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ORIGINAL PAPER

An integrated study for mapping the moisture distribution in an ancient damaged wall painting

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Abstract An integrated study of microclimate monitoring, IR thermography (IRT), gravimetric tests and portable unilateral nuclear magnetic resonance (NMR) was applied in the framework of planning emergency intervention on a very deteriorated wall painting in San Rocco church, Cornaredo (Milan, Italy). The IRT investigation supported by gravimetric tests showed that the worst damage, due to water infiltration, was localized on the wall painting of the northern wall. Unilateral NMR, a new non-destructive technique which measures the hydrogen signal of the moisture and that was applied directly to the wall, allowed a detailed map of the distribution of the moisture in the plaster underlying the wall panting to be obtained. With a proper calibration of the integral of the recorded signal with suitable specimens, each area of the map corresponded to an accurate amount of moisture. IRT, gravimetric tests and unilateral NMR applied to investigate the northern wall painting showed the presence of two wet areas separated by a dry area. The moisture found in the lower area was ascribed to the occurrence of rising damp at the bottom of the wall due to the slope of the garden soil towards the northern exterior. The moisture found in the upper area was ascribed to condensation phenomena associated with the presence of a considerable amount of soluble, hygroscopic salts. In the framework of this integrated study, IRT investigation and gravimetric methods validated portable unilateral NMR as a new analytical tool for measuring in situ and without any sampling of the distribution and amount of moisture in wall paintings.

Keywords IR thermography · Unilateral NMR · Wall paintings · Gravimetric tests · Moisture

This paper is dedicated to the memory of Prof. Annalaura Segre, who contributed highly to the development of unilateral NMR for monitoring cultural heritage.

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Introduction

As shown in the scientific literature on conservation science [1–3], moisture is one of the major causes of decay of masonry materials such as stones, bricks, mortar and plaster which are to some extent porous. In particular, the deterioration of wall paintings results from the open porous nature of their support, that is brick/stone masonry and plaster, and their interaction with the environmental conditions and microclimate. The porous mortar backing may provide a route for the flow of salt solutions which can be easily transported to the plaster underlying the painting. When these salts crystallize, the associated volumetric expansion may seriously affect the adhesion between the pictorial film and the plaster and the adhesion among the plaster layers, leading to the disintegration of the surface. Processes such as leaching, transport, accumulation, solute precipitation and fractionation, and the presence of biological patina tightly depend on the moisture content of the masonry, which is subject to seasonal variation and environmental conditions. Therefore, an accurate diagnosis of the cause and extent of the moisture is a fundamental step in the conservation of wall paintings [4]. Before any restoration or conservation treatment is performed, the amount and the distribution of the moisture in the plaster underneath the wall painting should be known. Nevertheless, the amount and the distribution of moisture within a wall painting is difficult to determine. The methods currently used for this determination are IR thermography (IRT) [5, 6], electrical conductivity [7] and gravimetric tests [8]. However IRT does not allow a quantitative evaluation of the moisture content, electrical conductivity may be affected by the presence of salts and gravimetric tests require the drilling of solid cores, which is highly forbidden in the case of precious artworks such as wall paintings. In particular, the major difficulty arises when the distribution of the moisture in large areas of a precious and ancient surface must be known.

In this paper, the study of the distribution and the amount of moisture in a very damaged wall painting located in San Rocco church, Cornaredo (Milan, Italy), is reported. The church walls have wall paintings that are heavily damaged owing to moisture diffusion. The monitoring of the moisture was performed using an integrated approach of unilateral nuclear magnetic resonance (NMR) and IRT supported by the traditional analytic gravimetric methods [9–11].

IRT is a non-destructive, non-contact method of detecting, gathering and evaluating information about historic buildings. It is a telemetric method to detect the temperature distribution on a surface.

IRT is often used to identify and monitor the diffusion of moisture on/or near the surface of masonry walls by determining the correspondence between the actual distribution of moisture and the visual damage. The passive approach to IRT is to thermally scan the surface under investigation without the application of any direct heat, either artificial or direct from the sun. It is possible to map the moisture distribution in plaster using IRT because of the cooling effect of evaporation on wet surfaces at the steady-state condition. The evaporation process is highly energy consuming (1 g of evaporating water absorbs 2,500 J). Where the cooling effect on the surface occurs due to evaporation, it overwhelms differences in temperature due to other factors, including emissivity. The phenomenon depends on the air temperature, the relative humidity levels, the air movement and the presence or absence of direct solar irradiation. Although research is ongoing [9–13], current IRT procedures do not easily supply quantitative data for the water content in a structure, and only a qualitative approach can be extensively applied. Despite this constraint, knowledge of the actual water diffusion is extremely helpful for establishing the sources of infiltration, and in this framework IRT can be successfully applied to map wet areas. Nevertheless, the visual state of the damage often does not match the current moisture distribution because the liquid evaporates from the damaged surface, and frequently the diffusion of water is greater than is apparent from visual inspection. The simplest way to obtain a quantitative analysis of the moisture diffusion is to integrate IRT with other procedures, such as gravimetric tests and moisture probes, which directly measure the water content [8, 14].

A unilateral NMR instrument is fully portable, allowing measurements to be performed in situ and in a fully non-destructive way. In contrast to application of conventional unmovable NMR instruments, which requires a sampling of the object, the use of this instrument does not require any sampling, fully preserving the integrity and the dimension of the object to be investigated [15, 16]. Unilateral NMR has been previously used as an analytical tool for monitoring early degradation in cellulose-based materials [17–19] and for evaluating the efficiency of protective and/or consolidating processes performed on porous materials [20]. Recently, it has been shown that unilateral NMR may be also used as an analytical tool for assessing the state of conservation of wall paintings [21–23].

The integrated approach of different techniques reported in this paper allowed a comparison between the results obtained with the unilateral NMR technique and those obtained with currently accepted techniques such as IRT and gravimetric methods.

The study case

San Rocco church is located in Cornaredo. It was built in the fifteenth century, and the interior was frescoed during the period from 1451 to 1524 [24]. In the twentieth century, during the 1960s to the 1980s, the building decayed owing to the lack of any kind of maintenance. As a consequence, much damage occurred to the structures, especially to the roof and wall paintings. During the period from 1987 to 1993, a complete restoration of the church was performed. Nevertheless, the condition of wall paintings got worse and worse. In 2007–2009, the preservation officer in charge of the conservation of ancient monuments ordered emergency intervention on the wall paintings and the necessary investigation of the cause of the damage. Restorers secured the poor pictorial layers by applying nanometric slaked lime. Nanometric slaked lime consists of nanoparticles of calcium hydroxide dispersed in an alcoholic medium; these act as consolidators for limestone and painted surfaces affected by different kinds of decay. Provided previous restoration products are removed, these nanoparticles are highly efficient in strengthening frescoes and decorated stones [25].

Experimental

Microclimate monitoring

The monitoring of the environmental conditions was performed with a Lascar Electronics EasyLog USB-2 data logger (range of measurements 0–100% relative humidity, -25 to 80 °C; resolution 0.5% relative humidity; sensitivity ±3.5% relative humidity, ±2 °C).

IR thermography

IRT images were collected with an AVIO TVS 700 LW instrument, a focal plane array microbolometric device with a thermal sensitivity of ±0.08 °C at 20 °C.

IRT allows the surface area under investigation to be divided into zones in which the temperature and, consequently, the water content, is consistent, that is measuring the water content at just one point of that zone gives information regarding all the area at the same temperature. As a consequence, the number of measurements may be drastically reduced, making it possible to repeat the investigation in different periods to establish the relationship of the moisture to the building features and environmental and weather-related factors [12].

Gravimetric tests

The thermogravimetric method allows the measurement of the difference in the weight before and the weight after the elimination of water by forced evaporation.

A first set of gravimetric tests was performed using a Sartorius MA 45 thermobalance (sensitivity ±0.001 g). A second set of gravimetric tests was performed with a Mettler PM2500 balance (sensitivity ±0.001 g). A Heraeus RT360 stove (sensitivity ±1.5 °C) was used. A third set of gravimetric tests was performed with a Sartorius BL150S balance (sensitivity ±0.001 g). A Binder oven was used. Samples were collected by core drilling (diameter 18–20 mm), and the powders obtained were placed into glass holders. Samples were weighed and then placed in an oven at a temperature of 105 °C until a constant weight was achieved. The difference between the initial weight and the weight after drying, expressed as a percentage of the dry material, was the water content in the sample. The uncertainty on data obtained by gravimetric tests was calculated using error propagation theory and was about ±1%.

Unilateral NMR

All measurements were performed in situ with a unilateral NMR instrument from Bruker Biospin. The probe head of the instrument is made of a U-shaped magnet assembled with two permanent magnets mounted on an iron yoke and including a radio-frequency resonator [15, 16]. The probe head detects the hydrogen signal from the moisture at a selected depth inside the wall. Because the magnetic field is inhomogeneous, the signal decays very quickly and, consequently, it must be recovered stroboscopically [16]. Accordingly, the signal intensity or integral is actually the intensity or integral of the signal resulting after application of a Hahn echo pulse sequence [26, 27]. Because the strength of the magnetic field penetrating the object decreases with the distance from the surface of the probe head, measurements can be performed at different depths in the object under investigation.

Measurements were performed using a probe head operating at 16 MHz and allowing the measurements within a slice of the plaster at a depth between 0.45 and 0.55 cm from the surface, fully disregarding the signal from the surface. The pulse width corresponding to the $\pi/2$ pulse was 4 µs and the dead time was 15 µs. In the Hahn echo pulse sequence, the echo time τ was set as short as possible (20 µs).

The integral of the NMR echo signal was calculated using a routine for numerical integration. With this routine the integral is obtained by summing the discrete amplitude values sampled under the echo.

Results and discussion

Microclimate monitoring

The microclimate monitoring over 1 year (November 2007 to December 2008) showed that the relative humidity was always very high between November and February, months in which the air temperature gradually decreased until the dew point and below [28]. It was also shown that external climatic conditions affected the microclimate inside the church only under the circumstance that these conditions were constant for at least 24–36 h. For instance, sharp and quick variations due to sudden air movement outside the church did not cause variations of the relative humidity inside the church. Moreover, owing to the location of the church (Padana plain), the humidity of the air is often high in any season. Another monitoring of the microclimate was performed in the period from December 16th 2008 to January 29th 2009. In Fig. 1 the daily average temperature and the daily average relative humidity measured in that period inside and outside the church are shown. During this period an external temperature between –2 and 8 °C and an internal temperature between 0 and 8 °C were recorded, whereas the external relative humidity varied between 80 and 98% and the relative humidity inside the church varied between 75 and 85%.

IRT and gravimetric tests

The IRT investigation was used to obtain qualitative information quickly on the distribution of moisture in the walls of San Rocco church. The composite thermograms

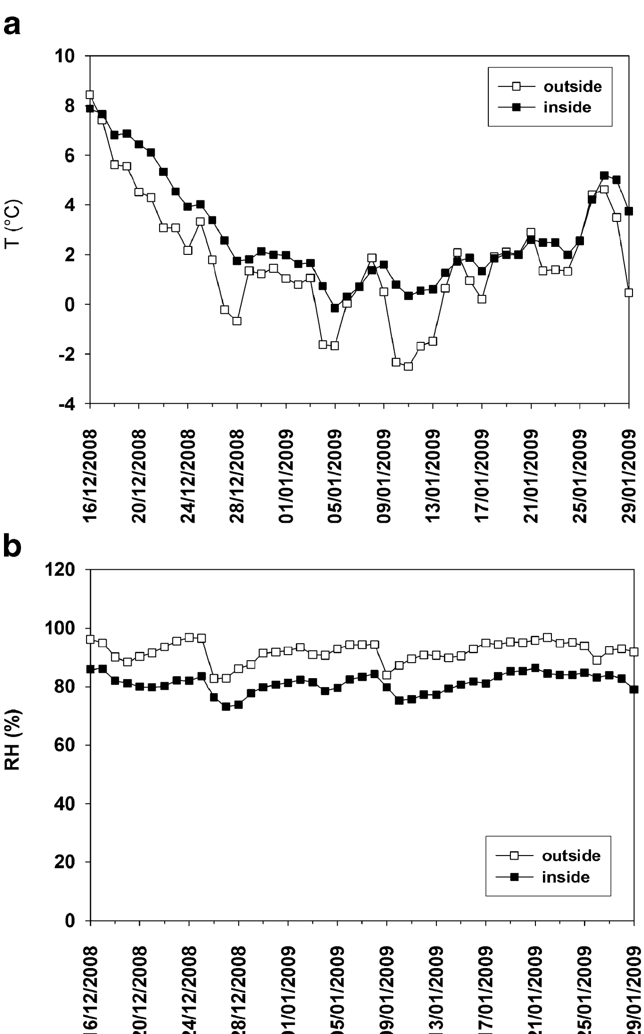

Fig. 1 Daily average temperature (**a**) and daily average relative humidity (RH; **b**) measured in the period from December 16th 2008 to January 29th 2009 inside and outside San Rocco church

obtained on the northern wall on April 16th 2008 and those obtained on October 24th 2008 are shown in Fig. 2. In Fig. 2a the presence of a thermal gradient of the surface temperatures is easily observable; the dark areas indicate lower temperature (at the basis of the masonry), which was higher after rainfall. Because evaporation of the surface moisture occurred during the summer, in the image captured on October 24th, after about 3–4 months without rain (Fig. 2b), the surface showed a lower thermal gradient (less than 1 °C) than that observed in the thermogram shown in Fig. 2a collected on April 16th (about 2 °C). Nevertheless, the location of the dark areas changed, and Fig. 2b shows a reduction of the blue strip at the base of the masonry, and dark stains popped up in the upper part of the image.

The survey of thermal anomalies of the surface allowed the choice of the points on the walls where the drilling of solid cores would be performed to obtain the samples for the gravimetric tests. For the sake of clarity, the locations of the points chosen (4.1, 4.2, 4.3) are also reported on the composite thermogram shown in Fig. 2a. Three samples were collected from the surface of the masonry (plaster) of the northern wall, and another three samples were collected at a depth of 20 cm inside the masonry (brick). A first set of samples was collected on April 16th 2008 (Table 1), and another set was collected on October 24th 2008 (Table 1).

The collection of powders from the interior layers of the masonry allowed the measurement of the water content of the materials constituting the structure (bricks and mortar) without any affect of microclimatic variations.

With the aim of finding out the source of water infiltration, for the samples collected at the bottom of the northern wall it was necessary to distinguish between the water content due to rain and the water content due to the water table by repeating the measurements before and after many weeks of heavy rainfall. It was found that the water content increased after a period of heavy rainfall. Therefore, rainfall had an important role in the rising damp occurring at the bottom of the northern wall. The cause of water entering the structure was localized where rainwater coming from the gutter piping was not adequately collected into a shaft.

The results obtained by IRT and gravimetric tests as well as the damage assessment performed by the restorers on the whole building showed that the worst damage was localized on the northern wall. Whereas the other sides of the church masonry were almost dry, in the northern wall a high water content was measured at the bottom of the wall, up to 60 cm from the floor and higher than 180 cm from the floor. In the horizontal strip between 60 and 180 cm from the floor, the water content was very low (below 1.5% of the dry weight); see also Table 1. The IRT investigation also clearly showed that the surface temperature of the interior northern side was lower during clear days, in any

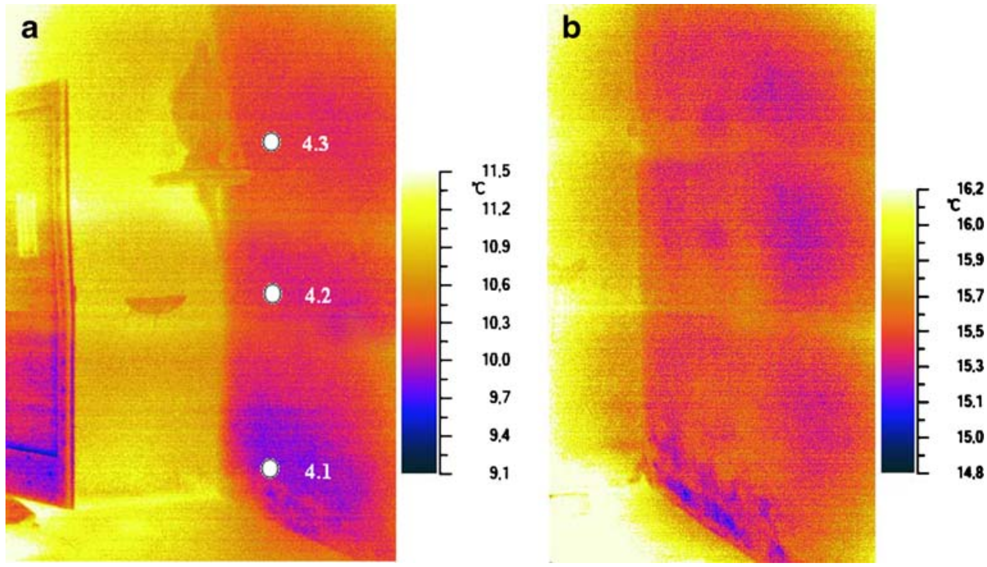

Fig. 2 Composite thermograms obtained on April 16th 2008 (**a**) and on October 24th 2008 (**b**). The locations of the points drilled to obtain samples for gravimetric tests are reported in **a** as *circles*

season, because of the lack of direct solar irradiation. Besides, the results of physicochemical tests [29] allowed the ascertainment of the presence of soluble salts, mainly sodium nitrate, especially at an elevation of 180–190 cm from the floor.

All these results pointed out the possible occurrence of condensation phenomena during the winter, early spring and late autumn, which caused the solubilization of salts and their spreading on the surface. Moreover, the high relative humidity could cause the spreading of salts which were in/on the surface, although the surface temperature was higher than the dew point (as calculated by the Mollier diagram), according to the scientific literature [30–32].

Unilateral NMR

Detailed knowledge of the distribution and the amount of moisture in the plaster underlying the wall painting on the northern wall of San Rocco church was obtained using unilateral NMR [33–35]. The campaign of measurements was carried out in the period from January 26th to January 31st 2009. With this equipment, the magnetic field is applied to one side of the object. As an example, the instrument while performing measurements on the northern wall painting in San Rocco church is shown in Fig. 3. As previously shown [20–23], the integral of the NMR echo signal is proportional to the water content.

Measurements were performed on a matrix of points chosen on the painted wall, each point being labelled with (x,y) coordinates. It is worth noting that each point actually covered an area of 10 cm^2, corresponding to the area detected by the probe head. The use of a purposely built grid gently leaning against the wall helped us to correctly label each area of measurement; see Fig. 4a. It was possible to measure many points covering almost the whole area of the wall panting. After the measurements had been performed, the data were processed to obtain a contour plot, which is a 2D representation of a 3D surface, where x and y were the coordinates of the measured area of the painted wall and z was the integral of the NMR echo signal. Given a value for z, lines were drawn to connect (x,y) coordinates where the z value occurred. Before the contour plot was drawn, the data were smoothed using a fraction of 20% of the total number of data points to compute each smoothed value. A Gaussian weight function and a quadratic fit were used to weight the data and a fourth-degree polynomial function was applied to the weighted data to compute each smoothed value [36, 37]. The contour

Table 1 Water content expressed as a percentage of dry material obtained by gravimetric tests performed on samples collected on April 16th 2008 and October 24th 2008 on the surface of the northern wall (plaster) and at a depth of 5 cm (brick)

Sample	Depth (cm)	Water content (%)		Materials
		April 16th 2008	October 24th 2008	
4.1	0	4.0	2.2	Plaster
4.1	5	8.6	7.9	Brick
4.2	0	1.4	1.4	Plaster
4.2	5	0.4	0.7	Brick
4.3	0	4.0	6.0	Plaster
4.3	5	4.5	5.4	Brick

Fig 3 Unilateral NMR instrument while measuring the moisture content of the northern wall in San Rocco church

plot obtained allowed easy visualization of the detailed distribution of the moisture in the wall painting; see the map shown in Fig. 4b. In this map the difference in the moisture level is represented as a gradient of colour: dark red indicates the lowest moisture content, whereas dark blue indicates the highest moisture content.

At a preliminary screening, the map showed a large dry area between 40 and 100 cm from the floor, whereas definitely more wet areas were observed at heights lower than 40 cm and higher than 120 cm; see Fig. 4b. The map confirmed the results obtained by previous IRT investigations and gravimetric tests. However, a successive step was necessary to associate with each point of the map an accurate amount of moisture. With this aim, the integral of the NMR echo signal was calibrated using a purposely designed analytical procedure.

Analytical procedure for calibrating the integral of the NMR echo signal

A calibration of the integral of the NMR echo signal was performed using three specimens obtained by drilling solid cores in the wall painting where the painted film was

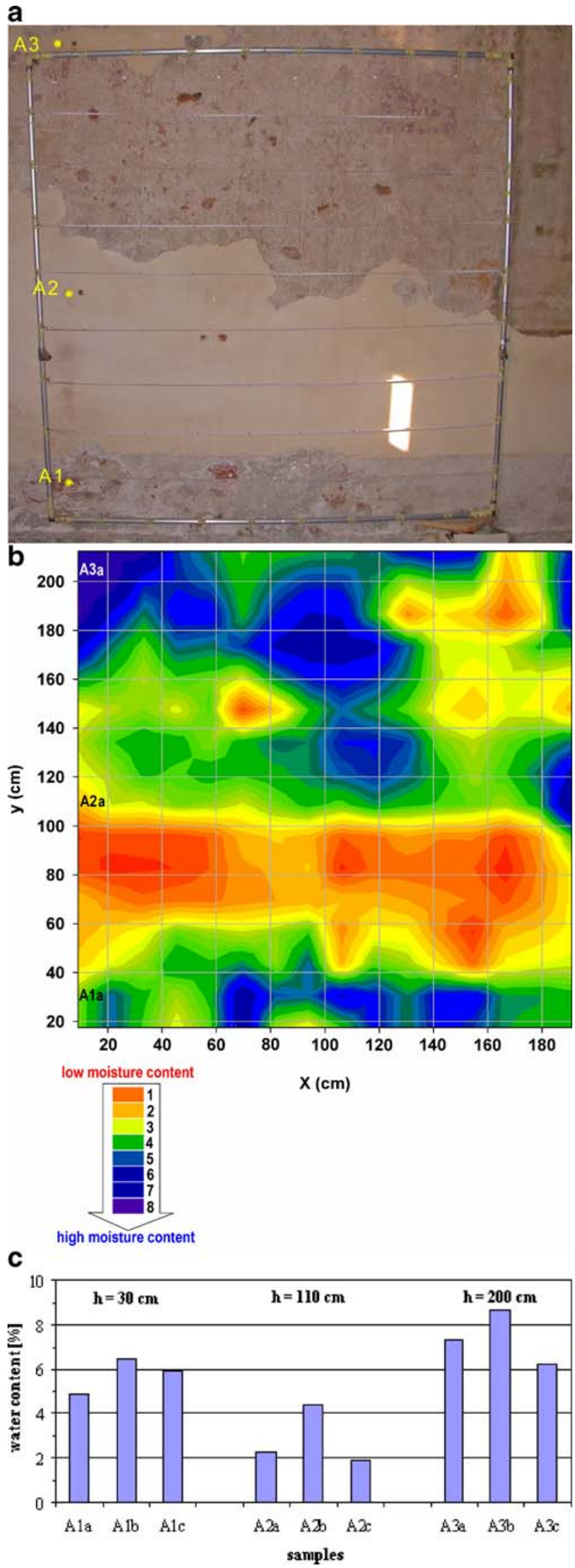

Fig. 4 **a** Northern wall painting with the grid gently leaning against the wall. The grid was used as a reference for labelling the areas of measurement. The locations of the drilling points on the northern wall painting were chosen at approximately 55 cm from the left corner at an elevation of 30 cm (*A1*), 110 cm (*A2*) and 200 (*A3*) cm from the floor. **b** Map of moisture distribution obtained by unilateral NMR. A gradient of colour was used to represent differences in moisture content: dark red was associated with a very low moisture content, whereas dark blue was associated with a high moisture content. The points drilled to obtain powders for gravimetric tests are reported as *A1a*, *A2a* and *A3a*. At the bottom, the scale of values of the moisture content obtained by calibrating the integral of the NMR echo signal is reported. **c** Moisture contents obtained by gravimetric tests reported as a percentage of dry material and represented as histograms

missing. The specimens were dried until they reached a constant weight Ps_d and were measured by unilateral NMR. As_d is the value of the integral of the NMR echo signal measured on the dried specimens. Then the specimens were fully saturated with water by total immersion according to the NORMAL protocol [38] up to constant weight Ps_w. As_w is the value of the integral of the NMR echo signal measured on the fully saturated specimens.

The imbibition coefficient ic, defined as the maximum amount of water adsorbed by the specimen compared to its dry weight, was calculated:

$$\mathrm{ic} = \frac{Ps_w - Ps_d}{Ps_d} \times 100.$$

The imbibition coefficient was 18.5%. Accordingly, a scale from 0 to 18.5% was used to calibrate the level of moisture in the wall painting investigated. The correlation between the integral of the NMR echo signal and the weight of the adsorbed water is summarized in Eq. 1:

$$M_i(\mathrm{NMR}) = (A_i - A_{\min})\left(\frac{\mathrm{ic}}{As_w - As_d}\right), \quad (1)$$

where A_i is the integral of the NMR echo signal measured on the wall painting and A_{min} is the lowest value of the integral measured on the wall painting.

According to this calibration procedure, each area of the contour plot map reported in Fig. 4b corresponded to an accurate amount of moisture. In particular, the maximum value of the integral of the NMR echo signal measured on the wall painting corresponded to about 8% moisture content. The maximum error on the M_i(NMR) values was calculated using error propagation theory and it was always lower than 10% of the nominal value.

Unilateral NMR and gravimetric tests

After the detailed moisture map had been obtained by unilateral NMR, two more sets of gravimetric tests were performed on January 30th, which was in the same period during which the NMR measurements were performed. The aim of this procedure was to check the results obtained with the new analytical non-destructive unilateral NMR technique applied directly on the wall painting with the gravimetric tests, which are well-known analytical tests to probe the amount of moisture, but which are destructive because they require the wall to be drilled.

The location of the drilling points on the northern wall is reported in Fig. 4a. Samples were collected at approximately 55 cm from the left corner of the wall at an elevation of 30 cm (A1), 110 cm (A2) and 200 cm (A3) from the floor. From every drilling point, three samples were collected: from the surface of the wall (A1a, A2a, A3a), at a depth of 5 cm (A1b, A2b, A3b) and at a depth of 20 cm (A1c, A2c, A3c). Two laboratories independently performed the measurements: Lab1 performed the measurements on samples collected at the surface of the northern wall and at a depth of 20 cm, whereas Lab2 performed the measurements on samples collected at the surface and at a depth of 5 cm. The moisture contents, reported as the percentage of the dry material, are shown in Table 2 and are also shown as histograms in Fig. 4c. The results clearly evidenced the presence of two wet areas at 30 cm and 200 cm of height, separated by a dry area at 100 cm.

Table 2 Water content expressed as a percentage of dry material obtained by gravimetric tests performed on January 30th 2009 by Lab1 (samples collected at the surface of the northern wall and at a depth of 20 cm) and by Lab2 (samples collected at the surface of the northern wall and at a depth of 5 cm)

Sample	Depth (cm)	Water content (%)		Materials
		Lab1	Lab2	
A1a	0	4.6	4.8	Plaster
A1b	5	–	6.5	Brick
A1c	20	5.9	–	Brick
A2a	0	2	2.3	Plaster
A2b	5	–	4.4	Brick
A2c	20	1.9	–	Brick
A3a	0	7.5	7.3	Plaster
A3b	5	–	8.7	Brick
A3c	20	6.2	–	Brick

The amount of moisture obtained by unilateral NMR and the amount of moisture obtained by gravimetric tests performed in the same areas are compared in Table 3. Because NMR measurements were performed at a depth of 0.45–0.55 cm, the comparison was done with the results of gravimetric tests obtained on the samples drilled at the surface of the wall. The values of the moisture content found at points A1a, A2a and A3a by gravimetric tests (Lab1 and Lab2) and by non-destructive unilateral NMR are in very good agreement; see Table 3.

Table 3 Comparison of the water content expressed as a percentage of dry material obtained by non-destructive unilateral NMR (depth between 0.45 and 0.55 cm) and by gravimetric tests performed by Lab1 and Lab2 on samples collected at the surface of the northern wall

Sample	Depth (cm)	Water content (%)			Materials
		Unilateral NMR	Lab1	Lab2	
A1a	0	5.3	4.6	4.8	Plaster
A2a	0	2	2	2.3	Plaster
A3a	0	7.3	7.5	7.3	Plaster

Conclusions

An integrated study of microclimate monitoring, IRT, gravimetric tests and portable unilateral NMR was applied in the framework of planning emergency intervention on a very deteriorated wall painting in San Rocco church. As is well known, detailed knowledge of the distribution and of the amount of moisture in plaster is mandatory before any restoring intervention.

The microclimate monitoring of the internal and external conditions of San Rocco church over more than 1 year strongly suggested that only in a few late spring/summer weeks the microclimate conditions may ensure the good conservation of the surface of the wall paintings.

The survey of thermal anomalies of the surface obtained by IRT investigation led to the choice of the points where the wall was to be drilled to obtain samples for gravimetric tests. The IRT investigation supported by gravimetric tests showed that the worst damage due to water infiltration was localized on the northern wall of San Rocco church, whereas at the other sides of the church the masonry was almost dry. Nevertheless, IRT did not supply quantitative and detailed information on the amount of moisture in the plaster underlying the wall painting. Quantitative information was obtained by drilling solid cores at a few, selected points on the wall. However, because drilling of the wall is very destructive, it was only done at very few points, carefully avoiding the painted areas.

A campaign of measurements was performed on the northern wall painting of San Rocco church using unilateral NMR, a new, non-destructive technique applied directly on the wall painting. With this technique a detailed map of the distribution of the moisture in the plaster underlying the fresco was obtained. A suitable calibration of the integral of the NMR echo signal allowed a quantitative determination of the moisture content. According to this procedure, each area of the map corresponded to an accurate amount of moisture. The results obtained by unilateral NMR were in very good agreement with the results obtained by gravimetric tests.

To summarize, IRT, gravimetric tests and the unilateral NMR technique applied to investigate the northern wall painting in San Rocco church showed the presence of two wet areas separated by a dry area. The moisture found in the lower area was ascribed to rising damp at the bottom of the wall due to the slope of the garden soil towards the northern exterior and the inadequate routing of rainwater from the gutter pipe. The moisture found in the upper area was ascribed to possible condensation phenomena associated with a marked presence of soluble, hygroscopic salts.

Finally, in the framework of this integrated study, IRT investigation and the well-known and accepted gravimetric methods validated portable unilateral NMR as a new analytical and non-destructive tool for measuring in situ the distribution and the amount of moisture in wall paintings as well as in any porous material.

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