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Unilateral NMR study of a XVI century wall painted

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Abstract

Wall paintings in the XVI century Serra Chapel in the "Chiesa di Nostra Signora del Sacro Cuore" Rome, have been studied using unilateral NMR. In order to map the distribution of moisture content in the wall painted, a large number of Hahn echo measurements, covering large areas of the wall painting were performed. Because the intensity of the Hahn echo is proportional to the amount of moisture in the area under study, the experimental data were transformed into 2D gradient colour maps which allowed an easy visualization of the moisture content of the wall.

The state of conservation of the wall painting was monitored using T_2 measurements specially with regards to outcropping salt.

Keywords: Unilateral NMR; Hahn echo; Moisture map of wall paintings; T_2 relaxation time distribution; Cultural Heritage

1. Introduction

In all areas of Cultural Heritage conservation there is an ever increasing focus on the interdisciplinary approach. This is particularly true for wall paintings, where indeed a number of special factors must be taken into account: their particular vulnerability, with respect to the extremely thin painted surface, which is itself the interface between the support, i.e. the plaster, and the surrounding environment; the difficulty involved in controlling potential agents of deterioration such as moisture and pollution; the proximity of crowded areas to most wall paintings. Moreover monitoring and/or restoring a wall painting is always complicated, expensive and requires extremely specialized and cultured people, the scale of intervention being usually large and performed under environmentally difficult conditions [1,2]. The preservation planning of wall paintings requires a precise knowledge about the microclimatic conditions of the painted walls both inside and outside. Changes of weather, humidity, variation of temperature, polluting agents, bacterial attack all affect the painted surface adhesion to its supporting wall. Temperature and humidity act as shearing forces between layers or portions of a wall, weakening the material and causing the appearance of micro-cracks, detachment and anomalous strains on the surface. Moreover plaster needs permanently the right amount of moisture to guarantee adhesion between it and the pictorial film. When this equilibrium is altered crumbling may occur leading to the destruction of the painting [2].

The evaluation of the moisture within the wall paintings is also a difficult issue. Usually the techniques used with this aim in mind are electrical conductivity [3], IR thermography [4,5] and also, in an highly destructive way, the drilling of a solid core necessary to measure directly the moisture content. All these methods are affected by intrinsic restrictions: electrical conductivity measurements can be very misleading due to the presence of salts which can affect the measurement giving too high values for the moisture level; the thermographic investigations can be affected by temperature and by the heat of evaporation which produces cooling effects. Even nowadays the drilling of a solid core used for direct moisture measurements is so destructive that it cannot be used on precious wall paintings.

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The moisture content measured directly on the wall painting is an important measurement which can be performed in a non invasive way using unilateral NMR and can give useful information to the restorer [6,7].

In a previous paper [8] we showed that unilateral NMR is a suitable non-invasive technique for assessing the state of conservation of wall paintings. In fact using unilateral NMR it is possible to evaluate the effect of consolidation treatments [8,9] and to monitor the detachment of the painted layer from its support, the plaster.

Another of the most common causes of degradation of wall paintings and building materials is the salt outcroppings. Salts can damage porous materials by chemical reactions or crystallization. Conventional NMR measurements have been performed to study the crystallization process in porous materials with well-defined pore size [10]. However we have used the unilateral NMR to observe the effect of salt outcropping performing *in situ* measurements of T_2 spin–spin relaxation times [8]. In fact the presence of hygroscopic salts on painted surface affects the T_2 spin–spin relaxation times.

This Article reports how the unilateral NMR can be used to monitor the effect of the rising moisture on a wall of an ancient church. In particular the results obtained on the wall paintings in the Serra Chapel, XVI century, in the Chiesa di Nostra Signora del Sacro Cuore in Rome are reported. The church located in Navona square, in the center of Rome, was built around 1450 B.C.; the Serra Chapel was built years later, during the Renaissance, between 1517 and 1519, and it was *frescoed* by Pellegrino degli Aretusi, a co-worker of Raphael. On the left side of the Serra Chapel wall paintings show a marked degradation with loss of the pictorial film, while on the right side they are in a much better state of conservation. On the left side of the chapel the main cause of the degradation of the wall paintings is thought to be due to the moisture rising from the ground.

The unilateral NMR study of the degraded wall paintings along with a suitable processing of the experimental data allowed the map of the distribution of moisture content of the whole wall to be drawn up. This map is useful to determine the source of dampness and to intervene with the aim of removing the excessive moisture content. Hahn echo [11] measurements in different regions of the wall were performed for this purpose, and, since the Hahn echo intensity is proportional to the amount of water, the moisture content distribution of the whole wall painted surface was obtained. Due to the high moisture content found in the degraded wall, it was possible to perform a large number of measurements covering large areas of the wall painting. After elaborating the experimental data, the Hahn echo data were directly transformed into a 2D gradient color map, such as those used in MRI [12]. Using this representation, the water content of different areas of the wall painting is shown as color gradients.

Note that, both in the bottom as well as in the top wall paintings of the left side of the chapel, wide areas show a noticeable salt outcroppings. According to a method previously outlined [8,9] T_2 measurements were also performed on several areas, with and without salt outcroppings, to evaluate the presence of hygroscopic salts in a fast and non-invasive way.

2. Experimental

2.1. Unilateral NMR relaxometry

Unilateral NMR has been used to monitor the state of conservation of the wall paintings on the left side of the Serra chapel, see Fig. 1. All measurements "*in situ*" were performed with a commercial unilateral NMR "ProFiler" from Bruker Biospin, which is a variant of NMR-MOUSE [6,7,13–15].

Two probe-heads were used; the 1 mm-probehead, operating at 18.153 MHz, allows the measurement on a ≈1 mm thick slice of "sample", hence the measurement is performed at a depth of 1 mm from the surface; the 3 mm-probehead, operating at 17.3 MHz allows the measurement on a slice of the "sample", i.e. the plaster, at a depth between 2.5 and 3.5 mm from the surface. The maximum echo signal corresponding to a $\pi/2$ pulse was obtained with a pulse width of 3 μs for the 1 mm- probehead, whereas for the 3 mm-probehead the pulse width for obtaining the $\pi/2$ pulse was 6 μs. The dead time was 15 μs in both cases.

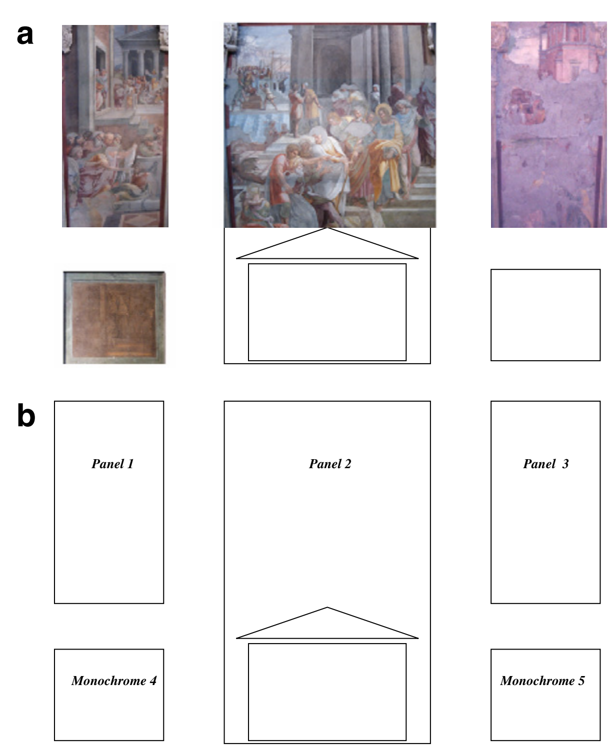

Fig. 1. (a) Details of the wall paintings of the left side of the Serra Chapel. (b) In order to allow an easy description of the different areas of the painted wall, they are referred to as labelled Panels and Monochromes.

To perform the measurement, the probe-heads were positioned very near to the painted wall, but never in contact with it.

The moisture content in the wall painting was obtained by performing single Hahn echo [11] measurements with an echo time of 20 μs, 2 K of scans. The data were collected using the 1 mm-probehead.

The salts outcropping was studied measuring the spin–spin relaxation time T_2 with the CPMG sequence [16,17] acquiring 2048 echoes; starting echo time 100 μs, $2\tau = 100$ μs, 32 K scans. T_2 measurements were performed using both the 1 mm-probehead and the 3 mm-probehead.

2.2. Analysis of relaxometric NMR data

Echo decays obtained applying the CPMG sequence were treated as multi-exponential decays:

$$Y = C_0 + \sum_i W_i \exp\left[\frac{-t}{T_{2i}}\right] \quad (1)$$

where i is the number of components, C_0 is the offset value, W_i is the spin density of the i component and T_{2i} is the spin–spin relaxation time of the i component.

2.3. Relaxation time distribution of multi-exponential decays

The distribution of relaxation times was obtained by numerical inversion of the Fredholm integral using a software implemented within the Matlab (The Math Works) framework [18,19]. In the resulting distribution, the abscissa provides the relaxation time value and the integral corresponds to the normalized spin density. The algorithm used to obtain the inversion of the experimental data was based on Regularized ILT and was previously tested on simulated and experimental data [18]: no artefacts were observed in the obtained distribution for signal/noise higher than 10.

2.4. Contour plot for mapping the moisture content of wall paintings

In order to measure the moisture content on the whole surface of the wall painting, single Hahn echo measurements were performed choosing a matrix of "points" on the painted surface. Actually, each experimental "point" covers an area of about $2 \times 5\ \text{cm}^2$ that corresponds with the area of the probehead. Due to the high signal to noise ratio it was possible to perform a large number of measurements covering almost the whole area of the wall painted. In particular, see Fig. 1b, on Monochrome 4, Panel 1 and Panel 3 a matrix of 25 "points", each one covering an area of $2 \times 5\ \text{cm}^2$, was measured, while on Panel 2, due to the larger dimensions of this wall painted, a matrix of 30 "points" was measured. Therefore, the investigated area in the case of Monochrome 4, Panel 1 and 3 was $25 \times 2 \times 5\ \text{cm}^2$, whereas in the case of Panel 2 was $30 \times 2 \times 5\ \text{cm}^2$. Afterward a contour plot was created applying an algorithm for smoothing sharp variations of the dependent variable values within the 3D data set. The contour plot was obtained using the elaborated data derived from the experimental ones. Here x and y are the coordinates of the strip surface of the wall painting and z is the intensity, in arbitrary units, obtained from the Hahn echo measurement. The graphic representation was obtained within the framework of the Sigmaplot 8.0 software. A contour plot is a graphical way for representing a 3-dimensional surface by plotting constant z slices, called contours, on a 2-dimensional format. That is, given a value for z, lines are drawn for connecting the (x, y) coordinates where z value occurs.

The smoothing method weighted the data contained in a window surrounding the smoothing location. A fraction of 40% of the total number of data points was used to compute each smoothed value. The weight assigned to each data value in the window was determined by its normalized distance (u) from the smoothing location. A Gaussian weight function and a quadratic fit were used to weight the data. A non-linear function (4th degree polynomial) was then applied to the weighted data for computing each smoothed value [20–22].

3. Results and discussion

3.1. Distribution map of the moisture content

With respect to other painted artifacts, wall paintings show a peculiar behavior which is due to their high and open porosity, in fact they constitute an essentially open physical system [2]; in other words their pores can easily intercommunicate among themselves, with the plaster and with the external environment. Therefore there is a wide specific surface exposed to degrading agents with a strong permeability to all fluids which may come in contact with the painted surface. Usually these fluids are liquids, that is water, but also diluted salt solutions acting directly on the painted wall, and/or gases, present in the form of water vapors or as other pollutants. As a consequence, even after any restoration treatment, wall paintings remain open systems in contact with the adjacent structures. Hence the wall painting takes part in the water dynamic of the whole system. For instance, in most cases contact with the ground or with the adjacent walls provides an efficient source for capillary percolation of moisture. This is the case of the Serra Chapel where the left side of the chapel is in a very bad condition due to the moisture rising from the ground. This high level of moisture is also ascribed to previous old restoration works, which caused evident alterations of the color of the wall paintings. Moreover a white marble socle (the socle is a projecting usually molded member at the foot of a wall or pier or beneath the base of a column, pedestal, or superstructure), used for decorating the lower part of the wall, does not allow any humidity exchange with its external surrounding environment, thus making the painted surface the only possible interface of exchange from the inner to the outer wall structure for the rising moisture.

The Monochrome 4, see Fig. 1b, is one of the most deteriorated wall paintings of the chapel. Due to the high stress produced by the crystallization of the salt outcroppings on the painted surface and the repeated cleaning procedures, the painting is hardly visible, having almost fully disappeared.

As previously reported [23,8,9] the intensity of the Hahn echo is proportional to the water concentration. Therefore, with the Hahn echo measurements and with a proper processing of the experimental data, it is possible to obtain a distribution map of the moisture content in the wall painting. In this wall painting, due to the high moisture content, it was possible to perform a large number of Hahn echo measurements. In particular, the Hahn echo measurements were performed on a matrix of 25 "points".

To show the NMR data, a contour plot representation was chosen, that is a two dimensional representation of a three dimensional surface graph, where x and y are the coordinate of the strip of the area of the painted surface and z is the intensity of the Hahn echo measurement. In this way the contour plot is used to create the distribution map of the moisture content in the wall painting.

The distribution map of the dampness obtained for Monochrome 4 is shown in Fig. 2. The difference in moisture content is shown with a gradient color: red indicates a very low water content, while dark blue indicates the highest water content. The map obtained shows the high moisture content found in Monochrome 4: the dampness rising from the ground is diffused throughout the wall painting, and the highest moisture content is located near the center of the wall painting.

Moving up to Panel 1, see Fig. 1b, the distribution map of moisture, see Fig. 3b, is completely different with respect to the one obtained in Monochrome 4. The intensities of Hahn echoes are definitely lower than those obtained in the case of Monochrome 4: as a consequence, in this part of the wall painting, i.e. Panel 1, a lower water content was found.

The distribution map of moisture regarding the wall paintings of Panel 2, see Fig. 4b, shows a water content definitely higher than that obtained in Panel 1, but lower than the one obtained in Monochrome 4. Finally, in Fig. 5 the moisture map of Panel 3 is reported. The water content of Panel 3 seems quite similar to the one obtained in Panel 2. To summarize, the Hahn echo measurements provide a detailed information regarding the distribution of moisture content of the whole painted wall of the left side of the chapel (see Fig. 6). This map is exactly what restorers need.

3.2. Salt outcroppings

Due to the high level of moisture, wide areas with salt outcroppings are present on the left side of the Serra Chapel both in the bottom as well as in the top wall paintings. Moreover over the centuries, the practice of fixing the pigments and preventing the crumbling of the pictorial films

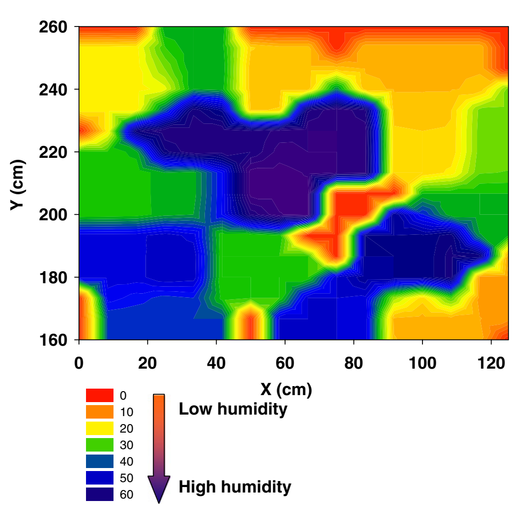

Fig. 2. Distribution map of the moisture obtained for Monochrome 4. In this representation x and y are the coordinates of the strip of the area of the painted wall and z is the intensity in arbitrary units, of the measured Hahn echo. A gradient of colour is used for representing the water content: the red is associated with a very low water content, while the blue color is associated with an high water content. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

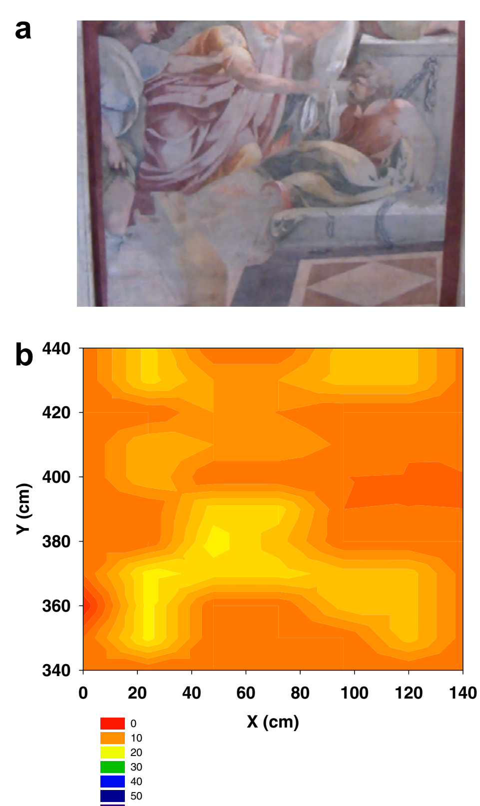

Fig. 3. (a) Detail of the wall painting of Panel 1 measured by unilateral NMR. (b) Distribution map of the moisture obtained for Panel 1.

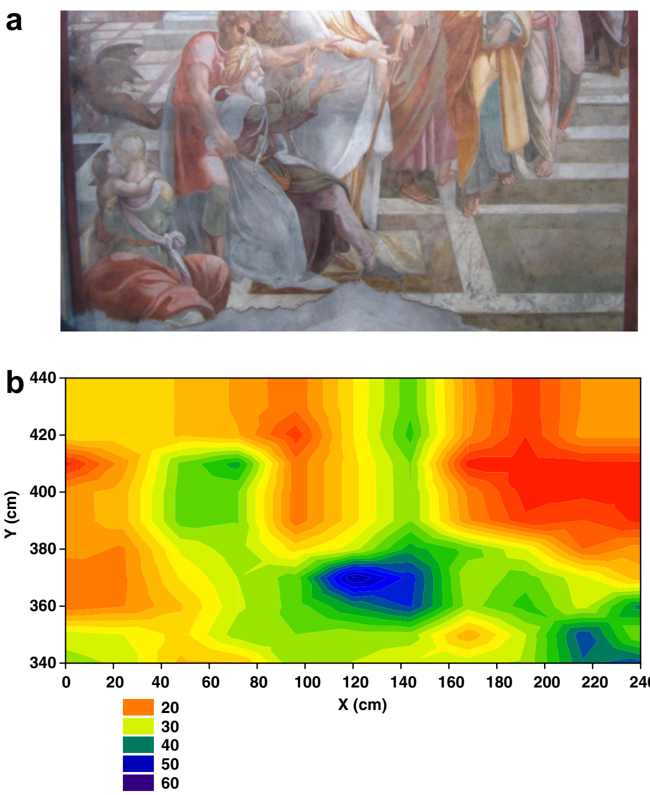

Fig. 4. (a) Detail of the wall painting of Panel 2 measured by unilateral NMR. (b) Distribution map of the moisture obtained for Panel 2.

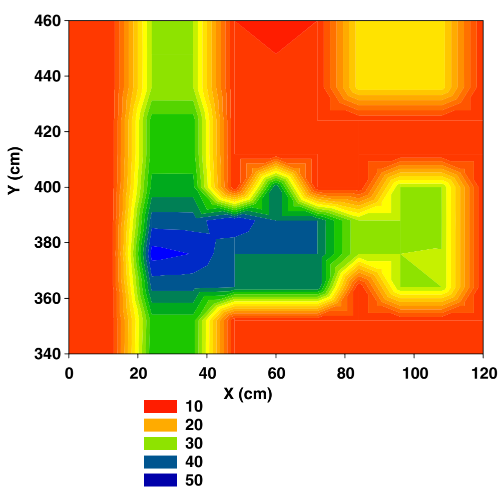

Fig. 5. Distribution map of the moisture obtained for Panel 3.

was a common procedure consisting in the application of a mixture of several biological substances such as egg-yolk, animal glue, wax and linseed oil; this practice worsened the situation. These organic substances changed the porosity and the permeability of wall paintings, consequently, the crystallization and the successive hydration of the salt outcroppings occurred under the fixative film. Salt outcroppings under these conditions damaged the pictorial film producing detachment and micro-fractures. To evaluate the presence of hygroscopic salts and their effect, T_2 measurements were performed in wall painting regions showing salt outcroppings as well as in regions free of salt outcroppings. In particular, T_2 measurements were performed both on the bottom panels as well as on the top ones, and in several regions of Monochrome 4. The T_2 distributions obtained by inverting the T_2 decays are shown in Figs. 7a, b and c, whereas the measured regions are shown in Fig. 7d.

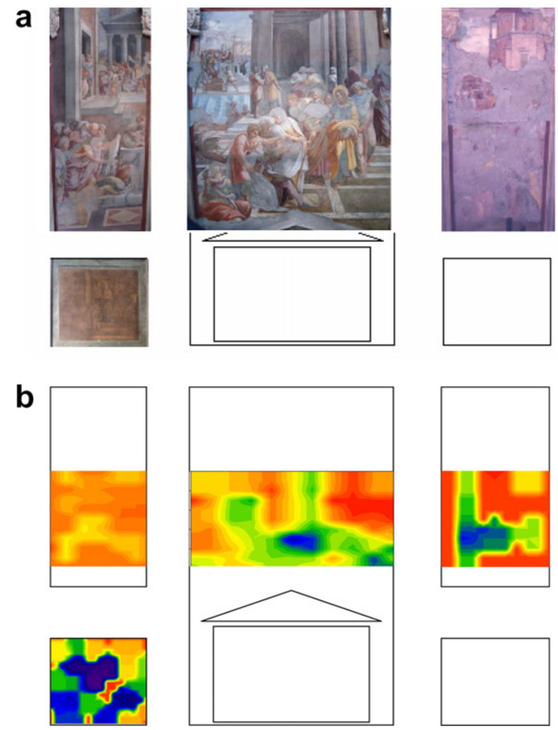

Fig. 6. (a) Details of the wall paintings of the left side of the Serra Chapel. (b) Distribution map of the moisture of the painted wall of the left side of the chapel.

In Fig. 7a the comparison between the T_2 distribution measured in region 1, with salt outcroppings, and the distribution measured in region 2, free of salt outcroppings, is reported. Region 2, free of salt outcroppings, was cleaned with water in previous restoration works (nine months before these measurements). As previously reported [8] in the presence of salt outcroppings the average T_2 value shifts to longer values. This effect can be attributed to the moisture adsorbed by the hygroscopic salts.

In Fig. 7b the T_2 distributions of region 2 and of region 4 are compared. Both these regions are free of salt outcroppings and were cleaned with water nine months before the measurements. Region 4 shows a higher moisture content, with three T_2 decays. One of these T_2 decays is rather slow with an average value of about 5 ms. It is well known that relaxation times, in particular spin–spin relaxation time T_2, depend on the degree of confinement of a fluid (water) within a porous structure [24]. Because the T_2 decay rate depends on the surface-to-volume ratio, water in small pores relaxes rapidly, whereas water in large pores relaxes more slowly [25]. The measurements of T_2 of a fluid

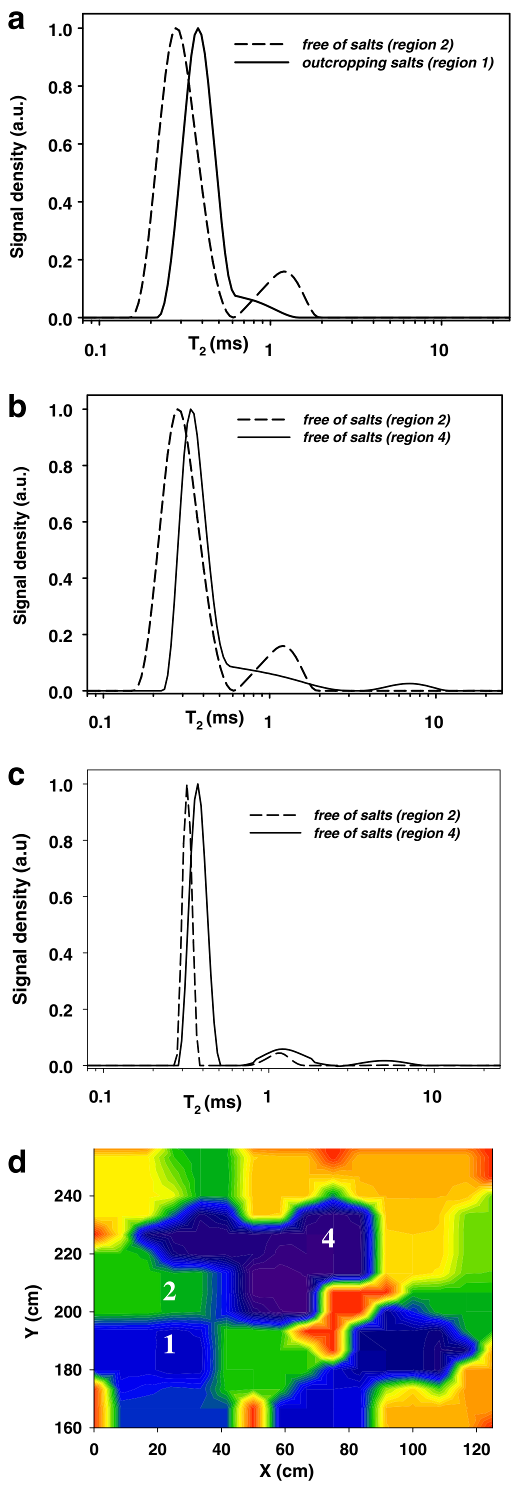

Fig. 7. (a) T_2 relaxation time distributions obtained by inverting CPMG relaxation decays measured in region 1 (with salt outcroppings), and region 2 (free of salts), of the wall painting of Monochrome 4. (b) T_2 distributions of region 2 and region 4 of the wall painting of Monochrome 4, both regions are free of salts. (c) T_2 distributions of region 2 and region 4 of the wall painting of Monochrome 4. The CPMG measurements were performed using the 3 mm-probehead. (d) Distribution map of the moisture obtained for Monochrome 4, the numbers correspond to the measured regions.

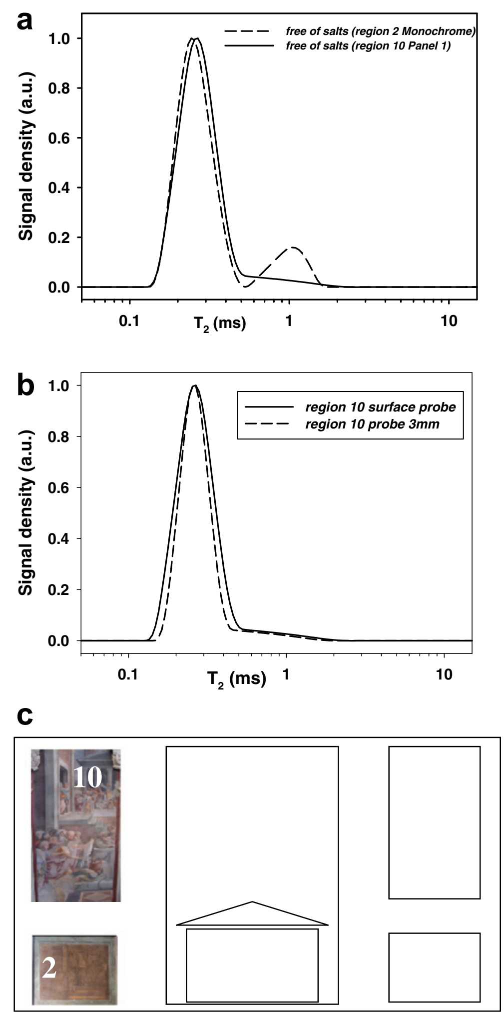

Fig. 8. (a) T_2 distributions obtained by inverting CPMG relaxation decays measured in region 2 of the wall painting of Monochrome 4 and region 10 of the wall painting of Panel 1, both regions are free of salts. (b) Comparison between T_2 distributions of region 10 of the wall painting of Panel 1 measured with 1 mm-probehead and with 3 mm-probehead, respectively. (c) Details of the wall paintings of the left side of the painted wall of the chapel where regions 2 and 10 are shown.

confined in a porous matrix [26] allows the pore size distribution and the effect of consolidation treatments on the capillary water absorption properties to be studied [23]. Therefore the slower T_2 decay found in region 4 is in accordance with the distribution map of moisture reported in Fig. 7d being region 4 the one with the highest moisture content and the longest T_2 values. It must be pointed out that we do not direct correlate T_2 distribution with the pore size distribution. In fact due the magnetic field produced with the unilateral NMR devices is rather inhomogeneous. Therefore, the T_2 spin–spin relaxation time is influenced by molecular self-diffusion [27]. Moreover the direct correlation between the T_2 distribution and the pore size distribution can be done only in the case of pores saturated with water; in this case or more generally in the Cultural Heritage field this condition is usually not fulfilled.

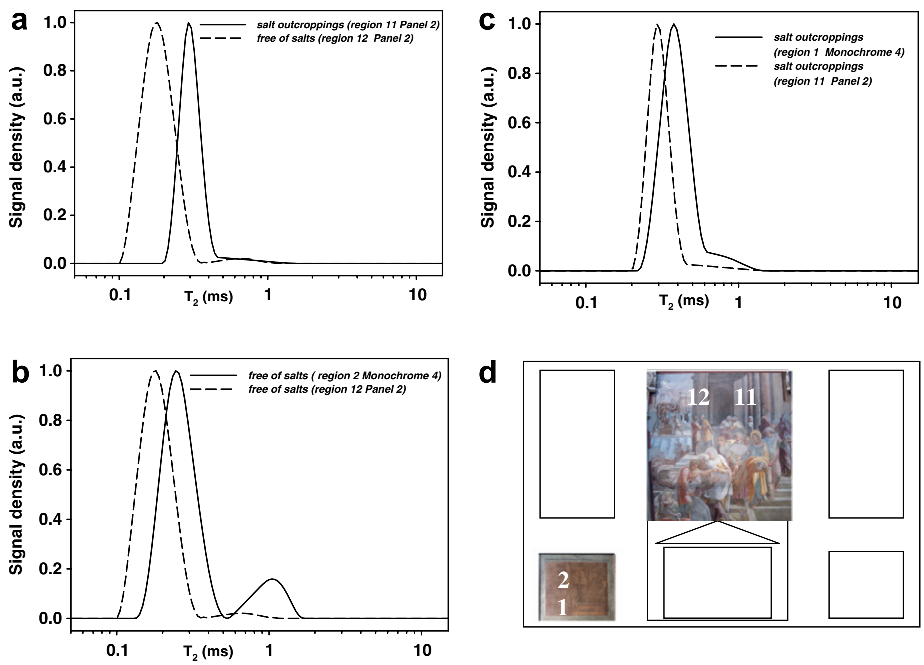

Fig. 9. (a) T_2 distributions of region 11 (with salt outcroppings) and of region 12 (free of salts) of the wall painting of Panel 2. (b) T_2 distributions of region 2 of the wall painting of Monochrome 4 and of region 12 of the wall painting of Panel 2, both regions are free of salts. (c) T_2 distributions of the region 1 of the wall painting of Monochrome 4 and of region 11 of the *fresco* of Panel 2, both regions show salt outcroppings. (d) Details of the wall paintings of the left side of the painted wall of the chapel where regions 1, 2, 11 and 12 are shown.

The T_2 distribution found in region 2 and in region 4 obtained with the 3 mm-probehead, are reported in Fig. 7c. The distributions are rather similar to those obtained using the 1 mm- probehead (surface probehead), see Fig. 7b. The T_2 distribution indicates the amount of small water aggregates is about the same as that one found in the plaster at a depth of 3 mm with respect to the distribution measured near to the surface.

In Fig. 8a the T_2 distribution found in region 2, belonging to the wall painting of Monochrome 4, and the distribution found in region 10, belonging to the wall painting of Panel 1, are compared. Both the regions shown in Fig. 8c, are free of salt outcroppings. In accordance with the dampness distribution map, in region 10 the amount of large water aggregates is definitely lower than that found in region 2 of Monochrome 4. In Fig. 8b the T_2 distributions found in region 10 on Panel 1, obtained by performing the measurements with the 1 mm-probehead and the 3 mm-probehead, respectively, are compared. The distributions are very similar in both cases, with a low amount of large water aggregates.

The T_2 distributions found on Panel 2 and the comparison between the T_2 distributions found on Panel 2 and on Monochrome 4 are shown in Fig. 9. The measured areas are shown in Fig. 9d. In Fig. 9a the T_2 distributions of region 11 with salt outcroppings and of the region 12, free of salt outcroppings, are compared. Again, as previously observed in the case of Monochrome 4, see Fig. 7a, in the presence of salt outcroppings the T_2 distributions shift to longer values. Note that the amount of large water aggregates in the wall painting of Panel 2 is lower than that of the wall painting in Monochrome 4. The T_2 distributions found in region 2 of Monochrome 4 are compared with the distribution found in region 12, Panel 2, both free of salt outcroppings, a definitely lower amount of large water aggregates is found in the latter case (see Fig. 9b). A similar trend is obtained by comparing the T_2 distributions found in region 11, Panel 2, with that found in region 1, Monochrome 4, both with salt outcroppings, see Fig. 9c.

4. Conclusion

A non-invasive investigation of the state of conservation of ancient wall paintings was performed using unilateral NMR analysis. The method allows a good characterization of the water content on the painted walls. A 2D representation of the moisture data is proposed allowing an easy to follow representation of the moisture content of the painted wall.

T_2 distributions obtained at different depths are sensitive to the wall painting porosity and to the presence of salt outcroppings.

The state of art of the distribution of moisture before and after any restoration process is a valuable information essential to the restorer. The *in situ* investigation above described and the proposed processing of the experimental data, can be considered as a general method for mapping the moisture on wall paintings.

Acknowledgments

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