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CHEMISTRY OF LIME MORTARED RUBBLE MASONRY IN BOHOL, PHILIPPINES

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Abstract

A combination of Energy Dispersive X-ray Fluorescence (EDXRF), Fourier Transform Infrared (FTIR) Spectroscopy, X-Ray Diffraction (XRD) and Thermogravimetric Analysis (TGA) provided a detailed chemical and mineralogical characterization of rubble mortars inside a coral stone veneer wall of 19th century Spanish Colonial Period structures in the Philippines. Evidence of possible raw materials (seashells/corals and beach sand), manufacturing techniques (dry and wet slaking), and type of lime binder (aerial and slightly hydraulic), was reported. Results of this study provides a needed baseline scientific data for promoting the proper restoration and conservation of heritage structures in the Philippines.

***Keywords**: Rubble mortar; EDXRF; FTIR; XRD; Thermal analysis; Philippines*

Introduction

In the last decades, the importance of applying chemical analysis in understanding the composition of historical building materials have been rapidly increasing [1]. With this chemical knowledge, a deeper awareness of the building's history is better appreciated. Before the development of modern Portland cement, lime mortars are an integral part of any building structure and widely incorporated as a wall plaster or a binder for stones, bricks, and rubble work materials [2, 3]. It is traditionally produced by firing lime (i.e., limestone or crush seashells) and mixed with various proportions of aggregates which may include sand, crushed bricks, broken pottery or seashells and volcanic materials [4, 5]

Even though lime mortars have been used for centuries, its efficiency as a building material is now beginning to be unraveled. Part of its complexity is the various mortar making traditions per country that also changes depending on the local culture and available raw materials. Chemical characterization is relevant to know the provenance of the raw material, assess the damage sustained through the years, address issues on the compatibility of replacement materials and to direct a proper conservation management scheme.

A common traditional technique of wall construction which relies on the binding properties of lime mortars is the core-and-veneer method [6]. This method involves the construction of two parallel veneer walls and a filling of rubblework is packed tightly in-between producing a thick solid core wall material. The rubble core may contain only plain aggregates (i.e., soil, sand, gravel or crush bricks) or mortared, wherein the aggregates are mixed together with lime [7]. This method has been utilized on historical structures such as civic and ecclesiastical buildings, palaces, bridges and fortifications [8, 9].

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Most of the studies describing historical rubblework infill on walls are mainly focused on its mechanical behavior and morphological characteristics [10, 11]. Model rubbleworks that approximate the physical properties of the old material were also reported. It was subjected to different tests such as compression tests, hole-drilling tests and sonic tests, to observe the performance of the binder and aggregates in a real mortar sample [12-14]. In contrast, few reports were directed on the actual chemistry of the mortars found in the rubblework core [8, 9]. The scarcity of chemical information on these core materials is what this research aims to address.

The materials for this study were sampled from 19th century mortared masonry walls found in different church complex buildings in the Province of Bohol, Philippines. These structures are associated with the Spanish Colonial Period. Specifically, the church convents in the Municipalities of Alburquerque and Maribojoc, and the mortuary chapel in the Municipality of Dimiao were investigated. An interesting structural feature of church complex buildings in Bohol is the introduction of tree-trunk posts that serves as the basic framework for constructing the wall pillars. The timber is usually encased in a wall of rubblework and mortar utilizing the core-and-veneer method. This represents an effective mingling of local and western building techniques which may also led to a possible local reinterpretation of preparing the mortars for rubblework [15]. Detailed chemical and mineralogical characterization of the lime mortars were accomplished using X-ray diffraction (XRD), energy dispersive X-ray fluorescence (EDXRF) and Fourier transform infrared spectroscopy (FTIR). The hydraulic nature of the samples was determined by thermogravimetric analysis (TGA).

Results from this study contribute to the technical knowledge on the church and chapel's construction as well as to provide valuable information on how the local artisans adapt colonial period paradigms on building structures. It is only during the Spanish Colonial Period in the Philippines that the core-and-veneer technique employing lime mortared rubble became prevalent. Records on how the core raw materials were originally prepared before in its poured inside the veneer wall are very limited. Furthermore, the baseline chemical data provided in this study can aid in future conservation and restoration works for colonial period structures in the Philippines [16].

Experimental part

Sample Collection

The rubble mortar samples were gathered from structures built during the Spanish Colonial Period in the 19th century. Figure 1 shows the site of the different coastal municipalities in the Province of Bohol where sampling was performed. The church convents at Alburquerque (ABQ) and Maribojoc (MBJ), and the mortuary chapel in Dimiao (DIM) are found within the perimeter of the town's respective historic church complex. In the case of the convents, both are adjoined to the main church structure, while the mortuary chapel is a few meters away from the main church. Table 1 provides a descriptive summary of the mortar samples used in this study and its provenance.

Table 1. Description of the mortar samples from Bohol

Mortar Code	Function	Date of construction [15]	Provenance	Veneer type
ABQ	Core mortar in pier	Between 1869-1879	Church convent in Alburquerque	Coral stone block
DIM	Wall core mortar	Early 1800's	Mortuary Chapel in Dimiao	Coral stone block
MBJ	Core mortar in pier	Between 1877-1884	Church convent in Maribojoc	Coral stone block

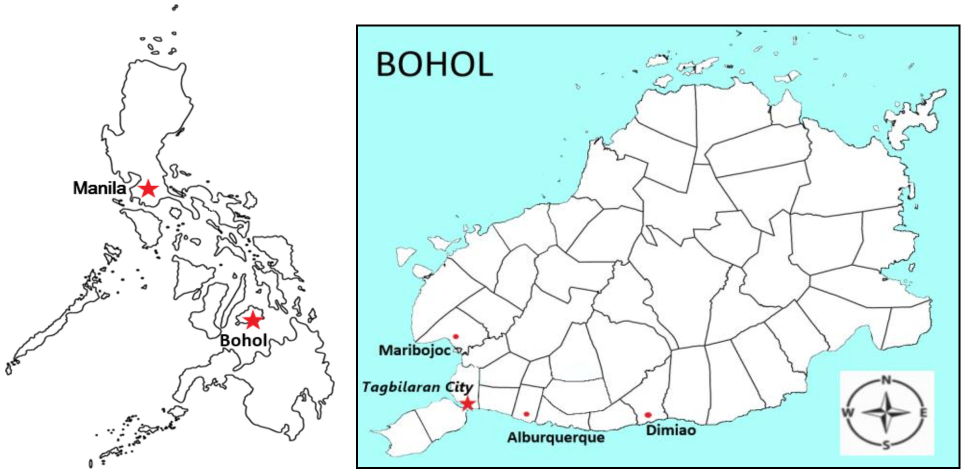

Fig. 1. Map of the Philippines showing the location of Bohol Province in relation to the country's capital, Manila. The geographical location of the Municipalities of Alburquerque, Dimiao and Maribojoc in the Province of Bohol where the samples are collected. Tagbilaran City is the provincial capital of Bohol

All the rubble mortars were collected in May 2017 by hand. The coral stone veneer walls fell off from the structure during a magnitude 7.2 earthquake in 2013 which exposed the inner mortar core [17]. This facilitated the easy removal of the mortar from the core without any need for a hammer and chisel to dislodge the samples. Upon closer inspection of the mortar materials, it was evident from the physical characteristics that it is made entirely of lime and not of modern-day cement. The wall enclosure is also located from an old part of the structure which is free from any intervention or restoration.

Fig. 2. The rubbled mortar samples used in this study from (A) Alburquerque church convent, (B) Dimiao mortuary chapel, and (C) Maribojoc church convent

This assures that the mortars obtained are a good representation of the original construction material used in the 19th century. To ensure that the core mortar is free of any contamination, the sample materials were obtained from a depth of about 1 to 2 inches from the exposed surface. A total of 15.27g of core mortar sample was taken from ABQ, 16.85g of mortar sample from DIM and 21.09g of sample from MBJ, respectively (Fig. 2). The amounts collected are considered very minimal and barely enough to perform the chemical analysis stated in this study. This guarantees the protection of the heritage material's historical authenticity.

Methods

Energy Dispersive X-Ray Fluorescence (EDXRF)

Shimadzu EDX-7000, Energy Dispersive X-Ray Fluorescence Spectrometer (EDXRF) was utilized to examine the elemental composition of ABQ, DIM and MBJ. Samples were analyzed using a polypropylene cup as a container. The collimator was set to 3 mm while the instrument was programmed to run a detailed analysis mode in a vacuum atmosphere. The results were reported as elemental oxide composition and undetected elements (i.e., carbon dioxide) are grouped under the loss on ignition (LOI).

Fourier Transform Infrared (FTIR) Spectroscopy

Infrared data for ABQ, DIM and MBJ was obtained by a Thermo Scientific Nicolet 6700 Fourier Transform Infrared (FTIR) Spectrometer. The samples were prepared using the KBr pellet method and scanned within the mid-infrared region (4000 to 500cm^{-1}) for 16 times at a resolution of 4 cm^{-1}.

X-ray Diffraction (XRD)

Mineral characterization of ABQ, DIM and MBJ were performed in an Olympus TERRA-248 InXitu portable X-ray Diffractometer. The X-ray source radiation came from a cobalt, Co-K_α peak with λ=1.7903Å. Samples were continuously scanned at a º2Θ range of 3.00 to 55.00. Phase data was interpreted using MATCH! Phase Identification from Powder Diffraction software developed by crystal impact (Version 3.9.0.158 downloaded on January 3, 2020). Results were also correlated with the XRD mineralogy database from www.webmineral.com.

Thermogravimetric Analysis (TGA)

Thermographs from ABQ, DIM and MBJ were collected on a TA Instruments Discovery TGA55. The parameters are set to a ramping temperature program from 22to 1,000°C at a rate of 10°C/minute in N_2 gas atmosphere. The platinum pan (Platinum HT) was used due to the high temperature. The mass of the sample was automatically weighted by the TGA and has the following initial masses: 18.038 mg for ABQ, 18.585mg for DIM, and 8.463mg for MBJ, respectively.

Results and discussion

Quantitative elemental composition

ABQ, DIM and MBJ are characterized as having an abundant amount of calcium ranging from 30.0 to 37.0% CaO and silicates from 2.0 to 4.5% SiO_2. The rest of the chemical elements are found to be less than 0.600% (Table 2). Having CaO and SiO_2 in relatively large quantities compared to the other elements in each samples are indicative of a predominantly lime and sand raw material origin. Lime acts as the binder in historical mortar preparations, while sand is generally classified as an aggregate. The sand is added deliberately or a result of manufacturing contaminations. The absence of magnesium on the EDXRF implies that the lime source is largely calcitic and dolomite rocks containing magnesium carbonate are not present. Computing for the weight ratios using the molar masses of CaO (56g/mol) and CO_2 (44g/mol), the lime material contains the following percentage of CO_2: 28.90% for ABQ, 30.26% for DIM and 23.91% for MBJ, respectively. If this is compared to the loss on ignition (LOI) values, CO_2

accounts to 50.37% for ABQ, 54.71% for DIM and 36.57% for MBJ, respectively. These values shows that the mortar material contain a large number of impurities and the unaccounted LOI values may be atttributed to organic matter or hydrated compounds [18]. Clays which consists of aluminum phylosillicate minerals are likely absent in all the mortar samples due to undetected Al_2O_3 and low amounts of Fe_2O_3 (< 0.450%).

Results obtained from the EDXRF data is analogous to an elemental XRF study performed on old mortars during restoration work of another Spanish Era (late 1800's) church in Dauis, Bohol damaged by the 2013 earthquake [17]. Aluminum and magnesium elements are also not detected in mortar samples from Dauis. It is speculated that the same building methodology may have been employed in Dauis and the rubble mortar samples in this study due to similarities in the construction period, management (i.e. Agustinian Recollects religious order) and coastal environment. Furthermore, these results give the impression that the lime used in the rubble mortar samples are sourced from crushed seashells or probably corals rather than limestones [19]. This is possible since the location of the structures are near the sea area and the local artisans can easily transport the raw materials into the site.

Table 2. Percent chemical composition of the rubble mortar samples using EDXRF

Sample	CaO	SiO_2	Fe_2O_3	K_2O	SrO	SO_3	Cl	TiO_2	MnO	CuO	Br	ZnO	NiO	LOI*
ABQ	36.786	3.341	0.231	0.253	0.507	0.294	1.193	-	-	0.008	0.006	-	-	57.380
DIM	38.508	4.448	0.443	0.206	0.525	0.498	-	0.034	0.016	0.008	-	0.001	0.002	55.308
MBJ	30.433	2.021	0.234	0.336	0.438	0.355	0.789	-	-	0.006	0.001	0.001	0.001	65.384

* LOI = loss on ignition

Mineralogical characterization

The dominant mineral component observed in the FTIR spectrum (Fig. 3) of ABQ, DIM and MBJ is the carbonate containing mineral, calcite ($CaCO_3$).

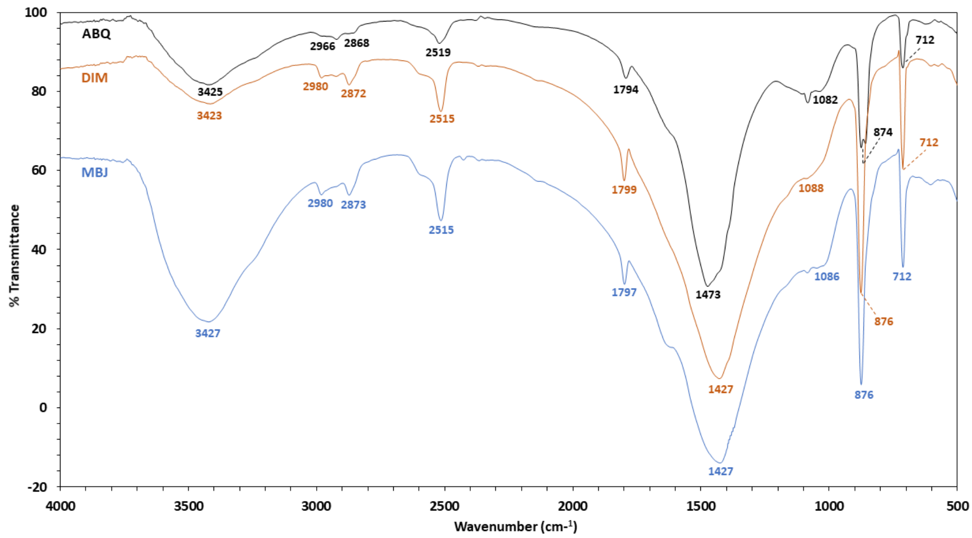

Fig. 3. FTIR spectrum of ABQ, DIM and MBJ

The fundamental vibration modes attributed to calcite's carbonate ion (CO_3^{2-}) are the prominent out-of-plane bending vibrations (υ2) at 876cm^{-1} (874cm^{-1} for ABQ) and the broad assymetric stretching (υ3) vibrations centered at 1427cm^{-1} (1473cm^{-1} for ABQ), respectively. The sharp absorption peak at 712cm^{-1}, assigned to the in-plane bending vibration (υ4), is the distinguishing feature of calcite as opposed to a dolomite ($CaMg(CO_3)_2$) [20]. EDXRF data also shows the absence of magnesium in all the samples. Combination modes confirm the peak assignments for carbonate as seen from the spectra at ~1797cm^{-1} (υ1 + υ4) and ~2515cm^{-1} (2υ2 + υ4). Furthermore, overtone and υ3 combination bands at ~ 2868cm $^{-1}$ and at 2980cm^{-1} (2966cm^{-1} for ABQ), respectively, provides additional supporting information [21, 22].

Indications of the mineral quartz in the samples are evident from the broad shoulder at ~ 1082cm^{-1} which is assigned to the Si-O assymetric stretching vibration. Other absorption features in quartz are not readily noticeable from the FTIR spectrum. Absorb water are assigned to the broad OH stretching band centered at ~ 3423cm^{-1} [5]. Eventhough there are accounts in the Philippines of organic additives (i.e. albumin, plant sap, molasses) being combined with the mortar mixture [7], the FTIR wasn’t able to detect any organic functional groups in the rubble mortar samples.

A more detailed mineral characterization is provided by the X-ray diffraction which shows crystal patterns for calcite, quartz and lime in the samples (Figure 4). These identified mineral phases are consistent with the results of the EDXRF and FTIR. Calcite is the most abundant mineral component in all the samples based on the high intensity peak at 34.30 (2Θ), 3.033 Å. This is supported by other prominent peaks attributed to calcite having d-spacing values at 3.846, 2.496, 2.284 and 2.095Å, respectively. Peaks for dolomite, which is another possible carbonate containing mineral in lime, is absent in the XRD.

Low intensity peaks assigned to small amount of quartz are seen mainly from the d-spacing values at 4.241 and 3.342Å. Among the three mortar samples, ABQ contains relatively more amounts of quartz compared to both DIM and MBJ due to the intensity of the small peaks. These small variations in amount are possibly due to production inconsistencies. Consisted with the EDXRF, mineral phases attributed to clays are not detected from the XRD. These information may run counter to the general soil composition of Bohol which largely contain clay minerals [23]. Since, importing soil from other places is not cost-effective, the best explanation is that beach sand may have been utilized as the source of quartz.

Traces of lime, refering to CaO or quicklime, are burned $CaCO_3$ that did not slaked or hydrated properly when water was originally added during the production process [24]. It can be due to bulk pieces that were not pulverized properly or to natural impurities in the raw material. This was observed from the diffraction patterns of ABQ and DIM. Table 3 provides a summary of XRD results obtained from the rubble mortar samples.

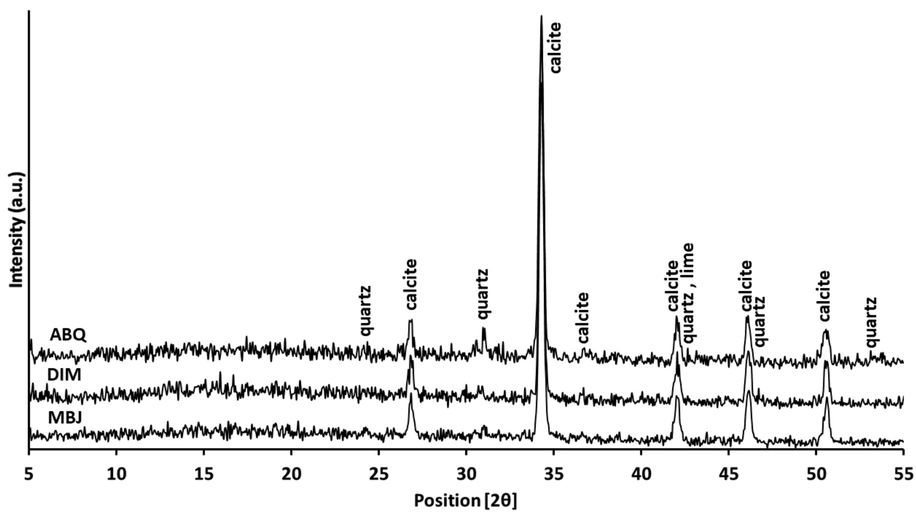

Fig. 4. X-ray diffraction patterns for the indicated bulk mortar samples

Table 3. Results of XRD analysis of investigated mortar samples

Sample	Crystalline phases		
	Calcite	Quartz	Lime
ABQ	+++	++	t
DIM	+++	+	t
MBJ	+++	+	-

Peak intensity: +++ abundant, ++ present, + small amount, t traces, - undetected

Thermal Analysis

The TGA recorded a continuous weight loss for ABQ, DIM and MBJ as the temperature increases from 22°C to 1,000°C (Figure 5). Calculated weight loss on specific temperature ranges enables a semi-quantitative identification of the mortar's components. Evaporation of hygroscopic water (until 120ºC) and crystallized hydrated salts (120ºC - 200ºC) [5] were exhibited by each sample with the highest combined weight loss for MBJ at around 3.4%. ABQ and DIM have total weight losses of less than 1.25% for these ranges, as shown in Table 4.

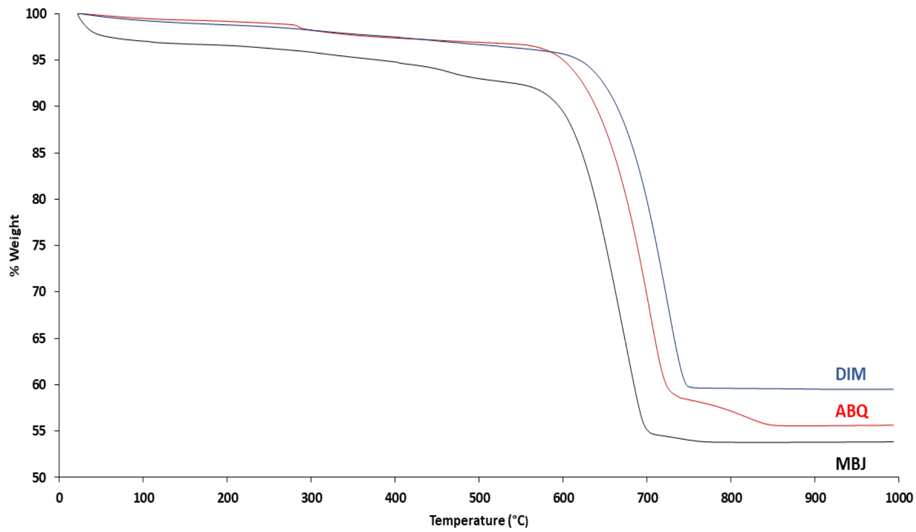

Fig. 5. Thermal analysis curves of the mortar samples

Table 4. Weight loss in % per temperature range of the sample

Sample	Temperature change (°C)					
	T < 120 °C	120 - 200 °C	200 - 600 °C	T > 600 °C	Total Weight Loss	CO_2/H_2O
ABQ	0.59 %	0.23 %	4.16 %	39.38 %	44.37 %	9.47
DIM	0.86 %	0.35 %	3.14 %	36.15 %	40.49 %	11.53
MBJ	3.13 %	0.28 %	7.16 %	35.58 %	46.15 %	4.97

These results suggest that two different slaking techniques may have been employed in preparing the lime raw material. MBJ due to its relative abundance of absorbed water is prepared by the wet slaking technique. This technique involves soaking the quicklime with excess water to form the lime putty [25]. It may be possible that the lime putty produced in this method was poured directly on the rubble core and was dried in time. Thus, water is retaining in the mortar sample. This observation coincides with the relatively broad OH absorption band in the FTIR spectrum of MBJ. On the other hand, ABQ and DIM are prepared using the dry slaking technique, where enough water was added to slake the quicklime and convert it into powdered form [25]. Eventually, as the slaked lime is poured in the rubble core, it contains lesser amount of water.

The temperature range between 200ºC to 600ºC corresponds mainly to the loss of chemically bounded water or can also be attributed to organic compound impurities in the samples [5]. DIM exhibited the least amount of weight loss at around 3.1%, while MBJ has lost around 7.1%. It is expected that MBJ will lose more water bound compounds due to the availability of more water during its preparation. A significant drop in each of the sample's weight, from 35.5% - 39.5%, occurred between 600°C to 800°C. These are characteristic patterns attributed to the decomposition of $CaCO_3$ by the release of CO_2. Specifically, the maximum weight loss occurred at 674°C for MBJ, 704°C for ABQ and 734°C for DIM, respectively, which implies that the samples are recarbonated [26]. By determining the ratio of CO_2/H_2O for each mortar samples, the hydraulic nature of the binder or its ability to harden and set under water can be assigned. This is computed based on the weight loss in the temperature range of > 600°C for CO_2 and the range of 200°C to 600°C for H_2O. Based on this ratio, ABQ and MBJ are considered as slightly hydraulic mortars (ratio between 4-9), while DIM is classified as typical air-lime mortar (ratio >10) [27]. These designations are generalized and the range of limits are not applied strictly. Note than production inconsistencies may have resulted to these differences. Furthermore since ABQ has a ratio of 9.47 which falls within the boundary of typical air lime to slightly hydraulic, it can be possible that the mortar may have hydraulic tendencies but not necessarily will harden in water [25]. Since clays are not identified from the analytical techniques used in this study, the hydraulic properties of MBJ may have been due to the amount of sand added to the lime mixture, imparting a certain hydraulic behavior in the mortar. Sand may have been pored separately into the veneer wall filings which eventually mix with the rubble lime mortar.

Conclusions

The chemical and mineralogical study of rubble mortar samples from the different colonial period structures in Bohol, Philippines have led to the understanding of the raw material origin and the possible manufacturing process. Baseline data of the composition was established from this study that can aid in future conservation work. It can also be used to compare data from 19th century Spanish Colonial structures in different locations.

The rubble mortars originated from seashells or corals and the sand additive is likely beach sand. This was evident by the dominant presence of calcite and quartz, while clay minerals are absent. Different slaking techniques were employed in making the mortars, a dry method (ABQ and DIM) and a wet method (MBJ). It is also possible that the slaked mortars were poured directly into the rubble core and was allowed to dry *in situ*. This would facilitate the construction of the wall masonry. The binder is classified as air lime (DIM) and slightly hydraulic lime (ABQ and MBJ). The hydraulic properties are possibly due to the beach sand additives and to the other bulk aggregates in the core. There were only slight differences in the chemical composition of the mortar samples studied implying that the general manufacturing process may have been similar.

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