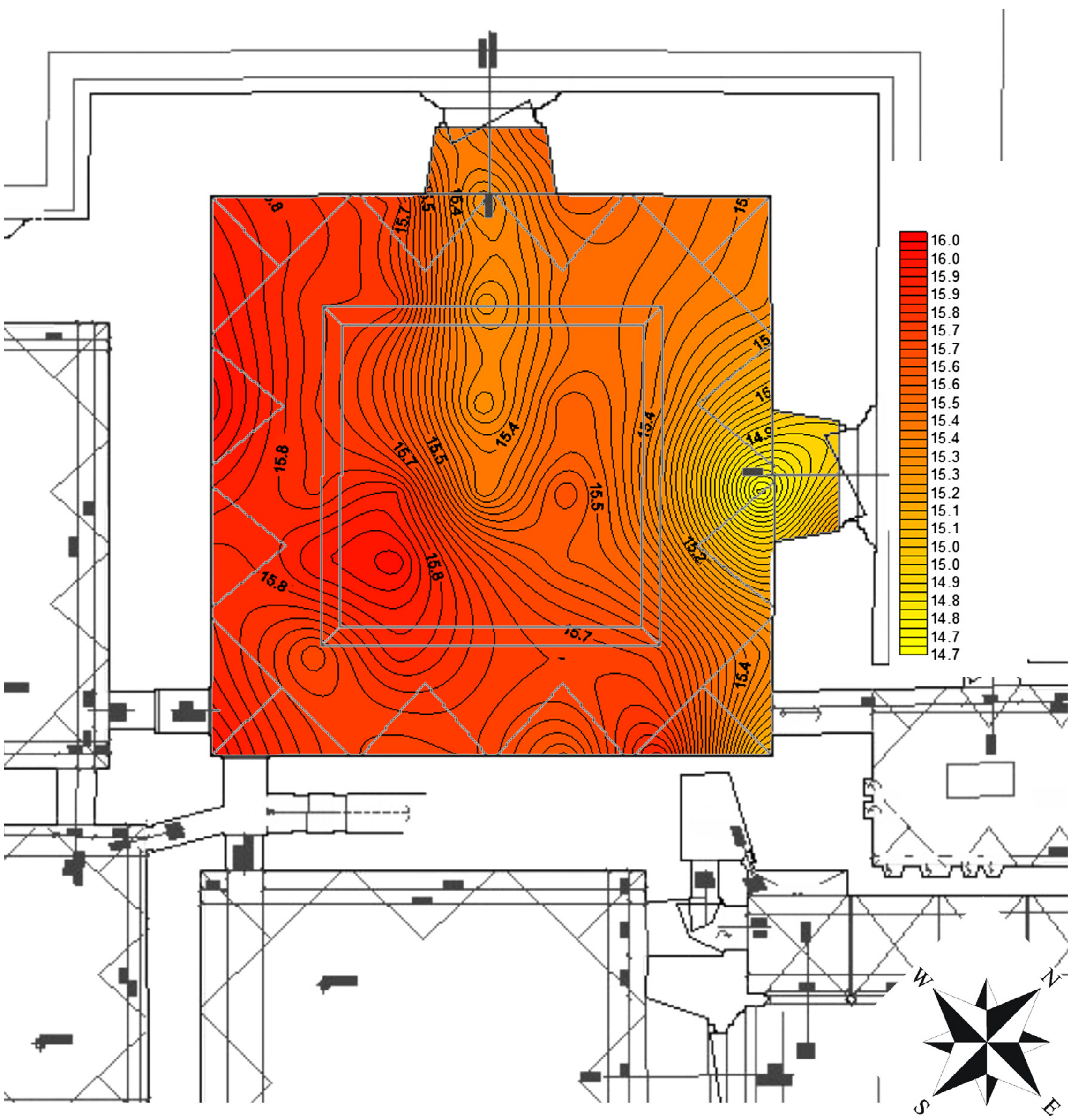


Fig. 8. Map of air Temperature distribution, December 19, 2013, h 4:30 pm.

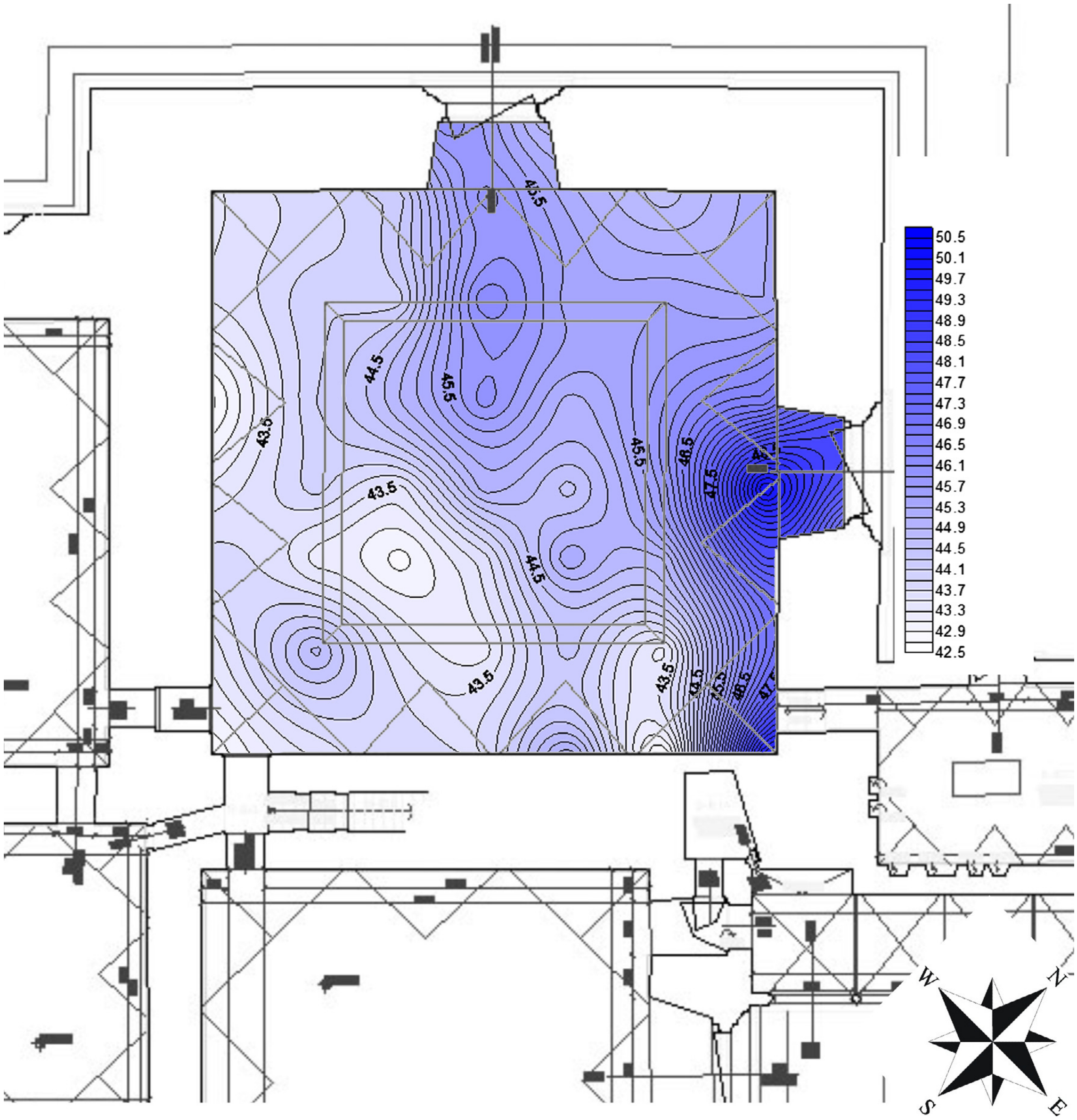


Fig. 9. Map of Relative Humidity distribution, December 19, 2013, h 4:30 p.m.

3.2. Microclimate monitoring

In the second phase of the investigation, as shown in the previous paragraph, the authors added a further eight probes at different heights and in the nearby rooms (Sala del Gonfalone and Salette Nere in Fig. 10).

The psychrometric maps showed that the highest unbalances are close to the windows and doors leading towards other rooms that have major air exchange with the exterior.

The probe installed in the northwestern window records variations very similar to the exterior air (with a delay of 2–3 h), but with a reduced span (daily excursion of about 3 °C in late spring, 7–8 °C in summer). On the contrary, the thermal difference of the values recorded along the walls, far from the windows, is about 1 °C, with very smooth and gradual variations.

The probe installed close to the monochrome and the one in the northeastern window measure values very similar to the ones measured far from the windows.

Therefore, the two probes in the two windows (north eastern and north western) measure very different values, despite their proximity to the opening, with the same kind of window frames.

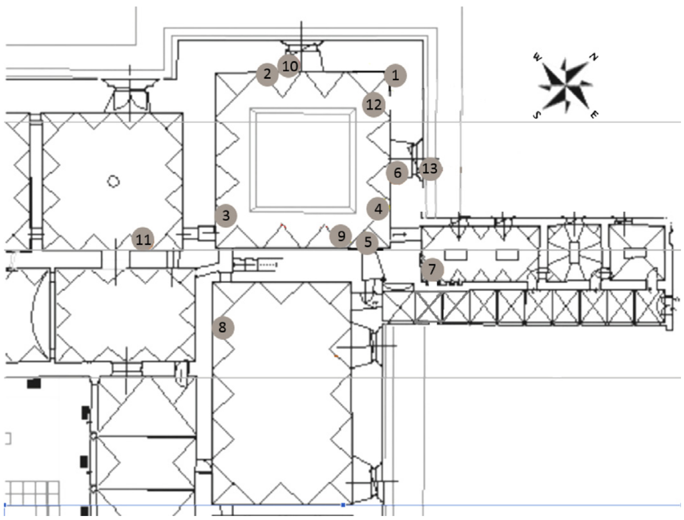
During the solar year, the values of RH vary between 30 and 70% inside the hall, the highest variations being close to the northwestern window, where the unbalance can be of 20% in few hours.

The other probes measure less frequent, smaller, and slower variations, up to 10%. Low values are frequent during the winter 2012–2013. From April 2013 up to September, the daily average values are higher, between 40 and 60%. On the contrary, in 2014 the values are very high also during the winter (up to 70% and often above). During summer 2014, often the values are higher than 70% due to prolonged rainfall, together with thunderstorms and high speed winds.

Finally, the measures of probes at 4 and 8 m elevation show a substantially homogeneous trend, temperatures are higher by about 0.5–1.5 °C than the others are.

In the small rooms beside the hall, the temperatures are the closest to the exterior ones (during the winter there are only 3–5 °C of difference between the interior and exterior values) and obviously, there are the highest variations of RH.

Therefore, the corridor between the small black rooms (Salette Nere) and the hall does not prevent the entrance of airflows from the exterior, with risk of conservation for the painting of the hall.



Probe n	Elevation (m)	Location
1	4	Sala delle asse
2	4	Sala delle asse
3	4	Sala delle asse
4	4	Sala delle asse
5	8	Sala delle asse
6	0	Sala delle asse
7	1	Salette nere
8	1	Sala Gonfalone
9	4	Sala delle asse
10	4	Sala delle asse
11	1	Sala 11
12	8	Sala delle asse
13	1	Sala delle asse

Fig. 10. Map of probe localization and their list.

Table 3
Frequency of the direction and speed of the wind higher than 6 m/s between July 20th and August 31, 2015.

Measures	Angle frequency	Speed > 6 m/s
0 < 15	17	2
15 < 30	13	0
30 < 45	17	0
45 < 60	22	0
60 < 75	32	0
75 < 90	34	0
90 < 105	74	1
105° < 120°	187	2
120° < 135°	688	42
135 < 150	50	1
150 < 165	8	1
165 < 180	5	0
180 < 195	4	0
195 < 210	4	0
210 < 225	5	0
225 < 240	1	0
240 < 255	3	0
255 < 270	5	0
270 < 285	5	0
285 < 300	10	1
300 < 315	29	2
315° < 330°	104	10
330 < 345	54	5
345 < 360	34	7
Total data	1405	

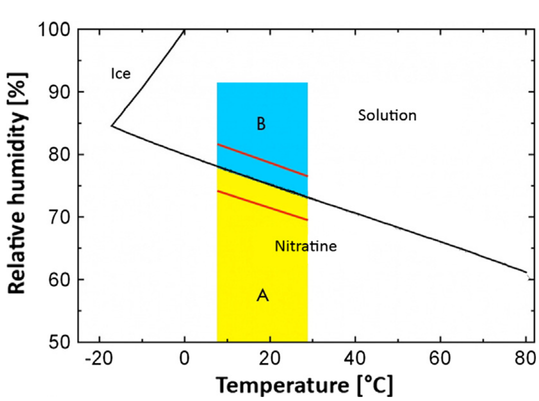


Fig. 11. Crystallization curve of nitratine (reference www.saltwiki.net/Hans-JuegenSchwarz).

Apart from this side and the northwestern window, inside the hall the conditions are almost homogeneous, at any height.

Climate records in Milan show the highest wind speed during the summer storms. In summer 2015, the authors placed an anemometer and a wind direction sensor on the top of the northern tower of the castle, exactly over the windows of Sala delle Asse, to find any correlation between the change of interior conditions and the outside wind speed and direction. The authors processed 1402 pieces of data, collected between July and August 2015. The data processing included the calculation of the frequency of the main direction and the threshold of wind speed above which the changes occurred inside the building.

The authors processed 1402 items of data, collected between July and August 2015. The data processing included the calculation of the frequency of the main direction and the threshold of wind speed above which the changes occurred inside the building.

Table 3 shows that the main direction of wind are the angles 105–135° and 315–330°, and most of the highest speed events occurred with these directions of the wind (see the red digit).

Hence, 0° is the northern direction, the main and faster wind has directions east-south east and north-west (almost the same alignment, opposite sides). Furthermore, the variation of RH and T °C inside Sala delle Asse occurred when the wind speed is higher than 3 m/s, and the area where the highest variation occurred are the square metres directly in front of the north-western window.

3.3. *Analysis of the change of salt phases and the mixing ratio of the air inside/outside the hall*

The results of the microclimatic monitoring show risky conditions for conservation due to prolonged rainfall (summer and late fall 2014), if considering the museum conservation standards for RH [2].

The next step of data processing is the extrapolation of the number of variations that are extremely risky for the cycles of salts solubilisation/crystallization on precious surfaces [16].

Particularly, the authors processed the monitoring data in relationship to the curve of crystallization of the salts most spread on the monochrome (nitratine $NaNO_3$, hexahydrite $MgSO_4{\cdot}6H_2O$ and epsomite $MgSO_4{\cdot}7H_2O$). Using the phase diagram of the highlighted compounds, the authors obtained the definition of the values of the critical threshold that delimits the passage into

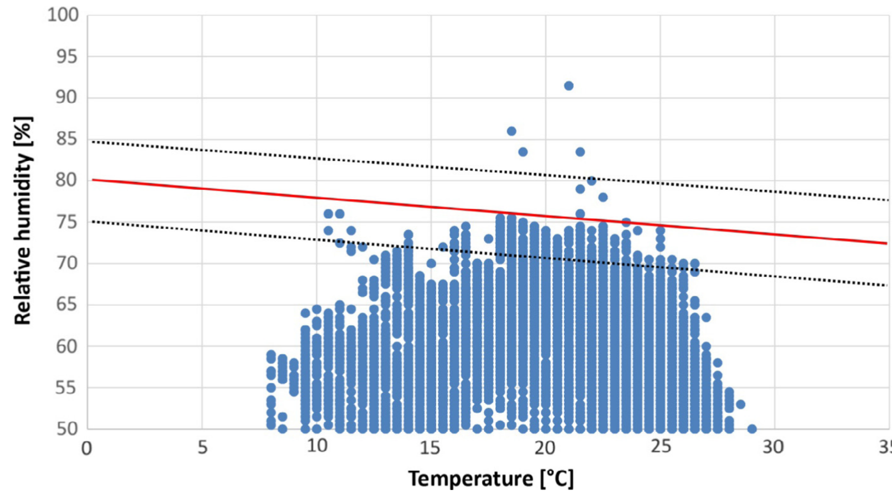


Fig. 12. Scatter graph of the data acquired from 23rd May 2012 to 10th October 2013 by probe n 2.

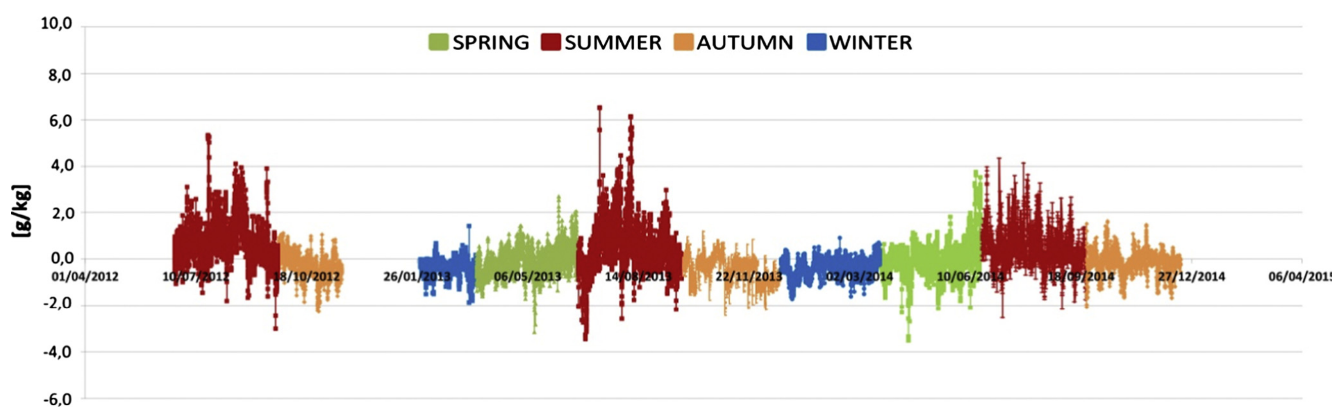


Fig. 13. Differences of mixing ratio versus time between exterior (probe n 10) and interior (probe n 2).

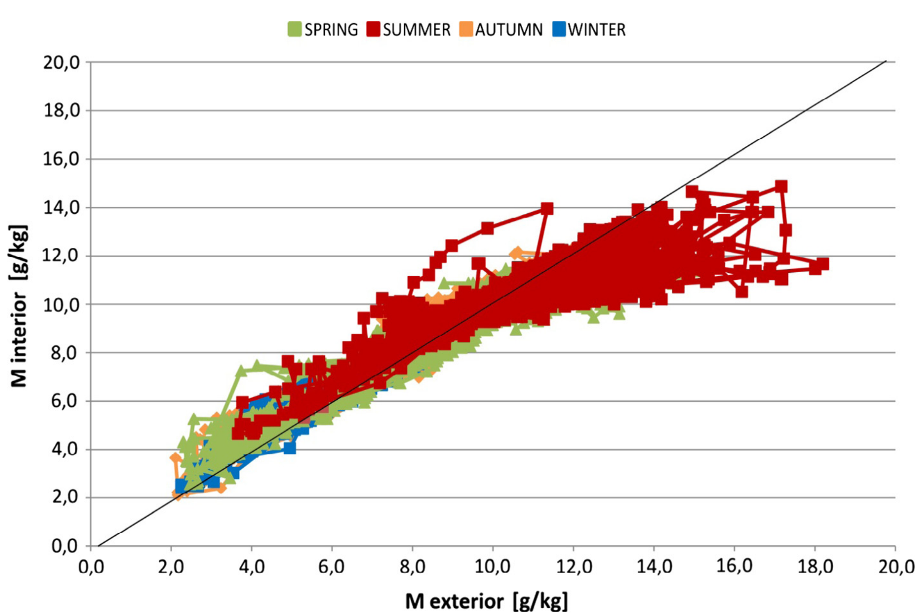


Fig. 14. Exterior (probe n 10) and interior (probe n 2) mixing ratio, year 2013–2014.

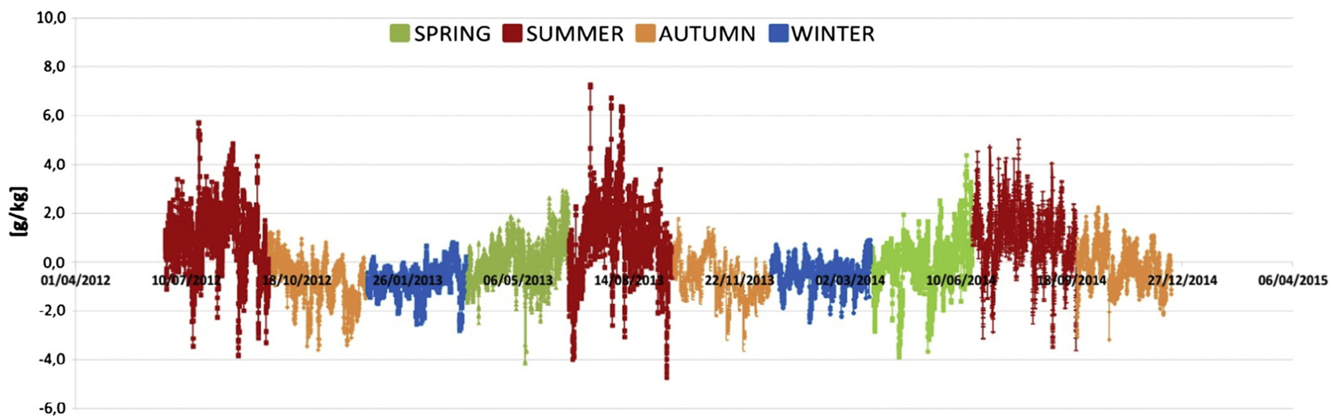


Fig. 15. Differences of mixing ratio versus time between exterior (probe n 10) and interior (probe n 8).

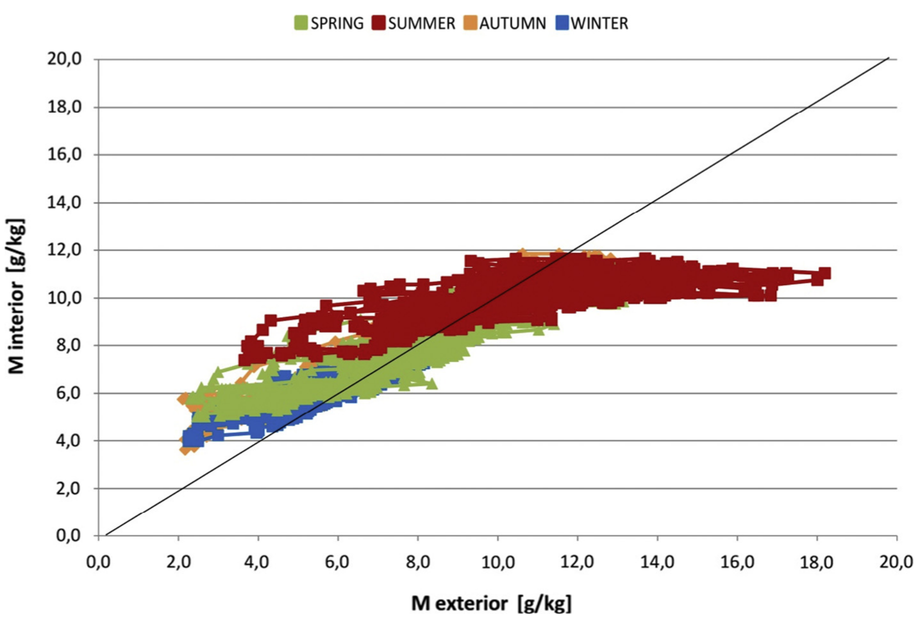


Fig. 16. Exterior (probe n 10) and interior (probe n 8) mixing ratio, year 2013–2014.

solution and vice versa, the precipitation of solute that causes the appearance of salt efflorescence on the surfaces.

The calculation of the frequency of these events is 10 in the monitoring year. Nevertheless, despite the most spread salt being nitratine, many other salts are present, in a mixture, and therefore their conditions of crystallization change [13,14]. Generally, the threshold of RH to obtain their deliquescence is lower, and the span of risk for occurring crystallization/solubilisation is larger.

Moreover, the curve useful to determine the number of risk events comes from the model of the materials behaviour during the change of the microclimatic conditions. Although the model is based on the experimental data, it remains laboratory data, and not data collected in Sala delle Asse.

Therefore, it is preferable to consider some additional possible events, as a wider span of the one previously defined, ranging±5% of the curve values, as the common sense of expertise suggests.

At the first step of the data processing, the authors filtered the picks and minimum values of T °C and RH in the first year of monitoring. Fig. 11 shows the colored area of the values, which are between 8 and 29 °C, 21 < RH < 91.5%.

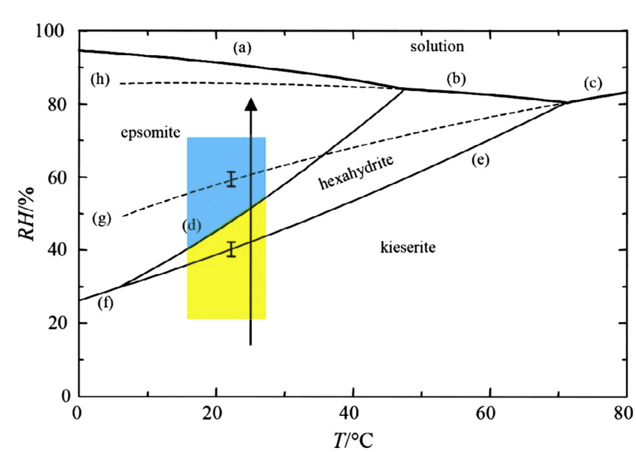


Fig. 17. Deliquescence behaviour in the system $MgSO_4$–H_2O. The relative humidity RH is plotted versus the temperature. The yellow strip indicates the span results of T °C and RH in Sala delle Asse between 2012 and 2015: the discussion of results will deal with the events occurring only at T > 21.5 °C (only at this condition, the phase is steady).

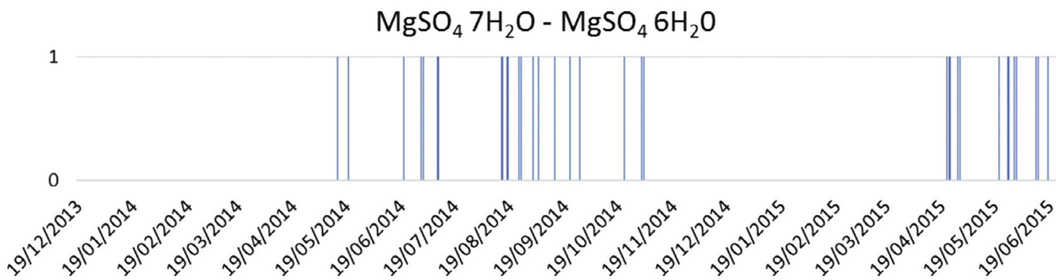


Fig. 18. Passage of phases of hexahydrite ($MgSO_4{\cdot}6H_2O$) and epsomite ($MgSO_4{\cdot}7H_2O$) at T °C > 21.5 °C (only at this condition the phase is steady), as an example this is the processed data acquired by probe n 1, between the Monochrome and the northwestern window.

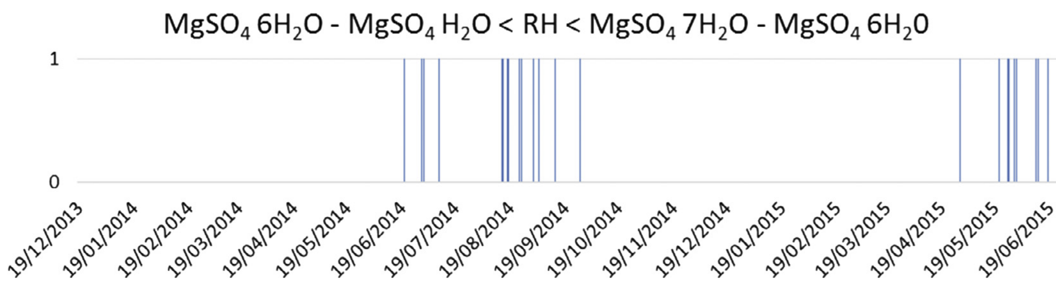


Fig. 19. Passage of phases of hexahydrite ($MgSO_4{\cdot}6H_2O$) and epsomite ($MgSO_4{\cdot}7H_2O$) at T °C > 21.5 °C, considering the points having $MgSO_4{\cdot}6H_2O$–$MgSO_4$ H_2O < RH < $MgSO_4{\cdot}7H_2O$–$MgSO_4$ $6H_20$; as an example, this is the processed data acquired by probe n 1, between the Monochrome and the northwestern window.

Major interest is in the phase transition between two values of T and RH, acquired in one hour, which belong to different sets (A solid, B liquid).

Moreover, the "buffer zone" is also visible (across the dotted lines) of ±5% of the known term of the crystallization line, that includes the points that should be events of the transition phenomena.

The graphic of Fig. 12 shows that the majority of the acquired points of probe n 2 are below the line of phase passage, (and below the security strip).

There are 261 points associated to the phase transition, localized in the critical area, often close to the critical events.

All 10 transition events are located between May and July, while the data of the critical strip are included between April and October.

Because the HVAC plant is never on, and the management and visiting procedures of the hall did not vary during the year under study, the authors concluded that the variations were caused by the exterior climate.

The comparison of the exterior and interior data shows that almost all the events occur for an increase of RH > 80% lasting for some hours.

On the contrary, there seem not to be connections when the variations are fast occuring. The calculations regarding the air-mixing ratio [2] are in Figs. 13–16.

The graphics show the values of mixing ratio with a similar trend: the peaks and major differences are in summer (SM black color) and at the end of the spring (SP). In fall (AU) and especially in winter (WN), the differences are lower.

These results agree with the localization of the critical events of salt crystallization, and confirm that the time of most frequent occurrence is between May and October.

The diagram of the magnesium sulphate phases (Fig. 17) shows the crystallization curves of all the magnesium compounds.

Because of the thermal dynamic instability of kieserite ($MgSO_4{\cdot}4H_2O$), the calculation of the changes of phase is limited to the upper line which divides light-blue and yellow areas, in the span of RH and T °C values resulting from the monitoring of the last 3 years, ignoring areas represented by metastable equilibrium (black dotted lines in the graphic).

The calculation of the events (crystallization and deliquescence) resulted in the following graphic. Fig. 18 shows that there are 60 changes of phase and 1217 points located in the buffer zone (±5%).

Despite of the high number of changes of phases, their occurrence is between April and October too, as shown for the nitratine. Moreover, because the instability of keyrsite prevents most of the changes to epsomite and hexahydrite, the researchers calculated the events only in the small area of the graphic where the changes between epsomite and hexahydrite occur [6]. From the calculation (Fig. 19) 242 critical points and 48 transitions across the black line in the small area result.

4. Conclusions

The investigations allowed so far ascertain that the prevention of further damage is strictly connected to the microclimatic control. The ongoing monitoring shows unbalances caused by exterior variations, especially during the summer when the main wind direction and speed affect the interior microclimate of the hall and nearby rooms. Therefore, the policy to prevent further damage regards the management of the hall, the improvement of the window frames and sealing any holes or cracks across the masonry to limit the exchange of air with the exterior, and obtain more even conditions inside the hall, especially close to the painted surfaces. The presence of magnesium sulphate phases is a worrying issue, especially for what concerns the interchange hexahydrite/epsomite. Although from the calculation, this did not result in high frequency of interchange; unfortunately very often in ancient times magnesium limestones or dolostones were used as a raw material in lime technology, therefore, it is impossible to completely eliminate magnesium from the surfaces and the substrate. Because of the lack of a certain source of water inside the masonry, the restorers plan to remove the salts from the surfaces by means of gel systems. The use of the gel is justified with the aim of confining the release of water in the outer layer of the plaster. On the contrary, soaked pads to extract the soluble salts could probably activate a dangerous dynamic of salt transport between the exterior and interior layers of plaster and masonry. The conservation plan for the future maintenance of the precious surfaces will take into consideration the restorers approach, and the further inspections will firstly check the critical areas where the salt content was higher, with the aim of early detection of any whitening of the surfaces before salt efflorescence will visually appear.

This case study stresses the importance of monitoring the contribution of RH in the interior environment, even in the case of almost dry masonry. The calculation of the air-mixing ratio was a very helpful tool to verify the influence of the exterior climate on the interior air conditions, together with the local measurements of wind speed and direction. The methodology used to connect microclimate variations and damage of the paintings was successful in identifying the main causes and planning an effective mitigation of their effects.

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