# Physical and chemical characterization of historical masonry materials from Yucatan churches, Mexico

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ABSTRACT: Yucatan churches are an important built heritage in Mexico from sixteenth to eighteenth centuries. They are massive structures, where loadbearing stone masonry walls and vaults represent the main structural elements. This paper presents the results of the experimental campaign carried out on stone units and mortars selected from six partially collapsed churches. The stone samples were collected from walls and vaults. Additionally, mortars samples were collected from joints and finishing mortars. The physical characterization was performed according to European standards and key physical properties were obtained, namely density ( $\rho b$ ), porosity (Po) and water absorption by immersion (Ab). Reasonable correlation between variables was achieved. The chemical characterization was carried out through X-ray diffraction test (XRD). A chemical composition of calcium carbonates showed almost all stones samples, only two stone samples show secondary compound as Magnesian and Ankerite. The mortars show a chemical composition mainly of calcium carbonates and few cases show additional traces of clays.

### **1** INTRODUCTION

In Mexico there are several buildings with historical value due to their architecture and constructive systems and materials describing the different stages of evolution of Mexican civilians.

Yucatan is a State in the south of Mexico with an extension of 3400 Km<sup>2</sup> approximately, where in the sixteenth century, the knowledge of the Mayan culture and the European continent were combined to give place to the Architectural evolution of the region. At this time, palaces, churches, convents, big houses, among others were built and the Mayan constructions were destroyed or forgotten. The religion started to be relevant and with it, the construction of churches, convents and similar was a priority.

Nowadays, Yucatan churches are an important built heritage in Mexico. They are massive structures, where loadbearing stone masonry walls and vaults represent the main structural elements. Both stone masonry walls and vaults were built with limestone and lime mortar. Nevertheless, regarding to limestone, six different limestones were identified in Yucatan and despite some papers in the literature, which present data on the physical, petrographic, and mineralogical properties (Alonzo & Espinosa, 2003; Alonzo & Espinoza, 1987; Carrillo, 1991; Estrada-Medina, Valdez, Zanatta, & Casolco, 2008; May-crespo et al., 2012), thermal effects (González-Gómez et al., 2015), environmental degradation (Maldonado, Veleva, & Díaz-Ballote, 2011) of limestone from Yucatan region, there is no document that specifies the typology of limestone used in Yucatan historic churches. In relation to historical mortars, several researches were developed on heritage religious buildings (from 14th to 17th century) around the world such as Portugal, Italy, Belgium and Spain (Balen & Hendrickx, 2008; Binda, Modena, Baronio, & Abbaneo, 1997; Calderini, Abbati, Cotič, Kržan, & Bosiljkov, 2015; Garmendia, Larrinaga, San-Mateos, & San-José, 2015; Magalhães & Veiga, 2009; Veiga, 2015). These works point out important data such binder: aggregate ratios from 1:1,5 to 1:5,5 (in mass). Few studies carried out on Mexican historical structures point out binder: aggregate ratios of 1:2.5 and 1:3 (volume) (Chávez & Meli, 2008; Chávez, Sánchez-Ramírez, & Meli, 2012). However, there are no quantitative evaluation of the binder: aggregate ratios of mortar from Yucatan historical constructions. It is important to note that the ratios in volume are different of ratios in mass, because, for example, volume ratios from 1:1 to 1:4 correspond to mass ratios of about 1:3 to 1:12 in mass (Veiga, 2015), as it depends on the density of the aggregate.

Presently, a research program has been developed to assess the structural condition of the most typical Yucatan historic churches. As a first step, it is very important to characterize the materials used in the construction of the churches so that compatible mortar can be used in the future in the experimental characterization of stone masonry, which can be representative of existing one. Therefore, an enlarged experimental characterization of the historical raw materials (limestone and lime mortar) has been defined.

This paper aims at presenting the results of the experimental campaign carried out on stone units and mortars selected from 6 historic churches from Yucatan region. In situ investigations were performed in six partially collapsed churches from sixteenth to eighteenth centuries. Stone samples were collected from walls and vaults. Additionally, mortar samples were collected from joints and finishing mortars. The physical characterization was performed in 111 cylindrical stone specimens according to European standards. The chemical characterization of stones and mortars was carried out thought X-ray diffraction test (XRD). This test was performed in both natural and decarbonated samples. The location of materials, possibilities of extraction, nature of the samples (historic samples) and all equipment's necessaries to carried out all tests in the characterization of the material represented a challenge in the development of this research.

# 2 METHODOLOGY

The methodology followed for the experimental characterization of buildings materials was organized in three main stages: (1) delimitation of influence area and selection of case of studies, from which it was intended to extract the building materials, (2) collection of material samples and preparation of test specimens; (3) definition of the experimental program and materials characterization.

# 2.1 Selection of case of studies and influence area.

The constructive typology selected in this research have an influence area of 146,120 Km<sup>2</sup> approximately, which is the area of Yucatan peninsula, located in South of Mexico. The selected churches for the in situ investigation are located in the Yucatan State, which has an extension of 38,400 Km<sup>2</sup>, see Figure 1.



Figure 1. Location and area of influence

Kikil, Timizin (Kik)
 Chancenote, tizimin (Cha)
 Tixhualactun, Valladolid (Tix)
 Ichmul, Chikindzonot (Ich)
 Petulillo, Peto (Pet)

6-Sisbic, Tixmehuac (Sis)



Figure 2. Distribution of selected churches as case studies.

The case studies selected to carry out the in-situ investigations are six partially collapsed churches from sixteenth to eighteenth centuries. The churches are distributed in the influence area, as it is shown in Figure 2. The selected churches have a constructive typology based on stone masonry in walls and vaults. The vaults are mostly barrel vaults and from in-situ inspection it was seen that the walls have three layers. In both walls and vaults the main materials are the limestone for masonry units, being linked with lime mortar.

# 2.2 Collection of historical samples and preparation of test specimens

It is important to note that the samples were collected from nationally protected historic buildings. Therefore, the collection of samples was supervised by the National Institute of Anthropology and History of Mexico (INAH it is the main representant of the Federal government for the heritage protection) and Commission of Ecclesiastical Properties and Sacred Art of the Archdiocese of Yucatan. Hence, only the samples allowed by both organisms were collected.

The guidelines for the samples collection were: (1) respect for the historical value of the monument, (2) selection of dismounted stones from the structure and do not remove from their original position in any case, (3) do not select stones with carvings, paintings or drawings, (4) the mortar samples must be collected with the minimum possible damage to the building.

Taking these guidelines into account, the stone samples were collected from walls and vaults. Additionally, mortar samples were collected from joints and rendering of the walls. The collected stone samples were from 12 walls, 4 vaults and the mortar was collected from 7 mortar joints and 6 rendering mortars.

All samples were identified with a code based on numbers and letters. The mortar samples were conducted at laboratory as they were collected. It was necessary to collect at least 30 gr of material in order to be used in the chemical characterization.

In order to carry out the physical characterization, the stone samples were cored according to standard ASTM D4543 (ASTM International, 2001). From 16 samples of masonry stone units collected in situ, 111 cylindrical stone specimens were extracted with three different diameters 5.715 cm, 5.08 cm and 2.54 cm. According to the Standard previously mentioned, the specimen shall have a length/diameter ratio of 2.0 to 2.5 and a diameter of not less than 47 mm. Nevertheless, the experimental conditions are never ideal and therefore, the specimens were cut in height to diameter ratios of 1:1 and 1:2. The diameters were measured three times and the average were used.

# 2.3 Experimental program

To determine the physical and chemical characteristics of the masonry materials, an experimental program was designed to be carried out at the laboratory. This program was structured in two stages, by considering the different materials: (1) mortars and (2) stones. In the mortar samples, chemical characterization was already performed. In the stone specimens, physical and chemical characterization was concluded. These results are part of the experimental results intended to be obtained from a wider ongoing physical and mechanical characterization.

# 2.3.1 Mortars

The chemical characterization of the mortars was carried out by X-ray diffraction technique in two type of samples, namely natural and decarbonated. The decarbonated samples were obtained

in order to identify other than carbonate components such as clay minerals in each sample. X-ray diffraction (XRD) test were performed in a Siemens D-5000 diffractometer. The conditions of the test were the following: (1) for natural samples; D-8 Advance diffractometer, range from 5 to 50 ° 2theta, time of step 0.5 seconds, step size 0.02 degrees and 40 KV 30 MA, copper tube wavelength 1.5818 Angstroms; (2) for the decarbonated samples the test conditions were: D-8 Advance diffractometer, range of 6 to 60 ° 2theta, step time 1 second, step size 0.02 degrees and 40 KV 30 MA, copper tube wavelength 1.5818 Angstroms. The decarbonated samples were run at a longer passage time because after the decarbonization have less sample volume.

The natural samples are the samples that came directly from the case studies and that were submitted to any chemical process before to introduce them at X-ray diffraction machine. From its dry and solids state, approximately 30 gr of natural samples were grounded with a ceramic mortar until to get a fine powder. After that, the powder was grounded again in a recipient from Agate mortar until obtaining a powder with the approximate fineness of talcum or gypsum. Finally, the sample was put it on the sample holder to insert it in the X-ray diffraction machine.

The decarbonated samples were subjected to dissolution of carbonates (CO3) with HCl 5% v/v. In this research, hydrochloric acid to 5% of concentration was used to decarbonate the samples. In the preparation, it was used hydrochloric acid ACS from Fermont company with a concentration of 37% in distilled water with 18 Mohm-cm.

This process was followed in case of the 13 mortar samples in order to carry out the X-ray diffraction test. Finally, 26 X-ray diffraction tests were performed.

#### 2.3.2 Stones

The physical characterization of limestone was performed in 111 stone cylinders according to EN 1936:2006 (European committee for Standadization, 2006) and NP-EN-13755:2008 (Norma Europeia, 2008). The key physical properties obtained were density ( $\rho_b$ ), open porosity (Po) and water absorption by immersion (Ab), according to equations 1 to 3 (Table 1).

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Equations	Symbology
$\mathbf{a}_{l}[\frac{Kg}{m_{d}}] = \frac{m_{d}}{m_{d}} \mathbf{x} \mathbf{a}_{l}$	Mass of the dry specimen
$m_s^{\mu}m_s^{3} = m_s - m_h^{\mu}m_s^{\mu} \cdots [1]$	Mass of the saturated specimen
$m_s = m_s - m_d \sim 100$ m <sub>h</sub>	Mass of the specimen immersed in water
$m_s - m_h \wedge 100 \dots [2]$ Ab	Absorption of water at atmospheric pressure
$Ms - Md$ $\rho_b$	Apparent density
$Ab[\%] = \frac{1}{Md} x100$ [3] Po	Open porosity
$ ho_{ m rh}$	Density of water at 20 $^{\circ}$ C is 998 Kg/m <sup>3</sup>

The chemical characterization was conducted in the natural and decarbonated samples similarly to what was described to mortars. About 11 stone samples were tested in both natural and decarbonated forms, meaning that 22 X-ray diffraction tests were carried out.

# 3 RESULTS AND DISCUSSION

### 3.1 Mortars

As previously mentioned, the mortars were collected from renders and joints of the masonry walls. In the terminology of mortar samples, AR means mortar and the last words relate to the local from which they were collected (Figure 2). In the process of preparation, different colours were identified in the samples. The most frequent was the yellowish, but whitish, light brown, dark brown and reddish were other colours observed in the samples. The render mortars showed only white (AR1SIS, AR8ICH, AR12TIX, AR8KIK) and yellow (AR4PE, AR11CHA) colours. While the joint mortars showed yellow (AR5PE, AR10CHA, AR6ICH, and AR13KIK), brown (light AR2SIS and dark AR3SIS) and red (AR9KIK) colours. In order to identify the mineral

phases in each mortar sample, X-ray diffractogram analysis was carried out. The results were divided in render and joint mortar results. Figure 3 shows the main diffractograms obtained in rendering and joint mortars during the analysis.

### 3.1.1 Render mortars

The X-Ray obtained in natural samples of render mortars showed only the presence of calcite and ankerite. Both compounds are carbonates. The calcite is a calcium carbonate (CaCO3) recorded in the six render mortars (AR1SIS, AR8ICH, AR12TIX, AR8KIK, AR4PE, AR11CHA) and Ankerite calcium carbonate with some traces of manganese and is iron (Ca(Mg0.67Fe0.33+2)(CO3)2) recorded in only one sample (AR8KIK). This means that the samples 22AR8KIK have both calcite and ankerite in percentages of 56.8% and 43.2% respectively.

As the predominant crystalline phase is carbonate in almost 100% of all render mortar analysed, the samples were decarbonized and further analysed aiming at identifying the mineral clay phases. From the decarbonate samples, the studies show the presence of clays such as kaolinite and mont-morillonite calcian in all cases of white finishing mortars. Additionally, 3 of 4 mortars in white colour show the presence of quartz (AR12TIX do not show). Also, minerals such as stilbite and phyllosilicates with micas such as Muscovite (only in AR12TIX) and Vermiculite (only in AR8KIK) were recorded as unique cases in white render mortars.

There were record only two render mortars in yellow colour (AR4PE, AR11CHA). From these, AR4PE sample show the presence of clay minerals as montmorillonite 2-0009, Kaolinite-montmorillonite and saponite. Sample AR11CHA presents kaolinite, kaolinite, palygorskite and halloysite.



Figure 3. Examples of typical diffractogram from natural mortar samples. (a) render mortars, (b) joint mortars. C=Calcite, A=Ankerite, H=Hydrotalcite, G=Gypsum, K=Kaolinite, Ar=Aragonite, Q=Quartz, Cl=Clinochlore, V=Vaterite.

#### 3.1.2 Joint mortars

In the natural samples of joint mortars, the X-ray diffraction shows a composition based on carbonates in almost all samples, meaning that the predominant crystalline phase in joint mortars is calcite (CaCO3), with very high concentrations (almost 100%). Only the mortar identified with a reddish colour (AR9KIK) recorded minerals of clays and phyllosilicates, as well as quartz and gypsum. Additionally, regardless to the colour of mortar, all samples recorded kaolinite.

Nevertheless, two of four mortars identified in yellow colour (AR6ICH and AR13KIK) and those identified in brown colour (both light and dark), showed the presence of tosudite, montmorillonite calcian and quartz. The difference in colour between them can be attributed to presence of calcite in yellow samples, and of rutile (TiO2) in brown coloured samples. Rutile (TiO2) is a mineral found in nature in red blood, bluish, brownish yellow, reddish brown or violet colours.

The AR6ICH sample also recorded traces of Rutile mineral (dark colours) in addition to the calcite (colourless or white, grey, yellow or greenish colours). Nevertheless, its colour is yellow-ish, so it is probably that the Rutile percentage was low.

Apart from the similarities between joint mortars described above, there are two cases of yellowish joint mortars with specific characteristics. The first corresponds to the sample AR5PE, which only contains clay minerals as montmorillonite 2-0009, palygorskite, ilite and kaolinite. The second mortar of joints with particular characteristics is AR10CHA, which contains only kaolinite and another phyllosilicate named clinochlore. Finally, the particular case of the reddish-coloured mortar (AR9KIK) shows the presence kaolinite and nacrite, as well as the presence of other two phyllosilicates: muscovite and chlorite-vermiculite-montmorillonite.

# 3.2 Stones

# 3.2.1 Physical characterization

The physical properties (specific weight, open porosity and water absorption by immersion) of limestone samples are in Table 2. The volumetric weight ranges from 1447.09 to 3128.76 Kg/m<sup>3</sup>, with a standard deviation of 347.15 Kg/m<sup>3</sup>, meaning that almost 65% of the samples tested are in the range between 1839.35 and 2533.65 Kg/m<sup>3</sup> (Figure 4a). The density shows values from 1459.09 to 2507.70 Kg/m<sup>3</sup> with a standard deviation of 305.81, nevertheless, being the greater frequency of values between 2300 and 2500 Kg/m<sup>3</sup> (see Figure 4b). The Porosity has values ranging from 2.46 to 41.56%, but as it was expected, the lower values are more frequent (see Figure 4c). Finally, the water absorption shows values from 1.07% to 27.41% with a greater frequency until 4%, see Figure 4d.

Table 2.	Physical	properties of	f stone	samples
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	Volumetric weightpb		Ро	Ab
	Kg/m3	[Kg/m3]	[%]	[%]
Minimum	1447.09	1459.09	2.46	1.07
Maximum	3128.76	2507.70	41.56	27.41
Mean	2186.50	2166.89	13.87	7.39
Standard deviation	347.15	305.81	11.55	7.43
Coefficient of variation15.88%		14.11%	83.31%	100.49%



Figure 4. Histograms under normal distribution of physical properties of stones samples. a) volumetric weight, b) density, c) porosity and d) absorption

Additionally, the relationships between porosity/absorption, porosity/density, density/absorption were analysed. A simple regression analysis was carried out and it was observed a good correla-

tion ratio between the variables, achieving coefficients of variation ( $R^2$ ) greater than 0.9, see Figure 5. It is important to note that the best trendlines accuracy was a polynomial curve of second order in the three graphics.

From the scatter diagrams of Figure 5, it is possible to divide the results in three groups of data, see Figure 6. Box plots were also used to group and analyse the results of all cylindrical specimens of the same stone sample (Figure 7). The result was a database with the information of the sixteen stones sample. This process was carried out for all variables, namely density, porosity and water absorption (see Figure 7). Group 1 includes P8Kik and P10Kik stone samples. Group 2 includes P2sis, P3Pe, P7Ich, P11Kin and P12Cha stone samples. Group 3 is composed of P1Sis, P4Pe, P6Ich, P9Kik, P13Cha, P14Tix, P15Tix, P16Tix and P17Tix stone samples.



Figure 5. Simple regression analysis. a) porosity vs density, c) density vs absorption



Figure 6. Values of density, porosity and water absorption in groups of data



Figure 7. Box plot for each variable. (a) density, (b) porosity and (c) absorption

For each group, the values of density ( $\rho_b$ ), porosity (Po) and water absorption (Ab) are shown in Figure 6 and Figure 7. The data shows from low to high dispersion, variable skewness in the data and only one outlier value, corresponding to the result obtained in specimen P9Kik-7V. As it is seen, group 1 has the lowest density but the highest porosity and water absorption and group 3 has the highest density and lower porosity and absorption. Additionally, it was found an overlap of ranges, between the group 2 and 3. In case of density ( $\rho_b$ ), the group 2 has an upper extreme of 2323.48[Kg/m<sup>3</sup>] and the group 3 has a lower extreme of 2263.61[Kg/m<sup>3</sup>]. In case of porosity (Po), group 2 has a lower extreme of 10.45[%] and the group 3 has an upper extreme of 14.94 [%].

Regarding to absorption (*Ab*), group 2 has a lower extreme of 4.49[%] and group 3 has an upper extreme of 6.59 [%], see Figure 6.

Therefore, from the physical characteristics review, it is possible to observe that in the Yucatan churches under analysis, three different types of limestone were used.

#### 3.2.2 Chemical characterization

The chemical characterization of stone was divided in stones from vaults and stones from walls. It is probably that the same typology of stones had been used in both structural elements, but it was decided to verify it. In order to identify the mineral phases in each stone sample, X-Ray Diffraction (XRD) analysis was carried out. Table 3 shows the detected mineral phases, while Figure 8 shows the three main diffractograms obtained during the analysis of natural samples.



# Table 3. Mineral phases identified in the samples

### 3.2.1.1 Stone vaults

Four stones were collected from the vaults (P15TIX, P9KIK, P11KIK and P14TIX). The results of the X-ray diffraction test carried out in primary samples showed a chemical composition mainly composed of carbonates in all samples. The samples P15TIX and P14TIX recorded almost 100% of calcium carbonates (namely calcite CaCO3). In other 2 samples calcite magnesia (Mg0.03Ca0.97)(CO3) and ankerite (Ca(Mg0.67Fe0.33+2)(CO3)2) were recorded. Nevertheless, the greater percentage of mineral calcite magnesia was recorded in the samples P11KIK with 79.7% and with only 20.3% of ankerite. The sample P9KIK recorded the greater percentage of ankerite (90%) and lower percentage of calcite magnesia (10%).

Additionally, to identify clay minerals, the samples were subjected to dissolution of carbonates with hydrochloric acid. The XRD analysis of the powders after the dissolution shows the presence of clay minerals as kaolinite (Al2Si2O5) and tosudite ((K,Ca)0.8Al6(Si,Al)8O20(OH)10 4H2O) in the four samples, nacrite (H4Al2Si2O9) in three samples (P15TIX, P9KIK and P11KIK) and montmorilonite calcian (Ca0.2(Al,Mg)2Si4O10(OH)2 H2O) was recorded only in the sample P14TIX. The phyllosilicate, Aluminosilicate and aluminium Muscovite ((H,K)AlSiO4) was present in the sample P11KIK and P14TIX. The presence of Gypsum (CaSO4 2H2O) and Quartz (SiO2) was detected only in the sample P9KIK. The salt halite (NaCl) was identified only in the sample P14TIX. In this sample, traces of calcite were identified.

### 3.2.1.2 Stone walls

From the walls, ten samples were selected, namely P4PE, P6ICH, P2SIS, P7ICH, P12CHA, P8KIK, P10KIK, P1SIS, P16TIX and P17TIX. From X-ray diffraction test was performed in natural samples, and it was observed that calcite (calcium carbonate – CaCo3) is present in almost 100% of the stones. Nevertheless, the samples were subjected to dissolution of carbonates to identify the clay minerals. After the dissolution, two samples (P4PE and P6ICH) showed no residues, meaning that its composition is really 100% calcium carbonates (CaCO3). The other samples show presence of clays minerals and quartz. The samples P2SIS, P7ICH, P12CHA, P8KIK, P10KIK, P1SIS, P16TIX and P17TIX present mineral kaolinite (Al2Si2O5(OH)4). Additional clay minerals such as tosudite ((K,Ca)0.8Al6(Si,Al)8O20(OH)10 4H2O) were observed in samples P8KIK, P10KIK, P1SIS, P16TIX and P17TIX; nacrite (H4Al2Si2O9) in sample P16TIX and montmorillonite calcian (Ca0.2(Al,Mg)2Si4O10(OH)2 H2O) in sample P7ICH. Three samples (P1SIS, P16TIX and P17TIX) show presence of muscovite ((H,K)AlSiO4) and others samples as P12CHA, P8KIK and P10KIK have traces of quartz (SiO2). Although the Quartz mineral is not the main component in these samples, it is important to note that it is directly related to some physical properties such as hardness and strength (Özkahraman & Işık, 2003).

In spite of previews investigation mention the presence of kaolinite and tosudite in all samples analysed (González-Gómez et al., 2015), in this research all stones from the vaults are in accordance with it but only five of ten wall stones samples show the simultaneous presence of these two clays.

# 4 CONCLUDING REMARKS

Regarding to the chemical characterization of mortars, it was seen that only three main crystallography phases were identified in natural samples of mortars. The mineral phase of calcite (CaCO3), a rhombohedral structure of calcium carbonate, was the most abundant compound in all lime mortar samples. The ankerite (Ca(Mg0.67Fe0.33+2)(CO3)2), a polymorph of calcium carbonate with iron and magnesium was found in only one sample (AR8KIK). The presence of this mineral in the mortar can be justified if for the aggregates used in the preparation of the mortar, stones with a high content of ankerite, such as the P9KIK sample, were used. Quartz is a common associated mineral with limestone and aggregates from limestones used in the mortars (Özkahraman & Işık, 2003).

It is important to remark that the rendering mortars presented only a yellow colour, whereas joint mortars were yellowish, brown and reddish mortars. No white mortars were found in the joint mortar samples. Even though the red coloration in the mortars is attributed to presence of hematite (González-Gómez et al., 2015; Maldonado et al., 2011), it was found only in one reddish mortar (AR9KIK). On the other hand, ankerite and vermiculite were recorded in mortars with dark colorations. In both stones and mortars, traces of kaulinite and montmorilonite were observed. It is important to stress that clays have the capacity to increase their volume when they are in contact with water. Regarding to mortar, it should be mentioned that additional studies are needed in order to identify the binder to aggregate ratio and possible additives.

Regarding to stone, it was identified three main mineralogical phases in the natural samples. After the carbonate dissolution, it was observed that almost all samples show kaolinite and tosudite clays. Additional minerals were record in some samples but were not representative.

The analysis of physical properties allowed to group the limestones in three groups. In addition, a really good statistical correlations (R>0.9) between physical variables (density, absorption and porosity) were found.

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