

2025

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M. E. Belgacem

University of Science and Technology Houari Boumedienne, USTHB, Algiers, Algeria,
ghazali2017@gmail.com

N. Rabahi-Touloum

National Delegation for Major Risks, Amar Mekid Street, Hussein-Dey, Algiers, Algeria AND National Center of Studies and Integrated Research on Building Engineering. Cité El-Mokrani, Soudania, Algeria,
touloumcnerib@yahoo.fr

R. Neves

Barreiro Technology School, Polytechnic Institute of Setubal, ESTB-IPS, R. Américo da Silva Marinho, 2939-001 Barreiro, Portugal, rui.neves@estbarreiro.ips.pt

Y. Ouldkhaoa

University of Science and Technology Houari Boumedienne, USTHB, Algiers, Algeria,
ouldkhaoayounes@outlook.com
ouldkhaoayounes@outlook.com at: <https://commons.yildiz.edu.tr/jscmt>



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Recommended Citation

Belgacem, M. E.; Rabahi-Touloum, N.; Neves, R.; and Ouldkhaoa, Y. (2025) "Scientific Characterization of Historic Mortars Dating From the 15th-Century of a Historical Building in Algeria: A Case Study," *Journal of Sustainable Construction Materials and Technologies*: Vol. 10: Iss. 4, Article 1.

<https://doi.org/10.29187/2458-973X.1199>

Available at: <https://commons.yildiz.edu.tr/jscmt/vol10/iss4/1>

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RESEARCH ARTICLE

Scientific Characterization of Historic Mortars Dating From the 15th-Century of a Historical Building in Algeria: A Case Study

M. E. Belgacem^{a,*}, N. Rabahi-Touloum^{b,c}, R. Neves^d, Y. Ouldkaoua^e

^a University of Science and Technology Houari Boumedienne, USTHB, Algiers, Algeria

^b National Delegation for Major Risks, Amar Mekid Street, Hussein-Dey, Algiers, Algeria

^c National Center of Studies and Integrated Research on Building Engineering. Cité El-Mokrani, Soudania, Algeria

^d Barreiro Technology School, Polytechnic Institute of Setubal, ESTB-IPS, R. Américo da Silva Marinho, 2939-001 Barreiro, Portugal

^e University of Science and Technology Houari Boumedienne, USTHB, Algiers, Algeria

ABSTRACT

This research investigates the historic mortars (Aggregate/Binder (A/B) ratio) of a historical patrimony building, dating from the 15th century, in Algeria, for restoration purposes. It is well known that thorough characterization is essential for successful restoration. To this end, several techniques, including Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), and X-ray Fluorescence (XRF), optical microscope were used to characterize the historic mortars. A mechanical test was used to characterize the compressive strength of a historic mortar. For a comprehensive characterization, A/B ratios were estimated using both Digital Image Analyses (DIA) and chemical analyses. The results obtained provided clear information on the fractions of aggregate and binder. The A/B ratios found of the historic mortars were in line with those found in the literature in historical building. It should be noted that chemical analyses were applied to all mortars, whereas the DIA method was limited to a few mortars (not applicable to earth-based mortars). The results of the A/B ratio for both methods are compared. Nevertheless, the estimation of the A/B ratios of historic mortars using the DIA method alone is insufficient and needs to be strengthened with robust methods such as the chemical one for more accurate and reliable estimation.

Keywords: Historical patrimony, Ancient mortar, Restoration, Characterization, Aggregate/binder ratio, Lime mortar, Digital image analyses

1. Introduction

Countries put a huge effort to preserve their heritage. Historical building is the heritage value in the region which makes the future generations knows its history [1, 2]. In this case, the structures feature of these buildings should be conserved [3]. To conserve them, restoration actions shall be adopted. Any random or inadequate intervention in these buildings using non-compatible materials can cause more damage than benefit [4, 5], which is observed in many

historical buildings where Portland cement was the restorer material of the historic building masonry [4]. This is due to the non-compatibility of the cement with the original materials (historic mortars, bricks, and stones) [4, 6, 7].

Historical buildings were designed based on walls of masonry (bricks or natural stones) connected to each other carrying horizontal and vertical loads, the materials linking the blocks are mortar [8]. The mortar plays an important role in the strength, durability and stability of the wall masonry [6, 9, 10]. Besides,

Received 4 May 2025; revised 25 September 2025; accepted 20 October 2025.
Available online 1 November 2025

* Corresponding author.

E-mail addresses: ghazali2017@gmail.com (M. E. Belgacem), touloumnerib@yahoo.fr (N. Rabahi-Touloum), rui.neves@estbarreiro.ips.pt (R. Neves), ouldkaouayounes@outlook.com (Y. Ouldkaoua).

<https://doi.org/10.29187/2458-973X.1199>

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Elevation: South-West (DLSO)



Elevation North-West (DLNO)



Fig. 1. Example of some of sampling areas (mentioned in Orange) selected to obtain and drill mortar samples: two photos above from elevation south west and North west, interior facade (a); exterior facade (facing the sea) (b).

mortars were also used for rendering. The mortars in historical buildings are often the material subjected to weathering and load impact which make it often the material that requires restoration [11, 12]. The choice of the replacement mortars in restoration shall be grounded the best possible characterization of the original one to ensure the best high-compatibility mortars [11, 13–16]. Besides to that lime mortar was the binder used in the past and first signs of the use of lime mortar were found in Palestine and Türkiye as reported by Murat Doğruyol [17].

The characterization of historical mortars is particularly complicated [18], this is due to their chemical complexity. For an archaeologist, characterization usually is limited to determining a small number of chemical parameters and often a visual examination is sufficient to satisfy one's claims. For a scientist who deals with historical mortars, the characterization must be more extensive and expected to carry

out a greater number of analyses [19]. If we want to carry out a reconstruction, it is advisable to characterize the mortar on its visual morphological, chemical, mechanical and physical appearance, so that the alternative mortar uses in the restoration does not differ too much and satisfy the compatibility requirement of the original one to attain the restorations objectives [20]. To do so, the aggregates binder ratio is one the most parameters that should be identified. Many techniques can be followed some of the them are simple and gives rapid results while others even are complicated but are efficient. in this purposes, the DIA today represents a quick and efficient way to characterize historic mortar samples [21] and has been used by many researchers in very important project as [22–26].

In this research study, mortars of one of the most ancient and high-importance historical buildings in Algeria are investigated. This historical building

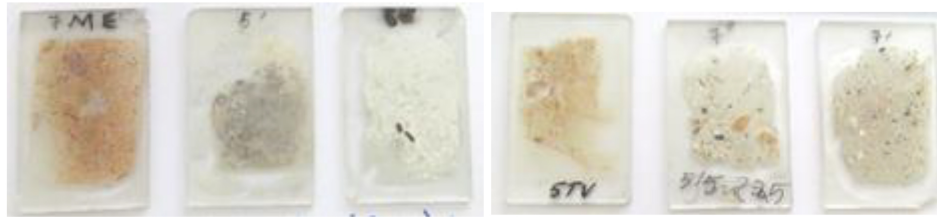


Fig. 2. Some of thin-sections prepared for the analysis.

belongs to the 15th century. For confidential purposes the location of the building is not communicated herein. In the aim to restore and conserve this historical patrimony, the present research study aims to help the restorer to redesign or reformulate the most compatible and adequate mortars to use in the rehabilitation. In this case, several samples were obtained and subjected to visual and microscopical analyses to gather as much of information, see Fig. 1. All the information obtained are used in order to determine the aggregate binder ratio (A/B), using two techniques the DIA and the chemical analyses.

2. Materials and testing methods

2.1. Sampling

In this investigation, to avoid abnormal parameters and for the best possible representativeness of the study's results, careful attention was paid to the selection and drilling operation of the different mortars to study. The mortars and the filling masonry materials were photographed and presented in the following table before the preparation of the thin-section, see Table 1. Each mortar used as coating or filling materials between blocks and stones were obtained from a place and has a name according to a location where it was drilled or obtained (mortars M, filling materials TV, coating E) and outside the bracket s their visual aspect in index (coarse (g), fine (f), earth (t), joint (j)). In total, sixteen samples of mortars were obtained each one from a location in the Fort. As DLNO: mortars obtained from Demi-Lune (half-Moon) place in North-West direction, DLSO: mortars obtained from Demi-Lune place in South-West direction, BSO: mortars obtained from Bastion place in South-West direction, BNO: mortars obtained from Bastion in North-West direction

2.2. Characterization and analysis methods/equipment

2.2.1. Visual and optical observations

The obtained samples of mortars, thin-sections see Fig. 2, were prepared for microstructures analysis as (petrography, identification of the filler or binder and the structure of the matrix), chemical analyses by FX,

X-ray Diffraction (XRD) for mineralogical analyses and Scanning Electron Microscopy (SEM) and EDX analyses. These analyses are a tool in order to identify the type of the obtained mortars and will be used to estimate the aggregates/binder ratios.

Two methods were used to determine the aggregate/binder ratio, the DIA method and the chemical analyses.

2.2.2. Digital image analyses and chemical analyses

DIA today represents a quick and efficient way to characterize historic mortar samples [21]. This method makes it possible to approximately estimate the aggregate/binder ratio, especially for lime-based historic mortar. Mainly there are two different methods for the quantitative estimation of the proportions of historic mortars. These methods are [27, 28] in TNO Report 2000-BTMK-R0081. While, The RILEM TC COM-C1 and C2 were developed based on the [27] method.

For the DIA, the method adopted is that described in the RILEM recommendations 'RILEM TC 167-COM/C2 [29] reported in [30].

In this case, thin sections were prepared for the microscopical analyses. Then, an optical microscope was used to take the images needed for the analyses, see Fig. 3.

In this investigation, several magnifications were used and the magnification that gives the best images were selected and processed. Therefore, the software program **ImageJ** open access source was used for the digital elaborations and the area quantitative measurements. Using ImageJ and after several trials and treatment (calibration) of the obtained images, the aggregates were separated from the binder using a thresholding operation (an example of the obtained image is given below in Fig. 4).

The ratios were determined and they are summarized in Table 5. However, the calculation of these ratios was not based just on the thresholding area obtained but based following the RILEM TC COM-C2 [29] where the ratio of the aggregates/binder is equal to F were:

$$F = \alpha \times \frac{\text{aggregate volume}}{\text{binder volume}} \quad (1)$$

Table 1. Macroscopical aspect of the mortars obtained for investigation.

Ref.	Visual aspect of the mortars and the filling materials			
	Sampling area: Half-moon (DL)			
(DLSO M) _g		(DLSO M) _f		
(DLSO) TV _i		(DLE M) ₄₋₃		
(DLE M) ₂₋₂		(DLE M) ₅₋₃		
(DLE M) ₁₋₁		(DLE M) ₃₋₃		
(DLNO)TV _g		(DLNO)TV _i		

(Continued).

Table 1. Contined.

Sampling area: Bastion North West (BNO)			
(BSO I)TV ₁ Doubted to be the most ancient		(BSO E)TV ₂	
Sampling area: Bastion North West (BNO)			
(BNO I)TV _g		(BNO I)E ₁	
(BNO E)M		(BNO E) _j joints	
Sampling area : carrière near the site			
Earth MC			

Where: α is a correction coefficient depending on the density of the paste and the aggregates.
Based on the hypothesis of historical mortars composed essentially of lime (confirmed next), the coefficient α is expressed by the following relation Eq. (2). Thus, for all mortars based on lime and siliceous aggregates, and according to RILEM TC COM-C2 [29] this coefficient α is found equal to 1.57.

Moreover, it worth to mention that F is based on 2D calculation where area of the aggregates and lime were determined and not volumes.

$\alpha =$

$$\frac{\text{density of aggregates} \times \text{molar weight (SiO}_2\text{)}}{\text{density of the binder} \times \text{molar weight (Ca (OH)}_2\text{)}} \quad (2)$$

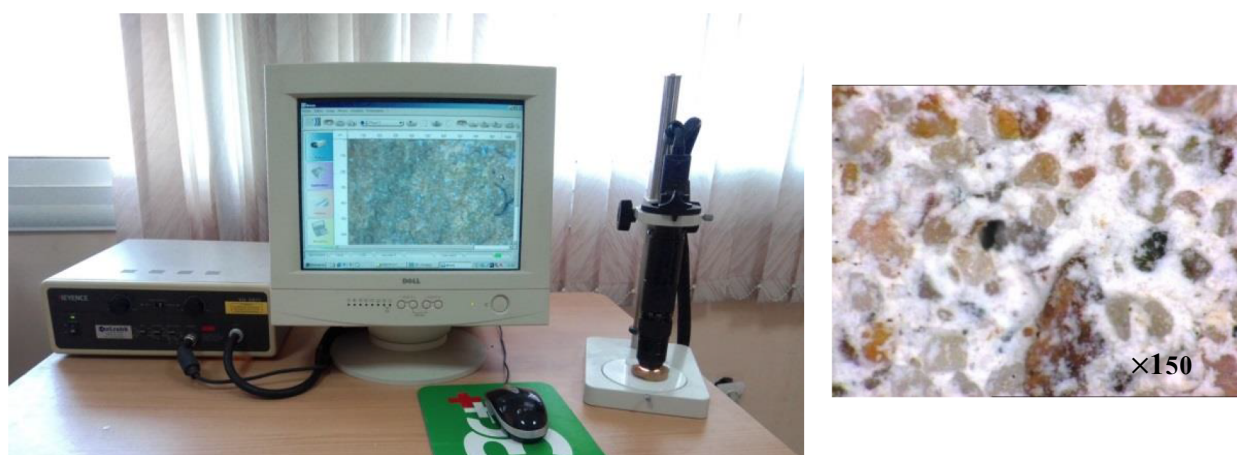


Fig. 3. Video microscope used for take the macroscopic images needed for DIA method and the corresponding image taken at magnification ($\times 160$).

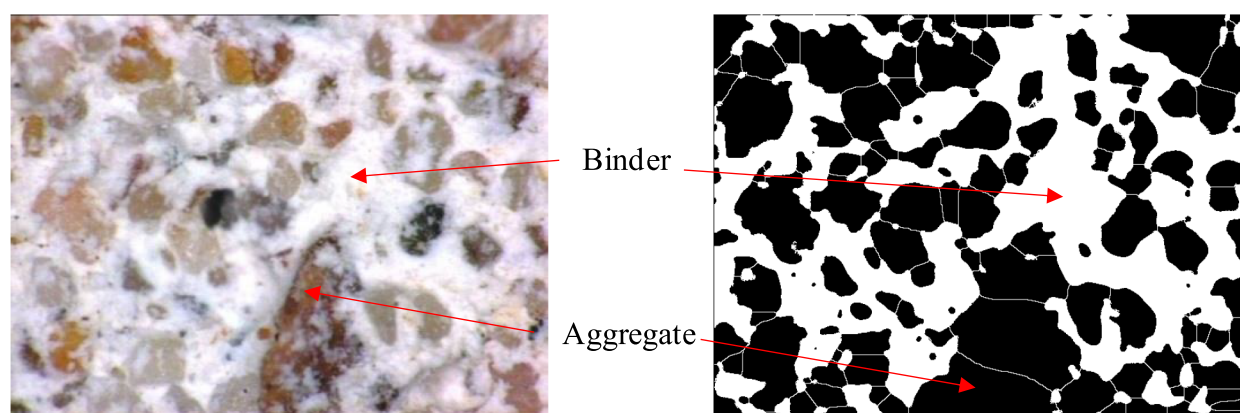


Fig. 4. Example application of the DIA method and the thresholding operation applied on $(DLSO M)_t$.

Where

The density of aggregates (sand) is estimated at 2.3 g/cm^3 ; the molar weight of SiO_2 is equal to 60.8 g/mol ; the density of the binder of a lime-based mortar is 1.2 to 1.3 g/cm^3 . The molar weight of Ca(OH)_2 is equal to 74 g/mol .

After determination of the ratios following the DIA method, and due to its sensitivity and limitation a second method based on chemical treatment then was performed.

In practice, many methods are available, the [31], which is usually used in the Nordic countries. However, other methods as recommended in the RILEM TC-167, proposed by [32]. The last one was selected to study the historical mortars of the investigated fort. The method is based on subjecting the mortars to an acidic solution to separate the binder from the aggregates. This is by using a chlorohydrin acid to attack the carbonates of the binder. Thus, the soluble silica is determined using sodium carbonates.

The adopted operating mode is summarized in the flowchart below, Fig. 5.

In order to evaluate the strength of the mortars it was possible to carry out compressive tests on one type of historic mortar. The test was carried out using a sophisticated universal machine MTS criterion C45-305E, the equipment used is a $1/1000\ 000$ precision machine with 300kN force sensor (see Fig. 6). The rate of loading used in this test was a constant load of 0.5 MPa/s on samples having a 50 mm diameter with double slenderness.

2.2.3. Microscopical analyses

In this investigation and to identify the nature of the tested samples microscopical analyses have been carried at both laboratories physical and chemical analyses scientific et technical research center and the national office of geological analyses and mines. The SEM used for the analyses was type Quanta 250 of the FEI company and some points was analyzed

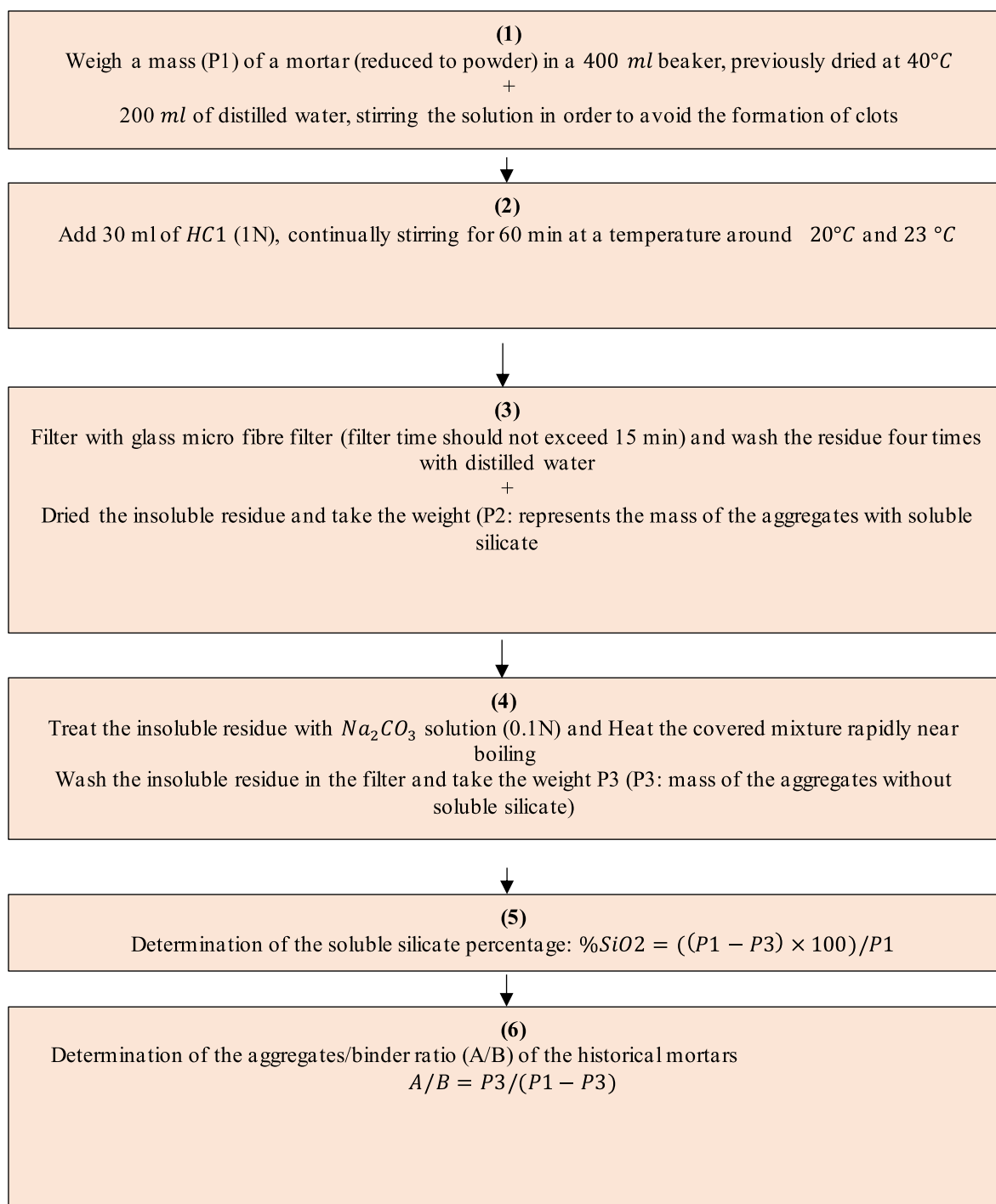


Fig. 5. Flowchart of the chemical analyses steps to estimate the historic mortar A/B ratio.

to identify their chemical composition using X-ray fluorescence. Besides, the equipment used for the X-ray diffraction analyses to identify the mineralogical phases is a Xpert-Pro diffractogram. The identification of the mineralogical phases was done using a DIFFRAC. EVA V4.0 software comparing the results obtained with the PDF-2-2004 and with the saved scan.

3. Results and discussions

3.1. Visual inspection of the historic mortars

Before starting the analyses, the mortars samples and their macroscopic aspect were described in Table 2. However, it is worth to mention that the whole mortar the aggregate distribution is heterogeneous.



Fig. 6. MTS machine used for the compressive test of the historic mortar.

3.2. Chemical/mineralogical composition and microstructure analyses

The chemical and mineralogical compositions of the mortars are summarized in [Tables 3](#) and [4](#), while representative SEM/EDX maps and XRD diffractograms are shown in [Figs. 7](#) to [16](#). The main oxides identified are SiO_2 , CaO , Al_2O_3 , and Fe_2O_3 , with minor Na_2O and MgO contents. These findings are consistent with the mineralogical phases revealed by XRD, including quartz, calcite, dolomite, muscovite, kaolinite, halite, and albite.

SEM/EDX elemental distributions provide further insight into the microstructural arrangement of these phases. Calcium is mainly associated with calcite (CaCO_3), resulting from carbonation of portlandite $\text{Ca}(\text{OH})_2$. Only trace amounts of free CaO or $\text{Ca}(\text{OH})_2$ appear to persist. This indicates that carbonation is an active process, producing a densified surface layer but at the expense of pore solution alkalinity. Silicon is largely detected as quartz (SiO_2), reflecting the contribution of siliceous aggregates, while minor signals correspond to aluminosilicate phases such as muscovite and kaolinite. The co-localization of sodium and chlorine signals confirms the presence of halite (NaCl). This salt may originate either from raw materials or environmental exposure and has important implications for durability, since chloride ingress can depassivate steel reinforcement and crystallization cycles may induce microcracking.

The mineralogical assemblage exerts a direct influence on the hydraulic and durability behavior of the mortars. Quartz, being inert, enhances mechanical

stability but does not contribute to hydration or pozzolanic activity. Calcite is beneficial for filler effects and matrix densification, but its predominance also indicates carbonation, which may reduce long-term alkalinity. Dolomite can improve compactness but is potentially vulnerable to dedolomitization under aggressive conditions. The presence of clay minerals (muscovite, kaolinite) introduces additional aluminosilicates; these may display limited pozzolanic reactivity but are generally associated with higher porosity and weaker interfacial zones. Finally, halite is a key deterioration factor, favoring salt weathering and chloride-induced corrosion risks.

Overall, the combined XRD and SEM/EDX analyses demonstrate that while the mortars benefit from stable siliceous aggregates and carbonate-based densification, their long-term performance may be compromised by extensive carbonation and the presence of soluble salts. These microstructural features must therefore be considered in durability assessments, particularly for exposure to marine or chloride-rich environments.

3.3. DIA and chemical analyses results

3.3.1. DIA results

The A/B ratios of the investigated historic mortars are summarized in [Table 5](#). before discussing the results found, it is worth to mention that the DIA method is an effective and simple tool to obtain a rapid estimation of the A/B ratio of historic mortars. However, careful attention should be made when selecting the area because in some cases the results

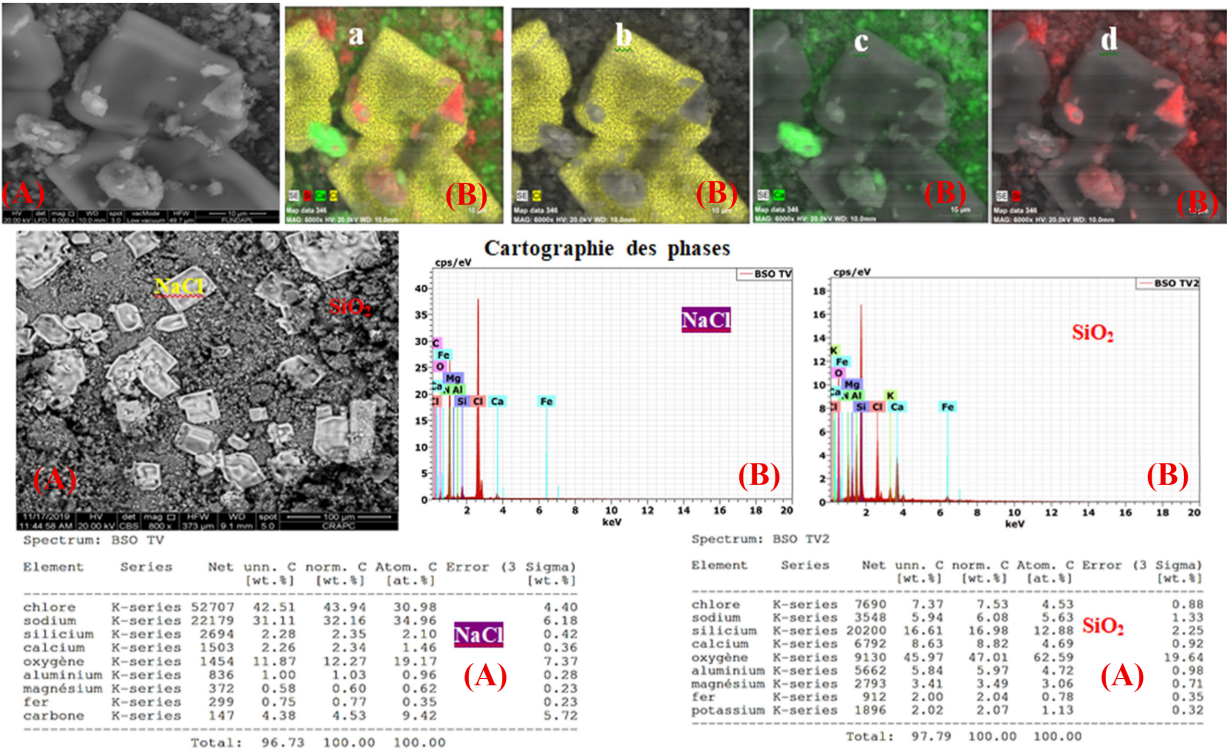


Fig. 7. (A) Combined microscopic analyses SEM (EDX) and (B) X-ray diffraction with cartography of the phases (Ca, Si et Cl) of BSO TV and BSO TV2.

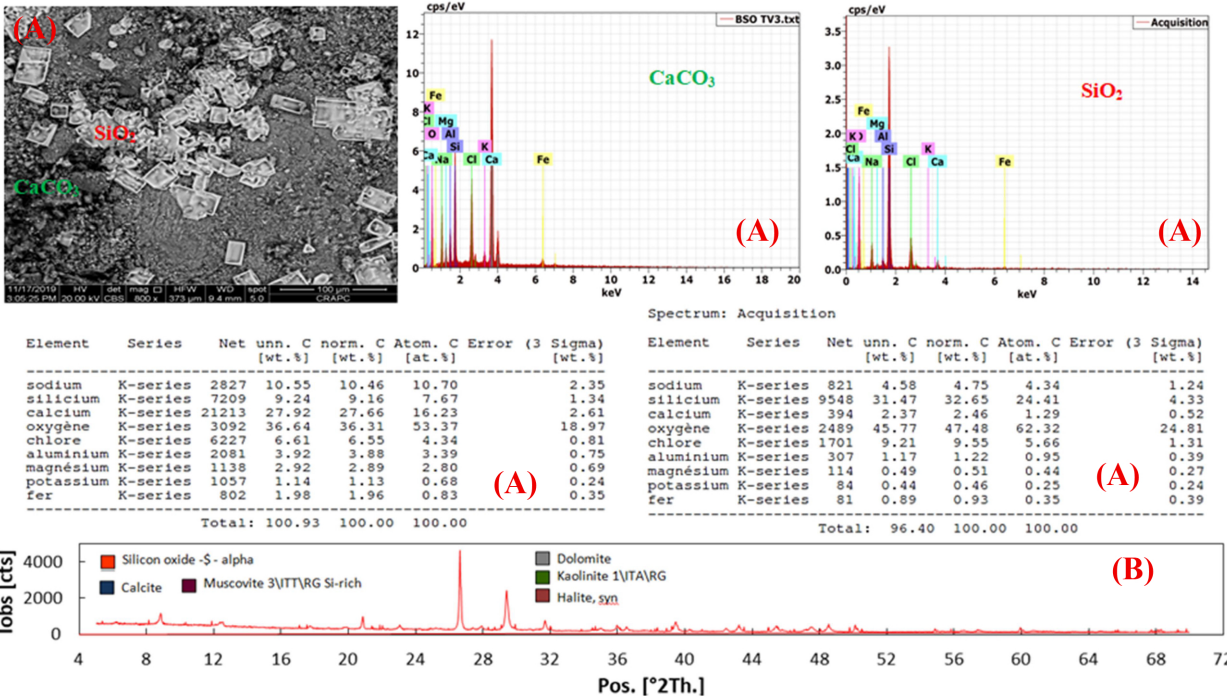


Fig. 8. (A) Combined microscopic analyses SEM (EDX) and (B) X-ray diffraction of the phases (Ca, Si et Cl) of (BSO E) TV₂, (BSO I) TV₁ et (DLNO) TV_g, (DLNO) TV_t.

Table 2. Macroscopic description of the mortars and the heterogeneous filling mortars.

N	Ref.	Probable designation	Color	Conservation state	Coarse aggregates average size, in mm	Visible sand	Gravel
01	(DLSO M) _g	Lime mortar with sand and aggregates	White - light gray	Soft/flexible	0 to 20	Black-gray-white grains (brilliant)	Alluvial -gray and white
02	(DLSO M) _f	Lime mortar with sand and aggregates	Beige	Soft/flexible	0 to 20	Gray grains (brilliant)	Rare Gray
03	(DLSO) TV _t	Earth-based filling materials	Red ocher	Soft/flexible	0 to 10	White grains (brilliant)	–
04	(DLE M) ₄₋₃	Lime mortar with sand and aggregates	White	Soft/flexible	0 to 3 0 to 20	White grains (brilliant)	Gray
05	(DLE M) ₂₋₂	Lime mortar with sand and aggregates	White	Pretty hard	0 to 2	White grains (brilliant)	–
06	(DLE M) ₅₋₃	Lime mortar with sand and aggregates	White – beige	Pretty hard	0 to 40 0 to 20	White and gray grains (brilliant)	Alluvial, white, beige, gray
07	(DLE M) ₁₋₁	Lime mortar with sand and aggregates	White – beige	Pretty hard	0 to 2	Brilliant white	–
08	(DLE M) ₃₋₃	Lime mortar with sand and aggregates	White - light brown - gray	Friable	0 to 5	Gray - black	–
09	(DLNO)TV _g	Filling materials based on earth, sand and aggregates	Beige - ocher	Pretty hard	0 to 40	Gray-black brilliant	Alluvial – white, ocher and gray
10	(DLNO)TV _t	Earth-based filling materials	Ocher and red	Soft	0 to 40	White gray-black Brilliant	Alluvial white gray and black
11	(BSO I)TV ₁	Matériau de remplissage à base de terre avec des grumeaux de chaux, gravillon et sable	Dark brown	Soft	0 to 20	White - black	Alluvial gray, black
12	(BSO E)TV ₂	Earth mortar- lumps of lime and coarse aggregates	Ocher and dark brown	Soft and crumbly	0 to 40	gray - white	Alluvial gray
13	(BNO I)TV _g	Mortier de terre riche en charges grossières	Beige – white	Pretty hard	0 to 20	Brilliant gray	Alluvial gray, white and black
14	(BNO I)E ₁	Enduit de chaux et de sable	White gray	Hard	0 to 3	brilliant white and black	–
15	(BNO E)M	Lime mortar with coarse aggregates (sand and gravels)	Beige -white	Crumbly	0 to 20	brilliant white and gray	Alluvial gray, white and black
16	(BNO E)j joints	Lime mortar with sand	White gray	Soft and crumbly	0 to 3	Brilliant white, beige and black	–
17	Quarry earth : MC	Earth with coarse aggregates	Red ocher	Soft	0 to 40	–	Alluvial white, gray and black

are too sensitive to the chosen points and the scatter between two different area for the same mortar sample can be high which is due to the high heterogeneity of the historic mortars. So, if this is observed it is recommended to have an estimation through scanning several areas and calculating then the mean value.

For the investigated historic mortars of the fort, the A/B ratios varied between 1.3 and 3.52. However, the majority the A/B ratio of the historic mortars such as (DLSO M)_f, (DLE M)₂₋₂, (DLE M)₅₋₃, (DLE M)₃₋₃, (DLE M)₄₋₃, (DLSO M)_g are between 2 and 3.52 which is in lines with those found for historic mortars

by [33, 34]. The obtained result confirms the truth concerning the formulation of mortars in the past where the volume of the aggregates is twice or three times higher than the binder. Meanwhile, for (BNO I)E and (BNO E)j the A/B ratio was less than 2. This is due that mortars exposed directly to the sea as BNO deteriorate more than the others that do not exposed directly to the sea as the case of the DLEM or DLSO mortars.

At the end, it is worth to mention that DIA method was not applicable for all mortars as the chemical one (in the next) specially in the case of earth-based fill

Table 3. Chemical analyses result of the historic mortars.

N°	Designation	Chemical elements rate (%)									
		SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	P ₂ O ₅	K ₂ O	TiO ₂	MnO	Na ₂ O
1	(BNO I)TV _g (DLNO) TV _t (DLNO) TV _g	43.54	9.43	5.82	16.41	2.99	0.25	2.04	0.63	0.09	2.10
2	(BSO I) TV ₁ (BSO E) TV ₂	43.42	7.22	4.46	19.41	2.06	0.24	1.93	0.62	0.06	2.78
3	(BNO E)M (BNO E)j	32.08	1.95	1.79	28.19	4.51	0.61	0.41	0.18	<0.05	2.79
4	(DLSO M) _g	16.91	1.82	2.54	37.25	2.17	0.49	0.37	0.16	0.05	6.67
5	MC	46.03	7.29	4.5	20.83	1.30	0.28	0.74	0.5	0.07	<0.05
6	(DLSO M) _f (DLE M) ₁₋₁ (DLE M) ₂₋₂ (DLE M) ₃₋₃ (DLE M) ₄₋₃ (DLE M) ₅₋₃ (BNO I)E ₁	17.15	1.22	1.6	41.54	1.55	0.63	0.27	0.17	<0.05	1.74

Table 4. Mineralogical nature of mortar samples.

N°	Designation	Mineralogical elements rate (%)						
		Quartz	Calcite	Dolomite	Muscovite	Koalinite	Halite	Albite
1	(BSO E) TV ₂ (DLNO) TV _t (DLNO) TV _g	57.4	14.9	6.9	11.9	6.9	2	–
2	(BSO I) TV ₁ (BSO E) TV ₂	61	27	7	–	–	5	–
3	(BNO E)M (BNO E)j	61.4	17.8	4	16.8	–	–	–
4	(DLSO M) _g	40	50	4	–	–	6	–
5	MC	57	24	2	–	–	–	17
6	(DLSO M) _f (DLE M) ₁₋₁ (DLE M) ₂₋₂ (DLE M) ₃₋₃ (DLE M) ₄₋₃ (DLE M) ₅₋₃ (BNO I)E ₁	48.5	43.6	5.9	–	–	2	–

materials because it is quasi-impossible to separate the granular filler from the matrix

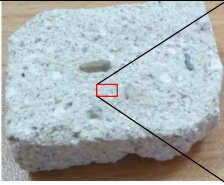
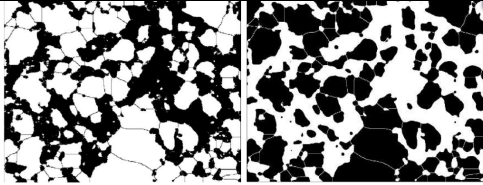
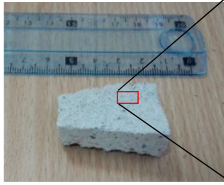
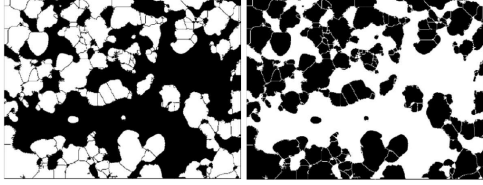
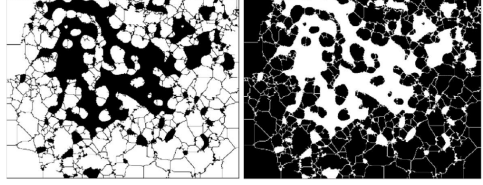
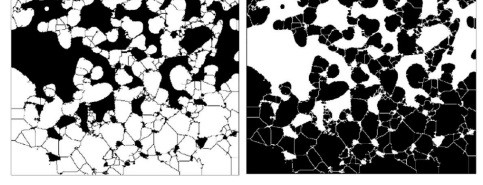
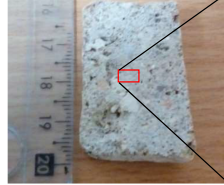
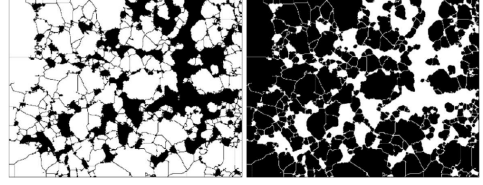
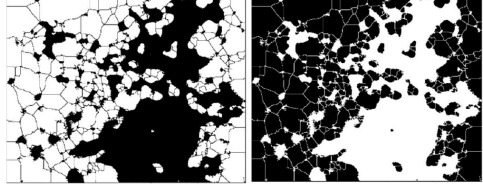
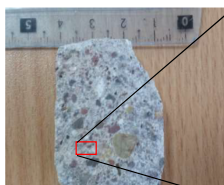
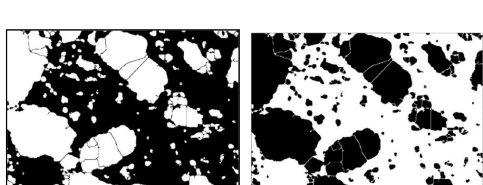
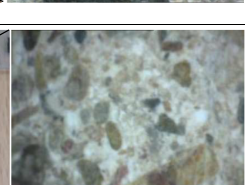
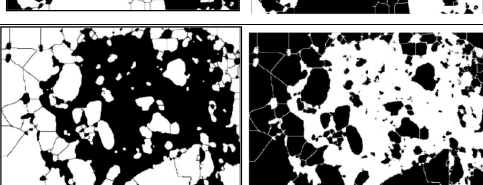
3.3.2. Chemical analyses

The results of the chemical analysis Table 6 show that most mortars present A/B ratios between 1.3 and 3, which is consistent with traditional practices of using two to three volumes of aggregates for one volume of binder. However, some mortars display ratios below 1.3 (down to 0.8), indicating mixtures with a relatively high binder content. These low values can be attributed either to specific construction practices (e.g., finishing or repair mortars intentionally enriched in lime to enhance plasticity and adhesion), to mineralogical characteristics (mortars poor in siliceous phases such as clays, resulting in binders almost exclusively lime-based), or to deterioration phenomena (loss of aggregates by weathering and

leaching). Conversely, very high A/B ratios (up to 4.0) correspond to lean mortars with abundant aggregates, generally used as bulk or bedding layers. This variability reflects differences in the functional role of the mortars, the raw material selection, and the effects of long-term alteration.

It is also noted that the samples often show considerable differences in coloring and texture, which can be related to the purity of the binder, the nature, size, and distribution of sand grains, as well as possible interactions with the surrounding environment. These factors, although evident, are difficult to quantify and interpret systematically. Regarding the percentage of fillers, it is expressed as the ratio of the insoluble residue to the total mass of the mortar sample. Finally, the soluble fraction of the matrix, in addition to calcium originating from carbonated lime and limestone, contains other soluble elements

Table 5. A/B ratio of mortars of the historical building.

Reference	Photo of the mortars and the corresponding macroscopic image		Digital Images Analyses		F : A/B
(DLSO M)f					2.0
(BNO I)E					1.5
(DLE M)2-2					3.2
(DLE M) 5-3					2.6
(DLE M)3-3					3.5
(DLE M)4-3					2.2
(BNO E)j					1.3
(DLSO M)g					2.0

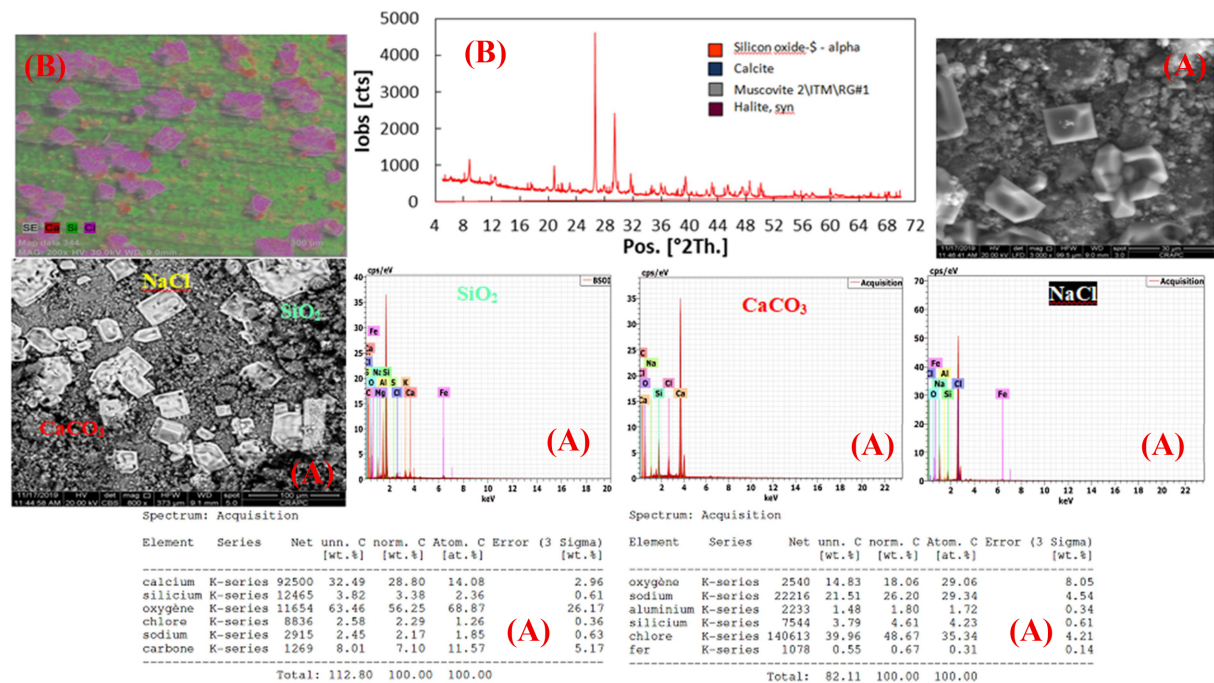


Fig. 9. (A) Combined microscopic analyses SEM (EDX) and (B) X-ray diffraction with cartography of the phases (Ca, Si et Cl) of (DLE M)₁₋₁ (DLE M)₂₋₂, (DLE M)₃₋₃, (DLE M)₄₋₃ et (DLE M)₅₋₃.

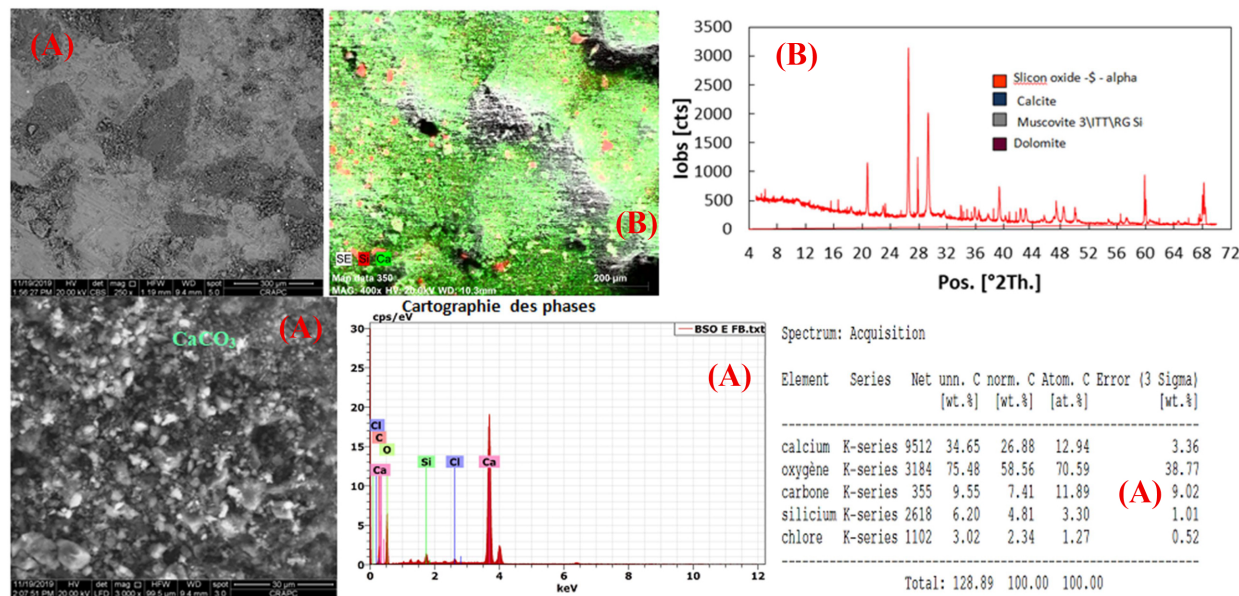


Fig. 10. (A) Combined microscopic analyses SEM (EDX) and (B) X-ray diffraction with cartography of the phases (Ca, Si) of (BNO I) E₁, (BNO I) TV_g.

mainly composed of silicates, which contribute to the hydraulic character of the mortar. However, a clear distinction between the calcite associated with the binder (CaCO₃) and that originating from limestone aggregates is not possible, which explains the relatively insignificant percentages obtained for the so-called “aggregates.”

In addition, a comparison between the results of both methods DIA and chemical one is presented in Fig. 17. From the results of the comparison, it is clear that for some mortars, approximately the same ratio was found. However, the DIA method is limited and it was not possible to apply it for all the mortars in comparison with the chemical method, which is

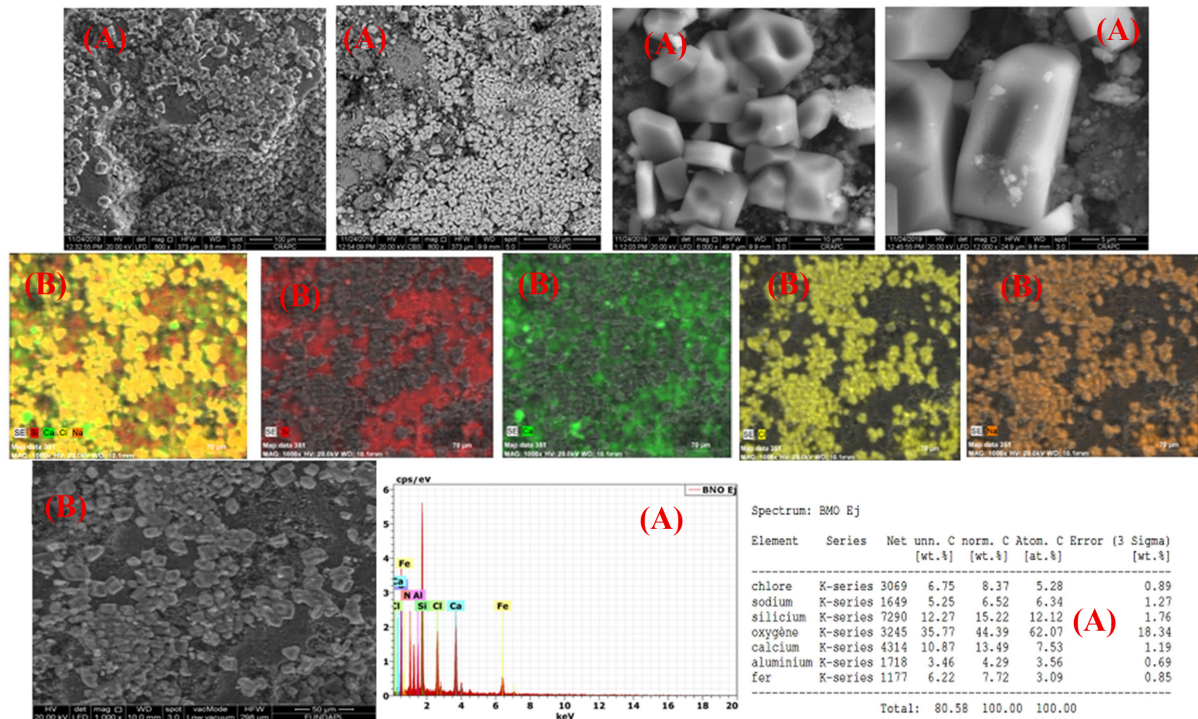


Fig. 11. (A) Combined microscopic analyses SEM (EDX) (A) and (B) X-ray diffraction with cartography of the phases (Ca, Si, Cl and Na) of (BNO E)_j, (BNO E)_M.

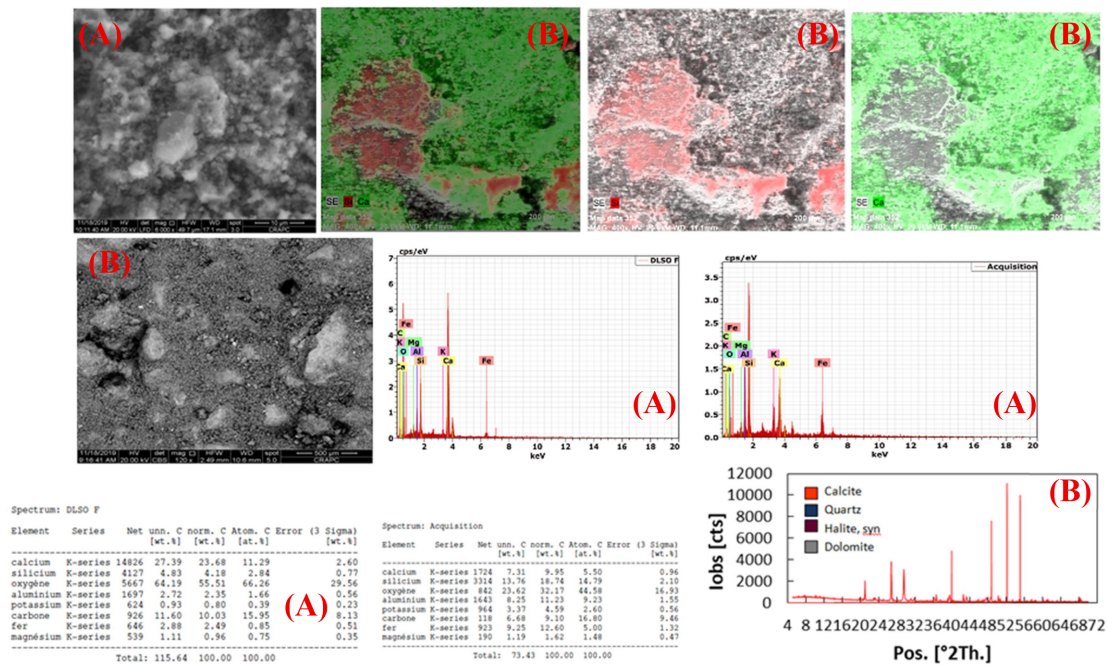
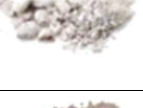


Fig. 12. (A) Combined microscopic analyses SEM (EDX) and (B) X-ray diffraction with cartography of the phases (Ca, Si) of (DLSO M)_g.

more efficient. In addition, the results based on the DIA method are based on a (2D) calculation (areas) while every particle has a 3D shape which leads to the conclusion that using the DIA method alone

is not sufficient and needs to be strengthened with other methods as the chemical one for more accurate and reliable estimation of the A/B ratios of historic mortars.

Table 6. The A/B ratios of the historic mortars based on the chemical analyses.

Mortars abbreviations	Dried mass of mortars P ₁ (g)	Mass of the aggregates + soluble silicate P ₂ (g)	Mass of the soluble silicate in the mortar's matrix (g)	Mass of the aggregates without soluble silicate P ₃ (g)	Mass of the binder (B) (g)	Macroscopic appearance of the residue at the end of the analyses	Macroscopic appearance of the filtrate at the end of the analysis	Aggregates (A) (%)	Binder (B) (%)	A/B ratio of the historic mortars	For 1 volume of the binder (B = 1) / A
(BSO E) TV ₂	10.4	7.5	1.3	6.2	2.9 clay			59.62	27.8	1.5	1 / 1.5
(DLNO) TV _B (DLNO) TV _t	10.1	5.1	0.4	4.7	5 clay			46.53	49.5	0.9	1 / 0.9
(BSO I) TV ₁	10.5	6.6	0.6	6	3.9 clay			57.14	37.1	1.54	1 / 1.54
MC	12.2	5.4	0.0	5.4	6.8 clay		Traces after decantation difficult to weigh	44.26	55.7	0.8	1 / 0.8
(BNO I) TV _g	17.6	13.4	0.0	13.4	4.2 clay		Traces	76.14	23.8	3.2	1 / 3.2
(DLSO M) _g	11	8.7	0.3	8.4	2.3 clay		Traces	76.36	20.9	3.65	1 / 3.65
(DLNO) TV _g	10.1	6.9	0.1	6.8	3.2 clay		Insufficient components to dry it and weight it	67.33	31.6	2.1	1 / 2.1
(BNO E) M	21.9	17.5	0.0	17.5	4.4		Insufficient components to dry it and weight it	79.91	20.09	4.0	1 / 4.0
(DLNO) TV _t	9.7	6.5	0.1	6.4	3.2 clay		Insufficient components to dry it and weight it	65.98	32.9	2.0	1 / 2.0
(DLSO M) _r	10.5	6.9	0.2	6.7	3.6		Insufficient components to dry it and weight it	63.81	34.2	1.8	1 / 1.8

(Continued).

Table 6. Continued.

Mortars abbreviations	Dried mass of mortars P ₁ (g)	Mass of the aggregates + soluble silicate P ₂ (g)	Mass of the soluble silicate in the mortar's matrix (g)	Mass of the aggregates without soluble silicate P ₃ (g)	Mass of the binder (B) (g)	Macroscopic appearance of the residue at the end of the analyses	Macroscopic appearance of the filtrate at the end of the analysis	Aggregates (A) (%)	Binder (B) (%)	A/B ratio of the historic mortars	For 1 volume of the binder (B = 1) / A
(DLSO M) _r	10.5	6.9	0.2	6.7	3.6		Insufficient components to dry it and weight it	63.81	34.2	1.8	1/1.8
(DLE M) 2-2	10.1	7.6	0.34	7.5	-		Insufficient components to dry it and weight it	74.26	24.7	3.0	1/3.0
(DLE M) 4-3	31.5	20.8	0.51	20.8	-		Insufficient components to dry it and weight it	66.03	33.9	1.9	1/1.9
(BNO E) _j	15.8	8.1	1.02	7.8	-		Insufficient components to dry it and weight it	49.37	48.7	1.0	1/1.0
(BNO I) E ₁	10.2	6.9	0.5	6.8	-		Insufficient components to dry it and weight it	66.67	32.3	2.0	1/2.0
(DLE M)3-3	33.2	24	0.38	24	-		Insufficient components to dry it and weight it	72.29	27.7	2.6	1/2.6
(BNO E)E ₂	10.1	5.1	1.02	5	-		Insufficient components to dry it and weight it	49.50	49.5	1.0	1/1.0
(DLE M) 1-1	14.9	8	---	--	-		Insufficient components to dry it and weight it Mortars made just with lime (lime paste) without aggregates				

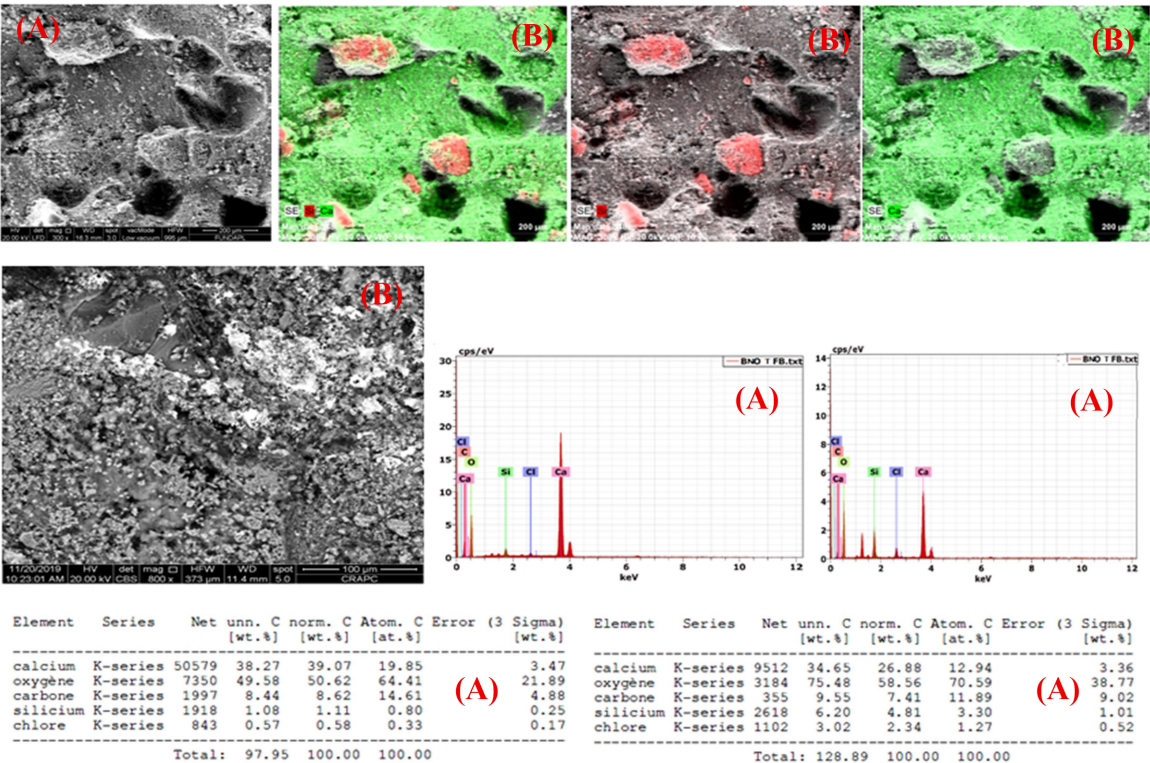


Fig. 13. (A) Combined microscopic analyses SEM (EDX) and (B) X-ray diffraction with cartography of the phases (Ca, Si et Cl) of (BNO)E1M, (BNO)IE1.

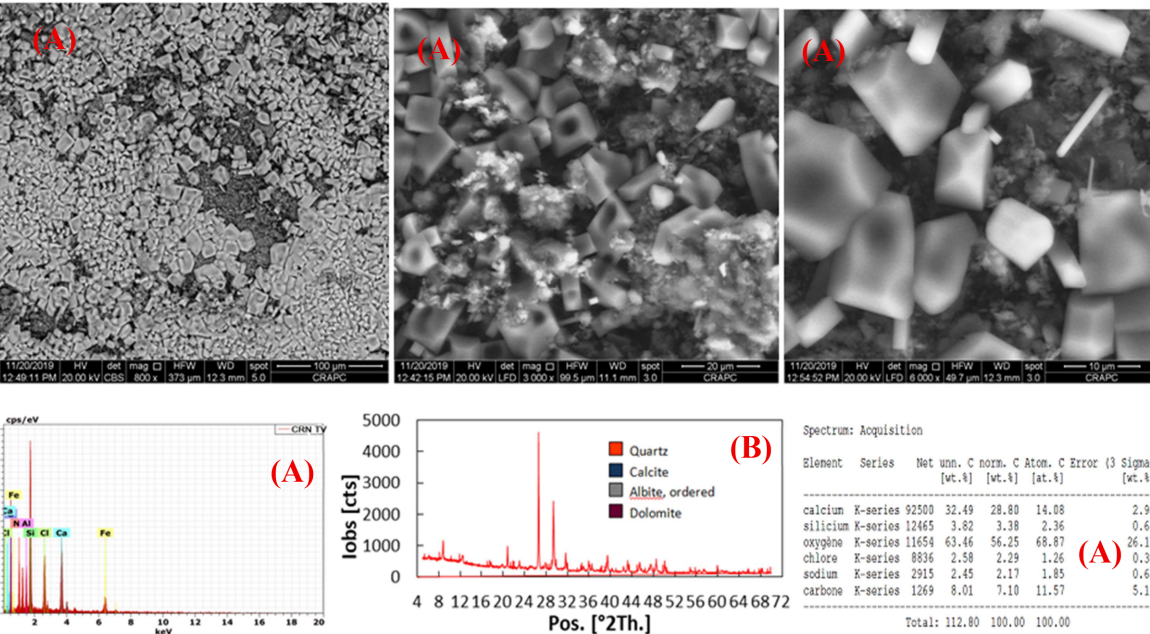


Fig. 14. Combined microscopic analyses SEM (EDX) and X-ray diffraction of (DLSO)TV1.

3.4. Historic mortar compressive strength

The stress-strain curves of the compressive test of the historic mortar samples are presented in Fig. 18.

The average compressive strength was around 6 MPa, which is the same results found by [35]. In addition, the tested mortars all present a quasi-brittle behavior. Meanwhile, it is worth to mention that the mechan-

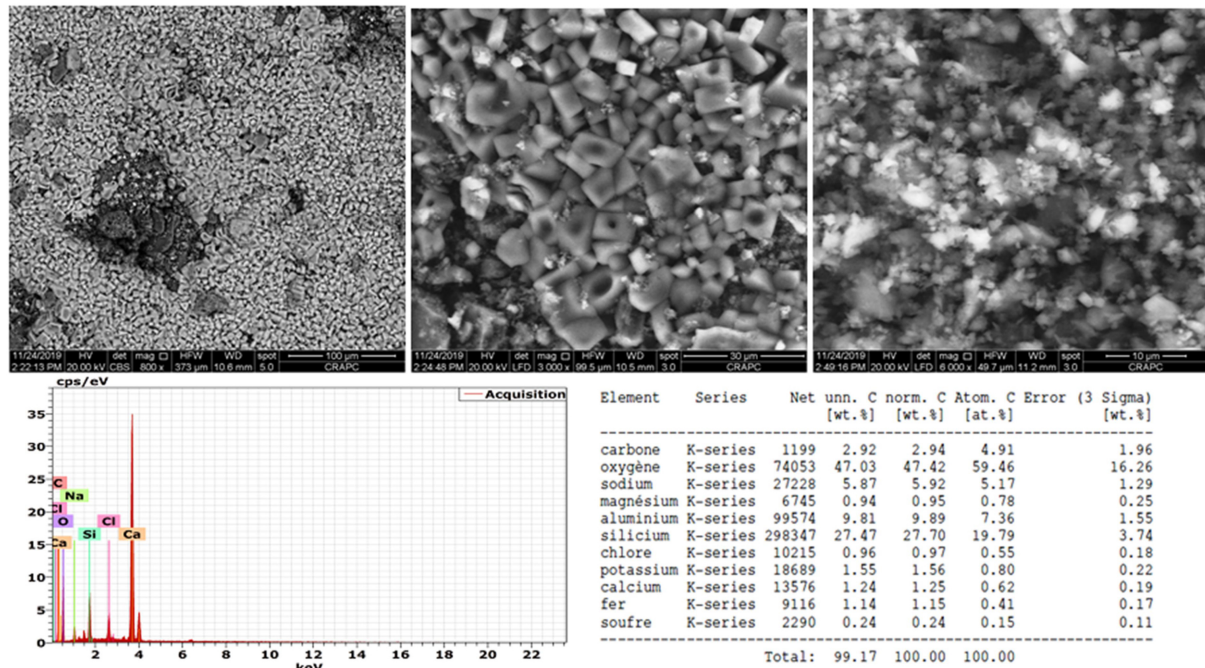


Fig. 15. Combined microscopic analyses SEM (EDX) of (DLNO)TV_g, (DLSO M)_g.

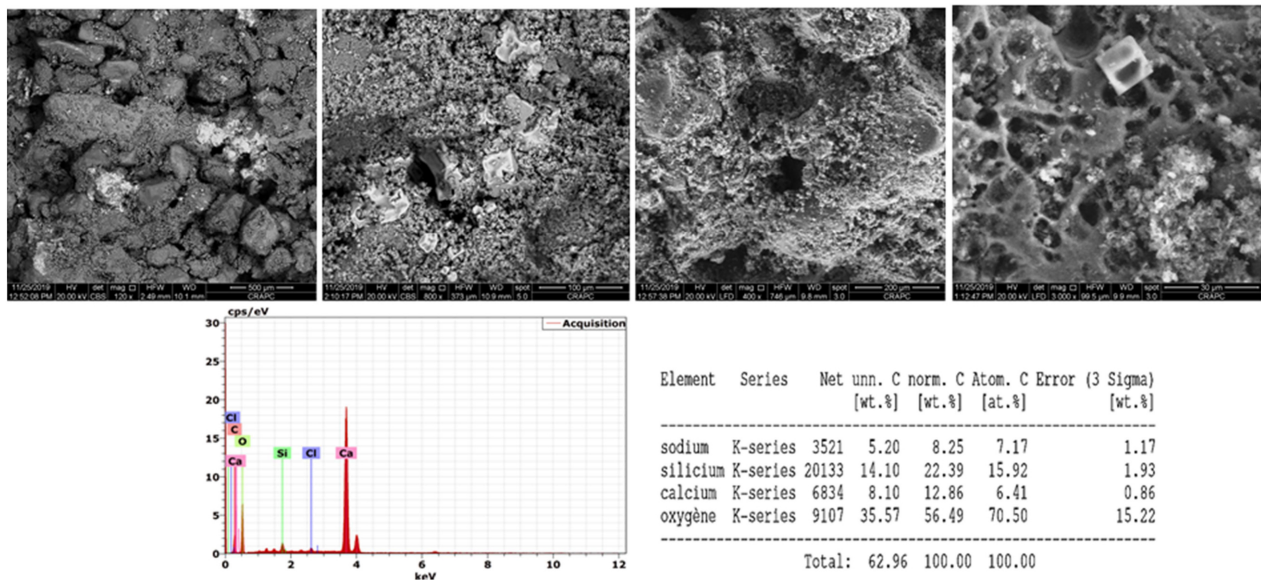


Fig. 16. Combined microscopic analyses SEM (EDX) of (DLE M)₂₋₂ (DLE M)₃₋₃, (DLE M)₄₋₃, (DLE M)₅₋₃.

ical properties of a mortar depend of the composed materials, their quantity, their chemical composition and particle size distribution of the aggregates. In the past, historic mortars were based on lime, various aggregates (often sand) and water. Thus, the quality and durability of these types of mortars depend of the type of lime used (hydraulic or non-hydraulic). Furthermore, it is well known that non-hydraulic lime-based mortar is characterized by a low com-

pressive strength [16], which varies between 0.5 and 3 MPa [36]. While, for hydraulic-lime-based mortar the compressive strength can range between 11 and 15 MPa [37]. Others scientific findings stated that the compressive strength of hydraulic lime-based mortar can be quite low which depends on the quality of the materials used and other uncontrolled factors [36].

In the same context, historic mortars of a Turkish building in Becin [38] that belongs to 14th century

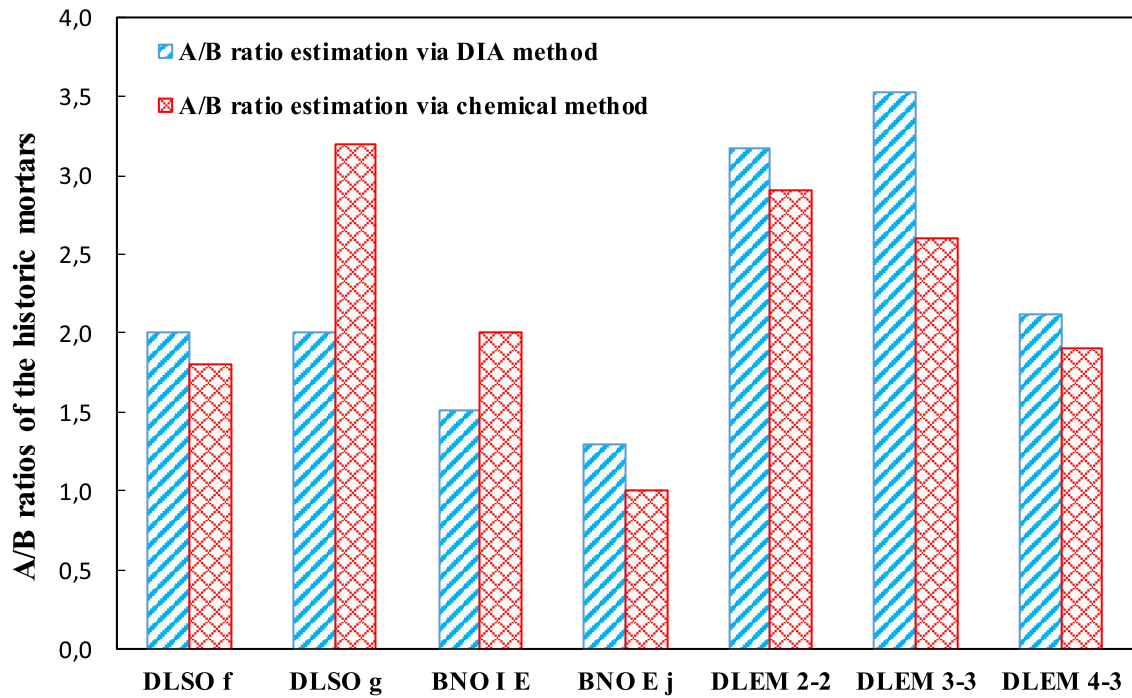


Fig. 17. Comparison of the Aggregate/binder ratio of the same studied mortars of the historical building using both DIA and chemical methods.

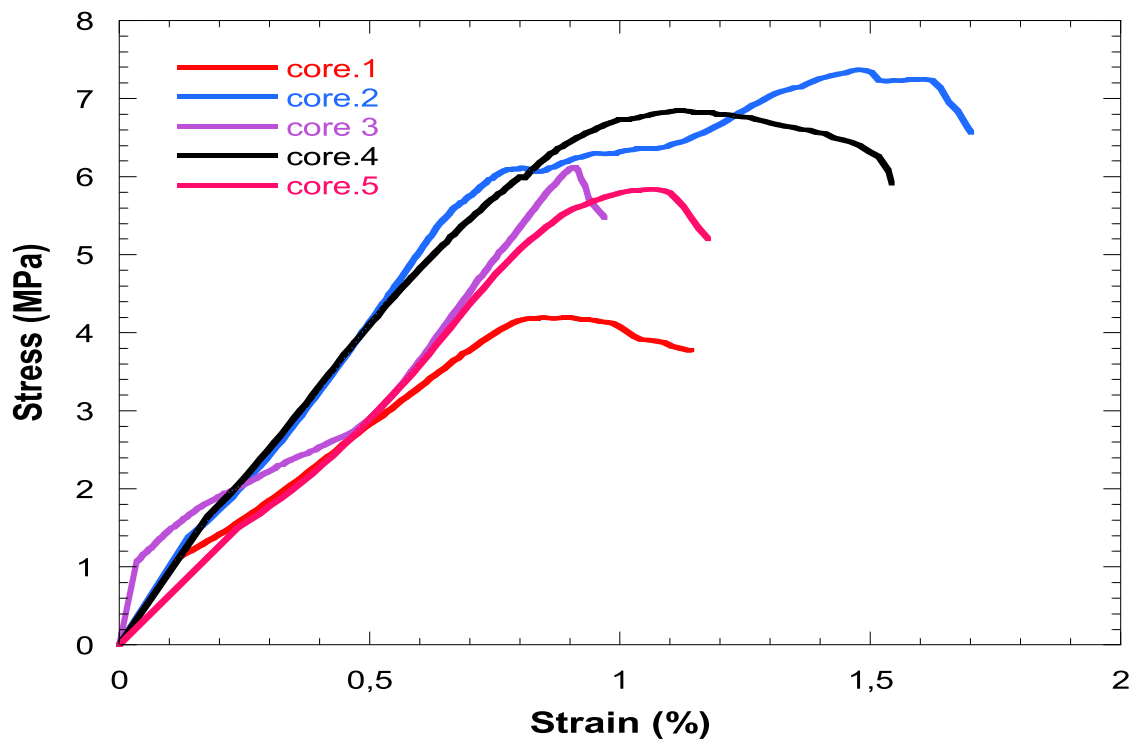


Fig. 18. Stress- strain curves of the tested historic mortar compressive strength.

found that the compressive strength of hydraulic lime based-mortars are around 1 and 3.86 MPa. Likewise, it is mentioned that the average strength values can ranged between 4.2 to 7.4 MPa. Some research as

[36] found that a lime-based mortar containing aggregates with a fraction of 0 to 4 mm in diameter has high compressive strength compared to mortars containing aggregates with a fraction of 0 to 2 from

0 to 8 or from 0 to 16 mm in diameter. However, due to small size of the tested cores the size effect is a parameter that should not be ignored by practitioners. Finally, we can state that the value of the compressive strength of the historic mortars tested are quite high which probably indicates the use of hydraulic lime.

4. Conclusions

To the best knowledge of the authors, this is the first investigation carried out on such an important patrimonial building in Algeria. The B/A ratios of the historic mortars are estimated. Experimental campaigns to characterize the mortars are carried out. Based on the results of the characterization and the two methods followed to estimate the historic mortars A/B ratios, the following conclusions can be derived.

- Based on the SEM (EDX), X-ray Diffraction, petrographic, chemical and mineralogical analyses of the historic mortars and filling materials the mortars are mainly composed of binders based on lime and earth, limestone aggregates and quartz sands. Chloride contamination of materials is also noted due to the site's geographical location (Gulf of Oran).
- The A/B ratios of the historic mortars of the historical building were approximately between 1 and 3 which lines with the truth about the formulation of the historic mortars used in the historical buildings in the past where the mortars were designed mixing nearly two until three volumes of aggregates for one volume of the binder.
- The A/B ratios of the historic mortars following the DIA method and the chemical analyses were compared. Based on the results, The DIA could be used as an alternative method that gave preliminary and approximate results. However, The limitations of DIA can be compensated by techniques such as thermogravimetric analysis (TGA) or FTIR for soil-based mortars.
- The A/B ratios of the historic mortars were determined using the DIA and chemical methods. Both methods can be used together to have the best characterization. However, the DIA method is limited which has not been effective in the case of earth-based fill materials. It is virtually impossible to separate the granular filler from the matrix.
- The ratios of the mortars determined through the DIA method were too sensitive to the area selected, which suggested taking more points during the analyses. The results based on the DIA method are based on a (2D) calculation (areas) while every particle has a (3D) shape which leads to the conclusion that using the DIA method alone is

not sufficient and needs to be strengthened with other robust methods as the chemical one for more accurate and reliable estimation.

- For restoration purposes, the results highlight the importance of using lime-based mortars compatible with the original ones. It is therefore recommended to design repair mortars with similar binder-to-aggregate ratios and mineralogical compositions to ensure structural and chemical compatibility with the historical fabric.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Author's contributions

All of the authors have contributed equally to the article. Further, all of the authors have validated and approved the final manuscript.

Acknowledgements

The first author acknowledges the support of the National Center of Studies and Integrated Research on Building Engineering, Algiers, Algeria. In addition, special thanks go to the personnel of the national laboratory center for their efforts and contribution.

Ethics

There are no ethical issues with the publication of this manuscript.

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