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USE OF FTIR AND THERMOGRAVIMETRIC ANALYSIS OF ANCIENT MORTAR FROM THE CHURCH OF THE CROSS IN GERASA (JORDAN) FOR CONSERVATION PURPOSES

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ABSTRACT

The present study intends to assess the performances of two kinds of ancient mortar in the Church of the Holy Cross Gerasa in Jordan. The conservation of ancient construction requires a worthy characterization of the most significant mortar components before starting the conservation, after the results obtained, and during the restoration process of the ancient site to decide how to react. The objective of this work is through FTIR perform experimental contribution to study the influence of organic part on the mechanical behavior of mortar (construction and conservation) and to minimize the risks of low tensile strength. Nowadays it is becoming increasingly important to apply the new techniques for sustainability and compatibility issues to conserve the historical monuments, particularly from increasing deterioration in the mortar's joints of the church. New results obtained concerning the identification of the binding materials of mortars, in particular the proteinaceous material (Egg-White) used in the church, and to collect more information to be used for the compatibility, which will be applied for the first time in Jordan. Furthermore, the results will make a new approach in Jordan to accept it in the field of restoration. It concludes that it is easy to identify all the organic part components of the mortar using the Fourier transformation infrared spectroscopy (FTIR) in the band of the organic material between 1480-1420 cm^{-1}), supported by thermogravimetric analysis (TGA)/ (DTA peak at around 250 °C). The investigations of both methods yielded precise information about mortar production technology and revealed an additive proteinaceous material in the mortars.

KEYWORDS: Mortar, Conservation, Gerasa, Decapolis, Egg-white, Church of Cross, TGA, FTIR

1. INTRODUCTION

The oldest evidence of the use of lime in the construction field came from the Levant during the pre-pottery Neolithic B (PPNB) (7500-6000 B.C.) (Grissom, 2000). In this area, it can be found in plasters that were used to cover walls and floors. Some believe that the development of the proto-pottery (ceramics) (PP), made from burnt lime and ashes, can be traced back to the use of these patches (Mazar, 1992). The Levant is an important archaeological area because of the wealth of prehistoric finds in its territory. A study of early pyrotechnics and lime plaster production in the region concluded that lime mortar and gypsum were used extensively in the Levant and were considered both as a commodity and a symbolic product, as indicated by their use as a modeling agent on skulls and as a sealant at burials (Clarke, 2012). Goren in 2008 found a major development in essential mortar production in south Levant area by using an experimental pit-kiln in Kfar HaHoresh in the lower Galilee, he had proved that industrial quantities of quick lime could be produced with modest prehistoric pit-kilns; he concluded that three workers could produce 250 kilograms of quick lime plaster in three full working days (Goren et al., 2008). As Elsen explained in his article, in the days of the Byzantines, in the 6th century B.C., with the appearance of brick in Catal Hüyük, the lime was used as a binder (Elsen, 2006, Roberts et al., 2013). Even at that time, the use of mortar in the plaster form was rare, which still dominate in places like the Pyramid of Khufu and Egypt at Timna (1400-1200 B.C.) (Hughes et al., 2003). Greeks are the innovators of many construction materials, they are the first who created lime mortars, the Romans continued the process of developing of Lime mortars during the 1st millennium B.C. (Pavía et al., 2008, Carran, 2015). Now I will shed light on some definitions in the field of binding materials, building mortars are mixtures used to join bricks, stones, blocks, and the like. Mortar can be defined as a paste which can set and harden, obtained by adding water to a fine-grained mixture of aggregates such as sand and binder., e.g., clay, gypsum, lime or cement or their combinations (Duggal, 2009, 2017). A historic building is defined as a structure that has features such as materials, architecture, culture, and history of significant value that have to be appreciated and should be preserved for future generations. It is essential for the conservation of the national cultural heritage as it reflects the identity of a whole society. When investigating historical buildings, it is important to understand and to know the materials that have been used in the past centuries and earlier decades to learn the processes of preparing the mortars according to the traditional methods. These ancient architectural structures were built with mortar, which in its composition consists of at least two components, the binder is calcium lime (air lime or dry hydrated lime), the aggregate (sand), may also contain pozzolanic ingredients or organic material (Margalha et al., 2011, Allen, 2015). However, the components used in the production of mortars have changed considerably in their composition and form over the centuries. Despite these changes, it is still necessary and of utmost importance to know the original material to meet the requirements of compatibility and durability in terms of building conservation. For this reason, it is essential to carry out a historical evaluation of the building, intending to study the availability of interventions, the time at which they were carried out, and the type of material used (Hughes et al., 2000).

The abundance of inappropriate materials and techniques, as well as poor decisions, have contributed to the process of losing the originality and beauty of cultural heritage resources. Today, conservation depends on portland cement and sometimes polymer by trying to reveal the originality of heritage resources. However, many of these measures do not meet the basic requirements concerning physico-chemical consistency and can cause great damage. The characterization of the ancient mortars with FTIR analysis will identify the existing materials in the samples, the combination with TGA will provide the exact composition of the ancient mortars, which should optimize the conservation of monuments and the preservation of our cultural heritage (Jordán et al., 2019). The importance in this study is to track the development of the binding material and use the best in the context of restoration consistent with the laws of protecting human heritage. One of the main problems arising from the use of incompatible materials is the deterioration of ancient structures and monuments in the form of detachments, superficial breaks or "gaps". It is becoming more and more common to check anomalies due to the use of mortars based on the use of Portland cement. This happens when an application is made on surfaces with mortar coverings with very different characteristics, which can lead to cracking and detachment of the new coverings. Because of this, current investigations emphasize attitudes toward using materials that are compatible with the original support materials for current and future Conservation strategies (do Rosário Veiga et al., 2010). Conservation strategies are consistently the best option that could be considered when deterioration is affecting ancient masonry: small repairs, filling the gaps, repairing cracks, and consolidation to restore the loss of integrity or liability. However, if there is general degradation or a major progression, it may be necessary to replace all or part of the old mortar. In these cases, the use of new, durable mortars that can be used with the existing masonry materials becomes essential for the appearance of the building and for preserving its integrity (Veiga et al., 2008). According to Martin's definition, hydraulic lime is a binder obtained from impure limestone, which contains 8 to 20% clay, its composition is dominated by calcium hydroxide (from the burning of calcium carbonate $CaCO_3$), which contains also silicates, calcium silicates, and calcium aluminates are attributed to the clay. A great advantage of hydraulic lime is its ability to obtain higher mechanical properties, better workability, and a smoother surface (Allen, 2015). To discuss the types of mortars used in conservation, therefore, some factors determine the type of Restoration materials in the cultural heritage sources, we explain below. The factors which determine the type of mortar to be used for a particular structure depend on the required strength of the masonry, rainwater resistance, immediate and long-term appearance, curing temperature, expected working conditions of the building, and costs. For most practical purposes, a building mortar falls into one of the following categories: The first is prepared from Portland cement or its variants, from sand and water. The second is a mixture of air lime which hardens on exposure to air or hydraulic lime, sand, and water. The third is made from gypsum or anhydride-binding materials (Duggal, 2017). Most studies mentioned the historic mortar in general, but in our study, we are interested in clarification to preserve the originality of the binding materials in the investigated church. Historical mortars are complex systems that use aerial lime supported or hydraulic lime or a mixture of them, aggregates (not at all times crystalline), and additives that interact with the binder (Gleize et al., 2009, Moropoulou et al., 2000). The process of obtaining the aerial lime starts with the calcination of the limestones, in furnaces. In the first stage of the process, the rock releases the water it possesses then calcium carbonate begins to decompose when it reaches the temperature of the decomposition. The calcination is the second stage of the limestone cycle (Fig. 1 and equation 1). It is the conversion process of a source of Calcium carbonate to calcium oxide (quicklime) by endothermal decomposition between 780ºC - 1340ºC (Alaabed et al., 2014). There are different types of calcium carbonate and each has a different temperature of burning; the purer the calcium carbonate is, the higher the moderate temperature required for calcination (Wingate, 1985; Holmes et al., 1997; Holmes et al., 2002; Kumar et al., 2007). Fig. 1 illustrates the process of calcination (burning about 900 ºC), slaking, and carbonation of the hydrated lime which reacts with carbon dioxide to produce calcium carbonate Fig. 1 and equations 1-5 explains the lime cycle, when $CaCO_3$ is heated, they decompose to form CaO (quicklime) and release (CO_2). Burnt lime is an unstable substance and reacts with water to hydrate to $Ca(OH)_2$. The re-absorption of CO2 from the atmosphere by $Ca(OH)_2$ and can take years to go back to $CaCO_3$).

This initial stage is represented by the equation;

$$\mathbf{CaCO_3 + heat \rightarrow CaO + CO_2} \qquad \textbf{eq (1)}$$

In the case of calcination, decomposition first begins on the outer surface of a sample to be examined before it is passed through the interior of the samples (Moffat et al., 2005, Turkdogan, 1980). To maintain the process of calcination, CO_2 must pass through the pore spaces of the rock; therefore, the decomposition temperature must be maintained until all the materials were calcinated. If the firing is interrupted before the decomposition reaches the center of the sample, then the core is considered underburnt. This causes the center to decompose and gases such as CO_2 to release, the surface temperature must exceed the dissociation temperature of the carbonate. The rate of Carbonation is primarily controlled by the rate of heat transfer through the calcined layer, which increases in thickness with the calcination time (Boynton, 1980). The reactivity of quicklime depends on the calcination conditions, preheating rate, temperature, and retention time) and its impurities (Kantiranis, 2004, Carran, 2015). As the temperature rises, the heat begins to penetrate the center of the samples; the dissociation of the carbonate begins in an atmosphere of pure CO_2, which can create increased pressure on the lump during the escape attempt. The higher pressure increases the temperatures and leads to an over-burning of the already calcined lump surface; this is preceded by a reduction of the sample size and the sealing of pores or interspaces (Oates, 2008). After completion of the calcination process, the CaO generated must be hydrated to convert it into a bulk product. The product is called slaked lime and is represented by the following equation:

$$\mathbf{CaO + H_2O \rightarrow Ca\ (OH)_2 + heat} \qquad \textbf{eq (2)}$$

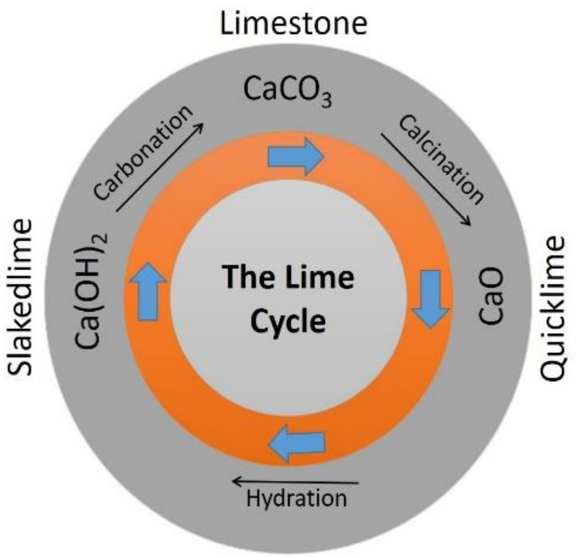

Figure 1. The lime cycle, during the burning process carbon dioxide (CO_2) escapes and quicklime (CaO) is produced, then the quicklime is slaked with water, ($Ca(OH)_2$) is formed, place by carbonation, i.e. by absorbing carbon dioxide (CO_2), after returning to nature, the lime absorbs (CO_2) from the air, which returns again to ($CaCO_3$).

When quicklime is combined with water, a strongly exothermic reaction takes place. The water migrates into the pores of the quicklime, where the heat that is generated creates an internal expansion force that disperses the particles into crystalline powder or colloidal suspension; a fresh surface is allowed to migrate into the further water, allowing the solubility of the particles, which then combine chemically to cause supersaturation of the calcium hydroxide, which leads to crystallization. Less reactive particles (e.g. those that are overburned) dissolve more slowly, resulting in the formation of crystals around a non-reactive core (Boynton, 1980, Carran, 2015). This entire reaction can be completed within a few minutes if the quicklime used is fine-grained and pure. The CaO can also react with moisture in the air; to avoid extinguishing the air, quicklime must, therefore, be stored in sealed containers in dry rooms before hydration (Wingate, 1985, Carran, 2015). Once the lime product is produced, it is blended with aggregates; the mortar begins to harden as soon as it is exposed to the air. The process of hardening called the carbonation, which is represented by the following formula:

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \qquad \text{eq (3)}$$

Equation 3 is a simplification of a two-phase process. Initially, the CO_2 dissolves in water within pore spaces to produce carbonic acid;

$$CO_2 + H_2O \rightarrow H_2CO_3 \qquad \text{eq (4)}$$

The lime in the mortar then reacts with the carbonic acid to produce calcium carbonate;

$$Ca(OH)_2 + H2CO_3 \rightarrow CaCO_3 + 2H_2O \qquad \text{eq (5)}$$

As mentioned before, the carbonation can take several months or years, the lime mortars with a high calcium content may contain different rates of carbonation. The carbonation rate of lime mortars is initially very low and gradually increases over time, as calcium lime absorbs CO_2 before the components are chemically bound; dolomitic mortars have a high carbonation rate from the outset (Hughes, 2003, Carran, 2015). Various materials can be added to the mortar to change the color or improve the properties of the mixture. Charcoal is a common inclusion, either added as a pigment to darken the mortar or an incidental inclusion from the burning process. Other fuel related inclusions include peat, wood, coal, and clinker. These were more common in early historical mortars, as from the 19th century the development of shaft kilns led to more efficient separation of the fuel from the binder (Elsen, 2006). Beer, blood, and urine may have also been added to mortars to increase frost resistance, however, such components would also delay the carbonation process and their presence cannot be verified (Carran, 2015, Hughes et al., 2003). Pozzolans are siliceous or aluminous materials that combine with hydrated lime in the presence of water to produce cementitious products such as calcium silicate hydrates called Pozzolanic Mortar (Carran, 2015, Elsen, 2006). Such materials include crushed ceramics, volcanic rock, and slags (Ingham, 2005). Often brick and tile were used because the clay minerals within had pozzolanic properties once-fired to 600 – 900 °C (Elsen, 2006). More modern pozzolans include Metakaolin, brick dust (Carran, 2015). The primary difference between standard lime mortar and pozzolanic mortar is their increased strength. We focus on previous studies to determine the components of mortar at different locations around the world to obtain the appropriate blend from the previous studies that are relevant to our study and the used analytical methods. The difference between pozzolanic and standard lime mortars; pozzolanic mortars displayed an 82% increase in compressive strength and a 42% increase in flexural strength when compared to standard lime mortar (Carran, 2015, Moropoulou et al., 2005). Elsen is shown in her study that the plasters and renders are alternative application methods of mortar, they tend to contain finer aggregates, animal hair, and straw to improve the tensile strength of the mixture (Elsen, 2006). The fat lime in putty form is used to produce internal plasters from pure lime with 95% or more calcium hydroxide (Watt et al., 2002, Holmes et al., 2002). Generally, In general, sands are used as aggregates; the most suitable are pit, river or washed sea sand with less than 6% impurities (Carran, 2015). They should be hard and sharp and must be washed from salt and sorted before use. For first coats of plaster, the grain sizes need not be too fine; the sands are sieved through a 5 mm sieve. For topcoats, a 1-2 mm sieve should be used to sort the sand. Alternatives such as stone, brick, tiles, gravel, stone, slag, volcanic ash, fly ash can be used as aggregates (Carran, 2015). The inorganic materials mainly influence the physical and mechanical properties of the mortars, are added to the mortars in very small quantities, it can also significantly improve their properties (Chandra et al., 1987, Rovnaníková, 2002, Krizova et al., 2018). The study investigates the addition of organic material (egg-white) to lime mortar to change its properties, the lime mortar with the addition of egg-white showed an increase in compressive strength compared to the control mixture of a lime mortar without egg-white, this was due to the effect of the proteins on the mortar. Egg-white can form binding interactions with other proteins and surfaces (Naceri et al., 2008, Mydin, 2017). The albumine (egg-white) and some other organic materials were often used as additives in the production of mortars in Roman and Byzantine periods (Kurugöl et al., 2012, Fang et al., 2014, Liu et al., 2016). Infrared spectroscopy is an interesting method for determining the binder materials, such as characterizing mortars and monitoring their hydration concerning possible species changes that may occur in mortar composites, especially in the organic part of it, Corti did not mention any of the organic parts in the ancient mortar study (Corti et al., 2013, Rampazzi et al., 2013). Although the scope of FTIR is sometimes difficult to interpret the different bands and sometimes is limited, the applications of our study have shown the usefulness of this analytical tool for characterization of physicochemical properties of the mineral part of the mortar with its organic and inorganic components, such as protein present in ancient Roman mortars, the monitoring of hydration by FTIR of the mortar matrix in the case of organic materials can be easily characterized. Infrared spectroscopy is a tool analysis of both organic and inorganic materials, which can be interesting to characterize ancient additive egg-white to the mortar, especially in the case of expert appraisals, and mortar-organic composites. However, there are still some difficulties in the identification of some of their components. Archaeological Jerash contains many ancient buildings and possesses different types of mortar materials and we shall mention here some of them for the reader to appreciate the importance of Jerash. The objective of the article is to prove that FTIR analysis in absorption mode, which meets the requirements of the relevant mineral and organic determination, provides a quality assessment of the nature of the phases that make up the anhydrous and the hydrated contested matrix of the ancient materials: Mortar, composite materials of egg-white material, etc.

1.1 THE ARCHAEOLOGICAL SITE

The modern city of Jerash, located about 50 km north of Jordan's modern capital Amman, is the site of the ancient city of Gerasa (Fig. 2). The city, which during the Roman period belonged to the Decapolis (Plinny et al., 1991), flourished during the late Hellenistic, Roman, Byzantine, and early Islamic periods. Gerasa was a Roman city in 63 B.C. and become part of the Roman province of Arabia in A.D. 90, Jerash is characterized by an impressive Roman architecture. Among the buildings of the city are the Arch of Hadrian, the Temple of Zeus, and the Artemis, oval square surrounded by Corinthian columns of a narrow and open Ionic order in Colonnaded Street, Hippodrome of the South theater and the North Theater. The Roman architectural style of the city is one of the most exquisite, valuable, and exceptionally well-preserved remains of the old Civilizations that have lived in northern Jordan.

Therefore, Roman architecture Projects are present in buildings and homes. throughout the modern world. However, the Roman mortars have in the church of the cross, and architectural monuments of ancient Jerash are in attracting the attention of researchers of the study (Yaseen et al., 2013). In a quiet valley, near the mountains of Gilead, lie the ruins of Gerasa (Jerash), the city of the Decapolis, Fig. 3 shows the map revealed Gerasa (Jerash) among Decapolis cities. The significance of Jerash is highlighted by its power through its streets and monuments in a good state of conservation. It is situated in a valley with a stream called the Chrysorhoas (Golden River) (Klein, 1974), whose banks are full of green and refreshing poplars and walnut trees, ran right through the middle of Gerasa, while the surrounding mountains are reduced to a rugged arid. Gerasa lies on the hills that cross the Zerka River and enclose a "forest" of pine trees that cover the mountains of Ajloun. From there you can visit the ruins, with the triumphal arch in the foreground. The other route shows us views of the eastern wasteland between Zerka and Mafraq. It is more or less 570 meters above sea level, surrounded by pastures and fertile fields, near a stream (where once there was a dense forest) and was settled in very old times (Prehistoric - Paleolithic and Neolithic), but for lack of evidence, nothing is known for certain. Greek, have founded the city in which they were, and they built it, the Romans continued the settlement. Antiochus III. (223-187 B.C.) led to the rule of the Seleucids, which resulted in the creation of Antioch over the Chrysorhoea (which would be Gerasa), where it was settled. A Seleucid garrison was established on the site. The temple of Zeus might have been built around this time. Excavations at the site show the jars of Rhodes and the presence of the entire Pantheon of Greece. The city's name was of Semitic origin, but it took the Hellenised name Gerasa. However, its Semitic name Garshu has attested in a Nabatean inscription (Starcky, 1965) after classical antiquity the Semitic name gained prominence once again and the city was called Gerasa (Lichtenberger et al., 2016). According to legend, the city was established either by Alexander the Great or his general Perdiccas, but it is more likely that it was founded by Antiochus 111 or Antiochus 1V and therefore also named Antiochia at the Chrysorrhoas (the Golden River) (Lichtenberger, 2016, Lichtenberger et al., 2015, Holdridge et al., 2017, Lichtenberger et al., 2020).

Figure 2. Panoramic view of Jerash city (Gerasa)

The diversity of construction materials and technologies used in the production of mortar in Gerasa need more time to develop a technique for conservation in the city of Gerasa. And this diversity is associated with events that were going through it, such as the visit of Hadrian. Emperor Hadrian visited the city in the winter of 129-130 AD, which was a reason for new buildings, and an arch of triumph was erected in his honor. This would be the Golden Age of the city, a major construction project, the enlargement of the streets of the Forum, the temples of Artemis, and the replacement of the street chronicles with models from Corinth (Lichtenberger et al., 2015). The temple of Artemis, with large accesses to the east and its large points of departure (doors), was dedicated to our era in 150 AD. The temple of Zeus (new construction) was built in 163 AD, the nymphaeum in 191 AD (Kraeling, 1938). Many inscriptions of the time show shrines, pedestals, statues, buildings offered by rich citizens. Remains from the late Hellenistic period have been found. In this period a small settlement is attested to Camp Hill in the southern part of the city and across from one of the main sanctuaries, that of Zeus Olympios, which was also founded in the Hellenistic period (Kehrberg, 1989, Ostrasz, 1989, Seigne, 1989, 1992, Lichtenberger et al., 2015, 2016, Lichtenberger, 2008, Raja, 2009, 2013). In the Roman period, Jerash flourished differently and expand. The city was enclosed by more than 4 kilometers of city walls with towers, bastions and gates punctuating the walls at various points. Various dates for the city walls have been proposed by scholars, Kraeling would prefer to connect the city with Antiochus IV with suggestions ranging from the Hellenistic period to late Antiquity without a consensus having been reached so far. Inside the wall of the city and Along the main street, the typical public monuments of any Roman city have Temples, macellum, theaters, shops, Nymphaeum, tetrapylon, public paths (Kraeling, 1938, Radford, 1941). Churches are constructed, such as that of the Prophets Apostles (464 - 465 AD) and the Church of St. Theodore (496 AD). Many churches are constructed with Justinian

(531 to 565), as well as many public buildings, with a superficially beautiful design. The most important centers of the period were the Churches.

A special Thermae was constructed by Bishop Placo, next to the church of St. Theodorus, for the use of his priests (perhaps one of the first examples of cleansing before' the sacred). It is beautiful and comfortable concerning the houses in general (which were made in a very crude way with those of the Roman period) (Gawlikowski et al., 1986, Kraeling, 1938, Smith, 1941, Boyer, 2016, Radford, 1941, Hachlili, 2009, Schick, 1995). The cultural diversity is linked to the events that pervaded the city, and the coming together of civilizations from East and West led to a great development in the field of urbanization, and even in Islamic times, they maintained the city buildings and churches. The city continues its prosperity during the Byzantine and Early Islamic periods as well as in the Early Mamluk period. More than 20 churches have been discovered inside the city wall, attest to the flourishing to the religious life of the city in the Byzantine Period (Kraeling, 1938, Boyer, 2016, Pierobon, 1983, March 2009, Walmsley, 2011, Shiyyab, 2013). The great buildings and temples possessed exquisite beauty and comfort. The last of the churches built dates from 611 AD and the Persian invasion in 614 AD marks the beginning of the end of the city. A series of earthquakes destroyed many churches and buildings without being reconstructed. The church of St. Theodore is a good example of this. The abandonment was gradual. The Church of the Cross was chosen in this study because it is in urgent need of immediate conservation, otherwise it will be difficult to restore it in the future. The study focuses on a scientific analysis of construction and restoration materials to preserve the compatibility, originality within the charters of conservation of the city heritage. In the northwest quarter of the city of Jerash, a new basilica church has been uncovered in 2010. It is located directly to the west of Genesius church and is adjacent to it.

The church was named the Church of Cross due to the lack of the inscriptions in it and the presence of a Cross on its mosaic floor. The floor of the church was decorated with Mosaics as well as marble and pavers. Portions of the mosaics pavement underwent later repairs. Due to the lack of inscriptions and the coins in the church, the date of the church still undetermined. but, based on the location of the church and the mosaic models on the floor, it can be said, that the church refers to the end of the 6th or the beginning of the 7th century AD, the glass mosaic was used to restore some of the damaged parts (Arinat et al., 2014).

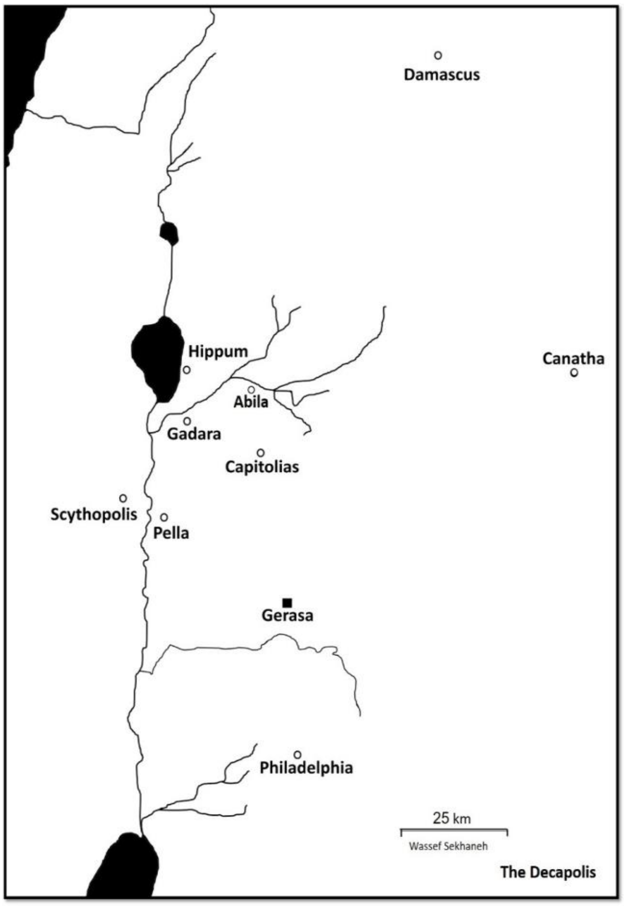

Figure* 3. *Decapolis cities location, the map revealed Gerasa (Jerash) among them.

2. MATERIAL AND METHODS

Five samples of Roman mortar materials (M1–M5) were collected from the boundaries from different places in the church between the bases of the columns (M1) and under the mosaics floor (M2) (see Fig. 4, 5), the samples which have been found at different locations in the church. Two samples are investigated in this article, Mosaic floor mortar (M2, Fig. 4), and between the column and its base (M1, Fig. 5) both have been investigated in this work. We took 100 grams for the study from each sample by chisel without destroying them. Their outer part was scraped and removed to ensure the collection of unaltered material. Approximately 6 mg was used for the thermogravimetric analysis (TGA) of the fine mortar powder; for the other tests (300 mg for XRD, 10 mg for FTIR, 5 g) the powder was dried at a temperature of approximately 60 °C to avoid the removal of organic matter. TGA was used to classify mortars as typical lime mortars, brick lime mortars of crushed lime, quarry masonry mortars, cement mortars, and mortars with gypsum, Thermogravimetry Analysis (TGA) is a method of measurement in which the mass variation of a sample is measured as a function of time or the temperature the Recording of weight loss of a sample is carried out under a controlled temperature impact. The weight changes can be caused by physical and chemical reactions (Heide, 1989). The temperature in a nitrogen atmosphere was measured from ambient temperature to 1000 °C at a constant heating rate of 10 °C/min. Fourier transform infrared (FTIR) spectrometry was used to detect the presence of salts and organic compounds and to confirm the results obtained from thermal analysis and XRD. Fourier Transform Infrared Spectrometry (FTIR) experiments need few milligrams of mortar, the samples were mixed gently and ground manually in an agate mortar and pestle. About 0.1 mg mortar sample was left in the mortar and mixed with about 0.5 mg potassium bromide (KBr), and pressed into a 3 mm diameter pellet with a machine presser. Each obtained in 32 scans over the spectral range of 400-4000 cm^{-1}. Phase identification was performed with OMNIC Software (version 2018).

Figure 4. General view of the mosaic of the church Hall, where the sample M1 have been collected. (Shiyyab).

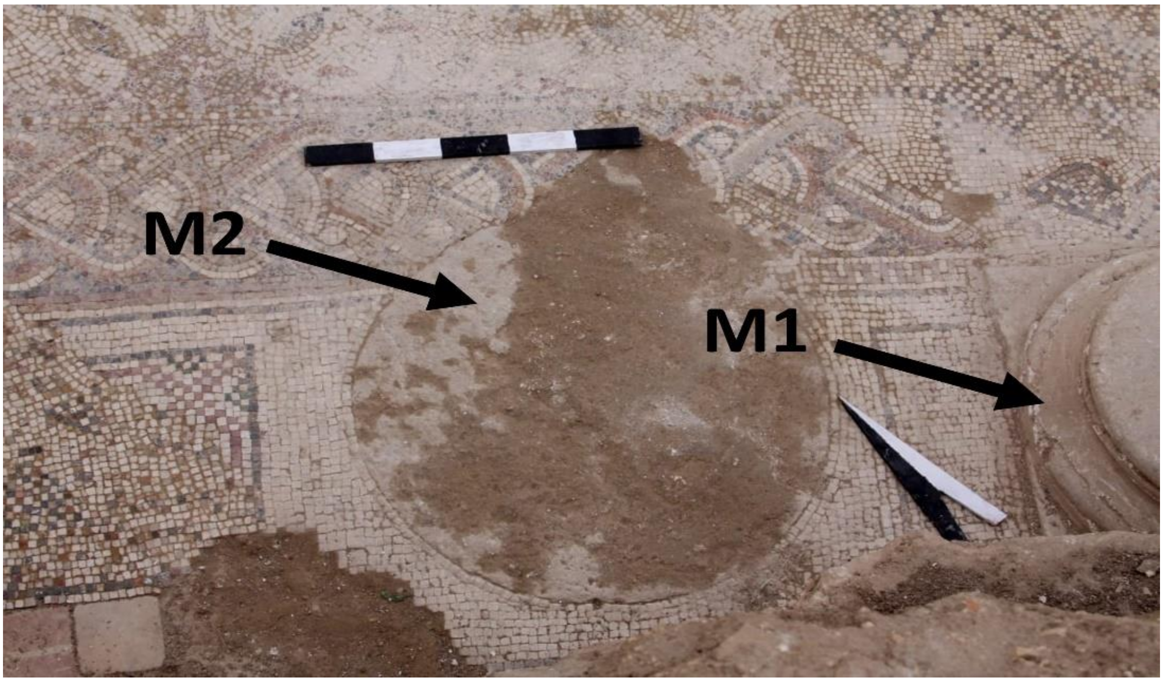

Figure 5. Detailed view of the mosaic of the church Hall, where the sample M1, M2 have been collected. (Shiyyab)

2. RESULTS AND DISCUSSION

2.1 FTIR spectroscopy characterization

The spectrum in Fig. 6 shows the typical bands detected in the mortar sample M1, i.e. it is attributed to calcite, quartz, and silicates, or iron oxide. Fig. 6 shows the infrared spectrum of the Church of the Cross column mortar and Fig. 6 shows the Mosaics floor mortar spectrum, In the church of the cross in Jerash.

The spectra of FTIR absorption bands (Fig. 6 and Fig. 7) corresponding to inorganic and probable to organic part in mortar samples. All are evident from the absorption peaks of the binder components, mainly the calcite $CaCO_3$. It is seen in the sharp peaks of C-O in-plane bending (υ_4) at 712 cm^{-1} and C-O out-of-plane bending (υ_2) at 875 cm^{-1} (Palacios et al., 2007). The sharp peaks confirm the identification of carbonates in the ancient mortar sample spectra of the church. Furthermore, the broad peak for the C-O multiband for organic materials, centered at asymmetric stretching (υ_3) at 1448 cm^{-1}. There is a sharp peak at 1795 cm^{-1}, and 2514 cm^{-1}, peaks are attributed to $CaCO_3$. Pozzolana is recognized in the spectra, it is present in small quantity in the analyzed mortar and is characterized by the vibration mode of the symmetrical vibration mode of strong band Si-O of silica at 1039 cm^{-1} (wide and intense). Saravanapavan and Hench have found in their study that the strength of the main maximum of the Si-O-Si stretching vibrations has increased and shifted with a decrease in the amount of calcium in the calcium-silicate structure. This means that there is more silicate material in the mortar of the mosaic floor in the church sample M2 (Saravanapavan et al., 2003). The absorption bands centered around 797 and 692 cm^{-1} are attributed to Si-O mode of quartz in the samples (Tyagi et al., 2006). The sand is a major component of the ancient mortar, absorption peaks for the mineral quartz should be present in the measured spectrum. These were identified in both spectra, it is based on the Si-O stretching peaks at around 604, 775 cm^{-1} and 1086 cm^{-1}, respectively. There is another Silicate phase that is relevant for the band at 472 cm^{-1}.

There is a fraction that 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 527 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 (Derrick et al., 1999).

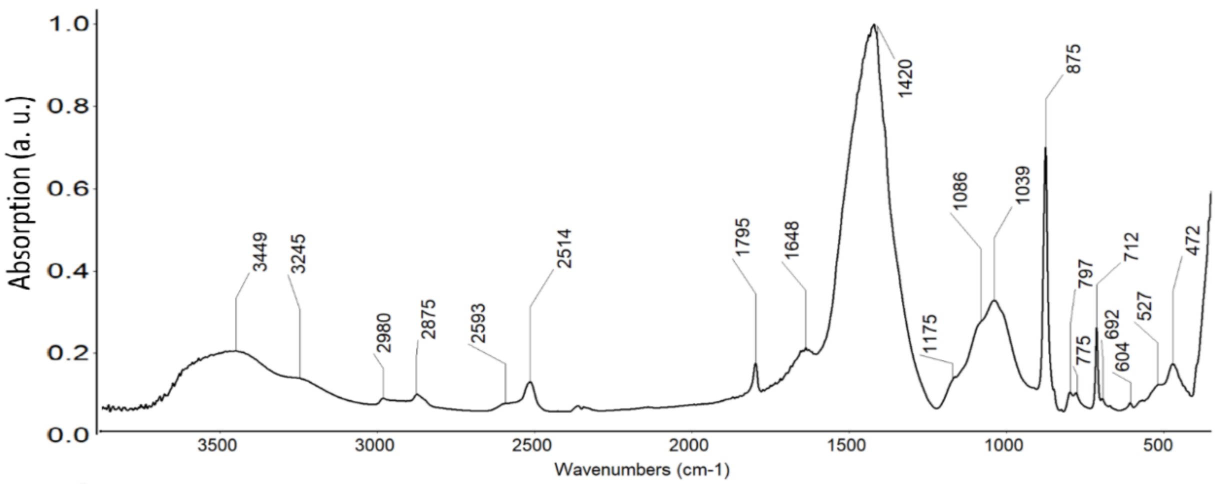

Figure 6. FTIR Spectrum of the mid-infrared region (4000-400 cm-1) of the mortar sample M1.

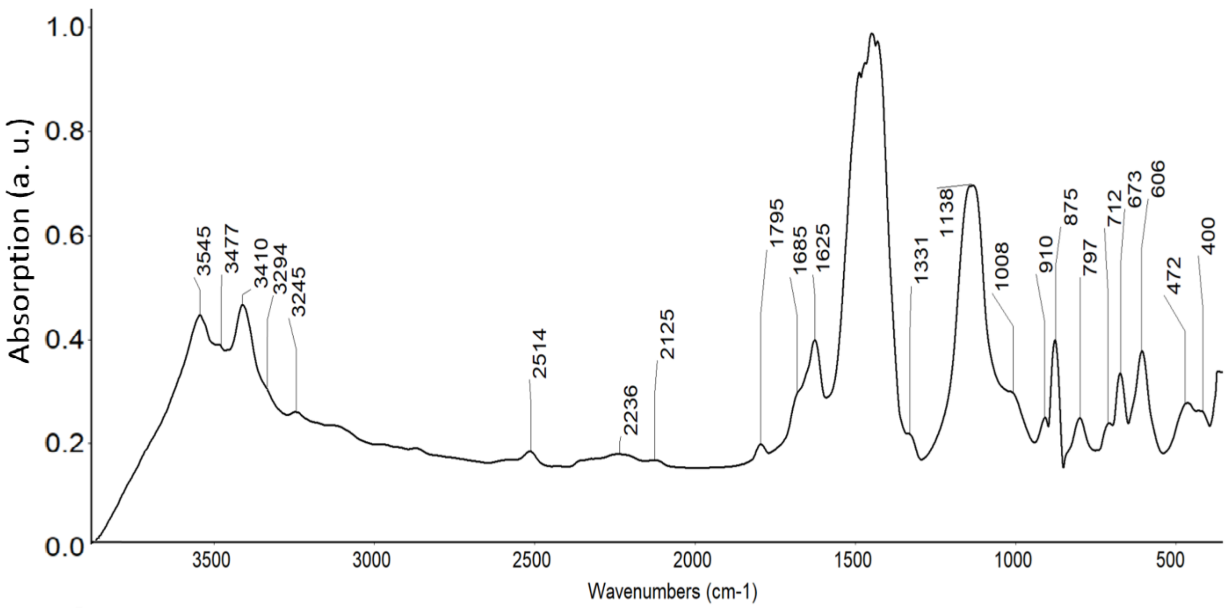

Figure 7. FTIR spectrum of mid-infrared region (4000-400 cm-1) of sample M2

The possible presence of proteins in the mortar sample was established by the characteristic peaks for the amide group at 3294 cm^{-1}, which includes also the broad bands centered at 3449 cm^{-1}, assigned to the N-H stretching vibration band, and peaks among (1625-1648 cm^{-1})(Fu et al., 1999) for the C=O stretching vibration, respectively, both bands are showing the existence of primary and secondary amides from protein-based materials, this indicates the addition of some protein material to the mortar (Singh et al., 2014). Since proteins, usually in the form of egg-whit, are added in low concentrations, peaks from inorganic components of the sample may likely interfere with its detection [5]. Further qualitative tests for proteins should be performed to confirm these assumptions. The FTIR spectrum of M1 confirmed also C–H stretches around 3500–2800 cm^{-1} (3449, 2980, 2875 cm^{-1}). The C–H stretching and bending by absorbance signals are at 2980 and 2875 cm–1 respectively, most literature of FTIR starts from left with high wavenumber, or higher to lower wavenumber, On a typical IR spectrum, wavelengths increase and wave numbers decrease from left to right (Fig. 6, 7 and 8). Infrared spectroscopy (FTIR analyses of mortar absorbance peaks. Generally, the C–H weak bond in comparing in the spectrum of M1, it is suggested by stretching and bending absorbance signals, respectively, at 2980 and 2875 cm–1. The stretching absorbances of C=O and C–O and N-H stretching vibration banda were observed with high intensity and resolution as an organic probably egg-white amid with comparing with M1, around 1625 1685, and 1795 cm–1, respectively. M2 spectrum shows Some peaks are due to residues of the inorganic fraction; that is, calcite and silicates. Gypsum ($CaSO_4$) is detected in the analysis of the Mosaics floor mortar spectrum highlights, on the one hand, two doublets to 1138 cm^{-1}, 1108 cm^{-1}, and 673 cm^{-1} which correspond to the stretchings of the S-O bond of sulfates (SO_4^{-2}) indicate the clear presence of gypsum in M2. On the other hand, the bands specify of the constituent water that appears above 3000 cm^{-1} as O-H bond. The peak at 2514 (overtone), 1795, 875 (v_2), and 712 cm^{-1} (v_4) are assigned to the carbonate phases. In contrast, the Mosaics floor mortar (Fig. 6) only shows traces of carbonate phases. of carbonates (smaller) bands at 1420, 1369, and alike at 875 cm^{-1}. In both samples, there are stronger bands that are associated with the presence of bound water about 3450 cm^{-1}. The water-containing may be bound to hydraulic components such as silicate and Aluminate hydrates, Church of Cross column and Mosaics floor mortar samples present strong silicate band (Si-O vibration) at 1039 cm^{-1} or 1138 cm^{-1} are in both samples, this is the results confirm the pozzolanic ingredients silicate phases which are also relevant for the band at 472 cm^{-1} in both spectra.

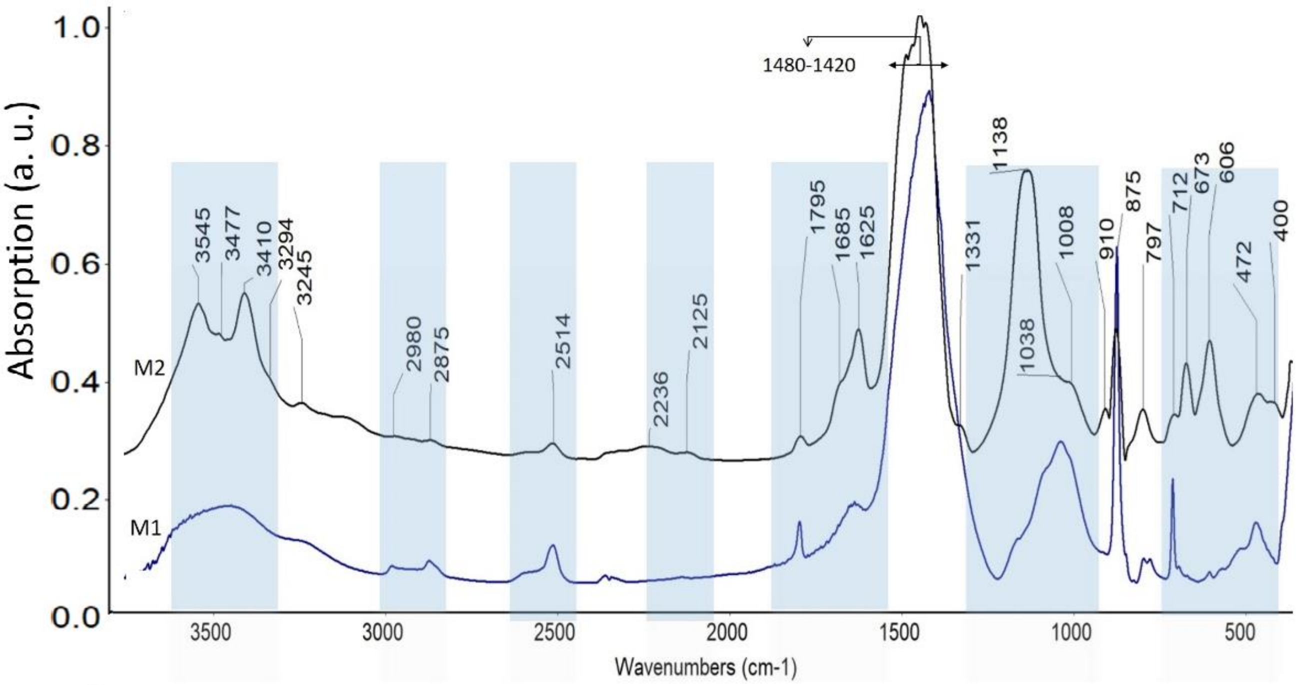

Figure 8. FTIR spectral comparison of mortar samples (M1, M2)

2.2 *Thermogravimetry (TGA/DTA)*

The analyses were carried out using a thermogravimetric balance (TGA-TGS2 Perkin Elmer) at Physical Chemistry Laboratory at Duisburg-Essen University in Germany. A fine powdered sample (60 mg) was brought from room temperature to 1000°C at a relatively slow speed with a linear heating rate of 5°C/min. TGA measures the weight loss in the samples, while DTA characterizes the heat effects on the samples. They were heated in platinum crucibles under oxygen flow at a rate of 20 ml/min to monitor and optimize the weight loss phenomenon. TGA and DTA test results are presented in (Fig. 9: of (M2), Fig. 10 of (M1)), a quasi-linear hydrated weight loss recorded at near 100°C as small damping in the curve of M2 and M1 associated with endothermic reactions is correlated to the moisture present in sample M2 and M1. A loss was detected between 100-250 °C, it is related to bound water. At temperature between 300-400 °C, there is a decomposition of the organic part of the mortar, it is estimated as egg-white dried materials in the mortar. A considerable loss detected between 450°C and 550°C with endothermic reactions corresponding to the elimination and transformation of the most common materials present in clays like illite and kaolinite. The results of DTA were quite expressive: both samples showed clear losses within the mentioned range. It is illustrated in Fig. 9 and Fig. 10 of the samples M2 and M1, respectively.

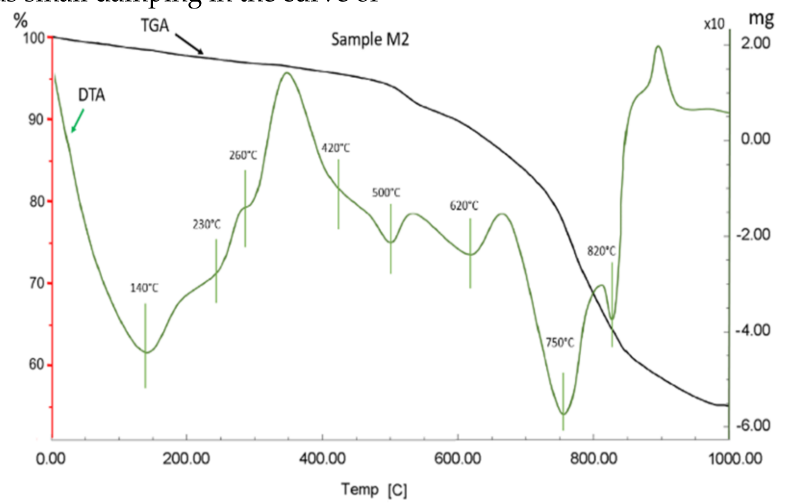

Figure 9. TGA/DTA diagram of lime mortar of M2 from the mosaic mortar floor.

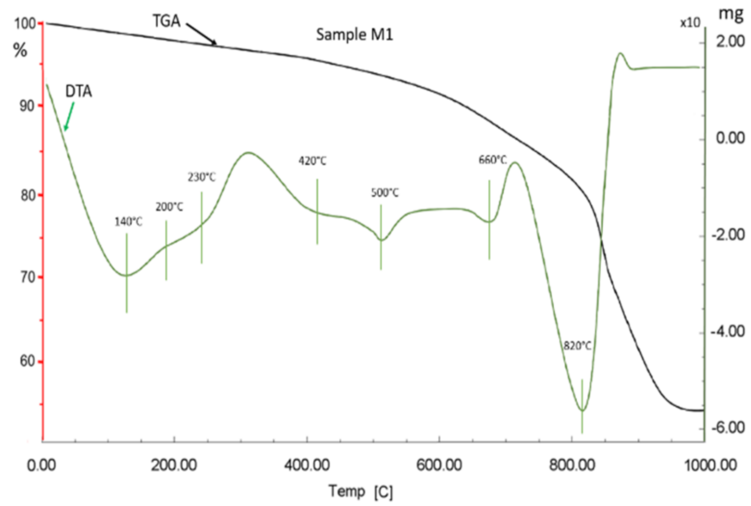

Figure 10. TGA/DTA diagram of lime mortar between the column of the church and its base M1.

There is a marked mass loss peak at about 750°C, it is for the decarbonation of calcite, between 700°C and 850 °C related to endothermic reactions in the mortar samples caused by the existence of carbonate accounts for 65 to 75 % of the total mass loss is recorded during this process in the linked region of heating. The TGA experiments were implemented on the same samples M2 and M1 analyzed by FTIR were confirmed the presence of protein material, as an organic fraction expected to be a minor component of the mortar sample, it is expressed through the band proportion. In particular, a weight loss was of organic part observed as a peak around 340°C. The signal was weak, as the organic fraction was expected to be a minor component of the mortar sample. Fig. 9 and 10 show the TGA/DTA curve of the sample M2 and M1, where M2 represents the ancient lime mortar collected under the mosaic floor of the church and M1 is the lime mortar between the column of the church and its base. TGA/DTA curve of both samples M2 and M1 in Fig. 9 displays eight peaks for M2 sample, at 140°C, 230°C, 260°C, 420°C, 500°C, 620°C, 750°C and 820°C and Fig. 10 shows seven peaks for M1, at 140°C, 200°C, 230°C, 420°C, 500°C, 660°C, and 820°C. The differential thermogravimetry thermogram in Fig. 9 shows a peak around 230°C, which may be related to the exothermic transition characteristic of the decomposition of organic materials as protein material and free bound water leave. The TGA and DTA analysis have been described as the behavior of the main components of the mortar, they can be identified by their weight loss, i.e. the loss of bound or free water from 100 °C to 260 in the samples. It is also known that water bound to SiO_2 can be removed by heating above 200°C (Iler et al., 1979, Zhuravlev, 2000). The loss of weight from above 260 until 420 °C, it related to the organic part in the samples, and we expect that this weight loss is due to the archaeological egg-white in samples. The decomposition of the carbonates is the main reaction occurred with a distinctive endothermic reaction releases CO_2 (Izzo et al., 2015). Further work is needed to investigate the isotope levels of the mortar from Theater's Structure, other churches, and buildings in Gerasa (Jerash) and to identify potential limestone feedstocks.

3. CONCLUSIONS

The main purpose of this study was to characterize the binders of mortars used in the church to collect innovative information on this important site in Gerasa that will be devised as compatible to restore archaeological buildings in the site. It concludes that it is easy to identify all the peaks of carbonate and silicate materials. However, the strength of the FTIR absorption peaks in the archaeological samples are very clear and represented by comparison of the intensity, broadening and shifting components materials, it means that FTIR is an excellent approach to study the ancient binding materials and comparing it with new restoration materials that have been used in the site through the time. The results of the FTIR method gives and an excellent indication that there is a large amount of amorphous calcium carbonate in the sample in addition to crystalline calcite and other components. The silicate bands are stronger in the mosaic floor mortar than in the wall and under column structure samples, which may indicate a higher proportion of silicate phases in the mosaic floor mortar. It concludes that the gypsum is present in both samples in different quantities. The organic materials which added as egg-white to the mixture have been well confirmed by FTIR and TGA/DTA, it can be also concluded that the samples consist mainly of various forms of pozzolanic and organic materials, namely organic matter like protein-based materials. Further sampling of mortars from along the line of the excavated places in 2019 and associated buildings. The thermal analysis method was applied to mortars and adhesive pastes to find the phase of decomposition as a function of temperature. The results showed significant development in the color of the test substances caused by heating. The main reasons for these changes were attributed to the physico-chemical transformations within the main components of the mortar and aggregates. The mineralogical composition of the ancient mortar (i.e. siliceous, calcareous) plays an important role in the overall color change of the heated mortar due to their chemical interaction and the new components transformed by the reaction. It also concludes in this study, aggregates containing organic compounds undergo a significant color change when heated to a temperature above 260 °C in both samples. The oxidation of iron components (i.e. minerals such as limonite, hematite, and goethite) was detected by observing FTIR bands in the spectra but not detected by TGA/DTA.

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