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RESEARCH ARTICLE

Lime-based mortars with added silica fume and bioproducts for restoration and preservation of heritage buildings

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Abstract

Restoration of heritage buildings requires an appropriate choice of materials, as inappropriate materials can lead to degradation rather than preservation. Hydrated lime and sand mortars are the most suitable for masonry cladding. However, they have low durability regarding current requirements. This work investigates the use of silica fume and/or biopolymers (egg albumen and additive derived from the reuse of milk unfit for human consumption (complexes of whey protein with κ-casein) in producing these mortars. Some mortars were characterized by mechanical properties (compressive and diametric strength, absorption rate) and chemical properties (SEM, Infrared spectrum). The research revealed that silica fume addition allows an increase of more than 50% in the mechanical strength of the mortars when associated with animal protein. The value may be higher than 60%, especially for biopolymers (complexes of whey protein with κ-casein). Finally, the proteins in the mortar mixture provide intense air entry that results in the formation of more pores. This increase in voids allows more CO_2 to enter, directly contributing to a faster carbonation process and performance mortar.

Keywords Hydrated lime · Mortar · Biodegradable material · Restoration and conservation · Sustainability

1 Introduction

Cultural heritage is directly related to the general principles of sustainable growth. Therefore, heritage buildings restoration allows future generations to know and identify the history and heritage culture of cities, and the interest in this topic also includes social and economic values Innocencio et al. [18], Oliveira et al. [25], Ayat et al. [5].

Lime (i.e., lime putty, aerial lime, or hydraulic lime) is one of the oldest binders used in construction to produce mortars. In this sense, the requirements established for the use of coating mortars for old buildings are different from those prescribe for the use in new buildings, due to physicochemical, mechanical, and microstructural compatibility criteria, with hydrated lime being the most adequate, as reported by several authors Innocencio et al. [18], Verdum et al. [32], Branco et al. [7].

There are some problems associated with lime mortar used the repair of heritage buildings and new constructions. They usually are associate with different factors, such as water permeability, absorption rate, pores size, and others, that negatively contribute to its performance (Ravi et al. [34]) since the higher porosity in the lime matrix is an index in the degradation process.

For lime mortars, curing time, type of aggregate, and binder-aggregate ratio affect the mortar performance Frankeová and Koudelková [14]. Therefore, many researchers have diversified the binder-aggregate and concluded that the binder-aggregate ratio of 1:3 offers

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better mechanical properties. In addition, the recommended to avoid rounded aggregates since the shape is another important factor regarding the quality of the fresh and hardened mortar. Regarding the size of the particles, in the case of a small diameter, with finer particles, it's possible to fill the pore space between lime paste and aggregates without affecting workability (Manoharan and Umarani [22]).

In this context, the design of new mortars, durable and compatible with authentic materials, becomes essential for maintaining the integrity of the old building. The material used for the repairs must meet requirements for compatibility and strength with current environmental factors. In several countries, rehabilitation on old buildings has seen a notable increase in recent decades, consequently, the research on suitable and durable mortars too. Special attention has been provided to the compatibility of lime with different binders, such as pozzolans. Oliveira et al. [25], Branco et al. [7], Ayat et al. [5].

Therefore, silica fume is an important addition, as it is a pozzolanic material with an average diameter of around 200 nm–1 μm and has high reactivity with calcium hydroxide. These characteristics result in more cohesive mixtures. In the hardened state, mortars with additions of silica fume present good results, providing mortar mixtures with high mechanical properties Kang et al. [20].

Furthermore, several authors Ventolà et al. [31], Thirumalini et al. [33], Mydin [24], manoharan and Umarani [22], Jayasingh and Baby [19] mention the use of natural additives in lime and sand mortars that used in old buildings, which enhance their properties, such as those based on plants and organic ones, such as brown sugar, cactus extract, lentils, among others,and which enabled gains in mechanical properties through the reduction of total porosity and, consequently, ensuring good durability to the composite. Therefore, it is necessary to analyze the role of organic additives in the lime matrix from the point of view of strength and durability.

The bio-additives should regard since it supports the cement hydration process. Souza et al. [28] and Souza et al. [29] concluded provides greater flexibility in setting time, increasing the workability time of the binder and the mechanical properties performance. For lime, the organic materials resulted in a higher structure, i.e., the self-healing capacity of lime mortar with bio-additives makes it time-resistant MYDIN [24], Manoharam and Umarani [22]). Recently, García-González et al. [15] investigated bioproducts obtained from the fermentation of biodiesel's crude glycol with lime mortars that have a positive effect on workability, mechanical performance, and absorption.

In this context, this research aims to test lime and sand mortar where silica fume with/without egg albumen MYDIN [24] with/without biopolymer (complexes of whey protein with κ-casein) SOUZA et al. [28] were incorporated. The experimental quality program measures the mortars made with the proposed materials. The results were compared with the reference mixture (without the addition products).

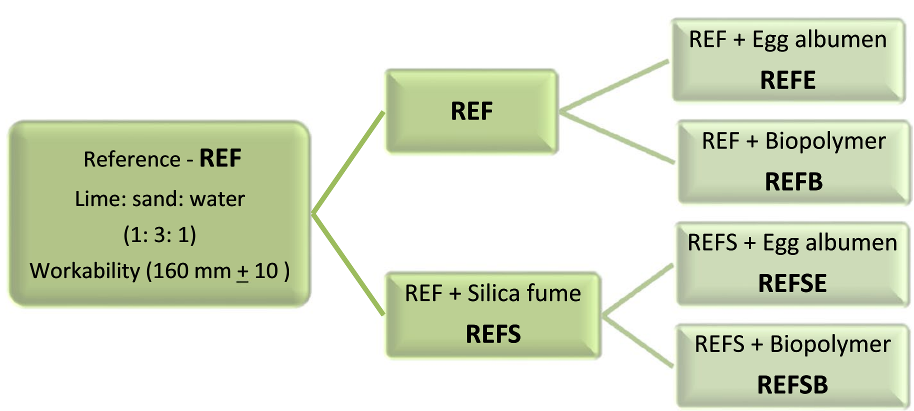
The experimental program stated where was mechanical properties (compressive and diametric strength, absorption ratio) and chemical properties (SEM, Infrared spectrum) of the different mortars.

The highlight of this research lies in the opportunity for a union between different industrial sectors (construction and agribusiness) aiming at the development of more sustainable products, such as, for example, additives (biopolymer), enabling effectiveness gains in the durability of materials in favor of safeguarding the built historical heritage.

2 Experimental procedure

Mortar ratio of 1: 3: 1 (hydrated lime: sand: water/lime factor, proportion in weight), workability = 160 mm ± 10 mm (Flow table (NBR 13276 [12]) was researched MYDIN [24], Manoharan and Umarani [22]). Particularly, 0.5 kg of silica fume, 6% of egg albumen, and 6% of biopolymer (complexes of whey protein with κ-casein) were employed. Figure 1 presents the nomenclature adopted for the specimen tests.

Fig. 1 Nomenclature adopted in the research samples



2.1 Materials

2.1.1 Hydrated lime

The Table 1 shows the characteristics of the lime used in this research.

2.1.2 Aggregate

River sand with the following physical characteristics: maximum diameter of 2.40 mm; specific mass: 2.62 kg/dm^3; fineness modulus of 2.52; powdered material content of 1.33% and organic impurity < 300 ppm.

2.1.3 Silica fume

% SiO_2 > 90%, specific surface: 20.000 m^2/kg, specific mass: 2220 kg/m^3, particle diameter 200 nm–1 µm.

2.1.4 Bioproducts

2.1.4.1 Egg albumen The clear or misty substance that surrounds the yellow yolk was used (MYDIN [24], since this is the part of the egg that contains different types of proteins, which are mostly composed of amino acids. The egg was beaten until it was foamy to increase its ability to mix with the mortar mixture.

2.1.4.2 Biopolymer The complexes of whey protein with κ-casein: κ-casein is a phosphoprotein found in milk, obtained from milk that was unfit for human consumption (SOUZA et al. [28].

2.2 Methods

2.2.1 Mechanical properties

The Compressive strength (($\mathbf{f_c}$) (NBR 7215 (2019))), diametric tensile strength (Brazilian test) (($\mathbf{f_{ct,sd}}$) (NBR 7222 (2011)), absorption by immersion ((**A**) (NBR 9778 (2009)) and capillary absorption ((**C**) (NBR 9779 (2012)) were verified by the Brazilian standard [8–11] for all mixtures (see Fig. 1). As many 6 (six) specimen tests were made, for each test and each mortar ratio, which was evaluated at 42 days of age (test age was defined as a function of restrictions on the use of laboratories due to COVID-19).

Table 1 Physical and chemical characteristics of hydrated lime

Chemical characteristics	Physical characteristics
Carbonic anhydride ≤ 5%	Fineness (% accumulated retained)—# 0.600 mm ≤ 0.5%
Calcium and magnesium oxide, unhydrated ≤ 10%	Fineness (% accumulated retained)—# 0.075 mm ≤ 10%
Total oxide, non-volatile ≥ 90%	Water retention ≤ 75%

2.3 Chemistry analysis

The chemistry analysis of the mixtures, at 42 days of age, consisting of *Scanning Electron Microscopy* (SEM images were obtained using a Hitachi Tabletop TM 3000 scanning electron microscope equipment, with magnification from 50 × to 10.000×, with no sample preparation (no metallic recovery of the samples), Back-scattered Electron Detector (BSE) and 15 keV of observation condition mode) and *Fourier transform infrared spectroscopy (FT-IR)* (The spectra of the samples after 1, 7, 14, and 28 days of hydration were obtained using a Perkin Elmer FTIR spectrometer operating in the absorption mode. The spectra were obtained in the range of 400–4000 cm^{-1} with a resolution of 4 cm^{-1}, and 32 scans were obtained using KBr pellets).

3 Results and discussion

Figure 2 shows the image in a digital microscope with 1600 × magnification of the mortars research (with 42 days of age), and Table 2 shows the results for the mechanical properties. The coefficient of variation (CV) has been calculated for the results presented in each property evaluation. If CV ≤ 25%, the results are accepted as adequate.

Analyzing the results, see Table 2 and Figs. 3 and 4, the silica fume allows an increase of more than 50% in the compressive strength of the mortars when associated with animal protein which may be higher than 60%. The addition of albumen MYDIN [24] provides strength gains due to the ability of the interacting proteins with the mortar components. However, the proportion interferes directly with the process since amounts greater than 6% generate a decrease in strength, as egg white is an alkaline solution, and this can be harmful regarding the mechanical properties of aggregates not susceptible to alkali-silica reactions.

In the case of milk protein (complexes of whey protein with κ-casein), the results are more expressive, around 70% since k-casein influences the crystallization form of calcium carbonate crystals. That is, it increases the binding capacity of lime, resulting in favorable impacts on the physical properties of the mortar and, consequently, on its mechanical properties.

Regarding the diametric tensile strength (Table 2 and Fig. 3), the albumen or biopolymer (complexes of whey protein with κ-casein) addition alone does not significantly change this mechanical property. However, with the addition

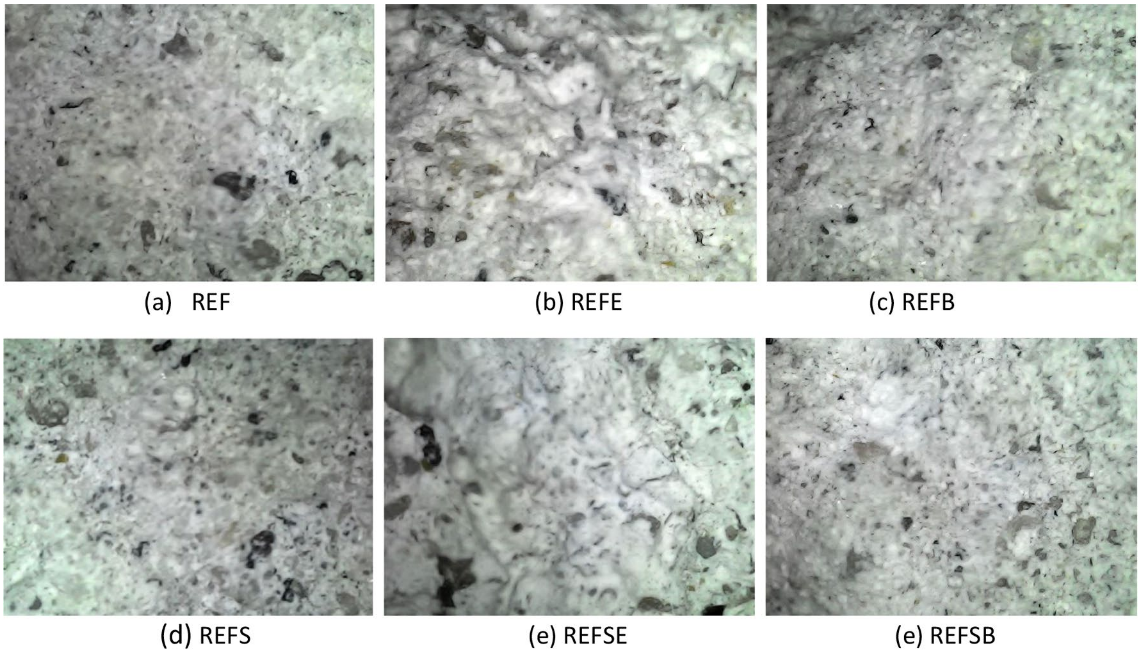


Fig. 2 Microscope image of the mortar research with 42 days of age

Table 2 Average results of properties research

Property	Without silica fume			With silica fume		
	REF	REFE	REFB	REFS	REFSE	REFSB
f_c (MPa)	0.67 (CV = .2.2%)	0.61 (CV = 18.0%)	0.71 (CV = 10.5%)	1.26 (CV = 10.3%)	1.65 (CV = 3.25%)	2.37 (CV = 9.2%)
$f_{ct,sd}$ (MPa)	0.03 (CV = 0%)	0 (CV = 0%)	0.125 (CV = 13.6%)	0.3258 (CV = 8.6%)	0.068 (CV = 11%)	0.87 (CV = 24%)
C (g/cm^2)	2.66 (CV = 3.0%)	2.88 (CV = 4.4%)	2.46 (CV = 3.3%)	3.46 (CV = 2.0%)	3.44 (CV = 9.0%)	2.27 (CV = 9.1%)
A (%)	17.85 (CV = 3.4%)	18.9 (CV = 2.8%)	18.31 (CV = 4.4%)	25.41 (CV = 8.8%)	23.14 (CV = 6.4%)	23.86 (CV = 5.1%)
Void index (%)	27.31 (CV = 13%)	29.7 (CV = 6.0%)	30.15 (CV = 0.54%)	25.42 (CV = 3.6%)	40.45 (CV = 14.1%)	36.14 (CV = 8.7%)

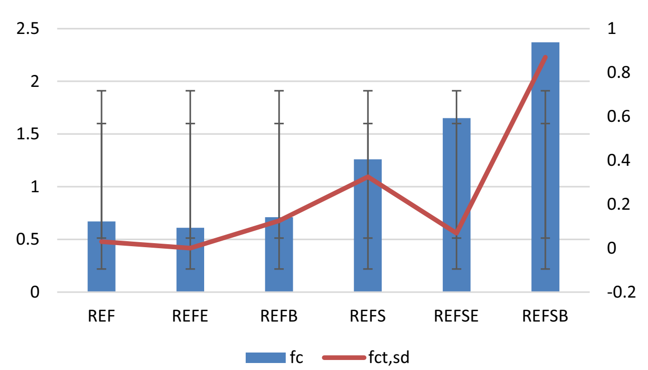


Fig. 3 Average results of compressive strength (fc) (MPa) × diametric tensile strength (Brazilian test) (($f_{ct,sd}$) (MPa)

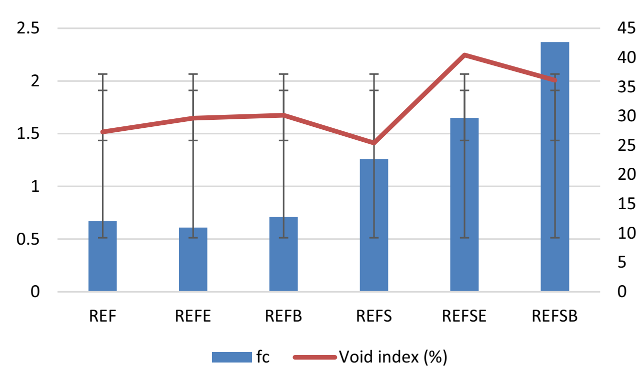


Fig. 4 Average results of compressive strength (fc) (MPa) × Void index (%)

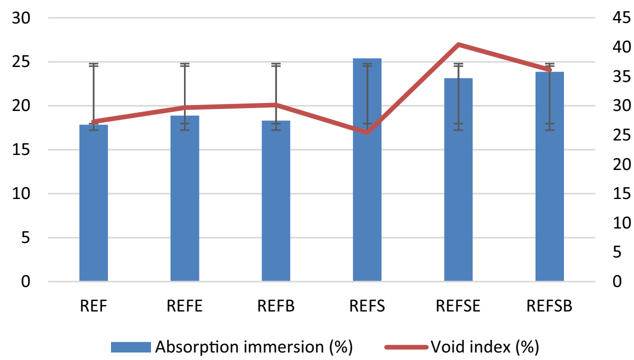


Fig. 5 Absorption immersion (%) × Void index (%)

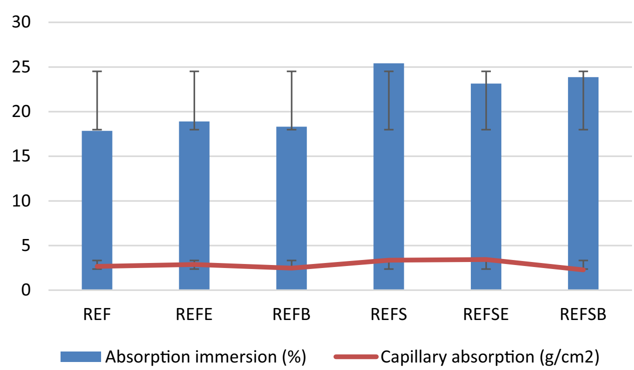


Fig. 6 Absorption immersion (%) × Capillary absorption (g/ cm.^{2})

of silica, a significant increase in strength to mechanical is observed, especially when this is combined with the biopolymer.

Aalil et al. [1] researched pozzolanic-lime mortars that reduced shrinkage and increased mechanical properties. Branco et al. [7] studied the addition of silica fume in lime mortar. They obtained better results in mechanical properties and the adequacy of the studied mortars in the rehabilitation of masonry elements. Thirumalini et al. [34] studied the influence of organic additions on the mechanical properties of lime mortar. The additions' mortars do not reduce total mortar porosity but increase the mortar strength. The bio-additive self-healing capacity of lime mortar makes it time-resistant (Manoharan and Umarani [21]).

For the case of absorption (Figs. 5 and 6 and Table 2), it is observed that:

(i) The addition of silica fume increases the water absorption in the mixtures, despite causing an increase in compressive strength.
(ii) In mixtures with the addition of egg whites into the snow, despite having a higher void index resulting from the material itself Mydin [24], the absorption does not change. The increase in porosity (void ratio) is related to the content of lime used since lime allows the formation of open pores due to the water demand Souza et al. [28], and the use of albumen and biopolymer increases these pores. However, these materials associated with silica fume favor the packing of the grains of the materials that compose the mortar.

In summary, the silica fume addition, albumen and k-casein prove beneficial for lime mortars, improving the mechanical properties, especially regarding compressive strength. This fact is associated with the proteins in the preparation of the mortar that provides intense air entry, resulting in the formation of more pores. This increase in voids allows more CO_2 to enter and directly contributes to a faster carbonation process. In the specific case of the k-casein and the whey protein complex, this use favors the formation of circular aragonite crystals, which improve the consistency of the mixture and explain the increase in compressive strength Ventolà et al. [31].

Besides, all additions are sustainable components since silica fume consists of mining waste and k-casein comes from milk unsuitable for consumption. In the case of albumen, due to the limitations of the research, perfect eggs were used. However, it is valid to analyze the results with eggs unusable for human consumption.

To relate the mechanical properties of the samples obtained with the chemical reactions that occur in the mortar curing process, infrared spectroscopy was used, see Fig. 7.

Analyzing Fig. 7, it is possible to observe, for hydrated lime, the characteristic bands of the substances that are part of its composition. The bands at 713, 874, 1454, and 1794 cm^{-1} are related to the presence of calcite and can be attributed, respectively, to the vibrational modes: δ_d(OCO), o $\gamma(CO_3)$, ν(C-O), and $\nu(C = O)^{1-3}$. In addition to these, the band at 2513 cm^{-1}, attributed to the stretching of CO_3^{2-} ions, can also be related to calcite CIZER et al. [13], HWIDI et al. [17]. The bands at 3643 and 3429 cm^{-1} are attributed to ν(OH), due to the presence of $Ca(OH)_2$ and hydration water molecules, respectively Cizer et al. [13], Roschat et al. [26]. The bands at 857 and 1084 cm^{-1} are attributed to the $\gamma(CO_3)$ and ν(C-O) of aragonite. Finally, the bands at 2872/ 2923/ 2978 and 2963 cm^{-1} can be associated with the presence of calcite and dolomite, respectively Stanienda-Pilecki [30]. For the infrared spectrum of silica fume, bands are observed at 3450, 1629, 1100 and 806 cm^{-1}, which are attributed, respectively, to ν(OH), δ(OH), siloxane vibrations in $(SiO)_n$ and ν(Si–O) Azarshin et al. [4], Khan et al. [21], ANSARI et al. [3]. Table 3 presents the bands observed in the spectrum of sand and the minerals responsible for their

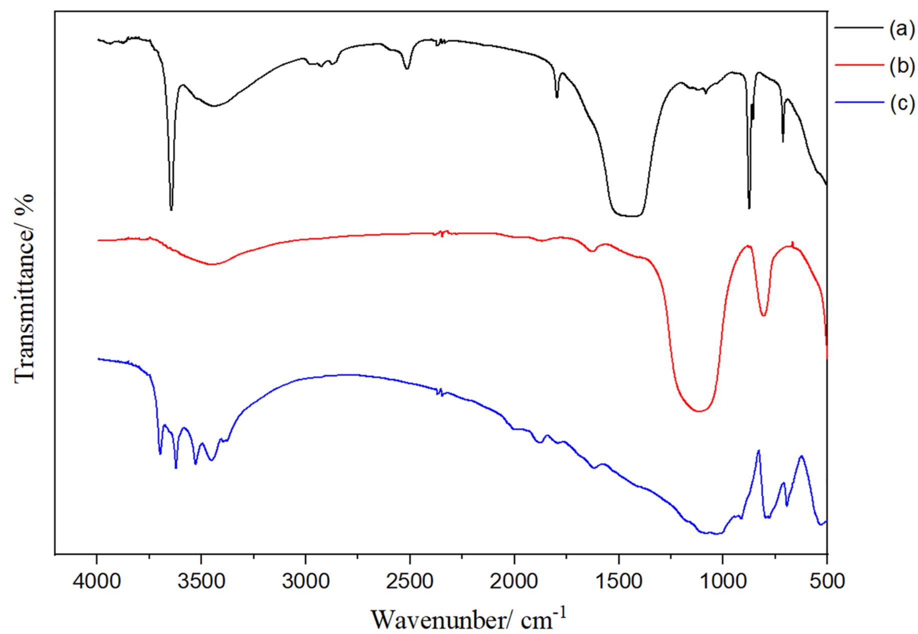


Fig. 7 Infrared spectra of **a** Hydrated lime; **b** Silica fume; **c** Sand

Table 3 Bands observed in the infrared spectrum and minerals related to them

Wave number/ cm^{-1}	Mineral
3697	Kaolinite
3618	Kaolinite
3448	Palygorskite
3390	Halloysite
1877	Quartz
1793	Aragonite
1171	Hectorite
1089	Quartz
1032	Kaolinite
915	Nacrite
795	Quartz
778	Quartz
688	Quartz

presence (Gnanasaravanan and Rajkumar [16], Meftah and Mahboub [23].

During the curing process of hydrated lime, used in the preparation of mortars, the carbonation process occurs. This consists of the reaction between calcium hydroxide and carbon dioxide, generating calcium carbonate and water, a process that is responsible for the final mechanical properties Billong et al. [6]. In this sense, to evaluate the carbonation process of the samples obtained, the ratio of the areas of the bands in the region between 800–900 and 3630–3670 cm^{-1} was calculated, as they are associated with the presence of $CaCO_3$ and $Ca(OH)_2$, respectively. Therefore, a higher ratio will be indicative of a higher carbonation process. The data obtained are presented in Table 4 and the infrared spectra of the specimens produced are shown in Fig. 8.

Table 4 Band areas

Sample	$A_{900-840}$	$A_{3630-3670}$	$A_{900-840}$/ $A_{3700-3600}$
Hydrated lime	14.4	20.1	0.7
REF	9.5	3.7	2.6
REFS	8.9	–	–
REFE	13.0	4.7	2.8
REFSE	19.4	1.7	11.3
REFB	7.6	7.4	1.0
REFSB	11.8	0.2	50.4

Analyzing the data presented in Table 4 and Fig. 8, it is observed that the area ratio of all samples is greater than that of the hydrated lime used, indicating the occurrence of the carbonation process in the samples. Only for the sample REFS it was not possible to calculate the ratio, as the band at 3643 cm^{-1} is absent in the spectrum, however, this indicates an advanced process of calcium hydroxide consumption. The samples REF, REFE and REFB present similar values regarding the areas of the bands, which indicates that the carbonation process is not influenced only using albumen or by the complex of whey protein and κ-casein. On the other hand, the samples where silica fume was used presented higher values of the ratio between the bands (RESB and REFSE) or no longer presented the presence of the band referring to calcium hydroxide (REFS). This difference between the samples with and without silica fume can be

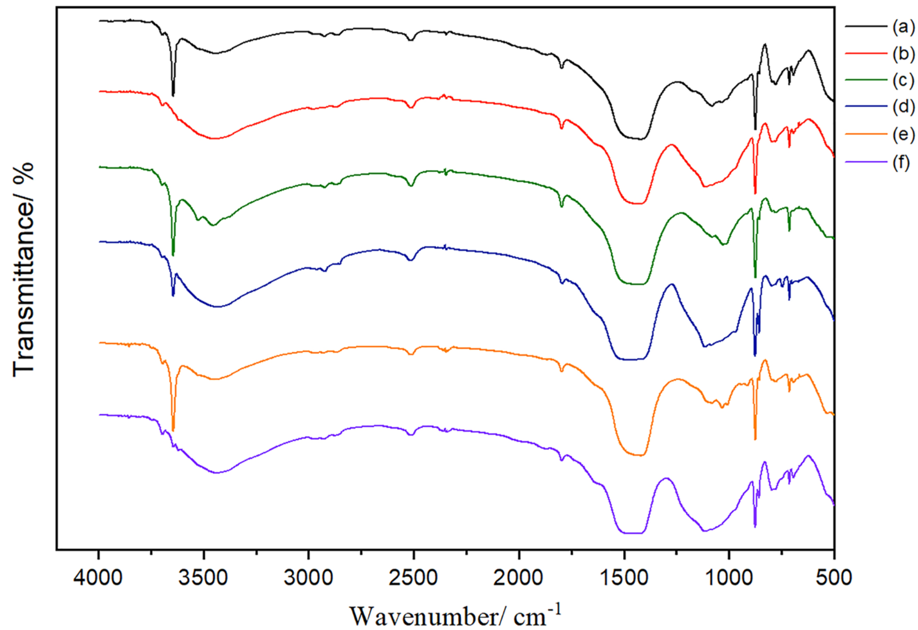


Fig. 8 Infrared Spectra of **a** REF, **b** REFS, **c** REFE, **d** REFSE, **e** REFB and **f** REFSB

explained by the chemical reaction that occurs between calcium hydroxide and silica, $Si0_{2(s)} + Ca^{2+}_{(aq)} + 2OH^{-}_{(aq)} = {}_{n1}CaO \cdot Si0_{2\ n2}H_20_{(s)}$[14]. Therefore, for the samples with silica fume, in addition to the carbonation process, this reaction contributes to the decrease in the concentration of calcium hydroxide, which is reflected in a smaller area of the band at 3643 cm^{-1}. In addition, the compound formed from this reaction is similar that produced in the cement curing process, and its formation can be verified by the greater intensity of the band at 1114 cm^{-1}, which is related to the greater compressive strength of these samples Adesina and Olutoge [2].

The use of silica fume together with the albumen and the complex of whey protein and κ-casein was the one that showed the highest compressive strength. This may be linked to the fact that the use of proteins in the mixture allows the intense entry of air into the mortar, which favors the formation of more pores that will allow the entry of more CO_2 and, consequently, contributes to a faster carbonation process Meftah and Mahboub [23], Billong et al. [6]. In addition, using of the complex of whey protein and κ-casein contributes to the formation of circular aragonite crystals, which improves the consistency of the mortar and results in better compressive strength.

Figure 9 shows the scanning electron microscopy image of the interior of the samples after breaking. It is observed that the topography of the sample REF is flatter and more compact, while in the samples where there was the addition of albumen, biopolymers, the topography is rougher and with the presence of more grooves or furrows. Thus, with the help of the ImageJ Software, the percentage of the area related to the grooves was calculated for each sample, and the values 1.267, 17.307, 16.510, 27.927 and 10.040% were obtained for samples REF, REFS, REFC, REFSC and REFB, respectively. This result corroborates the data on the void ratio and water absorption capacity by immersion, which indicated that the addition of silica fume, albumen and biopolymer collaborated to increase the porosity of the samples, which, consequently, contributed to the increase in the carbonation level of the samples and the increase in the mechanical strength.

4 Conclusions

Hydrated lime and sand mortars were studied with different additions: silica fume with/ without animal protein (egg albumen or whey with κ-casein) and examined mechanically and chemically. For analysis of the lime hydration process, it concluded that the animal protein is favorable, enabling significant gains in the performance of the mortars, especially in those with the addition of silica fume. It is possible the products manufacture with differentiated properties and is part of the concept of sustainable development, adding environmental, social, and economic

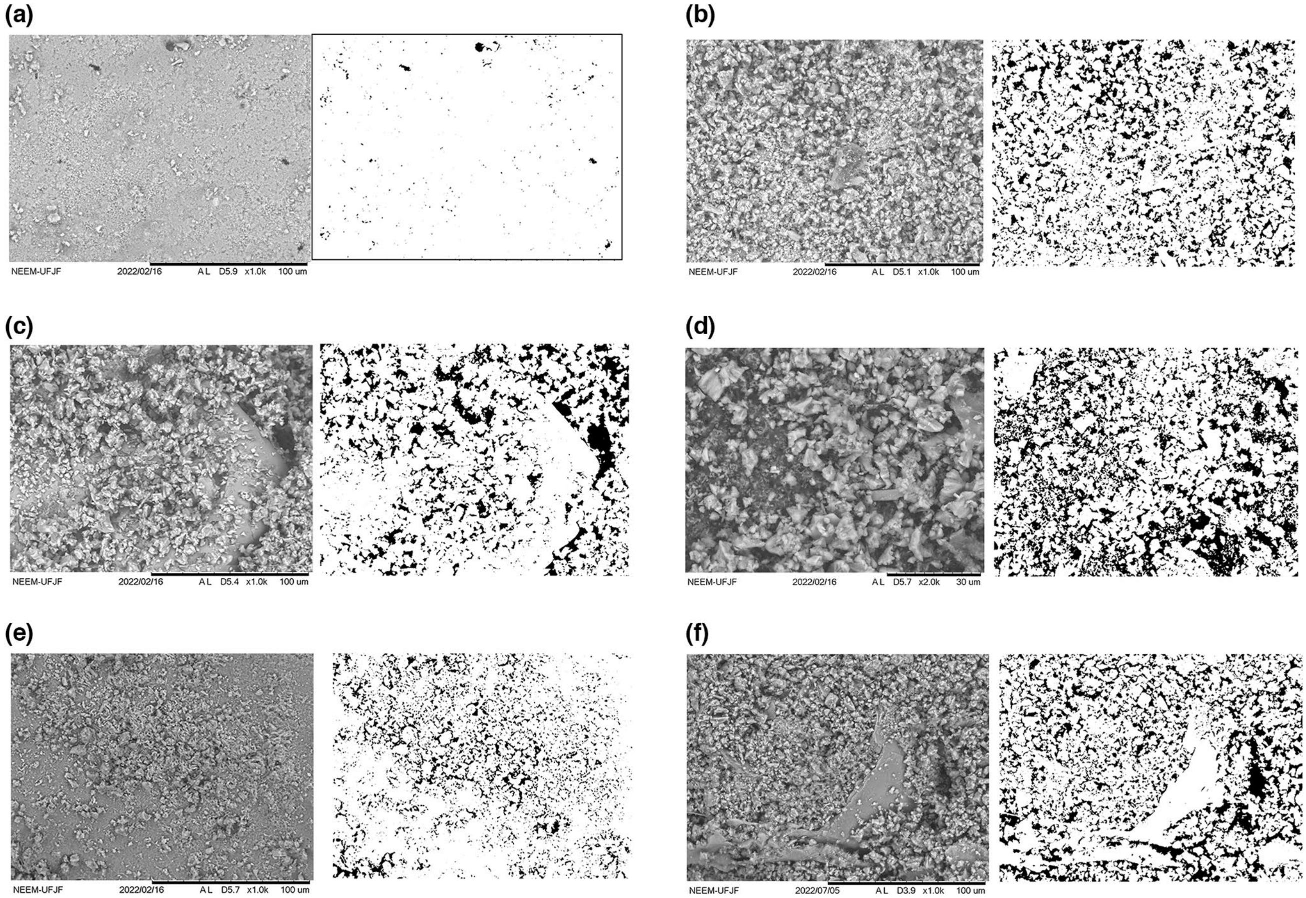


Fig. 9 SEM imagens: **a** REF, **b** REFS, **c** REFE, **d** REFSE, **e** REF and **e** REFSB

indicators and providing higher durability to the materials used in the restoration of historical buildings.

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Author contributions Conceptualization: CRI and MTGB; Methodology CRI, LOS, TSSP and MTGB; Formal Analysis, CRI, LOS, TSSP, MTGB, NLGDS, CDS, LFCO; Writing (original preparation) MTGB; Writing (Review and Editing), MTGB, NLGDS, CDS and LFCO; Project Administration CRI and MTGB. All authors reviewed the manuscript.

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Data availability The databases generated and analyzed during the current study are available from corresponding author on reasonable request.

Declarations

Conflict of interest The authors have not disclosed any competing interests.

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