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Mechanical and Compositional Characterization of Ancient Yemeni Qadhad Mortar for Sustainable Heritage Conservation

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Abstract

Qadhad is a traditional Yemeni slaked-lime mortar renowned for its exceptional waterproofing properties, particularly in cisterns, reservoirs, and masonry structures that have remained functional for centuries. Despite its historical significance, the mechanical properties and precise mix proportions of the ancient Qadhad remain insufficiently quantified. This study investigates the mechanical properties and compositional characteristics of ancient Qadhad mortar samples collected from four archaeological sites in Yemen. Compressive strength testing, thermogravimetric analysis (TGA), and chemical analyses were conducted to determine the material's performance and composition. Experimental results show that the compressive strength of the ancient mortars ranges from 2.77 to 4.39 **MPa**. Furthermore, the binder-to-aggregate ratio of the ancient mortars was determined to range between **1:2 and 1:2.5 by weight**. These findings establish a quantitative scientific basis for reproducing traditional Qadhad mortar for the development of compatible repair materials in heritage conservation. The study also highlights the potential of Qadhad as a durable and sustainable lime–pozzolan construction material suitable for both conservation and contemporary ecological building applications.

Keywords: Qadhad mortar, composition, slaked lime mortar, compressive strength, heritage conservation, Thermogravimetric Analysis, sustainable material

1. Introduction

The preservation of ancient Qadhad buildings in Yemen, the enduring performance of centuries-old Qadhad structures in Yemen serves as a testament to its durability and advanced hydraulic properties related to stone adhesion and water resistance, demonstrating the advanced material knowledge and craftsmanship of historical Yemeni builders (Figure 1). Qadhad is an ancient Yemeni lime-based mortar known for its long-term waterproofing performance. However, its mechanical properties and quantitative mix composition remain insufficiently characterized. It emerged as the preferred material for wall lining, roofing, and stone binding in ancient structures, many of which remain operational after several centuries. Its formulation consists of a sophisticated mixture of a calcium hydroxide (slaked lime) binder and a carefully selected aggregate composition, frequently incorporating volcanic scoria or ash, sand, and pebbles. The production process was elaborate and labor-intensive, necessitating thorough slaking and grinding of slaked lime with the aggregates to yield a dense, homogeneous, and impermeable layer. Lime is produced through the thermal decomposition of limestone (calcium carbonate, CaCO_3) at temperatures around 900°C , during which calcium carbonate (CaCO_3) dissociates into quicklime (CaO) and carbon dioxide (CO_2): The quicklime is then subjected to a slaking process, wherein it is mixed with water to generate hydrated lime (calcium hydroxide, Ca(OH)_2). The generation of hydrated lime is accompanied by the release of thermal energy. The resulting slaked hydrated lime is converted into a creamy lime putty, which is subsequently combined with additional materials such as sand, ash, and crushed aggregates to produce Qadhad mortar or plaster. The hardening of

lime mortar occurs through carbonation, wherein moisture evaporates, and the lime interacts with atmospheric carbon dioxide. This process initiates a chemical transformation in lime, with carbonation reverting it to calcium carbonate, akin to the original limestone, a phenomenon commonly known as the lime cycle, particularly regarding non-hydraulic lime. Conversely, hydraulic lime benefits from the incorporation of pozzolanic additives (such as volcanic ash or scoria), which promote a hydraulic setting, culminating in the formation of intricate hydration stable products, including C-S-H and C-A-H phases, thereby reducing the porosity of lime and rendering it denser and less permeable to water. [1-4]. It has traditionally served as a binding agent in construction for stones. Additionally, it is considered a material for the restoration of historical edifices.



a) Sample AC: Aden Cisterns (Aden Governorate)



b) Sample SF: Seera Fort (Aden Governorate)



c) Sample HP: Harawa Pool (Amran Governorate) d) Sample SP: Salama Pool (Sana'a Governorate)

Figure 1. Site photographs of Qadhad structures in ancient buildings and water tanks.

Studies of ancient construction materials from civilizations such as the Roman, Byzantine, Ottoman, and Indian cultures [5–13] provide valuable insight into the performance of slaked hydraulic lime mortars containing volcanic pozzolanic materials. Mineralogical and microstructural investigations using X-ray diffraction (XRD), X-ray fluorescence (XRF), and scanning electron microscopy coupled with energy-dispersive spectroscopy (SEM–EDS) have identified the principal hydration products formed within these mortars. The reaction between slaked lime and pozzolanic constituents promotes hydraulic hardening and leads to the formation of calcium silicate hydrate (C–S–H) and calcium aluminate hydrate (C–A–H) phases. These reaction products contribute to the long-term durability of the mortar by refining the pore structure and reducing permeability [1, 3, 4, 8, 14–16].

Previous studies on ancient slaked lime mortars [1, 16–19] have shown that SEM observations reveal a well-developed bond between the lime matrix and aggregate particles. This interaction, together with the formation of hydration products, partially fills internal pores and improves the compactness of the mortar. In addition, the combined particle-size distribution of sand, ash, and other aggregate fractions enhances particle packing, thereby

reducing void spaces. The resulting dense and interconnected microstructure lowers porosity and limits water penetration.

A considerable number of investigations have examined ancient lime-based mortars incorporating ash and mineral aggregates from Roman sites in Italy, Turkey, Egypt, Crete, and Bulgaria, as well as from historical structures in China and Mexico [5–12, 14, 19]. These studies have primarily focused on characterizing the composition, microstructure, and durability of the mortars to support their conservation and potential reproduction. To achieve this objective, researchers have employed a range of analytical techniques, including XRF, XRD, SEM, thermogravimetric analysis (TGA), and acid-solubility testing.

Although historic waterproof plasters have been widely researched around the world, there is a lack of scientific documentation of Yemeni Qadhad. Historical accounts of Qadhad were descriptive and mainly related to traditional building techniques. A significant contribution was made by Kulaib and Al-Jolahy (2006) [2], who recorded the traditional knowledge (*savoir-faire*) of the *muqadidun* (Qadhad applicators), and the time-consuming processes of lime slaking, pounding, application, and curing. Nevertheless, this work did not include a scientific analysis of the ancient Qadhad material, which has been used for hundreds of years and remains effective in historic cisterns and waterworks. Other studies, such as that by Weiss and Gerlach (2009) [20], provided valuable mineralogical and microstructural information about mortars from the Marib Dam. Despite such important work, prior experimental investigations into Qadhad reproduction were usually based on trial mixes with assumed binder-aggregate ratios, rather than ratios based on the composition of ancient materials. As a result, the mix proportions in these studies may not reflect the original historical mix proportions.

Algorafi et al. and Al-Gawi et al. [21-23] established the first comprehensive scientific characterization of four samples of ancient Yemeni Qadhad as waterproofing plaster, identifying it as a sophisticated hydraulic lime–pozzolan composite intentionally engineered for superior durability and hydraulic resistance. Analytical results demonstrate that Qadhad exhibits an extraordinarily low coefficient of permeability (6.55×10^{-9} to 26.2×10^{-8} m/s), values that are significantly lower than those typically reported for standard lime-based mortars. This performance is underpinned by high bulk density values (1,690–1,740 kg/m³), reduced porosity (19.6–25.0%), and a remarkably high Hydraulic Index (HI) reaching up to 2.16, all of which indicate a highly compact and well-consolidated internal matrix. XRD analyses identify Calcite as the primary binder phase, resulting from the carbonation of slaked lime. The detection of Quartz, Microcline, and Albite confirms the use of volcanic feldspathic aggregates, while the presence of iron oxides, specifically Hematite (Fe₂O₃) and Magnetite (Fe₃O₄), suggests a complex mineralogical profile. Furthermore, XRF and SEM–EDS investigations confirm the synthesis of Calcium Silicate Hydrate (C–S–H) phase through lime–pozzolanic reactions. These hydraulic products are the fundamental drivers of the material's high internal cohesion and its characteristic impermeability. However, Qadhad is also considered a construction binder, and its mechanical values have not been determined. There is also a need to confirm the ratio of the binder to the aggregate in a more precise way.

Consequently, the objectives of this research are to investigate the mechanical characteristics and determine the ratio of the binder to the aggregate precisely that uses in the ancient Qadhad mortar for four samples in Yemen reported in [21].

The mechanical characteristics, with the physical, hydraulic, chemical, and microstructural characteristics of Qadhad as reported in [21], are essential for informing sustainable

conservation strategies. Such an approach enables a more comprehensive understanding of the material's behavior and supports the development of compatible restoration practices. The scientific characterization and careful reproduction of Qadhad not only ensure material compatibility in conservation interventions but also promote its use as a durable and environmentally responsible solution rooted in local construction traditions. Notably, this study establishes an initial scientific baseline for recognizing Qadhad as a high-performance pozzolanic lime mortar. In doing so, it provides a conceptual and methodological framework for its future replication as a sustainable and compatible conservation material in Yemen.

However, the findings of this study should be interpreted within the context of its limitations. The analysis is based on a restricted sample set obtained from four archaeological sites in Yemen—Aden Cisterns, Harawa Pool, Seera Fort, and Salama Pool—dating to different historical periods (16th century for Aden Cisterns and Seera Fort and 19th century for Harawa Pool and Salama Pool). As such, the results may not fully capture the variability of Qadhad formulations across other regions or time periods. But it is sufficient to produce a Qadhad with excellent mechanical and hydraulic properties.

2. Research Methodology

2.1. Sampling

As previously articulated, the present investigation constitutes an extension of the study conducted by Algorafi et al. [21], wherein the identical four samples were selected as shown in Figure 2: Sample AC: Aden Cisterns (Aden Governorate, early 16th century AD) Sample HP: Harawa Pool (Amran Governorate, early 18th century AD) Sample SF: Seera Fort (Aden

Governorate, early 16th century AD) Sample SP: Salama Pool (Sana'a Governorate, early 18th century AD).

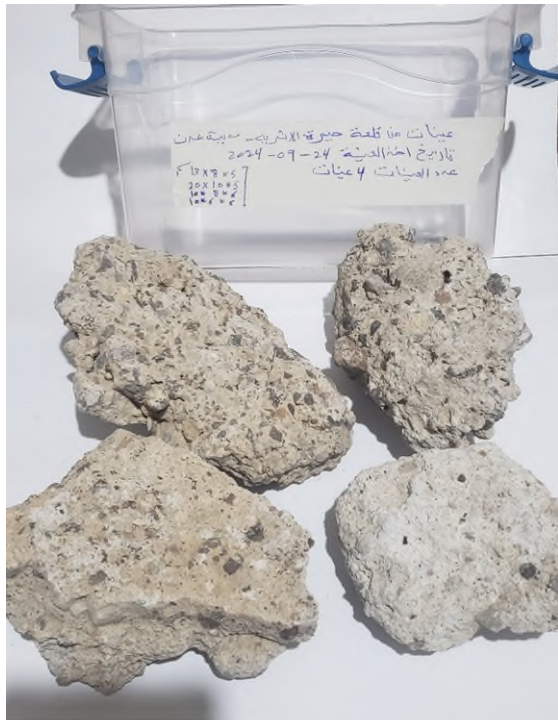
The samples were meticulously prepared for compressive strength, TGA, and Acid Digestion testing in accordance with the prescribed specifications for each respective test, as shown in Figure 3.



a) Sample AC



b) Sample HP



a) Sample SF



b) Sample SP

Figure 2. Original Qadhad samples collected from the archaeological sites [21].

2.2. Mechanical Test

Compressive strength is considered one of the most important mechanical properties used to evaluate the performance of building materials.

The compressive strength of the mortar samples was determined in accordance with ASTM C109 [24]. The tests were conducted on three cylindrical specimens with diameters ranging from 40 to 45 mm and heights ranging from 15 to 40 mm, as well as two cubic specimens with lengths and widths between 40 and 75 mm and heights between 22 and 54 mm for each

mortar type. During testing, the maximum compressive load sustained by each specimen was recorded using a constant displacement rate of 1 mm/min.

According to ASTM C109, the standard specimen size for compressive strength testing is a cube measuring 50 × 50 × 50 mm. However, archaeological constraints prevented the preparation of specimens with these dimensions. The limited thickness of the Qadhad layer used in the tank, together with the difficulty of obtaining specimens with a length and width of 50 mm, made compliance with the standard size impractical. In addition, some coarse aggregate particles protruded from the material surface and required removal during sample preparation, as illustrated in Figure 3.

The determination of tensile strength presented additional challenges, as it was difficult to prepare specimens suitable for either direct tensile testing or the splitting tensile test. Consequently, the tensile strength was estimated based on the classification of the mortar as a hydraulic lime mortar, as discussed in Section 4.2.



a)

Compressive Strength samples



b) Chemical analysis samples



c) | Grain size distribution samples



d) | TGA samples

Figure 3. Qadhad samples after preparation for mechanical and chemical tests.

2.3. Thermogravimetry Analysis (TGA)

Thermogravimetric analysis (TGA) was conducted utilizing a simultaneous thermal analysis TGA. The weight of the samples is 5 grams, powder, sifted through a 0.125 mm sieve. It was subjected to drying at 40°C within a drying oven for a duration of 24 hours, as shown in Figure 3. The thermal analysis was executed over a temperature spectrum of 25–1000°C, employing a heating rate of 10°C/min in a Nitrogen atmosphere, with the sample mass being recorded at increments of 100 °C. The carbonate content (expressed as % CaCO₃) was computed using the following equation:

$$\% \text{CaCO}_3 = Q(\text{CO}_2) * M(\text{CaCO}_3) / M(\text{CO}_2) \quad \text{Eq 1}$$

where $Q(\text{CO}_2)$ represents the mass loss occurring within the temperature range of 600–1000°C, indicative of the decarboxylation of calcium carbonate; $M(\text{CaCO}_3)$ denotes the molar mass of calcite, which is equivalent to 100, and $M(\text{CO}_2)$ refers to the molar mass of carbon dioxide, which is valued at 44.

2.4. Acid Digestion Analysis

An acid digestion analysis was performed to facilitate the separation of the binder from the aggregates present in the historical mortar samples. Four distinct types of mortar specimens were characterized through wet chemical analysis, in accordance with the specifications of ASTM D3042 [25]. The selected mortar samples were meticulously hand-crushed to prevent any damage to the aggregates. A quantity of 90–150 g of the processed mortar sample was introduced into a 1000-mL beaker containing 600 mL of distilled water along with 200 mL of

1M hydrochloric acid solution, and the mixture was agitated continuously for one hour to isolate the siliceous aggregate (insoluble residue) from the binder, as shown in Figure 3.

The insoluble residue underwent a washing process 4–5 times utilizing distilled water to filter the aggregates and was subsequently air-dried for a duration of 24 hours. The fractions that passed through the sieve were deemed to be enriched in binder. Particle size analysis on the fine aggregates derived from the acid digestion analyses was conducted by oven drying at 100°C and sieving through a series of sieves (4.75 mm, 2.36 mm, 1.18 mm, 0.6 mm, 0.3 mm, 0.15 mm, 0.75 mm) on a sieve shaker for 10 minutes according to ASTM C136 [26]. The percentages of aggregate, sand, and clay contents were calculated from the gradation curve.

The simplified compositions of the mortars were determined following the “Jedrzejewska method” for ancient lime mortars, as mentioned by Borsoi G. et al. [1]. This method considers three components of the mortars: aggregate (corresponding to the insoluble residue in acid, IR), carbonates (% mass of CaCO_3 , derived from TGA), and the acid “soluble fraction” (soluble fraction compounds in acid, excluding carbon dioxide formation). The content of the “soluble fraction” was calculated using the formula:

$$\text{Soluble Fraction} = 100 - \text{Sum (IR + Carbonates)} \quad \text{Eq 2}$$

The binder/aggregate ratio was computed with the assumption that carbonates function as a binder, while the aggregate comprises the aggregate (insoluble residue in HCl acid) and the acid (soluble fraction). The aggregate can be classified into coarse aggregate, sand, and ash. The coarse aggregate corresponds to the residue retained on the 4.75 mm sieve, the sand corresponds to the residue retained on the 0.15 mm sieve, and the ash corresponds to the material that passed through the 0.15 mm sieve.

3. Results and Discussion

The analysis of the four ancient Qadhad samples (AC, HP, SF, SP) yielded results from the compression stress test, TGA test, and Acid Digestion Analysis. The findings are subsequently summarized below.

3.1. Compressive Strength

The mechanical performance of Qadhad mortar, particularly its compressive strength, provides essential quantitative information for assessing its structural integrity and long-term durability. The use of non-standard specimen dimensions for compressive strength testing was dictated by the fundamental conservation principle of minimum intervention. Standard testing procedures, such as ASTM C109, require specimen sizes that cannot be obtained from historic materials without causing unacceptable damage to the original fabric. Given the fragility and limited availability of the historic mortar, extracting standard-sized specimens would have compromised both the physical integrity and authenticity of the heritage structure. Furthermore, the limited thickness of the Qadhad layer used in the tanks,

Therefore, non-standard specimens were employed to enable the necessary laboratory characterization while preserving the architectural fabric. Because of the difficulties associated with preparing specimens that conform to standard dimensions, only a limited number of studies have investigated the compressive strength of ancient lime mortars. To the best of the author's knowledge, Watsantachad [27] evaluated the compressive strength of ancient lime mortar using non-standard cubic specimens measuring 25 × 25 × 25 mm, with only two samples collected from each site.

In the present study, although the specimen dimensions deviated from standardized specifications, they remain suitable for consistent relative comparisons among the samples investigated in this study. In the present study, to enhance the reliability of the results, two specimen series—cubic and cylindrical—were prepared for each mortar sample. Furthermore, the specimens were manufactured to dimensions as close as possible to the standard 50 mm size. The dimensions, individual compressive strength values, average compressive strength, and standard deviation were determined for both cubic and cylindrical specimens of the four ancient mortar samples, as presented in Table 1a. Table 1b summarizes the average compressive strength and standard deviation of the four mortar groups.

As shown in Table 1a, the difference between the average compressive strengths obtained from cubic and cylindrical specimens was smaller than the corresponding standard deviation for each sample. Moreover, the standard deviation values exceeded any potential correction factor associated with specimen geometry. This observation suggests that the variations in compressive strength were primarily attributable to the intrinsic heterogeneity of the mortar rather than to deviations from the standard specimen dimensions.

As shown in Table 1b, the average compressive strength of the ancient mortars ranged from 2.77 to 4.39 MPa, with standard deviations of ± 0.89 to ± 1.63 MPa. Sample SF exhibited the highest average compressive strength (4.39 MPa), followed by samples HP and AC, with average strengths of 3.55 and 3.48 MPa, respectively. Sample SP showed the lowest average compressive strength (2.77 MPa). The relatively high standard deviation values observed for all samples indicate substantial variability within the mortar matrix, suggesting a heterogeneous material composition.

Table 1a: Average Compressive Strength of Ancient Mortars

Sample	Width cm	Length cm	Height cm	Compressive Strength MPa	Average Compressive Strength MPa	SD. Compressive Strength MPa
AC	4	4	4	2.50		
	5.5	4	3	3.86	3.19	0.97
	---	Ø 4	1.7	3.58		
	---	Ø 4	2	4.77		
	---	Ø 4.5	1.5	3.77	4.04	0.64
HP	7.5	4	2.4	2.83		
	6.5	4	2.2	3.08	2.96	0.17
	---	Ø 4.5	1.5	6.29		
	---	Ø 4	3.5	3.58		
	---	Ø 4	3.5	1.99	3.95	2.17
SF	5	5	4	4.00		
	4	4	5.4	2.19		
	5	5	3.7	4.00		
	4	4	4	6.88	4.27	1.94
	---	Ø 4.5	3.5	4.72		
SP	---	Ø 4	3.5	3.18		
	---	Ø 4	4	5.73	4.54	1.28
	5	5	3.32	1.60		
	5.5	4	3.82	2.73	2.16	0.80
	---	Ø 4	3.5	3.18		
	---	Ø 4	4	4.77		
	---	Ø 4.5	3.7	1.57	3.18	1.60

SD.: Standard Deviation.

Table 1b: Average Compressive Strength of Ancient Mortars

Sample	Average Compressive Strength MPa	SD. MPa	CV. %
AC	3.48	±0.89	25.60
HP	3.55	±1.63	45.98
SF	4.39	±1.57	35.71
SP	2.77	±1.32	47.68

SD.: Standard Deviation, **CV.:** Coefficient of Variation.

Although the sample dimensions differ, the observed compressive strength values for the ancient lime mortar align with those previously documented by Mahasuwanchai et al. [28] in the range of approximately 2.12 to 3.7 MPa, and by Wonganan et al. [29] in the range of around 1.88 to 2.71 MPa.

3.2. TGA Result

The thermogravimetric analysis (TGA) included the following temperature intervals: 25–100 °C, 100–200 °C, 200–600 °C, and 600–1000 °C, during which notable mass reduction was recorded (refer to Table 2 and Figure 4). The mass reduction within the temperature interval of 25 to 100 °C fluctuated between 1.1% and 1.9%. The reduction in mass associated with the bound water in the presence of porous volcanic aggregates can be attributed to the dehydration of hygroscopic moisture (humidity) or absorbed water. No substantial mass reduction (<1.1%) was recorded in the interval from 100 °C to 200 °C, which correlates with the loss of crystallization water.

The mass reduction occurring within the 200–600 °C interval arises from the loss of chemically structurally bound water (SBW) (dehydroxylation) of clay minerals [1] (for instance, Goethite

in sample SF and Siderite in samples HP & SP) as corroborated by X-Ray Diffraction (XRD) analysis and hydraulic products (e.g., Calcium-Silicate-Hydrate (C-S-H) and Calcium-Aluminate-Hydrate (C-A-H)) [21].

The mass reduction observed between 600 and 1000 °C is associated with the release of CO₂ (12.25%-15%), linked to the thermal decomposition of calcium carbonate. The magnitude of mass loss in samples HP & SP exceeds that in samples AC & SF, which aligns with the Calcite values identified in the XRD analysis [21].

According to Borsoi G. et al. [1], the CaCO₃ content of the samples can be determined by multiplying the CO₂ value presented in Table 2 by (100/44). Consequently, the carbonated lime content of the samples was found to range from 28% to 34% by weight. Mortars AC and SF exhibit comparable carbonated lime content (28–29%, by mass), whereas a significantly elevated binder content (31–34%) was observed in mortars HP and SP.

The hydraulic characteristics of the mortar can be inferred from the CO₂/H₂O ratio, which is calculated based on the losses noted in the 600 - 1000 °C temperature range in conjunction with the losses from structurally bound water in hydraulic phases (200 - 600 °C) [1] across all samples, with the range being from 4.34% to 8.2%.

As stated by Moropoulou et al. [30], all samples were classified as hydraulic lime mortars. These mortars comprise approximately 1% of physically bound water, coupled with low percentages of structurally bound water (approximately 3.5–6.5%). The CO₂ values for the four samples range from 12 to 15%, which is below the 24% threshold (refer to [30]); however, as noted, the CO₂ content falls below 24% when silicoaluminate aggregates are present in excess, a finding corroborated by the XRD results (Quartz, Microcline, and Albite) [21]. The CO₂ to

structurally bound water ratios across all samples range between 4.5 and 9.5, contingent upon the hydraulicity degree of the lime and the composition of the aggregate.

The hydraulic characteristics of mortars AC and SF are superior to those of HP and SP, which is consistent with the findings of the hydraulic index (HI) for the samples [21].

Table 2: Sample mass losses obtained by TGA

Samples	Mass Losses %				Total Losses %	CaCO ₃ %	CO ₂ /H ₂ O%
	(25-100)	(100-200)	(200-600)	(600-1000)			
AC	1.59	0.60	1.49	12.25	15.94	27.86	8.20
HP	1.89	1.09	3.18	13.82	19.98	31.42	4.34
SF	1.49	0.80	2.29	12.74	17.31	28.96	5.57
SP	1.09	0.20	3.17	14.98	19.44	34.06	4.72

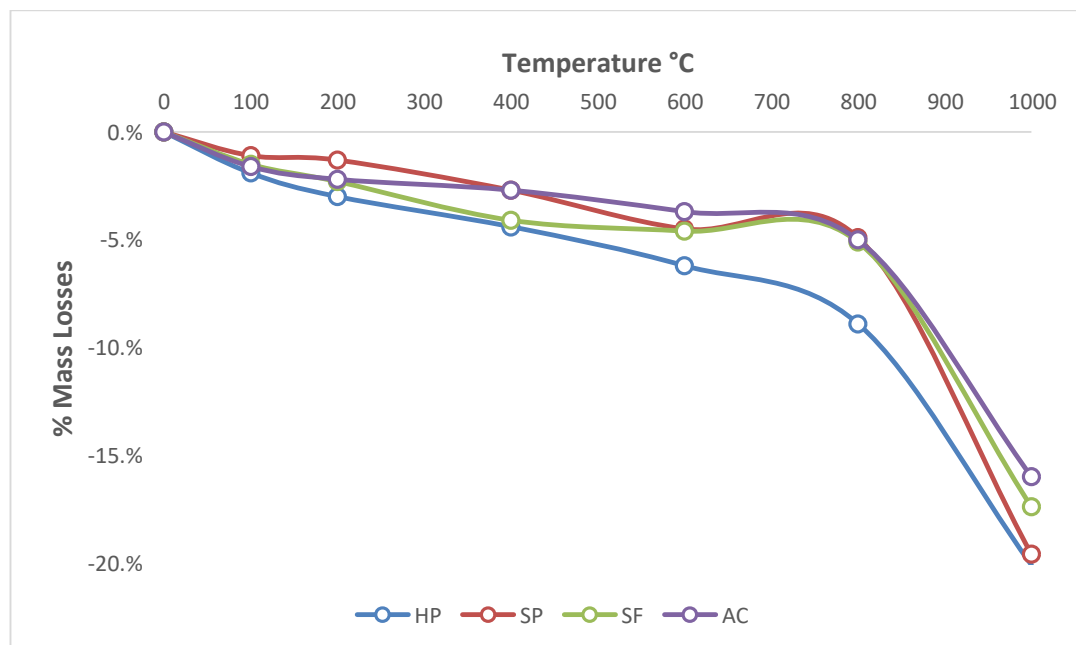


Figure 4: The Temperature losses of the samples by TGA

3.3. Chemical Analysis and Grain Size Distribution

The chemical analysis of the samples, as illustrated in Table 3, indicates that the insoluble residue (IR) for ancient lime mortar is 70% for sample AC, 67% for sample SF, 54% for sample SP, and 50% for sample HP, which corroborates the findings of Borsoi et al. [1] who reported values ranging from 46% to 68%.

The siliceous aggregates (including quartz, albite, and microcline) correspond to the insoluble residue in acid (IR), while the soluble residue (SR) compounds in acid correspond to carbonates such as CaCO_3 and clay minerals such as Siderite, Sylvite, and Goethite, as indicated by the XRD results of [21].

Table 3 Simplified Chemical Composition of the Mortars (% W_t).

Sample	IR%	SR%
AC	70.08	29.92
HP	50.05	49.95
SF	66.59	33.41
SP	54.35	45.65

IR: Insoluble Residue in acid
SR: Soluble Residue in acid

Figures 5, 6, and Table 4 illustrate the grain-size distribution and the cumulative percentage retained on the sieves of the insoluble residue present in the Qadhad mortar, determined through sieve analysis. This analysis demonstrates a well-graded distribution across the four samples.

Sample AC is composed of 40% coarse aggregate, 50% fine aggregate (sand), and 10% volcanic ash. In contrast, sample HP comprises 30% coarse aggregate, 65% fine aggregate (sand), and

5% volcanic ash. Sample SF is characterized by 60% coarse aggregate, 35% fine aggregate (sand), and 5% volcanic ash. Lastly, sample SP contains 20% coarse aggregate, 75% fine aggregate (sand), and 5% volcanic ash.

Samples AC and SF contain coarse aggregate, whereas samples HP and SP are predominantly fine aggregate. All samples exhibit a volcanic ash content of approximately 5%.

Sample AC is classified as a blended or well-graded aggregate, exhibiting a substantial ratio of both coarse and fine aggregates. Sample SF is definitely categorized as coarse aggregate, primarily consisting of gravel or crushed stone, with a minimal sand inclusion. Both samples are capable of yielding a robust, dense slaked lime mixture, serving as the foundational structural framework within the slaked lime composition.

Samples HP and SP are identified as fine aggregate sand, containing a negligible proportion of coarse aggregate. They are deemed appropriate for applications in plastering and masonry construction.

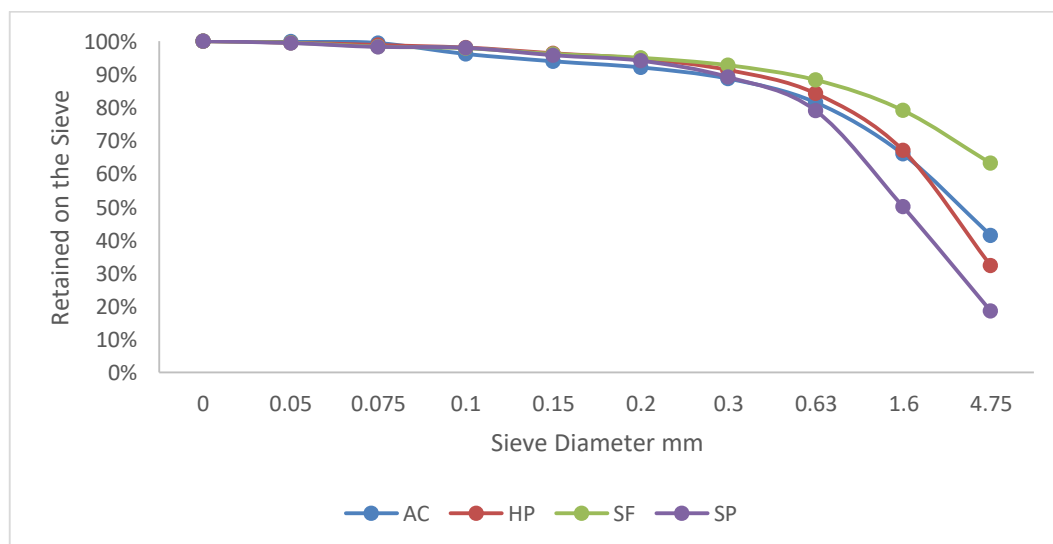


Figure 5: The percentage retained on the sieve of the insoluble residue in the mortars

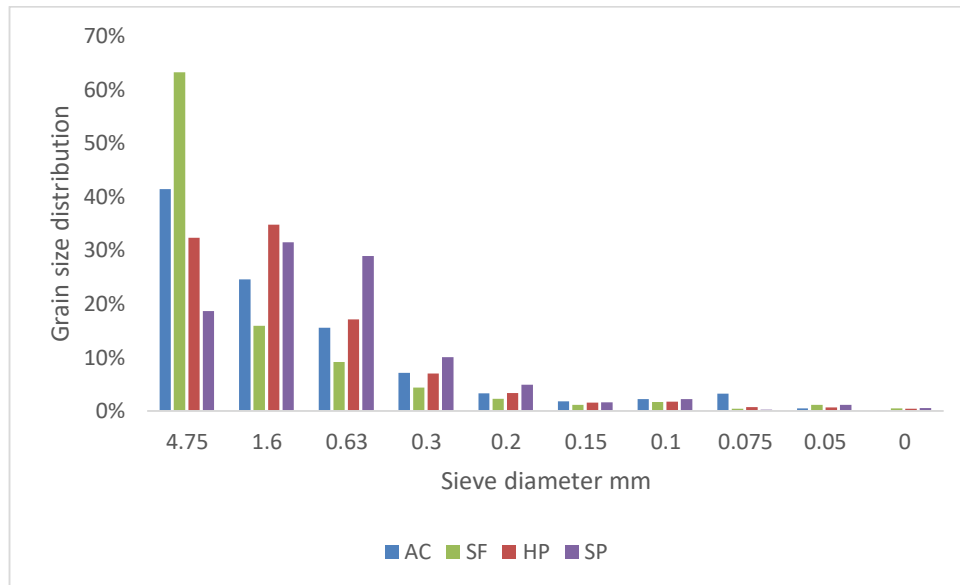


Figure 6: Grain-size distribution of the aggregates in the mortars

Table 4: The Distribution of the Aggregates in the Mortars

Sieve diameter mm	Type	AC	HP	SF	SP
>4.75	coarse aggregates	41%	32%	63%	19%
4.75-0.15	fine aggregates	52%	64%	33%	77%
<0.15	ash	6%	4%	4%	4%

4. More Discussion

4.1. Qadhad Composition

The compositional characterization of the mortars was carried out using the Jedrzejewska method for historical lime mortars [1], which distinguishes between two principal fractions: (i) the aggregate fraction, corresponding to the insoluble residue in acid (IR), and (ii) the soluble fraction (Sf), representing acid-soluble compounds excluding carbon dioxide-producing phases. However, the binder component is quantified as carbonate content (CaCO_3), derived from thermogravimetric analysis (TGA), as presented in Table 5.

The results reveal clear compositional differences between the studied samples. Specifically, the soluble fraction remains relatively low in samples AC and SF (below 4%), whereas it is substantially higher in samples HP and SP (ranging between 11% and 18%). This variation suggests differences in the presence of soluble mineral phases and potentially reflects distinct raw material sources or processing conditions. XRD analysis [21] indicates that the soluble fraction (Sf) is dominated by siderite and sylvite in samples HP and SP.

Mineralogically, according to XRD analysis [21], further indicates that the aggregate fraction (IR + Sf) is dominated by siliceous–feldspathic minerals, including quartz, albite, and microcline. These are accompanied by minor quantities of iron-bearing phases (hematite, goethite, and siderite) and trace evaporitic salts such as sylvite, while calcite constitutes the primary binder phase. This mineralogical assemblage confirms the hybrid nature of Qadhad as a lime-based mortar incorporating naturally pozzolanic volcanic aggregates. The aggregate fraction comprises a combination of coarse particles, fine particles, and ash-sized material, reflecting a deliberately graded system.

The calculated binder-to-aggregate ratios, based on CaCO_3 as the binder and (IR + Sf) as the aggregate, range from approximately 1:2.7 (AC) and 1:2.45 (SF) to 1:2.18 (HP) and 1:1.93 (SP).

These binder-to-aggregate ratios align with findings documented in other studies. The recommended binder/aggregate ratio, nearing 1:3 by volume, is supported by Borsoi G. et al. [1]. Additionally, Wonganan et al. [29] reported binder/aggregate ratios of 1:2, 1:2.33, and 1:2.5 by weight. The outcomes of this study corroborate previous research conducted by Watsantachard [27], which indicated that the binder/sand ratio fluctuated between 1:1.9 and 1:2.3 by weight. Furthermore, the binder-to-sand ratios of the ancient mortars were similarly

evaluated against the recommendations from Moropoulou et al. [30], which suggested that the binder/aggregate ratio (by weight) ranges between 1:4 and 1:1 for hydraulic lime mortars.

Table 5: Mortar composition and binder/aggregate ratio by weight.

Sample	IR%	CaCO ₃ %	Soluble fraction %	Aggregate ratio %	B/A ratio
AC	70.08	27.86	2.92	73.00	1:2.7
HP	50.05	31.42	18.53	68.47	1:2.18
SF	66.59	28.96	4.45	71.04	1:2.45
SP	54.35	34.06	11.59	65.94	1:1.93

IR: Insoluble Residue in acid

Soluble fraction in acid

Calculated CaCO₃ content from TGA analysis

Aggregate ratio %= (IR + Soluble Fraction).

When expressed as calcite/coarse aggregate/fine aggregate/ash ratios, the mortars exhibit measurable variability across samples. However, two compositional patterns emerge. Samples AC and SF display relatively higher coarse aggregate content, whereas HP and SP are characterized by increased proportions of fine aggregates. Averaged representations of these groups suggest ratios of approximately 1/1.35/1.2/0.15 for AC–SF and 1/0.55/1.45/0.1 for HP–SP. These distinctions indicate that Qadhad production was not uniform but adapted to locally available materials and construction practices.

4.2. Mechanical Properties

The mechanical behavior of Qadhad mortar is governed by the interplay of chemical reactions, microstructural development, and aggregate characteristics. Three primary mechanisms can be identified [14, 31, 32].

First, strength development is strongly influenced by the transformation of calcium hydroxide through both carbonation and pozzolanic reactions. While carbonation leads to the formation

of calcite, pozzolanic reactions between Ca(OH)_2 and reactive siliceous phases produce calcium–silicate–hydrate (C–S–H) and calcium–aluminate–hydrate (C–A–H), which contribute more substantially to long-term mechanical performance.

Second, microstructural densification plays a critical role. The formation of hydration products, combined with optimized aggregate gradation, leads to reduced porosity and improved internal cohesion. As a result, mortars with denser matrices generally exhibit enhanced mechanical resistance and durability.

Third, the proportion and intrinsic strength of coarse aggregates significantly affect load-bearing capacity. Coarse aggregates contribute to the formation of a rigid granular skeleton, which improves compressive strength and structural stability.

Although the XRD results [21] indicate that samples AC and SF exhibit a lower degree of carbonation than samples HP and SP, their microstructures appear denser and more compact. This apparent contradiction underscores the significant role of pozzolanic reactions and aggregate packing in the development of mechanical strength. SEM observations confirm the presence of C–S–H, C–A–H, and C–N–H hydration products in samples AC and SF. These cementitious phases, combined with a more favorable particle-size distribution, contribute to microstructural densification and compensate for the lower degree of carbonation. Therefore, the effect of the first and second factors on the compressive stress value is equal for all samples.

However, the proportion of coarse aggregates appears to be a key factor influencing compressive strength for these samples. Sample SF, which exhibited the highest compressive strength, contained the greatest proportion of coarse aggregates (63%). Samples AC and HP

contained intermediate coarse aggregate contents of 41% and 32%, respectively, while sample SP contained only 19%. This trend suggests that the higher coarse aggregate content enhanced load-bearing capacity and contributed significantly to the observed strength development.

The physical properties reported for the studied mortars are consistent with those of hydraulic lime systems. As noted in Section 4.1, typical hydraulic lime mortars exhibit porosity values between 18% and 40% and bulk densities ranging from 1700 to 2100 kg/m³ [30]. The agreement between these established ranges and the present results supports the classification of Qadhad as a hydraulic lime mortar. Therefore, the tensile stress value can be estimated from reported tensile strength values (0.35–0.55 MPa) [30], which provide a useful benchmark for contextualizing the mechanical behavior observed in this study.

4.3. Relationship Between Properties and Composition of Qadhad

A clear relationship emerges between composition and performance, allowing the samples to be grouped into two distinct categories: Group I (AC and SF) and Group II (HP and SP). This classification reflects not only similarities in material composition but also parallels in physical and mechanical properties.

Geographical proximity appears to partially explain this grouping. Samples within each group originate from relatively close locations, whereas the distance between groups is substantial. This spatial distribution suggests that regional geological conditions influence both raw material selection and mortar formulation.

Group I is characterized by a higher proportion of siliceous volcanic aggregates, particularly quartz and feldspar minerals. These components play a decisive role in promoting pozzolanic reactions, leading

to the formation of C-S-H, C-A-H, and C-N-H phases. The resulting microstructure is denser and less permeable, which enhances both strength and durability.

In contrast, Group II contains lower proportions of reactive aggregates and shows a greater dependence on carbonation processes. While carbonation contributes to mechanical strength through calcite formation, it does not provide the same level of long-term stability as pozzolanic bonding. This distinction explains the comparatively lower performance observed in these samples.

Differences are also evident in the distribution of accessory minerals. Group I is dominated by hematite and goethite, whereas Group II includes magnetite, siderite, and sylvite. Although these minerals are present in smaller quantities, they may influence durability and chemical stability.

Aggregate gradation further differentiates the groups. Group I exhibits a more balanced particle size distribution, which enhances packing density and reduces porosity. In addition, the higher proportion of coarse aggregates contributes to improved physical and mechanical properties by reinforcing the internal structure of the mortar.

Overall, the findings demonstrate that the performance of Qadhad mortar is not determined by a single factor but by the combined effects of composition, aggregate grading, and mineralogical characteristics. These results highlight the importance of material selection and proportioning in traditional construction technologies.

4.4. Production of Qadhad

The production of Qadhad mortar reflects a sophisticated empirical understanding of material behavior, integrating raw material selection, proportioning, and processing techniques. The reconstruction of its formulation begins with the characterization of original mortars, particularly their mineralogical composition and binder-to-aggregate ratios.

The use of hydrated lime as the primary binder, combined with siliceous volcanic aggregates, is essential for reproducing the hydraulic properties of Qadhad. Achieving appropriate workability requires careful control of the water-to-binder ratio, while long-term performance depends on the progression of carbonation and pozzolanic reactions. Consequently, extended curing and monitoring periods are necessary to evaluate durability and compatibility.

Based on the compositional characteristics identified in this study, together with documented traditional Yemeni construction practices [2], a representative production method can be outlined. Limestone with high calcium content is calcined at approximately 900°C for several days to produce quicklime, which is subsequently slaked to form hydrated lime. Volcanic aggregates are then processed by washing and sieving to achieve a well-defined particle-size distribution, typically comprising coarse, fine, and ash fractions.

The binder and aggregates are mixed in a ratio close to 1:2.5, producing a homogeneous mortar that is applied in successive layers. Each layer is compacted and finished through repeated mechanical treatment, which enhances density and reduces porosity. The use of angular coarse aggregates further improves mechanical interlocking within the matrix.

A distinctive aspect of Qadhad production is the prolonged surface treatment process. Maintaining moisture through lime washing and continuous polishing promotes gradual hardening, minimizes shrinkage cracking, and improves surface impermeability. The application of a protective layer, such as animal fat, further enhances water resistance, particularly during early exposure to environmental conditions.

This combination of material optimization and construction technique is central to the durability and performance of Qadhad. Importantly, it illustrates how traditional knowledge

systems achieved high-performance materials through empirical refinement, offering valuable insights for contemporary sustainable construction practices.

Despite these advantages, the production of Qadhad remains labor-intensive and technically demanding, which poses challenges for its wider application in contemporary construction. Accordingly, future research should focus on optimizing and standardizing production methods to improve efficiency and reproducibility while preserving the material's distinctive hydraulic and durability properties.

5 Conclusions

The findings of this study demonstrate that ancient Yemeni Qadhad mortar possesses mechanical characteristics that explain its long-term durability in historical hydraulic structures. The compressive strength values obtained in this research range from 2.77 to 4.39 MPa, which are consistent with the typical strength range of traditional hydraulic lime mortars used in historic masonry construction. These values indicate that Qadhad mortar provides sufficient mechanical capacity for use as a binding material in stone masonry while maintaining compatibility with traditional building materials.

The composition of Qadhad, inclusive of calcite as a binder combined with siliceous volcanic aggregates such as quartz, microcline, and albite, significantly influences its mechanical, physical, and hydraulic properties. The calculated binder–aggregate ratios range approximately 1:2.5 by weight, with the aggregate comprising 50% coarse aggregate, 40% sand, and no more than 10% particles smaller than 0.15 mm. This corresponds well with recommended proportions for hydraulic lime mortars used in conservation practices. Such

proportions appear to provide an optimal balance between mechanical strength, durability, and permeability control.

The results of this research are presented in conjunction with the results of algrafi et al. [21], providing an improved scientific understanding of the original composition and properties of ancient Yemeni Qadhad mortar, offering a valuable basis for reproducing compatible materials for heritage conservation. The reconstruction of this traditional mortar formulation can support the restoration and preservation of historic Yemeni architecture while also highlighting the potential of lime–pozzolan mortars as environmentally sustainable materials for modern construction applications.

6 Recommendations

1. In the production of Qadhad, in accordance with the results presented in this paper, particular emphasis should be placed on the workability and subsequent applicability of the mortars by optimizing the water/binder ratio. It is imperative to conduct mechanical, physical, and hydraulic assessments and validate the resultant data.
2. Investigate the potential applications of Qadhad in contemporary eco-construction, capitalizing on its durability, self-healing properties, and minimal environmental footprint.
3. Future Research: Undertake additional investigations to quantify the mechanical efficacy, self-healing capabilities, and carbon balance of Qadhad, while incorporating a cost-benefit analysis to delineate optimal production and application strategies for both preservation and sustainable construction use.
4. Streamlining the procedural steps of plaster manufacturing to render the process more straightforward and readily implementable for on-site construction personnel.

References

- [1] Borsoi, G., Silva, A.S., Menezes, P., Candeias, A., & Mirão, J. Analytical characterization of ancient mortars from the archaeological Roman site of Pisões, *Constr. Build. Mater.* 204 (2019) 597–608. <https://doi.org/10.1016/j.conbuildmat.2019.01.233>
- [2] Kulaib, M., & Al-Jolahy, A. Back to traditional building materials, *Yemeni Engineer, Faculty of Engineering-University of Aden* 6 (2006) 113–122.
- [3] Zicarelli, M. A., Ricca, M., Alberghina, M. F., Schiavone, S., La Russa, M. F., & Randazzo, L. Archaeometric investigation of artificial stone materials from the Theatrum Marcelli (Rome, Italy), *Heritage* 8 (2) (2025) 57. <https://doi.org/10.3390/heritage8020057>
- [4] Siddall, R. The use of volcanoclastic material in Roman hydraulic concretes: A brief review, in: W.J. McGuire, D.R. Griffiths, P.L. Hancock, I.S. Stewart (Eds.), *The Archaeology of Geological Catastrophes*, Geological Society, London, Special Publications, 171 (2000) 339–344. <https://doi.org/10.1144/GSL.SP.2000.171.01.24>
- [5] Degryse, P., Elsen, J., & Waelkens, M. Study of ancient mortars from Sagalassos (Turkey) in view of their conservation, *Cem. Concr. Res.* 32 (9) (2002) 1457–1463. [https://doi.org/10.1016/S0008-8846\(02\)00807-4](https://doi.org/10.1016/S0008-8846(02)00807-4)
- [6] Fort, R., Ergenç, D., Aly, N., de Buergo, M.A., & Hemeda, S. Implications of new mineral phases in the isotopic composition of Roman lime mortars at the Kom el-Dikka archaeological site in Egypt, *Constr. Build. Mater.* 268 (2021) 121085. <https://doi.org/10.1016/j.conbuildmat.2020.121085>
- [7] Jackson, M.D., Mulcahy, S.R., Chen, H., Li, Y., Li, Q., Cappelletti, P., & Wenk, H. R. Phillipsite and Al-tobermorite mineral cements produced through low-temperature water-rock reactions in Roman marine concrete, *Am. Mineral.* 102 (7) (2017) 1435–1450. <https://doi.org/10.2138/am-2017-5993>
- [8] Singh, S.K., & Singh, M.R. Characterization and compositional identification of Stepwell (Baoli) lime plasters from 16th Century Arab Ki Sarai, Delhi, *J. Build. Pathol. Rehabil.* 9 (2024) 77. <https://doi.org/10.1007/s41024-024-00427-1>

[9] Maravelaki-Kalaitzaki, P., Bakolas, A., & Moropoulou, A. Physico-chemical study of Cretan ancient mortars, *Cem. Concr. Res.* 33 (5) (2003) 651–661. [https://doi.org/10.1016/S0008-8846\(02\)01030-X](https://doi.org/10.1016/S0008-8846(02)01030-X)

[10] Qian, K., Song, Y., Lai, J., Qian, X., Zhang, Z., Liang, Y., & Ruan, S. Characterization of historical mortar from ancient city walls of Xindeng in Fuyang, China, *Constr. Build. Mater.* 315 (2022) 125780. <https://doi.org/10.1016/j.conbuildmat.2021.125780>

[11] Rodriguez-Juarez, M.E., Perez-Díaz, E., Lopez-Dominguez, G.I., Picazo, V.L., Valencia-Cruz, D., Millan-Malo, B.M., & Rodriguez-Garcia, M.E. Development and characterization of lime-based stucco for modern construction and restoration applications based on ancient stuccoes from the "El Cerrito" pyramid, Querétaro, Mexico, *Case Stud. Constr. Mater.* 16 (2022) e00875. <https://doi.org/10.1016/j.cscm.2021.e00875>

[12] Sanchez-Moral, S., Luque, L., Cañaveras, J.C., Soler, V., Garcia-Guinea, J., & Aparicio, A. Lime pozzolana mortars in Roman catacombs: composition, structures and restoration, *Cem. Concr. Res.* 35 (8) (2005) 1555–1565. <https://doi.org/10.1016/j.cemconres.2004.08.009>

[13] Vinay Kumar, G., Ramadoss, R., & Rampradheep, G. S. Study on various properties and behaviour of lime with hemp, metakaolin and carboxyl cellulose, *J. Build. Pathol. Rehabil.* 10 (2025) 19. <https://doi.org/10.1007/s41024-024-00525-0>

[14] Maravelaki-Kalaitzaki, P., Bakolas, A., Karatasios, I., & Kilikoglou, V. Hydraulic lime mortars for the restoration of historic masonry in Crete, *Cem. Concr. Res.* 35 (8) (2005) 1577–1586. <https://doi.org/10.1016/j.cemconres.2004.09.001>

[15] Shivakumara, M., & Selvaraj, T. A scientific study on the role of organic lime mortars of Padmanabhapuram Palace, Huckalay, Tamil Nadu, India, *Eur. Phys. J. Plus* 135 (2020) 923. <https://doi.org/10.1140/epjp/s13360-020-00896-6>

[16] Solak, A. Experimental investigation of lime mortar used in historical buildings in Becin, Turkey, *Mater. Sci.* 22 (1) (2016) 73-79. <https://doi.org/10.5755/j01.ms.22.1.9022>

[17] Haneefa, K.M., Rani, S.D., Ramasamy, R., & Santhanam, M. Microstructure and geochemistry of lime plaster mortar from a heritage structure, *Constr. Build. Mater.* 225 (2019) 538-554. <https://doi.org/10.1016/j.conbuildmat.2019.07.159>

[18] Işık, T., & Sağın, E. Characteristics of Byzantine-period lime mortars and plasters from the Anaia Church, *Mater. Tehnol.* 56 (5) (2022) 573–583. <https://doi.org/10.17222/mit.2022.524>

[19] Papayianni, I., Pachta, V., & Stefanidou, M. Analysis of ancient mortars and design of compatible repair mortars: The case study of Odeion of the archaeological site of Dion, *Constr. Build. Mater.* 40 (2013) 84–92. <https://doi.org/10.1016/j.conbuildmat.2012.09.086>

[20] Weiss, C., & Gerlach, I. Qadhat—a comparative study of ancient and new lime mortar from the Marib province/Yemen, *Archaeol. Anthropol. Sci.* 1 (2009) 87–94. <https://doi.org/10.1007/s12520-009-0007-2>

[21] Algorafi, M. A., Alwathaf, A. H., Al-Gawi, S., & Al-Nono, A. M. Characterization of Ancient Yemeni Qadhad Plaster: A Low-Permeability Lime–Pozzolan Material for Sustainable and Durable Conservation and Construction, *Int. J. Concrete Struct. Mater.* (2026) 20:62. <https://doi.org/10.1186/s40069-026-00930-4>

[22] Al-Gawi, S., Algorafi, M., & Al-Nono, A. The Physical, Hydraulic, and Mineralogical Characterization Study of the Traditional Qadhad Material of the Cisterns of Aden, *Journal of Science and Technology* 30 (1) (2025) 12-28. <https://doi.org/10.20428/jst.v30i12.3436>

[23] Al-Gawi, S., Algorafi, M., Kulaib, M., & Al-Nono, A. Unveiling the Secrets of Ancient Waterproofing: A Multi-Analytical Characterization of Traditional Qadhad Mortar from Sira Castle, Yemen, *Sana'a University Journal of Applied Sciences and Technology* 4 (5) (2026) 2103-2121. <https://doi.org/10.59628/jast.v4i5.2910>

[24] ASTM C109/C109M, Standard Test Method for Compressive Strength of Hydraulic Cement Mortars, ASTM International, West Conshohocken, PA, 2024.

[25] ASTM D3042-17, Standard Test Method for Insoluble Residue in Carbonate Aggregates, ASTM International, West Conshohocken, PA, 2017.

[26] ASTM C136/C136M-20, Standard Test Method for Sieve Analysis of Fine and Coarse Aggregates, ASTM International, West Conshohocken, PA, 2020.

[27] Watsantachad, N. The properties of historic mortar and plaster in the historic city of Ayutthaya world heritage site, *NAJUA Architecture Design and Built Environment* 33 (B) (2018) 43–92.

[28] Mahasuwanhai, P., Wonganan, N., Athisakul, C., Tangchirapat, W., Sahamitmongkol, R., & Leelataviwat, S. Engineering properties of ancient masonry materials in Thailand and substitution materials for historical structures preservation, *IOP Conf. Ser.: Earth Environ. Sci.* 463 (2020) 012077. <https://doi.org/10.1088/1755-1315/463/1/012077>

[29] Wonganan, N., Athisakul, C., Mahasuwanhai, P., Tangchirapat, W., Sahamitmongkol, R., & Leelataviwat, S. Ancient Materials and Substitution Materials Used in Thai Historical Masonry Structure Preservation, *J. Renew. Mater.* 9 (2) (2021) 179-204. <https://doi.org/10.32604/jrm.2021.013134>

[30] Moropoulou, A., Bakolas, A., & Anagnostopoulou, S. Composite materials in ancient structures, *Cem. Concr. Compos.* 27 (2005) 295–300. <https://doi.org/10.1016/j.cemconcomp.2004.02.018>

[31] Zacharopoulou, G. The Effect of wet slaked lime putty and putty prepared from dry hydrate on the strength of lime mortars, in: 2nd Conference on Historic Mortars–HMC 2010 and RILEM TC 203-RHM Final Workshop, RILEM Publications SARL, Prague, Czech Republic, 2010, pp. 1283–1291.

[32] Silva, B.A., Ferreira Pinto, A.P., Gomes, A., & Candeias, A. Effects of natural and accelerated carbonation on the properties of lime-based materials, *J. CO2 Util.* 49 (2021) 101552. <https://doi.org/10.1016/j.jcou.2021.101552>

Research Contributions

The principal contributions of this study are:

- (1) Quantification of the compressive strength of ancient Yemeni Qadhad mortar.
- (2) Determination of binder-to-aggregate ratios using combined TGA and acid digestion methods.
- (3) Establishment of a scientific basis for the reproduction of compatible conservation mortars.
- (4) Advancement of the understanding of traditional hydraulic lime technologies in the Arabian Peninsula.

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