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RESEARCH ARTICLE

Material Characterization of Byzantine Period Brick Masonry Walls Revealed in Istanbul (Turkey)

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Abstract
In this study, the characteristics of mortars and bricks used in the walls of the historical building, which were revealed during the subway station construction excavations, probably belonged to religious place, have been investigated. Results of the analyses indicated that the mortars are hydraulic but they have a low compressive strength and a high porosity. The bricks whatever used as masonry unit or aggregate were produced from calcium rich clay at a firing temperature of 850−900°C, they are of low apparent density, high porosity and relatively high compressive strength, and also show pozzolanic activity.

Keywords
characterization, byzantine mortar, byzantine brick, advanced analysis methods, mechanical properties

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1 Introduction

Conservation and restoration of historical monuments are important for preserving worldwide cultural heritage. Conservation of cultural heritage needs multidisciplinary working and specialized undertaking, while its constraints are defined by national/international laws, pacts and legislations. Intervention materials used in the conservation works of historical buildings must be compatible with original materials. Comprehensive experimental study should be performed in order to determine material characteristics. Not only basic esthetical, physical and mechanical compatibilities, but also the mineralogical and chemical compatibilities must be investigated for the characterization works to judge overall performance [1, 2].

In this study, the characteristics of ancient mortars and bricks used in the walls of the building, which was revealed recently during the subway station construction excavations in Uskudar (county of Istanbul), very close to the Bosphorus, have been investigated. Unfortunately, the investigation had to be limited to foundation remains that have reached today (Fig. 1). Plan of the building, the used materials and observations on the construction system indicate that the building was constructed at the late Byzantine period and utilized as a religious place, probably a chapel [3].

The most significant indicators about dating of the building are the walls of the building and the arrangement of the bricks. The wall construction was based on hidden brick row behind extraordinary thick bedding mortar and this method is also called as “recessed brick technique” (Fig. 2). The most important examples of this construction method exist in Istanbul and this technique was used from the second half of the 11th century to the end of the 12th century [3]. In-situ measurements show that the thickness of the mortars vary between 45 and 55 mm whereas the dimensions of the bricks are approximately 250 × 250 × 25 mm.

Fig. 1 (Left) An air view of the apsidal building remains (the apsidal building is circled by black dotted line) and adjacent region [3]; (Right) Revealed foundation of the apsidal building

Fig. 2 The brick-mortar walls of the building

2 Experimental Methods

In this study, four mortar samples, three brick samples and three brick-mortar-brick composites were collected from the walls of the building for the identification of their visual, mechanical and physical properties, and also mineralogical and chemical compositions. The districts where the samples were collected are marked on the architectural survey presented in Fig. 3. Mortar, brick and composite ternary samples were taken from each district, except 3rd point, from which only a mortar sample could be taken and all samples were numbered according to the district where they were taken.

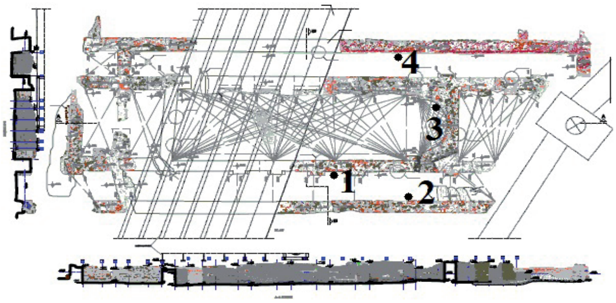

Fig. 3 The architectural survey of the building

Surface color was investigated for visual identification. Color measurements were carried out in accordance with EN 15886 [4] and the analysis was performed with a spectrophotometer device (8 mm diameter viewing aperture) by considering the CIELAB color space. The CIELAB color space is used to define the measuring protocol by considering the Cartesian coordinates: L*a*b*, where L* represents the lightness of the color, which varies from 0 black to 100 white, a* is the red-green coordinate on a red (+) to green (−) axis and b* is the yellow-blue coordinate on a yellow (+) to blue (−) axis. The total color difference (ΔE*) between two measurements ($L_1^*a_1^*b_1^*$ and $L_2^*a_2^*b_2^*$) is the geometrical distance between their positions in the CIELAB colour space and it is described in Eq. (1):

$$\Delta E^*_{2,1} = \sqrt{\left(\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}\right)} \qquad (1)$$

where ΔL*, Δa* and Δb* represent the difference between the measured values of L*, a* and b*.

The compressive strength was determined for mechanical characterization. Cubic mortar specimens with a side length of at least 3 cm were prepared for the uniaxial compression test. However, some of the mortar samples did not have suitable shapes and sufficient dimensions to prepare specimens required for the compression test, so point load test was performed to determine the compressive strength. The test was conducted by a hydraulic loading device with a precision of 0.1 kN in accordance with ASTM D5731 [5]. The size corrected point load strength index ($I_{s(50)}$) was calculated for each mortar sample and these indexes were converted to uniaxial compressive strength by using a correlation factor of 8 which has been the outcome of an experimental study previously carried out by the authors on the lime mortars [6]. The compressive strength of the bricks was determined in accordance with ASTM C67 [7].

Basic physical properties such as density, porosity and water absorption ratio were determined by weighing the specimens in dry state and in water saturated state [8, 9]. The cube or prismatic specimens with a minimum dimension of 3 cm were prepared for the apparent density and water absorption tests; however, pulverized and sieved to <63 μm fraction of the samples were used for determination of the real density by using ultra-pycnometer.

Pozzolanic activity of the bricks used as both masonry unit and aggregate was examined by a direct method, known as Frattini test [10, 11]. The Frattini test is based on the classical chemical titration and it was performed in accordance with EN 196-5 [12]. 20 grams of samples consisting 16 g cement (80%) and 4 g test material (20%) were prepared and then mixed with 100 ml distilled water. The samples were kept in sealed bottles and put in an oven at 40±1°C for 8 days. After 8 days, the samples were vacuum filtered through a 2 μm nominal pore size filter paper and the filtrates were analyzed for [OH^-] and [Ca^{2+}] (expressed as CaO) ions by titration with the help of solutions and indicators existing in the standard.

The samples were pulverized for mineralogical and chemical analyses. The mineral composition of this fine fraction (<63 μm) was identified using X-ray diffraction (XRD) and petrographic analyses, and also Fourier transform infrared spectroscopy (FTIR). Not only the mineralogical composition, but also probable organic based or biological additives like egg white, blood, urea in the mortars were investigated with FTIR. Thermogravimetric analysis (TGA) was performed to contribute mineralogical identification and indicate hydraulic characteristic of the mortars. The chemical composition of the samples was determined using X-ray fluorescence (XRF) which provides the chemical composition of the constituents in terms of oxide. Acid loss analysis for the mortars, which relies on acid dissolution/separation of the binder phase from the aggregate, was also carried out to obtain additional information about the chemical compositions of the binder and the aggregate and their relative proportions unless the aggregate is acid-soluble. In the experimental work, the acid loss analysis was conducted on at least 50 g mortar specimens by means of 10% diluted HCl. 3 ml of the solution was used per gram of the mortar specimen and time duration for the acid treatment was one hour [13]. Aggregate size distribution was determined by the sieve analysis and the test was performed on the grains remained after the acid loss analysis.

3 Results and Discussion

3.1 Characteristics of mortars

The L*, a*, b* values and the total color difference between the measurements for each specimen are presented in Table 1. L*, the lightness of the color varies between 77.1 and 85.8, is within the high-central part of the scale and exhibits a deviation of approximately 5% from the mean value of 81.1. The chromatic parameter a* as in a wide range between 4.7 and 8.3 with a deviation of approximately 30% from the mean value of 6.4, while the chromatic parameter b* varies between 12.8 and 17.9 with a deviation of approximately 15% from the mean value of 15.1. According to these results, the color of the mortars can be described as light-medium gray with a combination of slight red and intensive yellow tones.

The interfaces between the aggregate and the lime (matrix phase) are porous and very weak (Fig. 4). The aggregates are also porous and mostly angular including quartzitic sand and crushed bricks. The uniaxial compressive strengths of the mortars are very low, vary between 1.5 and 3.5 N/mm^2 and the mean value is 2.3 N/mm^2. The compressive strengths derived from the size corrected point load strength index ($I_{s(50)}$) are also very low and vary between 1.0 and 2.2 N/mm^2. The mortar samples have a low apparent density of 1.2 g/cm^3, a real density of 2.5 g/cm^3 and a high porosity of 53.6%. It should be noted that most of the pores are open pores considering the effective porosity of 52.0%.

Table 1 Surface color of the mortars

No.	L*	a*	b*	ΔE*
1	77.7±0.6	7.8±0.5	17.2±0.7	1.6±0.0
2	82.1±0.6	5.7±0.2	14.6±0.1	0.7±0.5
3	82.9±0.8	5.4±0.3	13.9±0.2	1.1±0.4
4	85.6±0.2	4.9±0.2	12.9±0.1	0.6±0.0

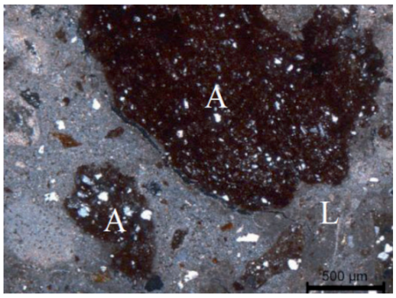

Fig. 4 Polarized microscope image of the mortar, (L: Lime; A: Aggregate)

The lime/aggregate ratios of the mortar samples vary between 1/4 and 1/5 by weight. It has been stated before that weathering, which washes out calcite, could cause the low lime/aggregate ratios as in this mortar samples and 1/3 is recommended for restoration works since it is compatible with Byzantine monuments in Rhodes and Crete from the same period [14].

The aggregates, which remained after acid loss analysis, were sieved in order to determine aggregate size distribution. The maximum aggregate size is 16 mm and the coarse aggregates with particle size greater than 4 mm constitutes the largest fraction of the entire aggregates (Fig. 5). It has been reported by the authors that the particle size distribution obtained after the acid loss analysis are mostly greater than the original one since the undissolved paste phase adheres to the aggregates and/or the aggregates adhere to each other [6].

XRD patterns of powdered lime lumps (Fig. 6) show that they are composed of calcite and calcium silicate hydrate (C-S-H). The presence of C-S-H is obviously a significant indication about hydraulicity of the mortars whereas the calcite is a product of the carbonation of slaked air lime.

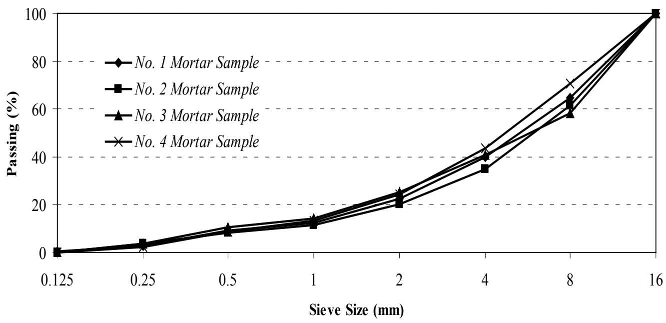

Fig. 5 The aggregate size distribution of the mortars

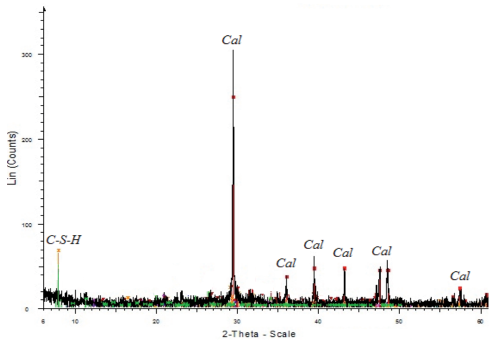

Fig. 6 XRD pattern of the no.1 mortar sample
(Cal: Calcite; C-S-H: Calcium silicate hydrate)

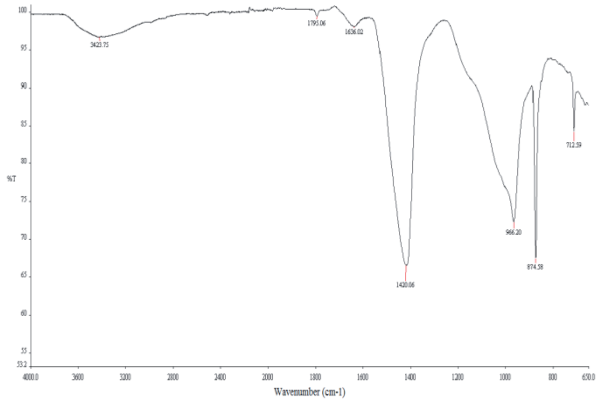

Fig. 7 FTIR spectrum of the no.1 mortar sample

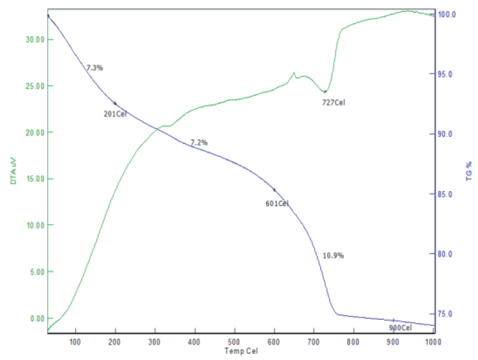

Fig. 8 Thermogram of the no.2 mortar sample

The chemical composition of the mortars (Table 2) indicates that they are mostly composed of CaO and SiO_2. The high CaO content and low MgO content probably mean that the lime was provided from calcareous stones rather than dolomitic stones. If the high content of SiO_2 and Al_2O_3 are interpreted with XRD results, these components may be products of the hydraulic reactions as well as the quartzitic or crushed brick aggregates.

Table 2 Chemical composition of the mortars

Constituent	%
CaO	36.1±0.6
SiO_2	22.3±0.7
Al_2O_3	6.0±0.5
Fe_2O_3	2.1±0.4
MgO	2.0±0.2
K_2O	0.2±0.1
Na_2O	0.1±0.0

In the FTIR spectrum of the mortars (Fig. 7), the main $CaCO_3$ bands at 713, 875, 1420 and also a minor peak at 1795 cm^{-1}; SiO_2 at 966 cm^{-1}; H–O–H at 1636 cm^{-1} and O–H peak at 3424 cm^{-1} are observed. It should be noted that any organic based or biological additives could not be discovered from the FTIR results.

The hydraulicity of the mortars was evaluated by thermogravimetric analysis. The weight losses between 200–600°C and 600–900°C are attributed to the structurally bound water and CO_2 elimination from the mortar, respectively. These analyses indicate that the loss CO_2/H_2O ratios vary between 1.4–1.6 (Fig. 8) and are less than 10. Therefore, these mortars can be considered as hydraulic [15], as also concluded by the XRD analysis.

3.2 Characteristics of bricks

The bricks were examined in two groups as masonry unit and crushed bricks existed in the mortars as aggregate. The brick samples, either masonry unit or aggregate, have a more uniform color distribution than the mortars; therefore the measurement results are presented in Table 3 regardless of sample variety. According to these results, the color of both types of the bricks can be described as medium gray with a combination of intensive red and yellow tones.

Table 3 Color of the bricks

Type	L*	a*	b*	ΔE*
Masonry Unit	55.8±0.4	18.0±0.2	24.3±0.6	1.0±0.6
Aggregate	56.0±1.0	16.7±0.4	20.6±1.0	1.2±0.5

The polarized microscope observations exhibit that the masonry unit bricks contain large and small pieces of broken brick fragments (grog) and they have a porous structure (Fig. 9). The masonry unit bricks have a low apparent density of 1.7 g/cm^3, a real density of 2.6 g/cm^3, total and effective porosity of 35.3% and 31.4%, respectively. The compressive strengths of the bricks vary between 9.2 and 11.0 N/mm^2, and the average compressive strength is 10.3 N/mm^2. However, the friction between the bricks and loading plates of the test machine may cause significantly higher strengths compared to the real strengths of the bricks due to small height of historical bricks [16].

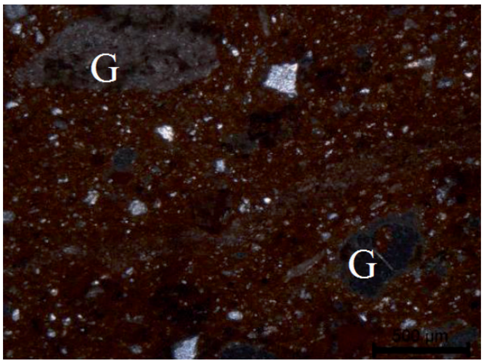

Fig. 9 Polarized microscope image of the brick (G: Grog)

XRD analysis results show that the masonry unit bricks and the aggregate bricks are composed of quartz, muscovite and anorthite minerals (Fig. 10). However, it is interesting to note that C-S-H peaks are seen in XRD patterns of the aggregate bricks and this is probably a result of the mortar grains that remained on the 'aggregate brick' surface, as also can be the product of the pozzolanic reaction between brick and lime.

The firing temperature range of the bricks was also estimated by XRD analysis. The presence of anorthite minerals imply that firing temperature is above 850°C [17]. However, the presence of muscovite, and also the absence of high temperature products of clay such as mullite and crystoballite indicate that the peak firing temperature does not exceed 900°C [18]. This temperature interval is consistent with the estimated temperature near the internal shelf in the brick kilns utilized in Roman period [19].

The chemical composition of the bricks (Table 4) indicate that they contain high amounts of SiO_2, CaO and Al_2O_3 and low amounts of Fe_2O_3, MgO, K_2O and Na_2O. The presence of high amounts of CaO determined in XRF analysis and the anorthite mineral revealed in XRD analysis mean that calcium rich clays were used as raw material.

Table 4 Chemical composition of the bricks

Constituent	%
SiO_2	48.4±1.1
CaO	23.1±0.8
Al_2O_3	14.0±0.4
Fe_2O_3	7.7±0.5
MgO	2.4±0.3
K_2O	2.0±0.3
Na_2O	1.1±0.2

The results of the Frattini test are presented as a graph of concentration of [CaO], in mmol/l, versus concentration of [OH^-], in mmol/l, in Fig. 11. The points lying below the saturation curve indicate the removal of [Ca^{2+}] from the solution which is attributed to pozzolanic activity and the points above the curve reveal no pozzolanic activity. The results shown in Fig. 11 indicate that all bricks, used as either the masonry unit or the aggregate in the mortar, show pozzolanic activity. This conclusion also supports the hydraulic characteristic of the mortars indicated by XRD and TGA results.

Since the production of hydraulic lime began in the 18th century at firing temperatures of 1000–1200°C [20], these hydraulic mortars were most likely constituted of air lime and pozzolanic brick particles. However, it has been concluded that the hydraulic lime could be produced at a relatively low temperatures as 850°C, e.g., by the calcining of limestone containing diatoms or fossils [21, 22]. The estimated brick firing temperatures (850–900°C) for the investigated building show that these firing temperatures could be obtained in that period and the hydraulic lime may be used if the raw limestone contained the specific impurities.

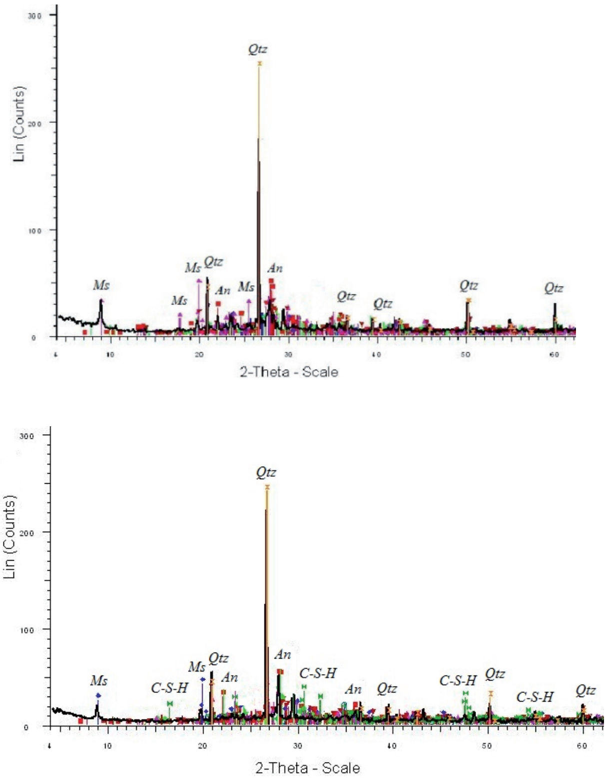

Fig. 10 XRD pattern of the (First) masonry unit bricks (Second) the aggregate bricks (Qtz: Quartz; Ms: Muscovite; An: Anorthite; C-S-H: Calcium silicate hydrate)

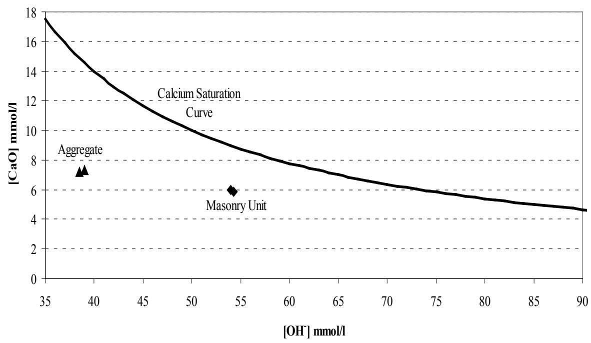

Fig. 11 The Frattini test results (Duplicate samples were prepared and the individual results are plotted)

3.3 Mechanical properties of brick-mortar-brick composites

The compression test was conducted on three brick-mortar-brick composites and the dimensions (width x length x height) of the prepared specimens were (78−100) x (100−182) x (80−88) mm. Upper and lower surfaces of the brick-mortar-brick samples were fixed with capping mortar and then, the prepared specimens were tested under uniaxial compressive loading in order to determine the mechanical properties. The displacements of composites were measured using linear variable displacement transducers (LVDT), as follows: two vertical LVDTs (one per each side) were used to measure axial deformations and one horizontal LVDT was placed on the mortar in order to record lateral deformations as shown in Fig. 12. The compression test was performed with a displacement controlled universal testing machine with a loading rate of 1 mm/min and each composite specimen was loaded until the failure occurred.

The stress-strain curves of the composite specimens are plotted in Fig. 13 and the test results, including the compressive strength (f_c), the ultimate axial strain ($\varepsilon_{a,ult}$), the modulus of elasticity (E) and Poisson's ratio (ν), are given in Table 5.

The compressive strengths of the specimens vary between 2.8 and 3.4 N/mm² and average compressive strength is 3.1 N/mm². These results are very close to the compressive strengths of the mortars since the larger part of the composites are composed of mortars with an extraordinary thickness of 45-55 mm. The ultimate axial strain values vary between 3.1% and 3.4% and similar strains have been recorded in the previous researches that carried out on either stone or brick masonry walls [23-26]. The modulus of elasticity was calculated by determining the slope of the stress-strain curve up to stress corresponding to 30% of the compressive strength and the average modulus of elasticity is 398.7 N/mm².

There are significant correlations ($R^2 \geq 0.98$) between the lateral strains and the axial strains for each specimen up to (0.3 f_c) stress level or any crack formation, whichever occurred first, and these results are shown in Fig. 14. Ratios of lateral strain to axial strain of the specimens, which are attributed to the Poisson's ratio, were calculated by determining the slope of the regression lines and these values vary between 0.28 and 0.31, and the mean value is 0.29 (Table 5).

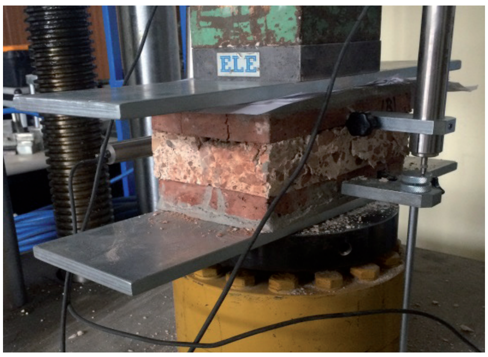

Fig. 12 A brick-mortar-brick composite and set-up for the compression test

Table 5 The mechanical properties of the composite specimens

Specimen	Compressive Strength (f_c, N/mm²)	Ultimate Axial Strain ($\varepsilon_{a,ult}$, %)	Modulus of Elasticity (E, N/mm²)	Poisson's Ratio (ν)
S1	3.1	3.4	380.2	0.29
S2	2.8	3.1	420.0	0.28
S4	3.4	3.3	396.0	0.31
Average	3.1	3.3	398.7	0.29

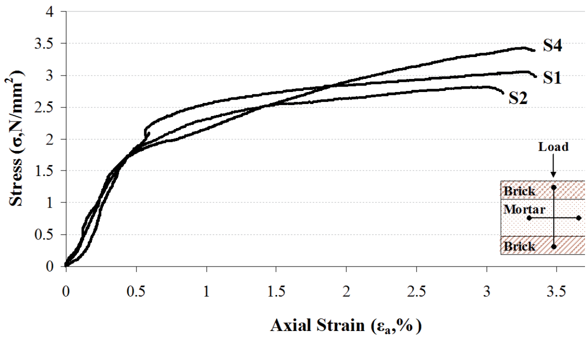

Fig. 13 The stress-strain curves of the composite specimens (The specimens are signed as S1, S2 and S4 according to the districts where they were taken)

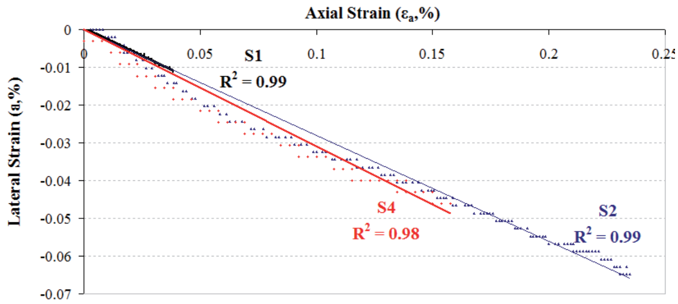

Fig. 14 The axial and lateral strains of the composite specimens (Rest of the strain distribution of the S1 specimen up to the (0.3 fc) stress level could not be plotted due to early crack formation)

4 Conclusions

The late Byzantine period (11–12th century) mortars can be described as light-medium gray with a combination of slight red and intensive yellow tones. They have a low apparent density and a high porosity, and most of the pores are open to the ambient. They have also a low compressive strength as a result of both the porous structure and the weak interface zones. The maximum aggregate size is 16 mm and the largest fraction of the aggregates is coarse aggregates.

According to the XRD and thermogravimetric analyses, the mortars can be considered as hydraulic mortar. Pozzolanicity of the bricks is another evidence for the hydraulic products indicated by the analyses. Although the mortars are considered as hydraulic and durable, low compressive strengths are confusing. This incompatibility and also the low lime/aggregate ratios may be explained with the loss of the calcite by weathering.

The bricks have a more uniform color distribution. They have a low apparent density, a high porosity, but have a relatively high compressive strength (~10 N/mm^2). The mineralogical and pozzolanic characteristics exhibit that masonry unit bricks and the aggregate bricks were most likely manufactured from the same raw material. The calcium rich clays were used as raw material and they were fired at a temperature probably in the range of 850– 900°C.

The uniaxial compression tests performed on brick-mortar-brick samples indicated that average compressive strength, modulus of elasticity and Poisson's ratio of the composites are 3.1 N/mm^2, 398.7 N/mm^2 and 0.29, respectively. These original material parameters play an important role to analyze the structural behavior accurately which makes a remarkable contribution to a successful restoration.

Acknowledgement

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