

CHARACTERIZATION OF HISTORICAL LIME MORTAR FROM THE SPANISH COLONIAL PERIOD IN THE PHILIPPINES

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Keywords: lime mortar, binder, analytical chemistry techniques, cultural heritage preservation, Philippines

1. Introduction

One of the enduring legacies of the Spanish Colonial Period (1521-1898) in the Philippines, is the grand Roman Catholic Church structures that still stand today and are strategically located in the central plaza of most towns throughout the country. The Philippines was governed by the Spanish empire for almost 380 years. It began with the arrival of Ferdinand Magellan in 1521, when he claimed possession of the entire island, and remained under the King of Spain until the outbreak of the Spanish-American War when the country was later turned over to American rule in 1898. During the early years of the Spanish Conquest, different religious orders i.e. Augustinians (1565), Franciscans (1578), Jesuits (1581), Dominicans (1587) and Recollects (1606), arrived with the Spanish contingent and initiated missionary work in different parts of the country. These missionaries were not only pastors and evangelizers, they were also church builders. In Europe, they had been eye witnesses to the magnificent European medieval and baroque cathedrals and desired to duplicate this architectural and artistic style as well as impart their knowledge to the locals [1]. However, Filipino masons, being more familiar with local environmental conditions, also contributed to enriching this church building process by integrating native elements, designs and local ingenuity. The product is an architectural style which is uniquely Filipino and quite different from what was seen in Europe or other Spanish Colonies at that time [2]. Hundreds of colonial churches were built during this period. Some have recently been destroyed by natural calamities, like earthquakes [3] and typhoons [4], others are in a state of neglect or decay, but most are now being preserved, albeit the conservation method applied may not be compatible with the original materials.

Studies on historical lime mortars in the Philippines are very limited. Eusebio tested the presence of egg whites on mortar samples using a simple qualitative Biuret test but yielded negative results [5]. Jose, Jr. presents an old parish account of records (*Libros de Cargo y Data*) that mentions the purchase of duck eggs for renovation work on the churches of Bacoor and Imus in Cavite, which was presumed to be one of the ingredients in making mortar [6]. In another study, Jose, Jr. also mentions the use of molasses, honey and chopped leaves from local trees, which were added to the mortar mixture for their supposedly binding and water proofing characteristics [7]. There was

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no detailed information on the exact amount or ratio of these organic additives included in the mortar mixture. Moreover, the absence of a specific set of standards, partly due to variations in the actual natural building materials available on-site and also in the local building skills, probably gave rise to different mineral and chemical compositions which affected the properties of the mortar. Studies done by Cayme *et al.* concluded that historical brick samples from various locations in the Philippines indeed have different chemical compositions [8, 9]. This lack of proper documentation on the exact methodology in making old building materials has resulted in the inappropriate use of modern day materials in preservation work. For historical lime mortars, cement is commonly added to facilitate the hardening of the mixture.

Mortars (*argamasa*) are used to hold the brick and stone together in old church constructions. They are made by kneading a mixture of lime, sand, water and organic additives. Since the Philippines is composed of many islands, the common source of lime would be crushed and powdered seashells (oysters) or corals. This would be burned constantly in a lime kiln by putting it between layers of firewood. The clump-like product was then moistened into a paste and stored until usage. It would eventually be mixed with the other ingredients of the mortar [10].

This study describes the composition of a lime mortar sample attached to a loose brick fragment from the ruins of an old church from the Spanish Colonial period in Manila, Philippines. The absence of a systematic and detailed baseline data on historical lime mortar composition in the Philippines makes this study innovative and very important in future conservation work, as it will ensure that mortar replacements are more compatible with the original material. The sample in this study was sieved in order to determine the distribution of aggregate (stones, rocks or shell fragments) and binder (lime). The amount of calcium, magnesium and iron were quantified using atomic absorption spectroscopy (AAS). These elements are very important in determining the durability of the mortar sample. The total elemental composition and morphology were obtained from scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX) techniques. Infrared spectroscopy (FTIR) was used to determine the bulk composition, while thermogravimetric analysis (TGA) provided information on the nature of the calcium carbonate binder in the mortar sample.

2. Materials and methods

2.1. Lime mortar sample

The mortar sample collected in October 2013 was attached to a loose brick fragment found within the ruins of an old 19th century Roman Catholic Church in Manila, Philippines (Figure 1). Upon careful examination, the mortar contains small shell fragments and stones of various sizes and irregular shapes. Based on its physical characteristics, the sample was made entirely of lime and not of modern-day cement which has a more massive texture. The inner portion of the mortar was gently scraped-off at an approximate depth of 0.5-1.5 cm from the surface, without being contaminated by the brick fragment. A total of 28.3 g of mortar sample was obtained and the remaining fragments were returned to the site. Sampling was accomplished ensuring that enough minimal sample amounts were taken from the collection site to perform the detailed analysis in this study.



Figure 1. Historical lime mortar samples from the ruins of a 19th century church.

2.2. Sieve analysis

About 16.0 g of mortar sample were crushed very lightly into small pieces and carefully disaggregated in a ceramic mortar with a wooden pestle. Since shell fragments mixed in the mortar are generally quite hard, they will not be pulverized during this process. The resulting material was sieved through a USA Standard Testing Sieve ASTM E-11 Specification, with sizes of 4.75, 2.36, 1.18, 0.600, 0.425, 0.250, 0.150 and 0.075 mm. Sieves were shaken using a mechanical shaker (US Tyler brand) for about 5 min to provide complete separation of the crushed samples. Each fraction was labelled according to the size of the respective sieve where it was retained. The fraction retained on the “pan”, <0.075 mm, was also included in the analysis.

2.3. Digestion with hydrochloric acid solution

The various sieved fractions were heated for 24 hrs in an oven at 110°C and weighed separately. Each fraction was then placed in separate 100mL Erlenmeyer flasks with 4mL distilled water and 30 mL 2M hydrochloric acid solution. Each mixture was stirred at room temperature (29°C) for 5 min, followed by heating in a boiling water bath for another 15 min and was filtered immediately [11]. The filtrate was transferred to a 100 mL volumetric flask and diluted to the mark with distilled water. These digested samples were subjected to atomic absorption spectroscopy (AAS) measurements.

2.4. Analysis by atomic absorption spectroscopy (AAS)

2.4.1. Preparation of standard solutions

Calibration curves were constructed from a series of standard solutions of known concentrations of calcium (ranging from 0.30 to 1.3 ppm), magnesium (ranging from 0.05 to 1.0 ppm) and iron (ranging from 0.10 to 8 ppm), prepared from 1000 ppm stock solutions (Fluka brand, analytical standard grade). The concentration ranges were selected based on the ideal straight line plot in agreement with Beer's Law. Since calcium and magnesium atoms are prone to chemical interferences, a 5.0% w/v $\text{Sr}(\text{NO}_3)_2$ was

added to each standard solution as a chemical suppressant. The blank solution used throughout the experiment is distilled water. AAS absorbance measurements were performed in triplicate [8, 9]. The detection wavelength for the target elements are: 423 nm for calcium, 285 nm for magnesium and 248 nm for iron, respectively. The absorbance was recorded in a Shimadzu AA-6300 Atomic Absorption Spectrophotometer in an air-acetylene gas mixture.

2.4.2. Preparation of unknown mortar fraction solutions

The amount of calcium in the mortar sample was determined initially by mixing together a 0.20 mL aliquot of the digested stock solution of each sieved fraction with 0.50 mL of 5.0% $\text{Sr}(\text{NO}_3)_2$ in different 100 mL volumetric flasks. Since the concentration did not fit within the standard curve range, the solutions were further diluted by pipetting 20 mL aliquot portions into another 100mL volumetric flask. The absorbance reading was measured in triplicate at 423 nm. To determine the amount of magnesium in the sample, a 1.5 mL aliquot portion of each sieved mortar fraction was added with 3.5 mL 5.0% $\text{Sr}(\text{NO}_3)_2$ and diluted to 100 mL in volumetric flasks with distilled water. The absorbance reading was again measured in triplicate at 285 nm. Lastly, the amount of iron was determined by getting a 1.0 mL aliquot portion from each sieved mortar sample solution which was then diluted with distilled water in individual 100 mL volumetric flasks. The absorbance was measured at 248 nm and was done in triplicate.

2.5. Analysis by infrared spectroscopy (FTIR)

An unsieved portion of the mortar sample of about 1 to 2%, relative to the KBr amount, was mixed and ground together with the KBr powder. Precautions were taken to ensure that the KBr powder would absorb minimum moisture from the surroundings. The spectrum was recorded on a Thermo Scientific Nicolet 6700 FT-IR Spectrophotometer and acquired over the frequency range of 4000-400 cm^{-1} using a resolution of 4 cm^{-1} with 32 scans per spectrum.

2.6. Analysis by scanning electron microscopy with energy-dispersive x-ray spectroscopy (SEM-EDX)

The sample was prepared by grinding an unsieved dry portion of mortar and dispersed on double-sided conductive tape attached to an aluminium stub. The sample was coated with gold to make it more conductive. The SEM profile was recorded with a SEM/EDX JEOL JSM-5310 scanning microscope combined with an Oxford Link Isis in the spot profile mode. Observations were made in the back-scattered electron mode. The elemental composition of the SEM sampling region was provided by the EDX profile which was taken at 62 eV system resolution.

2.7. Analysis by thermogravimetric analyzer (TGA)

A 6.50 mg mass of an unsieved mortar sample was heated from a temperature range of 25 to 900°C, at a ramping heating rate of 10°C/minute in dynamic air. The percentage by weight loss of the sample was plotted relative to the change in temperature. The thermograph was recorded in a Mettler Toledo TGA.

3. Results and discussion

3.1. Distribution of particles in the mortar

Sieve analysis was employed to obtain the particle-size distribution present in the historical mortar sample. The size of the soil particles in the mortar can influence its adhesion to bricks and other masonry characteristics, such as compressibility, shear strength and hydraulic conductivity [12]. Figure 2 shows the percentage by weight of each retained fraction in the sieve. Shell fragments are apparent from the sieve sizes of 4.750 mm and 2.360 mm as shown in Figure 3. Most of the weight retained from the 4.750 mm sieve is composed of shell fragments rather than gravel, hence digesting the entire mortar with acid to obtain the binder / aggregate ratio was not feasible [13]. The distribution of weight is highest at around 0.600 mm followed by 1.180 mm sieved sizes, which means that the sample is mainly composed of coarse to medium sand aggregates. The fraction retained in the pan (<0.075 mm) is associated with the binder and is mainly CaCO_3 [14].

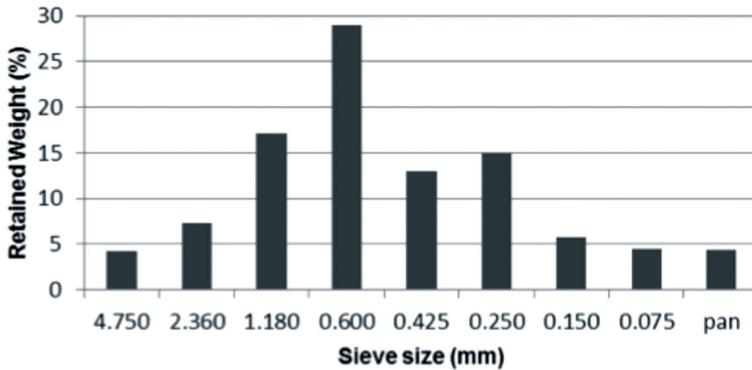


Figure 2. Weight distribution in percentage of the various sieved fractions in the historical mortar sample.

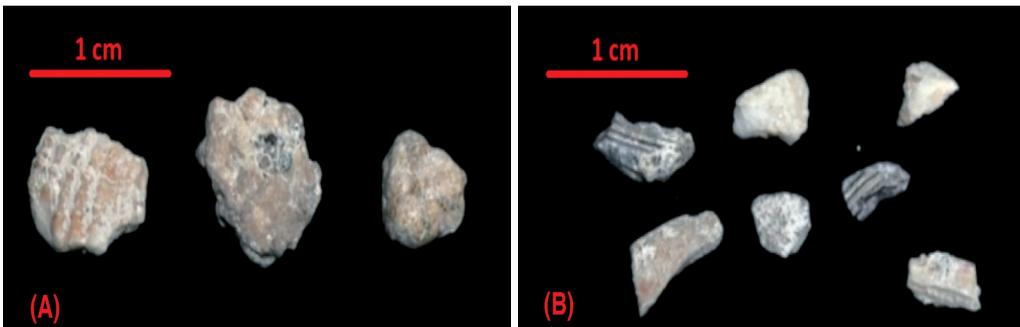


Figure 3. (A) Shell fragments retained from the 4.750 mm sieve; (B) shell fragments with other minerals retained from the 2.360 mm sieve.

The graph in Figure 4 is a logarithmic plot showing the cumulative percentage of sample passing through a certain mesh size. This percentage is called percent finer (% finer). The value for D_{10} is 0.17 mm, D_{30} is 0.43 mm, and D_{60} is 0.90 mm, respectively, and were obtained by extrapolation from the plot. These are the values for the grain size diameters (D) that correspond to 10%, 30% and 60% of the sample passing through the sieve by weight, respectively. From these parameters, the coefficient of uniformity (C_u) was computed to be 5.29 and the coefficient of curvature (C_c) was equal to 1.21. Since the value for C_u is greater than 4 and C_c has a value between 1 and 3, the mortar sample is considered to be well-graded. This means that the particle sizes are well represented over a wide range [15].

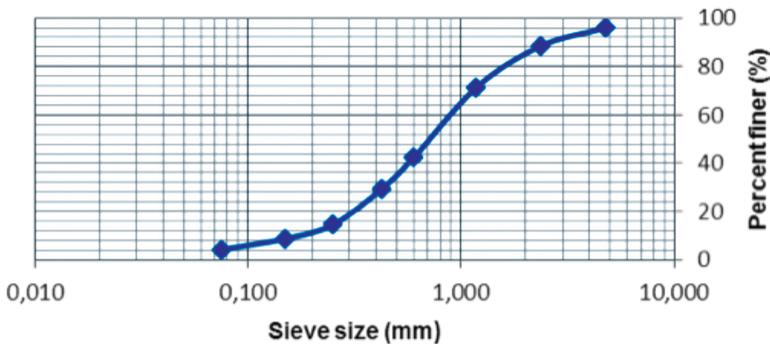


Figure 4. Grading curve distribution of the mortar sample.

3.2. Acid digestion and atomic absorption spectroscopy (AAS) analysis

The sieved fractions were digested separately with hydrochloric acid to find the distribution of calcium, magnesium and iron in the mortar sample. Since mortars are mainly composed of a mixture of lime and sand, the acid soluble fractions are considered part of the binder, while the acid insoluble residues are generally the aggregates. Table 1 shows that the amount of calcium is higher compared to magnesium and iron. This is expected since the binder is made of lime and would contain high amounts of calcium carbonate. The fractions at 4.750 mm and 2.360 mm have about 10 to 15% calcium which is due to the shell fragments observed during the sieving analysis. The sieved sizes at 0.075 mm and the fraction retained on the pan (<0.075 mm), respectively, have the highest percentage of calcium (about 18 to 26%), thus inferring that CaCO_3 is more concentrated in the smaller sized fractions. The presence of about 4 - 9% calcium in the sieve sizes ranging from 0.150 mm to 1.180 mm, respectively, implies that the lime is uniformly distributed within the different sieved fractions.

Results also show that magnesium is only present in trace amounts (< 0.480%), thus the lime content in the mortar is considered as being non-dolomitic [16]. The amount of iron is attributed to clay mineral impurities that are present in the aggregates. This explains why a higher percentage of iron (about 1.20 to 2.25%) was observed in the sieve fractions containing the sand aggregates (0.075 mm to 1.180 mm). It is also possible that small pieces of broken bricks may have been used as additives and contributed to the increased amount of iron. Moreover, another possible source

of iron impurities may have originated from the brick kilns, or “*horno*”, doubling as lime kilns in burning seashells to produce lime [17].

Table 1. Summary of the Atomic Absorption Spectroscopy (AAS) data for calcium, magnesium and iron content.

Sieve Size (mm)	Mass of Mortar Sample (g)	Average % Composition		
		Ca	Mg	Fe
4.750	0.6174	10.852	0.056	1.059
2.360	1.0718	15.080	0.146	0.065
1.180	2.5012	5.646	0.258	1.270
0.600	4.2310	4.390	0.223	1.348
0.425	1.8975	5.584	0.475	2.106
0.250	2.1765	5.380	0.440	2.235
0.150	0.8467	8.790	0.471	1.373
0.075	0.6475	18.944	0.211	1.606
< 0.075 (pan)	0.6268	26.082	0.144	0.818

3.3. Infrared (IR) spectroscopy characterization

IR absorption peaks (Figure 5) corresponding to inorganic minerals and possible protein content in the mortar sample are evident from the spectrum. Characteristic absorption peaks for the binder components, mainly containing the mineral calcite which is composed of CaCO_3 , are seen from the intense peaks for the C-O in-plane bending (ν_4) at 712 cm^{-1} and C-O out-of-plane bending (ν_2) at 877 cm^{-1} . Since only few organic compounds absorb strongly at these wavenumbers, the sharp peaks confirm the identification of carbonates in the sample spectrum. Furthermore, the broad peaks for the C-O asymmetric stretching (ν_3) at 1425 cm^{-1} , 1797 cm^{-1} , and 2514 cm^{-1} , respectively, are also attributed to CaCO_3 . Since sand is a major component of mortars, absorption peaks for the mineral quartz should be present. These were identified from the spectrum, based on the Si-O stretching peaks at 652 cm^{-1} , 786 cm^{-1} and 1060 cm^{-1} , respectively. Moreover, the AAS data for the different sieved fractions showed significant amounts of iron in the mortar sample, which was complemented by the presence of the Fe-O bending peak for the mineral hematite at 536 cm^{-1} [8, 9]. Overlapping absorption peaks cause difficulty in identifying the other mineral groups, which is expected because the raw materials used in making mortars contain different inorganic minerals [18].

The possible presence of proteins in the mortar sample was established by the characteristic peaks for the amide group which includes the broad bands centered at 3439 cm^{-1} , assigned to the N-H stretching vibration, and at 1627 cm^{-1} for the C=O stretching vibration, respectively [19]. Since proteins, usually in the form of eggs, are added in low concentrations, it is likely that peaks from inorganic components of the sample may interfere with its detection [5]. Further qualitative tests for proteins should be performed to confirm these assumptions.

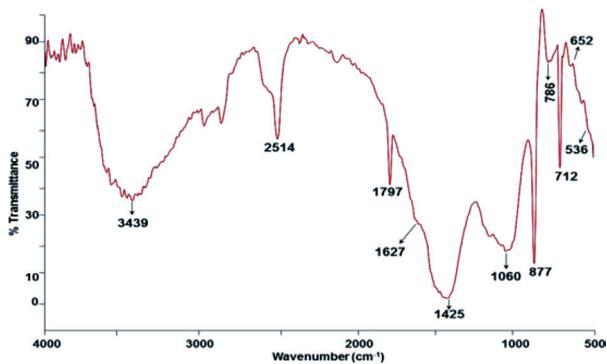


Figure 5. Spectrum of the mid-infrared region (4000-400 cm^{-1}) of the mortar sample.

3.4. Microstructure of mortar using scanning electron microscopy and energy-dispersive x-ray spectroscopy (SEM-EDX)

SEM provides information on the size, texture and distribution of aggregates and binder components in the mortar sample. Analysis of magnified images in Figures 6A and 6B shows that the mortar has a compact microstructure with no major pores. Aggregates, mainly in the form of quartz, were observed to be well-embedded in the sample matrix (Figure 6A). The presence of quartz was also confirmed by the EDX

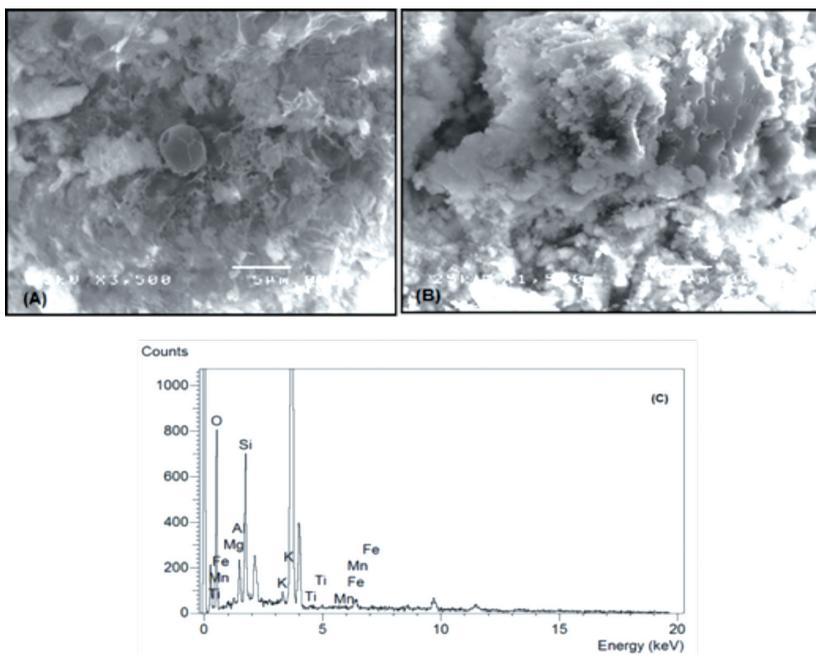


Figure 6. SEM image of the mortar sample magnified (a) 3,500x, and (b) 1,500x. The resulting EDX spectrum of these images is shown in (c).

spectrum (Figure 6C) which provided an atomic percentage for Si at 2.95% and the fourth most abundant element relative to the SEM image. The crystal structure magnified at 1,500x (Figure 6B), gives details on the mineral calcite which forms part of the binder [20, 21]. This was supported by the EDX spectrum in Figure 6C as having a high atomic percentage for Ca at 14.82%. Hence, the mortar sample is essentially composed of lime and is calcium-rich. The EDX data supports the results of the AAS in that magnesium is indeed present in low amounts (0.16%) implying that the lime is non-dolomitic. Table 2 summarizes the percentage composition of different elements from the EDX spectrum.

Table 2. Summary of elemental composition using EDX.

ELEMENT	ATOMIC %
O	58.72
C	21.81
Ca	14.82
Si	2.95
Al	0.89
Fe	0.37
K	0.21
Mg	0.16
Ti	0.04*
Mn	0.04*
Total	100.00

*trace amounts

3.5. Thermal analysis

Information on the binder composition was determined by measuring the weight loss of the mortar sample as the temperature was increased to 900°C. A continuous weight loss was observed from the thermograph (Figure 7) which became significant between 790°C and about 850°C. This non-linear drop in the sample's mass is a characteristic peak for the decomposition of calcium carbonate by the release of CO₂. Weight loss between 200°C and 600°C is due to the evaporation of water chemically bound to hydraulic compounds, while the decrease in weight above 600°C is attributed to the loss of CO₂ [22, 23]. Furthermore, the binder composition was classified based on the ratio of CO₂/H₂O from the thermograph. The 1st derivative plot shows that the weight loss of the sample was highest at 2.73% for the hydraulic H₂O temperature range (200-600 °C) and the maximum weight lost for the CO₂ temperature range (790-850 °C) was at 24.97%. Thus, the CO₂/H₂O ratio is equal to 9.15. This implies that the mortar sample shows binder characteristics for a typical air-lime with a slightly hydraulic character. The hydraulic nature of the sample was supported by the significant amount of iron found on the sand aggregates as recorded by the AAS. Air-lime mortar slowly hardens in air by reacting with CO₂ in the atmosphere through time. The hydraulic character may be attributed to clay impurities (i.e. iron) during the lime burning process, effectively making the binder carbonate faster [24].

Moreover, the TGA data gives consistent results with the AAS and EDX in which no significant amount of magnesite or dolomite decomposition was detected in the binder. This is due to the absence of major weight loss between 400°C and 650°C [25].

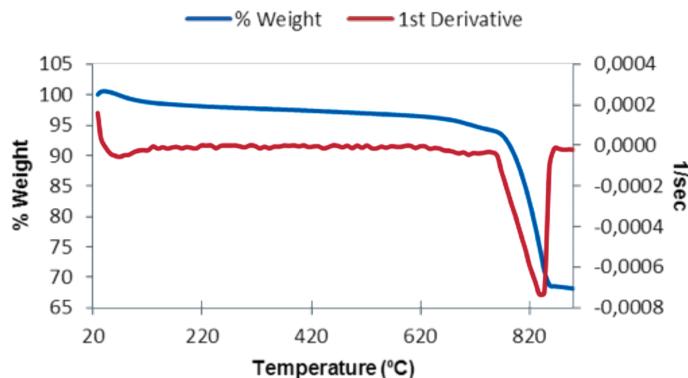


Figure 7. Thermograph of the mortar sample showing loss of weight as temperature is increased.

4. Conclusion

This paper presents different analytical techniques that effectively characterize the chemical composition of historical lime mortars in the Philippines and will serve as useful baseline information for renovation work to be employed for structures built within the Spanish-Colonial Period. The mortar material is calcitic and well-graded, with shell fragments used as aggregates. This implies that the source of the raw material may have come from the vicinity of the seashore, since the sampling site is near Manila Bay. Sand with silicates, quartz and hematite minerals were observed from the IR and SEM-EDX analysis and provided a basic mineralogical description of the sample. Organic protein compounds may have also been added to the mortar mixture. This will be explored further using protein extraction techniques. Furthermore, the binder has a typical air-lime characteristic with a slightly hydraulic property which allowed for effective carbonation. Signs of Portland cement were not observed on the sample; hence it is regarded as original and not affected by any modern restoration.

Acknowledgements

The authors would like to express their deepest gratitude for the generous support given by the Chemistry Department of the De La Salle University, Manila, Philippines. The helpful insights and valuable assistance of the following persons from the Chemistry Department are greatly appreciated: Dr. Glenn Alea, Mr. Michael Ajero, Mr. Irving Chiong and Dr. Maria Carmen Tan.

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