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Neutron tomography for the assessment of consolidant impregnation efficiency in Portuguese glazed tiles (16th and 18th centuries)

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ABSTRACT

Neutron tomography (NT) has been applied to visualize the inner structure of ancient Portuguese glazed tiles undergoing conservation treatments. Neutrons have the advantage of interacting strongly with hydrogen, so NT is able to map hydrogenous compounds with high sensitivity. The present study explores its potential for assessing the distribution of the consolidant Paraloid B-72 inside tiles, to evaluate the efficiency of two different methods of treatment: brushing and immersion in solution. Using a prototype NT setup at the Reactor Português de Investigação (Sacavém, Portugal), each two-dimensional image is obtained from a 90 s exposure, at a thermal neutron flux of 2×10^5 n cm^{-2} s^{-1} at the irradiation site. The neutron beam has a diameter of 5 cm, so fragments with outer dimensions up to 4.8 cm can be inspected. Samples are automatically rotated by an angle of 0.9° between successive images. Images were obtained before and after the application of the consolidant. The results obtained show that: (i) NT is a useful tool for visualization of the inner structure of ancient glazed tiles, and to assess penetration depth of consolidant and its distribution inside the tile; and (ii) brushing with 10% Paraloid® B-72 in acetone solution appears to be more efficient than immersion. Neutron tomography showed a greater and more uniform retention of resin inside the tile if the brush is used to apply the consolidant, to increase the cohesion of the object.

1. Introduction and research aims

Neutron imaging techniques have been applied to visualize the inner structure of cultural objects (archaeological and historical including museum artworks) to help understand manufacturing processes and preservation/degradation state (Deschler-Erb et al., 2004; Lehmann et al., 2005b, 2010a, 2010b).

Glazed tiles ("azulejos") have been manufactured in Portugal for many centuries and are present in many historic buildings of the 16th to the 19th centuries. These "azulejos" often present degradation/alteration features, including disintegration of the ceramic fabric. Consolidants are widely used in restoration and conservation interventions as a means of improving the structural strength of several materials such as stones and ceramics. Consolidation is an artificial way of repairing the bonds that normally hold the object together. A good consolidant should meet performance requirements concerning durability, depth of penetration, effect on porosity, effect on moisture transfer, compatibility with the object material, reversibility and effect on appearance (Clifton, 1980).

For tiles and other porous materials, the consolidant should have sufficient stability and penetration that the microstructural and mechanical properties of the tile will be improved, but must not seal the surface being treated: this must remain permeable to any existing humidity in the fabric, as well as protecting against further deterioration (Fleischer et al., 2005). Acrylic polymers and co-polymers are used as protective coatings due to their non-wettability, chemical inertness and environmental stability (Carreti and Dei, 2004). These properties have lead to the extensive use of these polymeric materials as consolidants and/or protectors for the conservation of artworks made of stone, ceramics, glazed tiles, wood and other porous material (Farinha Antunes, 1992; Ribeiro Carrott et al., 1997; Carvalho et al., 2006; Vaz et al., 2008; Constâncio et al., 2010).

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Penetration is a key factor in any treatment with a consolidant. Visualization of penetration depth and consolidant distribution inside a ceramic body enables conservators to assess the effectiveness of a treatment. The application of neutron tomography (NT) is highly appropriate for these purposes, since neutrons penetrate ceramic materials very efficiently, while their strong interaction with hydrogen can be used to detect and map hydrogen-rich compounds, such as acrylic polymers (Lehmann et al., 2005b; Kaestner et al., 2008). Regarding cultural assets, neutron tomography has been applied to visualize and quantify conservation measures for several types of porous materials including different types of stone (Kaestner et al., 2008; Masschaele et al., 2004; Cnudde et al., 2004; Hameed et al., 2009) and wood (Lehmann et al., 2005a).

In the present work NT is applied to visualize the inner structure of glazed tiles and explore its potential to assess the distribution of conservation products, namely Paraloid® B-72, inside the samples. The consolidant product is an acrylic co-polymer of ethyl methacrylate and methyl acrylate (70/30). Two types of Portuguese glazed tiles from the 16th and 18th centuries with distinct characteristics (glaze, ceramic body and mortar) were selected for this study. Identification and semi-quantification of mineral phases of the ceramic body was made by X-ray diffraction (XRD), to aid interpretation of the neutron absorption characteristics of the different sample materials.

The objectives of the present study were to 1. develop a visualization tool for the inspection of consolidant penetration in ancient glazed tiles, based on neutron tomography using the Portuguese Research Reactor, and 2. to use this to evaluate the efficiency of two methods of treatment with the consolidant Paraloid® B-72: brushing and immersion in solution. The overall aim is to contribute to the establishment of the best strategy for conservation of this type of cultural object.

2. Materials and methods

2.1. Samples, consolidant and treatments

Two fragments of Portuguese glazed tiles were chosen for study (Fig. 1): (a) sample NSA – a polychrome glazed tile of the 18th from the "Nossa Senhora da Conceição dos Aflitos" Church (Elvas, Portugal) and (b) sample MNA of the 16th century – a green glazed tile from the "Madre de Deus" Church, "Museu Nacional do Azulejo" (Lisbon, Portugal). On visual inspection sample MNA presents a serious state of degradation, with glaze detachment and exfoliation of the ceramic body; sample NSA does not present cohesion problems and mortar is still attached on the rear.

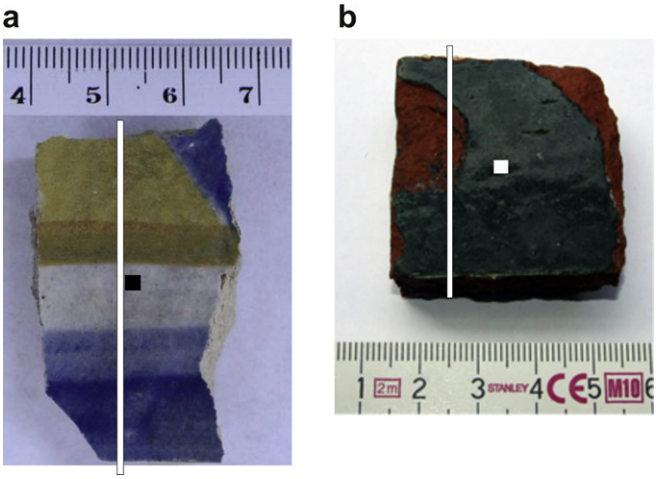

Fig. 1. Tiles fragments used in the present work: a- "Nossa Senhora dos Aflitos" Church, Elvas (sample NSA); b- "Madre de Deus" Church, Museu Nacional do Azulejo, Lisbon (sample MNA). Lines indicate the locations of the sections shown in Figs. 4 and 5, squares indicate the locations of the line profile analyses.

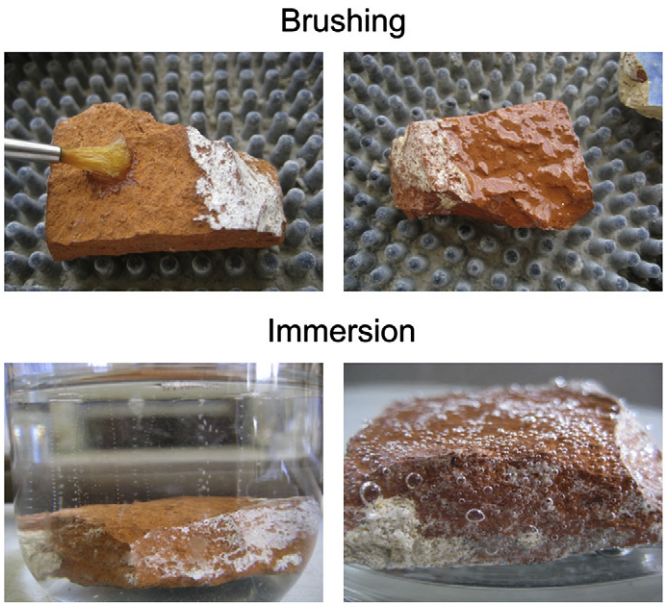

Fig. 2. Photographs of the brushing and immersion techniques for the application of resin in the MNA sample.

Consolidation treatments of ancient glazed tiles were conducted at the "Museu Nacional do Azulejo" following standard protocols.

Mortar attached to the rear of the tiles is partially removed using a scalpel and spatula: carbonate rich mortars are not removed completely due to the elevated risk of fracturing or damage the ceramic body of the tile. High reactivity solvents are avoided in conservation treatments, as well as those of strong or long retention. The solubility of dirt of the ceramic body of a sample is first tested in order to verify that the conservation treatment will not have a detrimental effect. At the "Museu Nacional do Azulejo" this is normally conducted using a 1:1 solution of distilled water and ethanol with a few drops of a neutral detergent.

In the case of dusty or friable ceramic paste it may be necessary to perform consolidation. This is conducted using Paraloid® B-72 (co-polymer of ethyl methacrylate and methyl acrylate and), a commercially available acrylic adhesive known for its stability and reversibility. It is dissolved in acetone (1:9) to produce a low viscosity solution that may be applied by brush or immersion (Fig. 2). To facilitate penetration by the solution, the piece is first soaked in solvent (acetone). Immersion is conducted in a hermetically sealed container to avoid increase in the viscosity of the solution due to evaporation of the solvent. After some hours the piece is removed and left to dry at room temperature. The film of resin that forms on the surface of the ceramic glaze during drying is removed using cotton soaked in acetone.

Table 1
Characteristics of the neutron beam at the irradiation position at the Reactor Português de Investigação (Sacavém, Portugal).

Neutron flux at the irradiation position	2.2×10^5 n cm^{-2} s^{-1}
Beam diameter	5 cm
Mean energy	25 meV
Spatial resolution (μm)	323 (11)

Fig. 3. Images of the neutron tomography (NT) setup of the Portuguese Research Reactor (RPI): A. general view of the RPI, with the NT setup (middle, bottom); B. NT setup; C. detail of the neutron window and the rotary table with the MNA tile sample.

2.2. X-ray diffraction

Mineralogical analysis of the ceramic body of the tiles was performed by X-ray diffraction (XRD) of non-oriented aggregate powders using a RX Philips diffractometer, CuKα radiation at 45 kV and 40 mA, a step size of 1° 2θ/min from 2 to 70° 2θ. The mineral proportions were determined by semi-quantitative XRD diagnosis of peak areas, according to procedures proposed by Rocha (1993).

2.3. Neutron tomography

The tomography equipment is installed at the horizontal beam – port of the thermal column of the 1 MW nuclear research reactor (RPI) at the Instituto Tecnológico e Nuclear (ITN) with a thermal neutron flux of about 10^{13} n cm^{-2} s^{-1} near the reactor core. The thermal column is a graphite stack of 90 cm between the core and the port (Fernandes et al., 2010). Inside the beam hole, a collimator provides a parallel neutron beam at the irradiation position. Its main characteristics are shown in Table 1.

In the present system the scintillator is a 0.42 mm thick NDg type scintillator (Applied Scintillation Technologies, 2000), based on ^{6}LiF/ZnS co-doped with Cu, Al and Au coupled to a cooled CCD camera (Proline – Finguer Lakes Instrumentation), equipped with a Kodak KAF-1001E (Eastman Kodak Company, 2001) grade 1 CCD, with 1024 × 1024 pixels (24 × 24 μm size). A Nikon 50 mm/f1.4 lens was coupled to the CCD camera and is normally used in the f/1.4 aperture.

A tomography is obtained according to the following procedures: the object is positioned on a rotary table and it is irradiated in the neutron beam; the image formed in the scintillator is captured by the video camera and stored in a computer; an electro-mechanical interface connects the table, the computer and the camera in a such way that at the end of the capture, the object is rotated and a new image is captured (Fig. 3). The stored images are used by the software Octopus to perform image reconstruction and, the software VG Studio provides a three dimensional view of the internal structure of the object. The system is managed by a PC equipped with an Intel Xeon 2.4 GHz processor, with 4 Gb of RAM, and dual redundant hard disk drives. The Fig. 1 shows pictures of the installed setup (Stanojev Pereira et al., 2010).

Neutron tomography of the NSA and MNA samples was performed in the following order: (i) untreated; (ii) brushed with 10% Paraloid B-72 in acetone, and dried at room temperature; (iii) immersed in 10% Paraloid B-72 in acetone and dried at room temperature. Resin was removed from the fragment after the image collection following the brushing treatment, by soaking in acetone, before the immersion technique was applied.

3. Results and discussion

3.1. Mineralogical composition (ceramic body)

The mineralogical compositions obtained by XRD of the ceramic bodies showed that sample NSA is mainly composed of quartz, wollastonite, gehlenite and similar amounts of feldspars and anatase (Table 2). The main mineral phases present in the ceramic body of sample MNA were quartz and phyllosilicates (micas), plus small amounts of calcite and feldspar. These results point to different firing temperatures: sample MNA was fired at lower temperatures (significant amounts of mica) than sample NSA (absence of phyllosilicates and significant amounts of wollastonite and gehlenite).

Table 2
Semi-quantification obtained by XRD of the minerals present in the ceramic body of the two glazed tiles – NSA and MNA (values are given in percentage).

	Quartz	Calcite	Phyllosilicates	K-feldspar	Plagioclase	Wollastonite	Gehlenite	Anatase
NSA	37	–	–	10	7	17	21	10
MNA	47	8	40	2	3	–	–	–

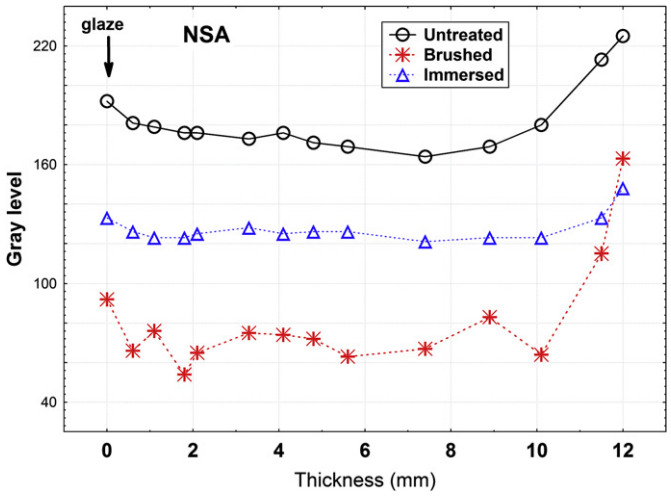

Fig. 4. Gray level variation across the NSA sample (glazed tile from Nossa Senhora dos Aflitos Church, Elvas, Portugal) obtained by neutron tomography: Untreated; Brushed and Immersed correspond to the tile after brushing and immersion in 10% Paraloid® B-72 in acetone, respectively.

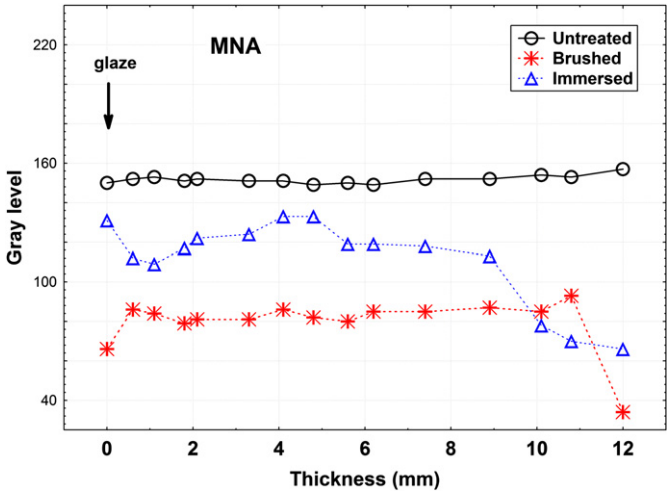

Fig. 5. Gray level variation across the MNA sample (glazed tile from Madre de Deus Church, Lisbon, Portugal) obtained by neutron tomography: Untreated; Brushed and Immersed correspond to the tile after brushing and immersion in 10% Paraloid® B-72 in acetone, respectively.

The mortar attached on the rear of sample NSA, not entirely removed during the treatment at the Museum, is mainly composed of feldspars, calcite, quartz, amphibole and gypsum.

3.2. Neutron tomography

The moving average of gray level through the center of the main face of each glazed tile (see Fig. 1), from the glaze to the rear, obtained by the "line profile" tool of VG Studio software, is shown in Figs. 4 and 5 for samples NSA and MNA, respectively. Each point corresponds to a volume of 6.27 mm^3 (13,216 voxels). The mean values and standard deviations of the gray level in five sections of the line profile are given in Table 3, together with the percentage variation of the gray level after immersion and after brushing, relative to the untreated sample.

The lower gray level found in untreated sample MNA when compared with NSA (see Figs. 4 and 5 and Table 3) can be explained by higher hydrogen content in the MNA ceramic body. The transformations of colloidal mineral grains that are inferred to have occurred in sample NSA, during high temperature firing, are expected to produce a significantly reduced capacity for hydration or water adsorption when compared to sample MNA, in which a significant amount of phyllosilicates (micas) were observed. High temperature phases (wollastonite and gehlenite) were observed in sample NSA and phyllosilicates were not detected (see Table 2).

The gray level across the NSA tile is higher in the outer parts, particularly in the rear of the ceramic body. This is a consequence of lower neutron attenuation in the outer parts of the tile and indicates higher porosity. The contrast is particularly clear between the ceramic body and mortar, where this is present. The same general tendency in the line profile was found before and after consolidant application using both brushing and immersion, but lower gray levels were observed following treatment, particularly by brushing (see Fig. 4). The lower gray level found after consolidant application is mainly due to the increase of the light elements inside the sample, particularly hydrogen. The results show that impregnation and retention of the resin inside the tiles was ~30% higher for brushing than for immersion, except at the rear faces where the final quantity of resin is similar. The application of the resin has no significant effect on the difference in porosity between the mortar and the ceramic body of sample NSA, as deduced by NT.

The gray level across the tile of the untreated MNA sample was approximately constant (Fig. 5). The brushing treatment produced higher neutron attenuation across the tile, but particularly close to the glaze and in the rear where the resin appears to have been more

Table 3
Mean values and standard deviations of gray levels in sections across the line profiles (from the glazed surface to the rear) obtained by NT of NSA and MNA glazed tiles. The line profiles in each fragment are indicated in Fig. 1.

NSA sample		Depth (mm)	Gray level (GL)						GL variation	
			Untreated (U)		Immersed (I)		Brushed (B)		Immersed (I − U)/U × 100	Brushed (B − U)/U × 100
			Mean	σ	Mean	σ	Mean	σ	c, %	c, %
Glaze		0–1.1	184	7.0	127	5.1	78	13.1	−31	−58
Ceramic body	Frontal	1.8–4.1	175	1.5	125	2.1	67	9.8	−29	−62
	Central	4.8–7.4	168	3.6	124	2.9	67	4.5	−26	−60
	Rear	8.9–10.1	175	7.8	123	0.0	74	13.4	−30	−58
Rear surface (mortar)		11.5–12.1	219	8.5	141	10.6	139	33.9	−36	−37
MNA sample		Depth (mm)	Untreated (U)		Immersed (I)		Brushed (B)		Immersed (I − U)/U × 100	Brushed (B − U)/U × 100
			Mean	σ	Mean	σ	Mean	σ	c, %	c, %
Glaze		0–1.1	152	1.5	117	11.9	78.7	11.0	−23	−48
Ceramic body	Frontal	1.8–4.1	151	0.5	124	6.7	81.8	3.0	−18	−46
	Central	4.8–7.4	150	1.4	122	7.2	83.0	2.4	−19	−45
	Rear	8.9–10.1	153	1.4	96	24.7	86.0	1.4	−38	−44
Rear surface		10.8–12	155	2.8	68	2.8	63.5	41.7	−56	−59

Table 4
Resin mass in each in each 6.27 mm^3 across the line profile obtained by NT of NSA glazed tile.

NSA		
Depth (mm)	Brushed (mg)	Immersed (mg)
0.0038	1.09	0.543
0.6278	1.46	0.498
1.0958	1.24	0.555
1.8173	1.74	0.577
2.1098	1.46	0.475
3.2798	1.20	0.429
4.0598	1.33	0.504
4.8398	1.22	0.432
5.5418	1.46	0.437
7.3943	1.35	0.475
8.8958	1.05	0.480
10.066	1.50	0.578
11.548	0.858	0.702
12.113	0.841	0.615

concentrated. After immersion, the gray level inside the tile was more variable. From the glaze to the rear there is a lower gray level in the first 2 mm after the glaze, followed by an increase up to the middle part, and a significant decrease in the rear (Fig. 5). These results point to higher concentration of the resin inside the tile using the brushing technique (~25% higher).

Quantitative estimates of resin mass in each volume slice (1.85 × 1.85 × 1.85 mm) represented by the points in Figs. 4 and 5 are presented in Tables 4 and 5. These were obtained from the density of resin required to produce the observed difference, after and before treatment, between spatially averaged gray scale index values for a given slice. Attenuation in each slice was calculated using a gray scale index range defined as 255 for no sample to 0 for total absorption, and the thermal neutron mass attenuation coefficient for hydrogen was assumed to be representative for the resin (Von Der Hardt and Roettger, 1981).

The differences found in the rear surface of the two tiles (see Figs. 4 and 5) may be explained as following: (i) in the case of NSA sample, with mortar attached, there is a lower resin concentration when compared with the rest of the tile, particularly after brushing; this may be due to removal of the resin (formed on the surface of the tile during drying) using cotton soaked in acetone from the porous mortar, particularly in the brushed sample; (ii) in MNA sample, where no mortar exists, the neutron attenuation of the rear part is similar to the rest of the tile. However after treatments there

Table 5
Resin mass in each in each 6.27 mm^3 across the line profile obtained by NT of MNA glazed tile.

MNA		
Depth (mm)	Brushed (mg)	Immersed (mg)
0.001	1.21	0.200
0.6	0.882	0.462
1.1	0.888	0.518
1.8	0.992	0.372
2.1	0.936	0.310
3.3	0.891	0.297
4.1	0.793	0.195
4.8	0.864	0.160
5.6	0.882	0.360
6.2	0.780	0.280
7.4	0.816	0.360
8.9	0.783	0.435
10.1	0.858	0.990
10.8	0.700	1.05
12.0	2.34	1.17

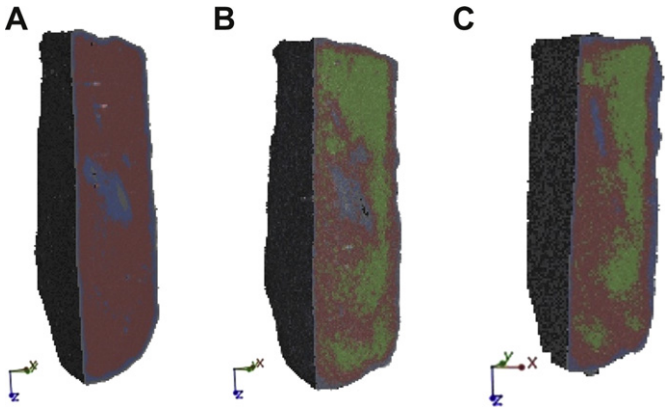

Fig. 6. NT images of NSA tile sample: A: before any treatment; B: after the impregnation of resin using brushing technique; C: after impregnation of resin using immersion techniques (red – ceramic body; blue – voids; green – resin). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

is a significant decrease of the gray level in the last 3 mm relative to the central body after the application of consolidant using immersion technique. When brushing is used the gray level is only lower in the outermost surfaces (1 mm).

These results point to a better efficiency of impregnation by brushing than immersion when using a 10% Paraloid® B-72 in acetone solution. This can be explained by a slower and deeper impregnation of the polymer inside the porous material applying the brush, and a more uniform retention after the application procedure. In the immersion technique, the lower retention of the polymer may be explained as follows: even impregnation of the resin may occur during immersion, but a significant loss may occur when the tile is removed from the acetone solution, or a concentration occurs in the outer parts of the object as it drains/dries, blocking further out-flow of the solution. A high-density distribution of Paraloid B-72 5% on the surface of stones was also found by Hameed et al. (2009). Also, the absence of mortar in the rear of the tiles leads to a higher impregnation of resin in the outer part of the tile, particularly if immersion is used (Figs. 4 and 5, Tables 3–5). Neutron tomography images of NSA and MNA tiles samples taken before and after the application of the consolidant by using brushing and immersion techniques are shown in Figs. 6 and 7, respectively. The primary images of the tiles can be used to distinguish between the ceramic body and the resin uptake.

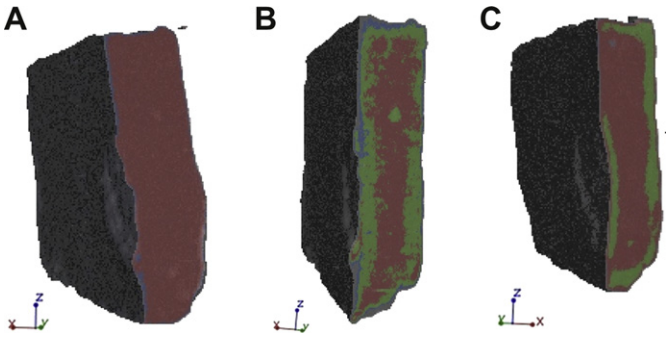

Fig. 7. NT images of MNA tile sample: A: before any treatment; B: after the impregnation of resin using brushing technique; C: after impregnation of resin using immersion techniques (red – ceramic body; blue – voids; green – resin). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

4. Conclusions

Neutron tomography permits visualization of the penetration depth and distribution of polymer-based consolidants inside ancient tiles, and can thus provide a significant aid to the development of strategies for their conservation or restoration. Neutron attenuation in the hydrogen-rich consolidant is much greater than in the mineral constituents of the ceramics: this is registered as sharply contrasting signal levels (gray levels) in images generated from slices through the tomographs. Tomography of two ancient glazed tiles, with different composition and texture, indicates that the application of 10% Paraloid® B-72 in acetone solution by brushing produces a higher and more uniform impregnation of the consolidant in the tile than does immersion.

In terms of conservation practices, for 10% Paraloid® B-72 in acetone solution, the brushing technique used at the Museu Nacional do Azulejo appears to be the most appropriate way to apply the consolidant in order to improve structural strength in an homogeneous way through ancient glazed tiles. This is most clearly demonstrated in the MNA sample, which required a conservation procedure due to serious degradation, related principally to exfoliation.

The results obtained for the immersion technique indicate that this might be improved if the concentration of the resin were reduced. Further studies using different resin/solvent ratios are foreseen.

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