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Restoration of decorative glazed ceramic from Morocco: Technical replicas

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Abstracts

Français English

Les bâtiments historiques du Maroc présentent des décors de carreaux de céramique émaillée appelés zelliges, qui doivent souvent être restaurés en raison de leurs dégradations. Les carreaux reproduits n'ont souvent pas les mêmes propriétés physico-chimiques que les originaux, ce qui entraîne divers problèmes de conservation. Cette étude examine les zelliges des Tombeaux Saadiens et du Palais El Badi (XVIe-XVIIe siècles), dans le but de développer des répliques compatibles. Des échantillons ont été préparés avec de l'argile de Fès, cuits, et analysés pour le retrait, la perte de poids et l'absorption d'eau. Les mortiers ont également été analysés. Une forte absorption d'eau a été observée, renforçant l'adhérence entre le zellige et le mortier.

Historical buildings in Morocco feature decorative glazed ceramic tiles known as zelliges, which often require restoration due to degradation. Reproduced tiles frequently lack the original physico-chemical properties, leading to various conservation issues. This study examines the zelliges from the Saadian Tombs and El Badi Palace (16th-17th centuries) to develop compatible replicas. Samples were prepared using clay from Fès, fired from 600°C and 900°C, and analyzed for shrinkage, weight loss, and water absorption. The mortars were also analyzed. A high water absorption was observed, enhancing the adhesion between the zellige and the mortar.

Index terms

Keywords : zellige, céramique, argile, réplique, bâtiment, conservation, restauration
Keywords: zellige, ceramic, clay, replica, historical, building, conservation, restoration

Full text

Introduction

1 Glazed colorful wall tiles, called “zellige”, are a distinctive aspect of Moroccan cultural heritage. In Morocco, the first forms of zellige appeared in the Almohad architecture (1130-1269) in the Casbah and the Koutoubia Mosque (Marrakech region). The manufacturing technique is developed and the decorations are in complex polychrome geometric patterns obtained by the juxtaposition of a variety of small shapes carved during the Merinid (1244-1465) and Saadian (1530-1660) dynasties. These decorations cover the surface of the ground and the lower walls of monuments, which symbolize wealth, prestige and power in place (madrasahs, mosques, palaces, etc.) (Fig. 1). Similar sets were developed simultaneously in Mediterranean cities such as Tlemcen (Algeria), Tunis (Tunisia) and more widely from the Iberian Peninsula to Uzbekistan. The technology of these decors originates from the lead-glazed and tin oxide opacified mosaics of the Roman and Byzantine Empire (Tite et al., 2008; Gradmann, 2016, Matin et al., 2018).

Fig.1. Wall tiles decoration

Zellige from the Saadian Tombs (left) and the Madrasah Ben Youssef (right).

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2 Conservation of several historical monuments remains a serious problem because of their strength degradation mainly due to human impact and weathering. In Morocco, zellige walls and floors of historical monuments in the Marrakech region have gaps where the shards have cracked or fallen out.

3 In some places, tiles are deeply damaged which makes it difficult to conserve and restore the original pieces of zellige. Two previous restoration interventions were conducted during the 20th century. Restorations have focused on replacing the altered tiles and filling the gaps with newly produced ones, in order to restore the aesthetic appearance. However, zellige tiles restored are technically very different from the oldest ones and do not have the same mechanical and chemical resistance to weathering. This generates heterogeneous behaviour within the restored surfaces. It seems that recent tiles produced deteriorate faster than the old zellige tiles still in place, which is pushing to increase interventions within these historical monuments.

4 The state of conservation of decorative ceramics is strongly influenced by the behaviour of the architectural ensemble and the surrounding materials (wood, plaster, zellige, mortar and clay) and human activity as well as environmental conditions (soluble salts, climate, pollution). While mechanical and chemical resistance of materials depends on the physico-chemical properties of ceramics and their manufacturing process. Accordingly, it is essential to understand the technical characteristics of traditional zellige manufacture and especially drying and firing

processes in order to improve historical building conservation procedures and to propose suitable material for restoration.

5 Although the published literature on tiles, glazes and colouring oxides of architectural and Islamic ceramics is abundant, a few studies deal specifically with zellige tiles and raw materials in Morocco (Gradman et al. 2015-2016; El Halim et al. 2018; El Halim et al. 2022; Damas et al. 2018).

6 Archaeological zellige samples from the two historic sites studied are porous, beige to light brown body, made up of calcareous raw material and lead glazed. Those archaeological zellige samples are homogenous and have almost similar chemical and mineralogical compositions. This fact suggests that the origin of raw materials used to manufacture archaeological tiles is almost similar (El Halim et al. 2018). The raw material from Fez, currently and traditionally used for zellige manufacturing, has mineralogical compositions close to the archaeological zellige samples, which suggests a possible origin of the raw materials (El Halim et al. 2018, 2022).

Multidisciplinary collaboration and contribution of the conservator-restorer

7 Research on zellige tiles in Morocco is extensive and has already been the subject of several studies, notably on physicochemical properties and raw materials (Gradman et al. 2015-2016; El Halim et al. 2018; El Halim et al. 2022; Damas et al. 2018). This study is part of this research and results of from multidisciplinary collaboration between ESA Saint-Luc Liège (conservation-restoration, ceramics and glass), the Geosciences and environment laboratory at Cadi Ayyad University in Marrakech and the Geology Department (AGEs) at the University of Liège.

8 The state of preservation of decorative ceramics is influenced by their mechanical and chemical resistance. It is essential for the conservator-restorer to understand the technical characteristics of traditional zellige manufacture, and in particular the drying and firing processes, in order to improve conservation procedures for historic buildings and propose appropriate materials for restoration.

9 The conservator-restorer plays a crucial role in this multidisciplinary research involving geochemists and geologists. As part of this collaboration, the team conducted fieldwork in various regions of Morocco to observe the zellige decorations within the architectural ensemble and to meet the craftsmen. During these field campaigns, the conservator-restorers examined the zellige decorations and tiles, the appearance of the shards, the color of the glazes, the repertoire of geometric patterns, the mortar, etc., to assess the state of conservation and note any alterations. The encounter with the zellige artisans, who are the bearers of this craftsmanship, was a rich exchange that provided a better understanding of the artisanal process, observing the gestures, the texture of the clay paste, the kilns, and the changing colors during drying and firing processes (Fig. 2).

10 To investigate the hypotheses regarding the firing temperatures of archaeological zellige, an experimental study was carried out as part of a master's thesis in conservation-restoration at ESA Saint-Luc Liège. To shape and fire the tile samples in the laboratory, the conservator-restorer prepared the raw materials and clay paste, shaped the test specimens, and observed the variations in weight and shrinkage during drying, as well as the changes in color texture, and mineral composition after firing. The testing protocol for this research was drawn up based on previous studies and artisan know-how, in collaboration with partner scientists. The results are presented in this study and may serve to develop new tests.

11 Each stage of the study progressed thanks to constant feedback between the geochemist's and geologist's knowledge, the laboratory analyses, and the interpretation of the obtained physico-chemical results, combined with the conservator-restorer's expertise in materials, diagnosis, manual practice, and tactile sensitivity.

Fig. 2. Artisanal process

The artisanal process highlights the raw materials from the Benjlik site in Fez, emphasizing the texture of the clay paste that changes during drying and after firing.

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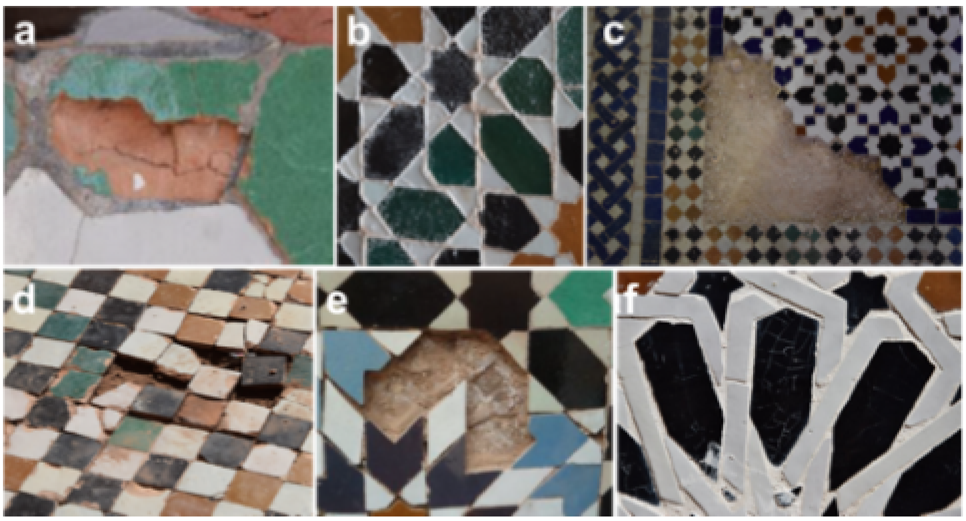
Description of pathologies

12 Archaeological zellige tiles show various pathologies, among the most common are the presence of grains of calcium carbonates that have not been transformed during firing, and saline efflorescence (Fig. 3a-b). The grain of lime comes from an incomplete oxidation of the grains of carbonates present in the raw material. After firing when in contact with air, lime grains increase volume and even cause fracturing of the ceramic body (Fig. 3a). The increase in the volume of these clay grains in contact with humidity can cause tensions within the tile. The crystallisation of soluble salts is an external deterioration factor that particularly affects porous ceramics such as zellige tiles (Fig. 3b). Degradation of the tile inevitably causes alteration of the glazed layer. Disbonding of tiles was observed on the restored zellige from the Badi Palace (Fig. 3 c-e). This is most likely due to the mortar composition used during the restoration process. Mortar seems to be responsible for an uplift of the restored zellige tiles from the Saadian Tombs resulting in the irregularity of the surface (Fig. 3f).

Fig. 3. Damaged archaeological tiles from the Saadian Tombs



Time grains (a); soluble salts and cracks (b). Loss of tiles from the restored tiles at the Badi Palace (c, d, e). Restored zellige tiles from the Saadian Tombs revealing fouled cracks and uplifted pieces, causing surface irregularities (f).

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Material and methods

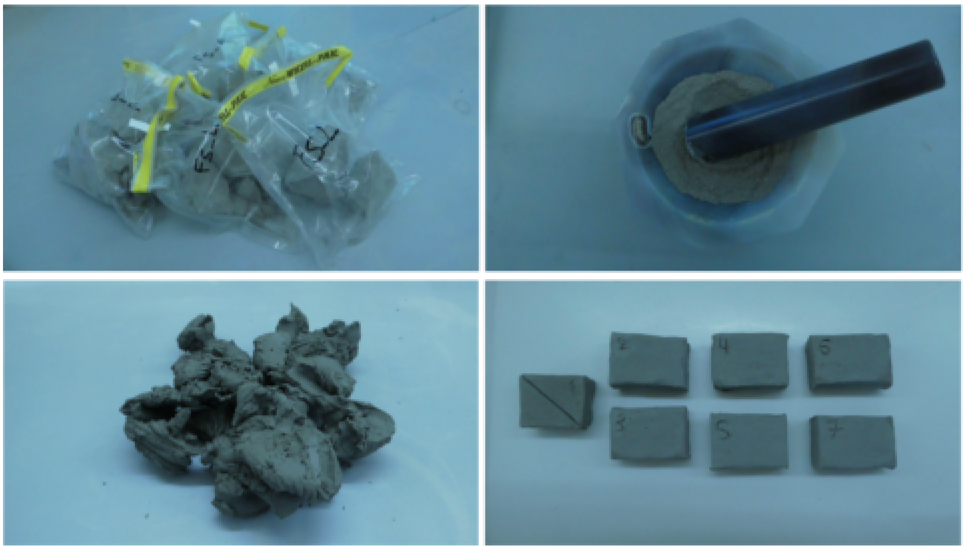
13 The test pieces were prepared from clay materials from Fez, taken from the Benjlik site. These raw materials are grey, comprising two facies: sandy (FS-R) and silty (FS-L) clays known as "rough" and "smooth", respectively. The two facies have almost similar chemical and mineralogical compositions. They are composed of 23% clay minerals (variable proportions of illite, kaolinite, chlorite and smectite), calcite (20%), dolomite (8%) and traces of K-feldspar, plagioclases (albite), muscovite, hematite and gypsum. FS-R clay is richer in smectite (15%) than FS-L (7%). FS-L is richer in quartz than FS-R clay (El Halim et al, 2019, El Halim et al, 2022).

14 Two samples of original mortars from El Badi Palace and the Saadian tombs, and two mortar samples used during previous restoration phases were sampled to determine the causes of early degradation that led to the detachment of the zellige tiles.

15 The plasticity of two raw clays FS-R and FS-L and the FS-L (1/3) + FS-R (2/3) mixture were obtained by determining the Atterberg limits: liquidity limit (LL), plasticity limit (PL), and the plasticity index (PI) calculated by the difference between LL and PL of the studied samples. These tests were carried out with a Casagrande apparatus according to standard NF P94-051.

16 The raw materials were first crushed, sieved at 250 µm mesh and mixed according to the proportions currently used by artisan from Fez: 1/3 of FS-L sandy clay and 2/3 of FS-R silty clay. Demineralized water was added to achieve the desired consistency of the paste. Afterward, seven specimens and one reference specimen of 2 cm thick, 15 cm width and 20 cm length were shaped (Fig. 4).

Fig. 4. Preparation of the raw clay material



Sample preparation in the workshop by the conservator-restorer

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17 Specimens were dried at room temperature (~ 25°C) during one week. Dimensions and weight of the reference specimen was monitored and measured every 30 min until stabilisation at about170 hours. Dimension change and weight loss were monitored to evaluate the shrinkage behaviour during drying.

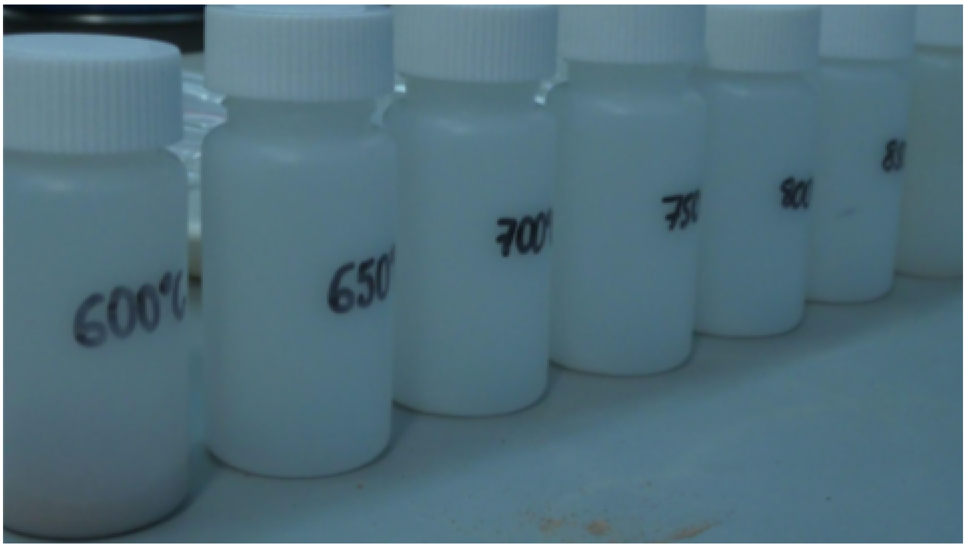
18 The seven other specimens continued to dry in an oven at 105°C during one week to ensure that all moisture was gone out. Then, the oven dried specimens were kilned in a Carbolite® electric oven at 600°C, 650°C, 700°C, 750°C, 800°C, 850°C, 900°C for 1

hour with a heating rate of 5°C/min. Appearance, dimension and loss of mass of the fired specimens were monitored to determine the shrinkage behaviour during firing.

19 Water absorption capacity (WAC) of the fired specimens calculated as $WAC = (G2 - G1)/G1 \times 100\%$, where G1 and G2 is the mass of dry and sample immersed in the distilled water during 24h, respectively.

20 To follow mineralogical transformations during firing and to compare the mineralogical results of fired bodies with those of archaeological zellige, fired bodies were analysed by X-Ray Diffraction (XRD) (Fig. 5). XRD analysis of powdered and sieved at 250 µm samples was performed using a Bruker diffractometer with CuKα radiation (Department of Geology, University of Liège). The angular range explored was between 5° and 60° 2θ. The X-ray spectra obtained were processed using the DIFFRACT PLUS EVA software. Mineralogical phases were quantified by the Rietveld method using Topas v5 software (Bruker).

Fig. 5. Sample



Preparation of powdered samples for XRD analysis.

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Results

Physical properties of raw clay

21 The clay sample FS-L has a high plasticity index (PI = 36%) compared to FS-R (PI = 16%; Table 1). This difference of plasticity is due to the occurrence of more smectite in FS-R (15%) than FS-L (7%) (El Halim et al., 2022). This is also justified by the smooth texture due to the small amount of quartz in FS-L clay. The FS-L (1/3) + FS-R (2/3) mixture used by the potters has a moderate plasticity index of 21% allowing better handling of the paste.

Table 1. Physical properties

Sample	Color	Texture	Liquidity limit (LL %)	Plasticity limit (PL %)	Plasticity index (PI %)
FS-L	Greenish-grey	Smooth	56	20	36
FS-R	Greenish-grey	Rough	36	20	16

FS-L (1/3) + FS-R (2/3)	Greenish-grey	Rough	48	27	21

Physical properties of raw clay samples from the Benjelik site and the mixture used for zellige manufacturing.

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Zellige properties

Drying behaviour

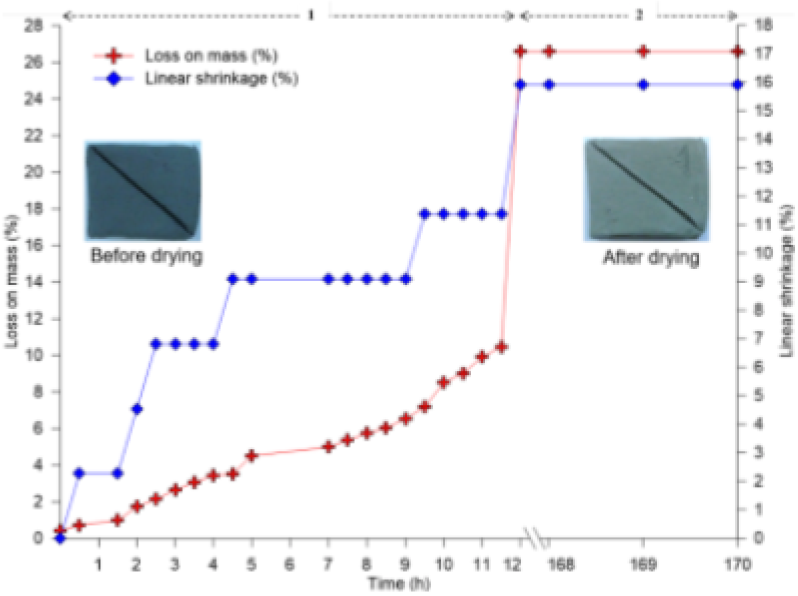
22 Drying process that took about 168 hours, the specimens have lost ~ 26.5% of their weight and have shrunk by 13.6% (Fig. 6). We observe that the elimination of the moisture during drying is not a linear process. Two stages can be distinguished: 1) the first stage (wet zone), representing ~10% of the total cycle time, shows small the loss of moisture and the drying shrinkage is small of the drying cycle

23 In the second stage (dry zone), the maximum loss of moisture and drying shrinkage take place. In this stage, the shrinkage stabilizes during drying with minimum tendency of crack generation in the dry zone.

24 Tile paste shows a slow drying process due to the occurrence of smectite rich clay used. Smectite retains a large volume of water within the interplanar spacing.

25 The grey colour of specimens became lighter after drying (Fig. 6). We did not observe any cracks or defaults during drying.

Fig. 6. Evolution of drying behaviour of reference specimens



Evolution of drying behaviour over time, 1 : wet zone, 2 : dry zone.

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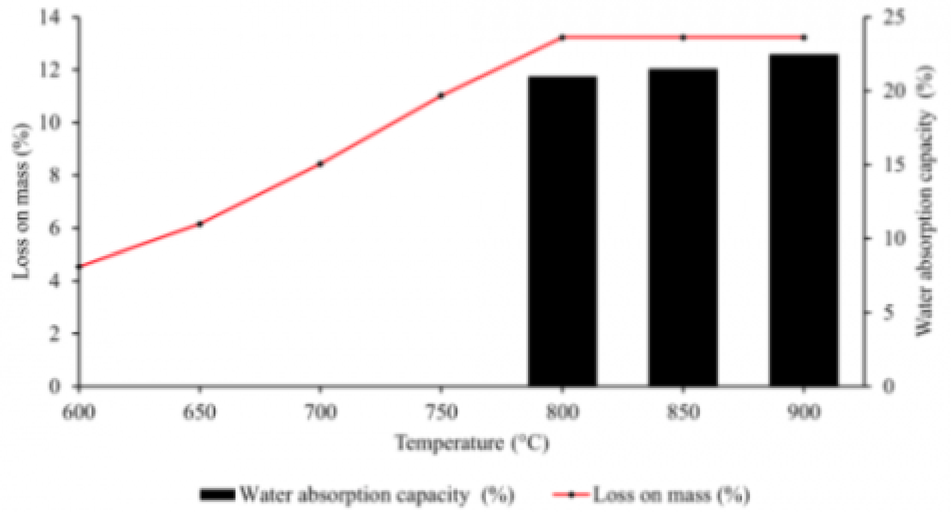
Firing behaviour

26 Linear shrinkage was not observed on fired specimens. This indicates that there may have been no significant vitrification process from 600°C to 900°C. The percentage of the weight loss gradually increases with the rise in temperature, which reaches 14% from 800°C. From 800°C to 900°C weight loss tends to stabilize (Fig. 7).

27 The water absorption capacity (WAC) values slightly increase from 21% at 800°C to 22.5% at 900°C. The colour of the specimens changed from grey to ochre-brown after firing, and becomes lighter as temperature increases (Fig. 7).

Fig. 7. Firing Behaviour



Evolution of loss on mass and water absorption capacity of the specimen in terms of firing temperature.

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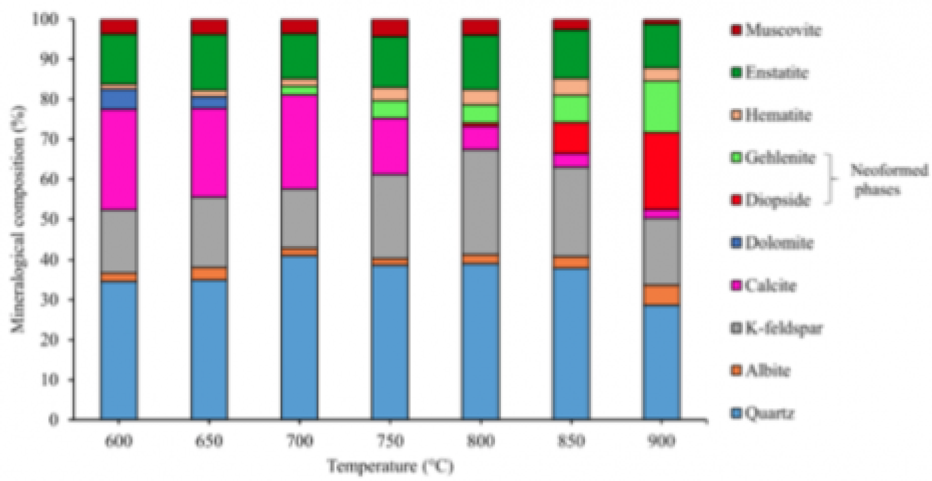
Mineralogical composition of fired specimens

28 The XRD results of fired specimens indicate mineralogical transformations within all specimens (Fig. 8). Mineralogical composition consists of calcareous phases already existing in clay (calcite and dolomite), neoformed phases of calcium silicate (gehlenite and diopside), clay minerals and hematite (Fe_2O_3).

29 The XRD spectra show a gradual decrease of calcite ($CaCO_3$) peak from 600°C to 800°C, which persists as small peak up to 900°C. Dolomite ($CaMg(CO_3)_2$) peak decreases to 650°C, while disappears at 700°C. Gehlenite ($Ca_2Al(AlSiO_7$) peak appears noticeably from 700°C and increases significantly up to 900°C. Diopside ($CaMgSi_2O_6$) peak appears around 800°C and increases up to 900°C. The intensity of hematite (Fe_2O_3) peak increases significantly from 600°C to 900°C. Total clay peak at d = 4.46 °A is still present at 600°C that slightly decreases but persists up to 900°C.

Fig. 8. Examination of fired samples



Mineralogical composition (XRD) of fired specimens

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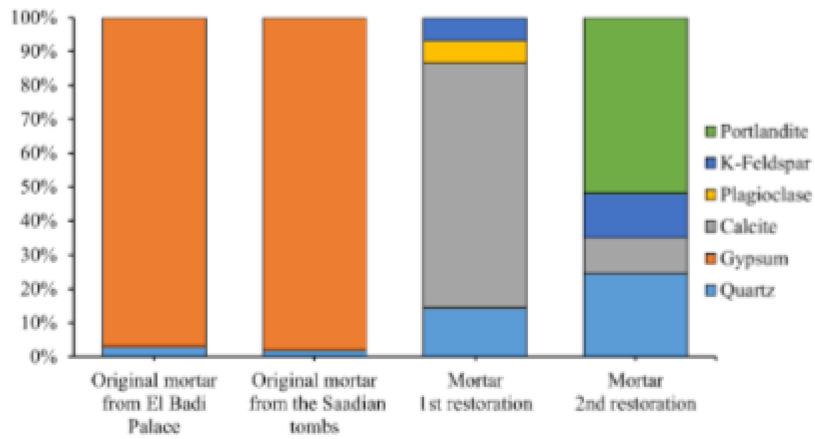
Mortar composition

30 The four mortars have different compositions and therefore different adhesion properties. The original mortars used to fix zellige tiles are made of gypsum, which

makes them more resistant and durable. The mortar used for the first restoration phase is composed of 65% of calcite associated with quartz and K-feldspath (Fig. 9). It seems more sensitive to capillary rise: the water which infiltrates into the mortar becomes loaded with calcium bicarbonates which leads to damage to the grains due to the solubility of the calcite. The mineralogical nature of the mortar is therefore an important factor responsible for the resistance of the zellige tile.

31 For the recent restoration phase, the mortar is composed of only 9% of calcite mixed with sand and portlandite. It is thus a portland cement, currently the most used in the field construction.

Fig.9 Mineralogical composition of mortar



Mineralogical composition (XRD) of original mortars and those used in previous restoration phases.

Discussion

Zellige characterization

Drying behaviour

32 Drying is an important step in the ceramic process that consists of the removal of moisture. Drying requires the control of the internal humidity of the specimens (Utlu et al., 2011). If drying is not carried out in a controlled manner, cracks occur decreasing the strength of the ceramic paste (Malizia and Shakoor, 2018). It is of upmost importance to stabilize the drying shrinkage even at the end of the wet zone (zone 1, Fig. 6), the final removal of moisture occurs only in the dry zone (Zaccaron et al., 2021).

33 Drying is accompanied by shrinkage due to the loss of moisture by the paste, mainly in the dry zone. The shrinkage in the dry zone was 16% due to moderate amount of coarse particles and moderate plasticity (Tabl.1). The paste used to manufacture zellige is therefore not suitable for the fast-drying process (Zaccaron et al., 2021). Otherwise, this can cause problems as cracks due to the occurrence of swelling clay minerals (smectite) that tend to be denser when shaped.

34 For a long and controlled drying, sand supplied by ARFL sandy clay, increases the amounts of sand (16%) and silt (58%) of the paste (El Halim et al., 2021) preventing cracks during drying. The shrinkage being stable in the dry zone, the coarse particles as free silica and non-clayey minerals assist in the diffusion of interstitial water. The high content of coarse particles, even with the occurrence of smectite prevent cracks development, due to the high content of free silica helped in the flow of moisture. Zaccaron et al., (2021) attest that plastic clays are not suitable for rapid drying cycles, as is the case in semi-arid areas where hot air ensures very rapid drying. The drying cycle

must be appropriate to the plastic behaviour of the paste; this means that the drying shrinkage must be the minimum in the dry zone to minimize the formation of cracks (Santos et al., 2018).

Firing behaviour

35 The slight increase in hematite content from 750°C (Fig. 8) relates to the release of iron oxide contained in the raw materials in the oxidizing firing atmosphere (Baccour et al., 2009). Hematite is responsible for a light reddish colour observed in tiles bodies from 800°C. Tiles intended for glazed zellige must generally have a light shade for better shine of the glaze. For this reason, the use of FS-L clay must not exceed 50 % of the mixture due to its high iron content (El Halim et al., 2022).

36 The presence of carbonates (calcite and dolomite) in raw clay (Fig. 8) strongly influences porosity during firing, causing the formation of micrometric size (< 1 μm) (Cultrone et al., 2004; Costa et al., 2016). The pore structure, in particular the presence of open porosity, increases water absorption capacity and can make the tile susceptible to weathering (Cultrone et al., 2004). However, when the water infiltrates by capillary, the lime particles contained in the mortar are introduced to the body of the tile through porosity. The carbonation of the binder creates the bond between the mortar and the tile and increases mechanical strength of the masonry (Lanas and Alvarez-Galindo, 2003). Therefore, the porosity of tile could be an advantage by creating an interface between the tile and the mortar and will limit infiltration in historical buildings (Lanas and Alvarez-Galindo, 2003; Santos et al., 2012).

37 During the firing process, in an oxidizing environment, the calcite and dolomite grains present in zellige paste decompose at temperatures ranging from 700°C to 790°C, yielding lime (CaO). The resulting lime remains unreactive if it does not integrate into the crystalline structure of the newly formed phases (diopside and ghelenite, Fig. 8). Only the outer part of these lime grains reacts with the vitreous phase during firing and undergoes silicification (Cultrone et al., 2001). The internal part of the transformed lime grain retains its CaO composition. Over time, unreacted lime reacts with ambient humidity at low temperatures and either hydrates ($CaO + H_2O => Ca(OH)_2$) or recarbonates ($CaO + CO_2 => CaCO_3$). Both of these processes result in expansion because the specific volume of $Ca(OH)_2$ and $CaCO_3$is greater than that of unreacted lime (and hence the density is much lower: 3340 kg/m³ for CaO, 2710 kg/m³ for $CaCO_3$, and 2340 kg/m³ for $Ca(OH)_2$). Hydroxide [Ca(OH)2] exhibits the most significant expansion, eventually causing surface cracking in the zellige piece (Fig.3).

Inhibition of defects associated with lime expansion

38 Various solutions have been proposed in the literature to inhibit the swelling and efflorescence of lime. Kornmann (2007) suggests wet sieving with a mesh size of 0.2 mm to prevent this problem. Laird and Worcester (1956) propose soaking the fired clay body in cold water immediately after firing and cooling, known as docking process (Ramachandran et al., 1960), to dissolve lime or portlandite. Rapid water absorption results in the immediate extinction of lime and reduces apparent volume. Consequently, no disruptive forces will develop within the piece, preventing lime swelling.

39 The addition of up to 0.5% NaCl to clay containing 10% carbonates inhibits the transition of calcite to CaO and then eliminates lime swelling and efflorescence (Blumen, 1943; Laird and Worcester, 1956; Rye, 1976; Stimmell et al., 1982; Bearat et al., 1989; Hoard et al., 1995). NaCl disappears at temperatures above 500°C, and HCl is formed during the dehydroxylation of clay minerals (Fabbri and Fiori, 1985). CaO, resulting from the decomposition of $CaCO_3$, reacts with HCl to form $CaCl_2$ between 500

and 600°C (Bearat et al., 1989). This $CaCl_2$ further reacts with clay minerals to form more stable calcium silicates at low temperatures. The addition of NaCl is an effective solution to prevent lime-related defects. However, this addition changes the color of the shard's surface to a light or white shade. This discoloration occurs only when the raw clay is rich in Ca and when the firing temperature ranges from 750 to 900°C (De Bonis et al., 2017). Additionally, some Tunisian potters use seawater for white pottery and freshwater for red pottery. This method is applied in other regions of the world, such as the Middle East, Pakistan, and Italy.

40 The use of fluxing agents is also recommended to address lime-related problems. Adding periclase (MgO) enables the incorporation of Ca from lime into the newly formed high-temperature silicate phases such as gehlenite, anorthite, and diopside. The reaction of the incorporated CaO with air in the newly formed phases no longer occurs as it does in the powdery form of this oxide. The addition of fluxing phases can help neutralize any lime through silicification (Kornmann, 2007), if the lime particles are small enough. During firing, it is advantageous to kiln at high temperatures (1100°C) for an extended duration to promote the silicification of calcium and consume lime particles. It also appears that residual lime treated at high temperatures is less reactive (Cultrone, 2001).

Conclusion

41 This study focuses on the technical replication of zellige tiles from historical monuments in Marrakech and investigates their similarities to the original archaeological tiles. The raw materials used for the archaeological zellige tiles are sandy calcareous clays containing iron oxide. Specimens were prepared using calcareous clay from the Fez region, following the traditional methods employed by local ceramic craftsmen.

42 A slow drying of the raw material is observed attributed to the presence of smectite-rich clay, which retains a significant amount of water in its interplanar spaces, was observed. To ensure controlled, prolonged drying and prevent cracking, additional sand from ARFL sandy clay was mixed into the paste, increasing the sand (16%) and silt (58%) content (El Halim et al., 2021).

43 X-ray diffraction (XRD) analysis suggests that the zellige tiles were likely fired at temperatures between 700°C and 900°C, as indicated by the evolution of calcareous phases (calcite and dolomite) and the emergence of neoformed phases (diopside and gehlenite). The presence of carbonates in the raw clay explains the natural high porosity of the tiles. This porosity allows for environmental exchanges (e.g., humidity, pollutants), which can accelerate degradation but also enhances the bond between the zellige and the mortar.

44 Mortar analysis revealed different compositions across various periods. The older mortars used to set the zellige tiles consist mainly of gypsum, with a small amount of quartz, making them more resistant and durable. The quality of the zellige-mortar interface and the mineralogical nature of the mortar are crucial factors for the conservation of both the decoration and the structure.

45 The contributions of scientific analyses were considerable in the in-depth characterization of materials, their production processes, and raw materials. The specific knowledge of the conservator-restorer played a crucial role in studying of alterations and conservation issues. He thus placed the research within an experimental context, with a view to propose suitable restoration material for zellige decorations. Additionally, the study provides material insights that can guide the production of replicas capable of performing similarly to the originals, thereby aiding in the development of new restoration products.

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List of illustrations

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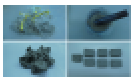

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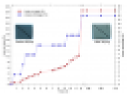

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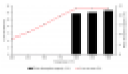

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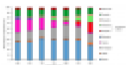

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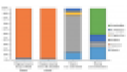

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