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Archaeometric Study of Masonry Red Bricks and Pottery Water Pipes from the Tanks of A Coptic Water Wheel, Sheikh Hamad, Athribis, Sohag, Egypt

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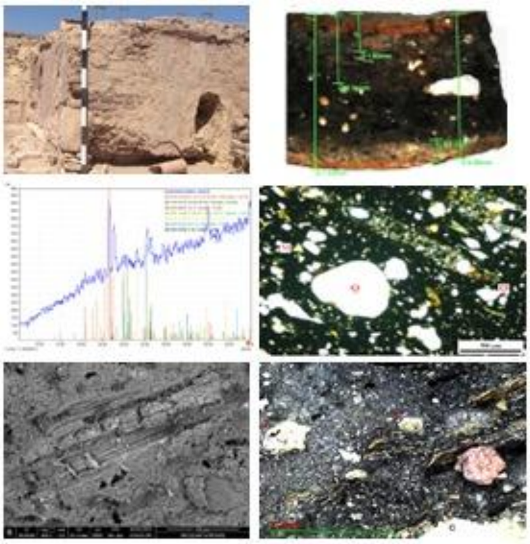
HIGHLIGHTS

- Characterization of red brick and water pottery pipes which were built on the ruins of Ptolemy XII temple Sheikh Hamad, Athribis, Sohag, Egypt.
- The archaeometric study was performed using a USB digital microscope, Polarizing Light Microscope (PLM), Powder X-ray (XRD) diffraction, and Scanning Electron Microscopy (SEM) coupled with EDX.
- Red bricks used in the construction of water wheel tanks were highly burnt and more durable.
- The detection of calcite in the components of water pottery pipes confirms the incomplete process of its burning. The chemical composition of red bricks and pottery pipes confirm that Nile clay was the resource of their raw materials.

GRAPHICAL ABSTRACT



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ABSTRACT

This research will shed light on the compositional characterization of red bricks and pottery water pipes used in the tanks of a Coptic water wheel. This water wheel was built on the ruins of Ptolemy XII temple Sheikh Hamad, Athribis, Sohag, Egypt. Representative samples were analyzed using USB digital microscope, Polarizing Light Microscope (PLM), Powder X-ray diffraction (XRD), and Scanning Electron Microscopy (SEM) coupled with energy dispersive X-ray analyses (EDX). The physicochemical study of these samples indicates that the red bricks and pottery water pipes consist of orthoclase, mullite, quartz, spinel, diopside, hematite and halite.

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That mineralogical composition reflects the well burning temperature of the studied red bricks. While the detection of calcite and some organic residues of chopped straw in the pottery pipes revealed that they were burnt under low temperature when they were compared with the red brick samples. The chemical composition results of all samples confirms that Nile clay is the resource of clay raw materials used for the manufacturing of the studied red bricks and pottery water pipes.

1. Introduction

With the premise that water is a necessity for human existence and is vital for survival; the Nile river has been the focus of the life of Egyptians throughout the different eras. If the Nile does not meet its annual flood, disturbances and famines will occur. Hence the urgent need to build water structures such as cisterns, dams, water wheels, arches, aqueducts, and canals in order to ensure the distribution and sustainability of water [1, 2].

Among these structures are the water wheels tanks which played an important role in the Mediterranean region for water supplying and distributing, whether drinking water or irrigation [3]. Water wheels are an Alexandrian invention from the Ptolemaic period used for the purposes of irrigation, and then their uses varied in the Mediterranean region [4].

The construction of waterwheels tanks was associated with the implementation of water structures such as canals, tanks, and cisterns. These structures required the use of special building materials like red bricks, lime mortars with hydraulic properties and pottery water pipes, etc. The lime mortars and plasters of the Coptic waterwheel- subject of the research were studied in a previous research, while in this research the red bricks and pottery water pipes will be studied. The subject of the study is Coptic water wheel tanks, excavated in the temple of Ptolemy XII (Fig.1) in Athribis [5]. Athribis is an old Egyptian city situated on the west bank of the Nile river, it belongs to the 9th district of Upper Egypt “Akhmim”, Sohag [6, 7]. Many building materials were used in the construction of the water wheel tanks such as red bricks, lime mortars, plasters and pottery water pipes. This paper will study the archaeometric characteristics of the red bricks and pottery water pipes.

Mud bricks are the oldest environmental building materials used in Egypt, as its industry dates back before the dynastic era [8]. This may be due to the abundance, the cheapness, and the accessibility of soil as a raw material. However, the urgent need for more durable and resistant bricks to fire and ambient conditions, with the possibility of using in damp environments; made the burnt red brick more favourite [9].

Red bricks and mud bricks have the same components and manufacturing methods, however red bricks are fired at high temperatures [10]. The heating cause dramatic change in the physical, chemical and mechanical properties of the fired bricks making them more suitable building material for water structures which spread in Egypt from the Greco-Roman era [11, 12].

The earliest known pottery water pipes were discovered in the Bronze Age in Mesopotamia, Mohenjo-Daro in the Indus Valley and in Minoan in Crete [13]. Although the ancient Egyptians relied mainly on simple systems of water transport that depended on their manpower [14], they had used more advanced water supply systems for villages and mines far from the Nile, with the help of digging wells, water constructions and water pipes made of various materials, including pottery pipes . These pipes had widely spread in the Greco-Roman era [15, 16].

There is a diversity of opinions about the way in which pottery water pipes were made, it was mentioned that they were likely formed as flat slices of clay and then wrapped around a central wooden form, or by rolling the clay by hand as rings and then joining them together, pottery pipes may also be formed on the potter's wheel. The third hypothesis may be enhanced by the fact that the discovery of the potter's wheel coincided with the earliest known pottery pipes [13, 17].

Clay is the main raw material used in the manufacture of bricks and pottery products. The Nile river is the most important source of clay in Egypt. The mud used in making bricks and pottery water pipes in ancient Egypt consists of several inorganic and organic materials; mainly clay minerals, sand, lime, iron oxides, aggregates, and animal dung with chopped straw as binding or cementing materials, as well as grog and stone remains which were added during the manufacturing process to enhance the mechanical properties [18]. All of these ingredients are added to water, then they are formed and dried. Finally, the dried clay products are placed in a kiln with temperatures between 500:700^{o}C, it may sometimes reach up to 1000^{o}C. In the early kilns, straw and wood were used as combustible materials, and the burning process may take more than three days [10, 19].

Heating process of clays causes noticeable changes in their mineralogical, chemical and physical properties. This process leads to the appearance of mineral phases such as metakaolin, mullite, cristobalite, gehlenite, diopside, wollastonite and hematite; that is according to the used temperature degrees and the chemical composition of the raw materials [12].

This study aims to identify the characteristics of the red bricks and pottery water pipes used in this Coptic wheel tanks - the topic of the research - by identifying the mineralogical composition and by revealing some important information about the raw materials used in the studied materials. This helps in developing a strategy for conservation procedures of the Coptic water wheel and its associated constructions, Fig.1.

2. Materials and Methods

2.1. Sampling

Two samples were collected from the bricks used for tanks building and one sample from pottery water pipes using a micro scalpel from the deteriorated and separated parts from the Coptic waterwheel to identify the chemical composition, and to reveal some information about the raw materials and the used techniques. (Table 1) (Fig. 2).

2.2. Examination by USB Digital light Microscope

A digital light microscope (Model (USB): X4, Magnification 1600X, Image sensor CMOS, Image resolution up to 640×480, Focus range 15mm-40mm, Frame rate up to 30 FPS, Adjustable illumination 8 Built-in LED Diodes) was used to study the microscopic aspects of the red brick and the inner core for the water pipe sample.

2.3. Polarizing Light Microscope (PLM)

The study of thin sections under the polarizing microscope is a very effective analytical technique to study the properties of pottery products. Many features can be observed by this analysis such as the nature and characteristics of different minerals, the shape and quantity of voids and the relationship between pottery body and its surface treatment [20]. The samples were prepared and studied by Optiphot2 Nikon.

2.4. X-Ray diffraction (XRD)

X-Ray diffraction Analysis (XRD) was used to identify the mineralogical phases and chemical compounds of the burnt clay body and any crystalline phases [21]. XRD device with the following operation conditions was used: Diffractometer type: PW1840, Tube anode: Cu, Generator tension (KV): 40, Generator Current (mA): 25, Wavelength Alpha1 (Å): 1.54056, Wavelength Alpha2 (Å): 1.54439, Intensity ratio (Alpha2/Alpha1): 0.500, Receiving slit: 0.2, Monochromator used: No, 2θ scanned range (5 - 60°).

2.5. *Scanning Electron Microscopy (SEM) coupled with EDX*

A scanning electron microscope coupled with Energy Dispersive X-ray spectroscopy unit (Model Quanta 250 FEG (Field Emission Gun) coupled with EDX Unit (Energy Dispersive X-ray Analyses), with accelerating voltage 30 K.V., using 14-x up to 100000 magnification and resolution for Gun. was used to examine the microscopic features of red brick and pottery pipes, as well, to identify the chemical composition of these materials.

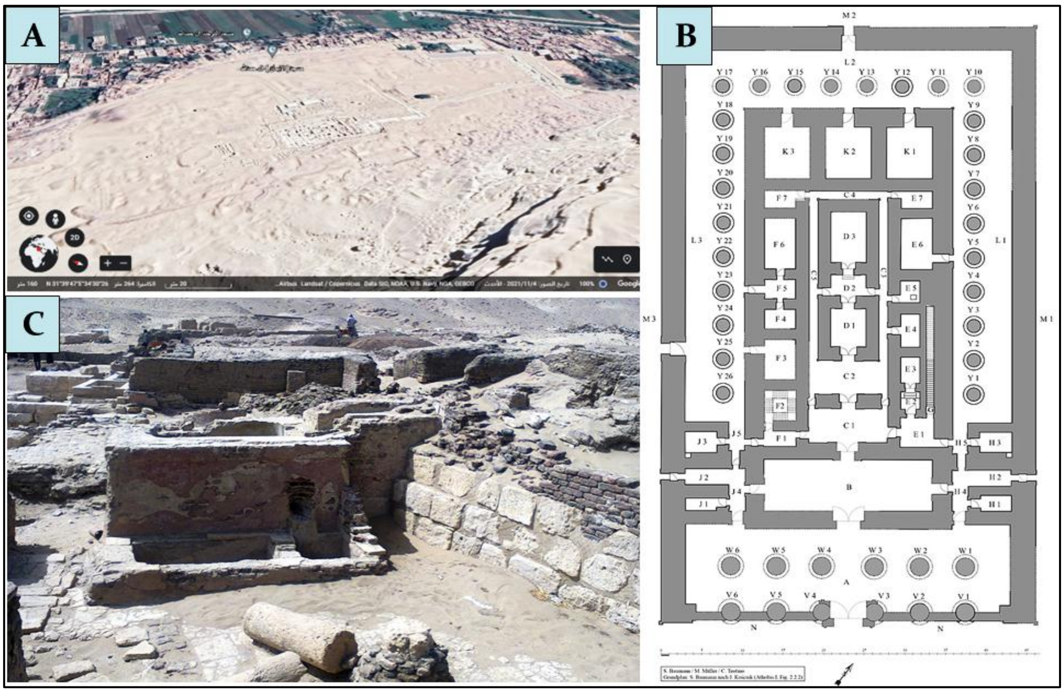


Fig. 1. The location of the Athribis temple, El-Sheikh Hamad, Sohag, Egypt,(A) Map of the site (from Google map 10-9-2022),(B) Plan of the temple ([www.wikiwand.com/en/Athribis_Project 8-10-2022](www.wikiwand.com/en/Athribis_Project)), (C) Location of the Coptic waterwheel.

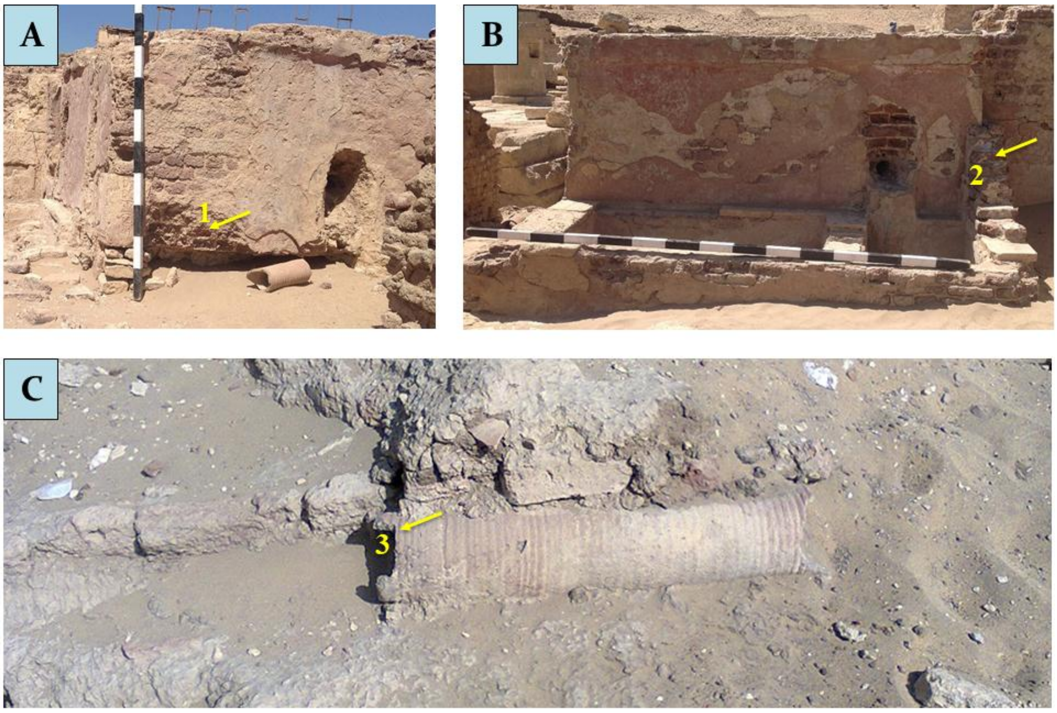
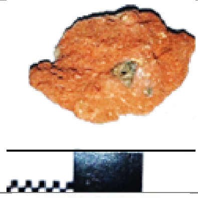
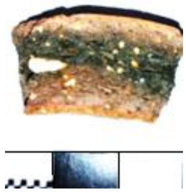


Fig. 2. Locations of the Coptic waterwheel samples, (A) Red brick sample from the lower part of the south wall of the big tank, (B) Red brick sample from the north upper part of the small water tank, (C) Sample of pottery water pipes used for the transportation of water among tanks.

Table 1. Description of the brick and water pipe samples collected for the study

Sample No.	Sample description all (samples' locations were shown in Fig 2.	Figure
(1)	Red brick sample from the lower part of the south wall of the big tank.	
(2)	Red brick sample from the north upper part of the small water tank.	
(3)	Sample of pottery water pipes used for the transportation of water among tanks.	

3. Results and discussion

3.1. Digital Microscope

The results of the examination of red brick and pottery samples by digital microscope showed the following results:

The texture of the brick sample No. 1(Fig. 3 A) is rich in iron oxides, the grains and components are homogeneous and contain some calcite grains. In the second sample of bricks No.2, some melted components of bricks are observed which may be silica, because of the high burning temperature and the long burning time. That was observed through black conglomerates inside the inner core of the brick sample (Fig. 3B). On the other hand, calcite and pottery powder(grog) were observed in the pottery pipe sample, which were used as inorganic additives for clay during the preparation process to improve the properties of the product. Moreover, the core of the sample is not well-burned and that was deduced from the black color, which is due to the formation of carbon. This indicates that the used burning temperature was not high, or the burning time was not sufficient to complete the burning process for the thick pottery water pipes. In addition to the above, many voids have been observed due to the use of straw/hay as an organic additive material. Also, some spots of salt efflorescence were observed on the surface of pottery pipe sample [22] (Fig. 3C).

3.2. Polarizing Light Microscope (PLM)

The results of the petrographic study of sample 1 of red brick revealed the obvious diffusion of quartz (Q) grains of similar size. In addition to the presence of calcite(C) (Fig. 4A-B) (1a, 1b) with parallel nichols (2a, 2b) with crossed nichols. Sample 2, red brick from the north upper part of the small water tank, also included quartz(Q) and calcite(C)inside a fine-grained texture, and a relatively minor quantity of Plagioclase (Pl) [23]. The grains of quartz are accurate and homogeneous. This indicates that the components of the bricks are well prepared, and the bricks produced are of high hardness.

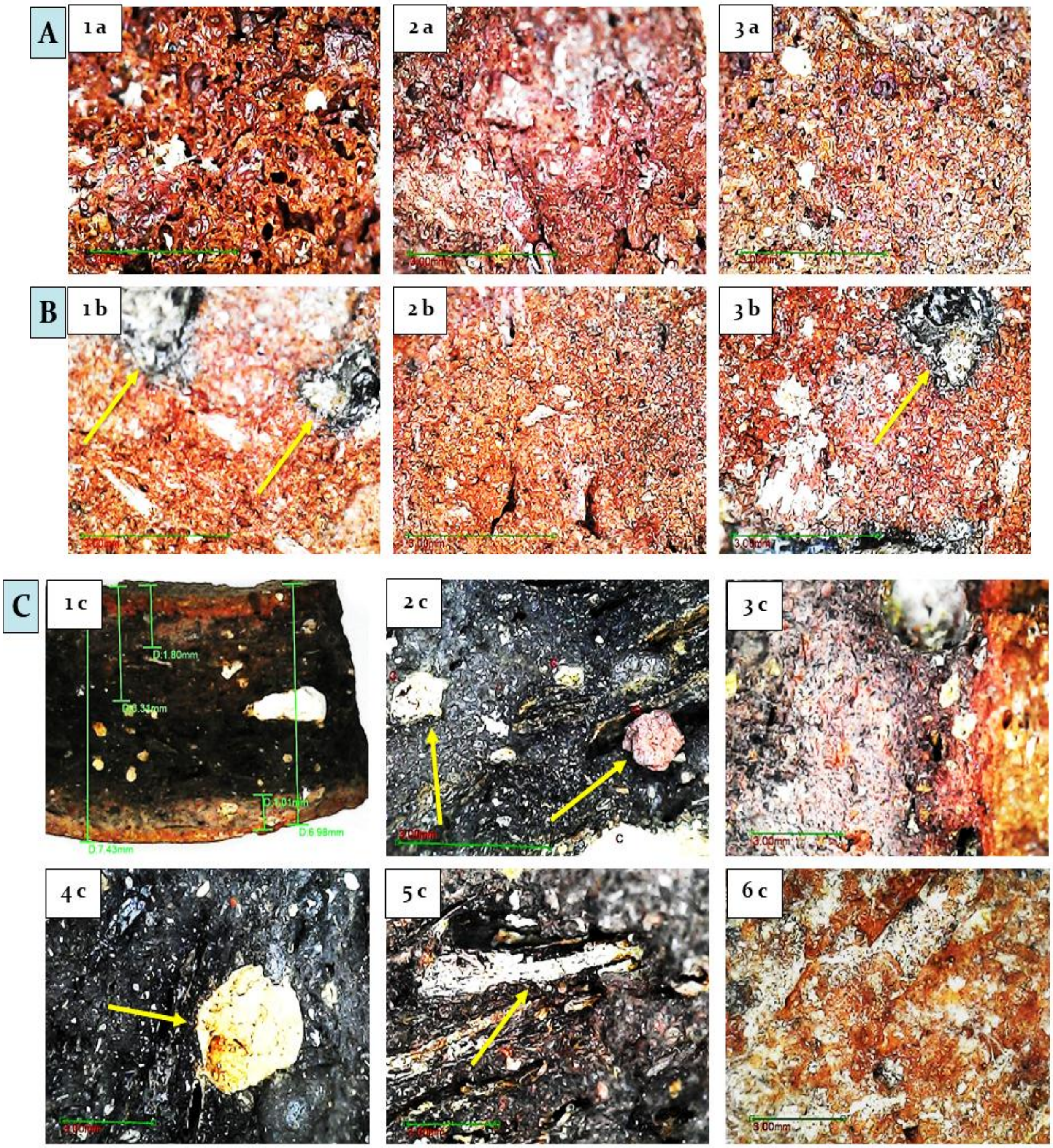


Fig.3. The investigation results of the samples by USB digital microscope. (A) Red brick from the lower part of the south wall, (B) Red brick from the north upper part of the small water tank, (C) The pottery water pipes used for the transportation of water among tanks.

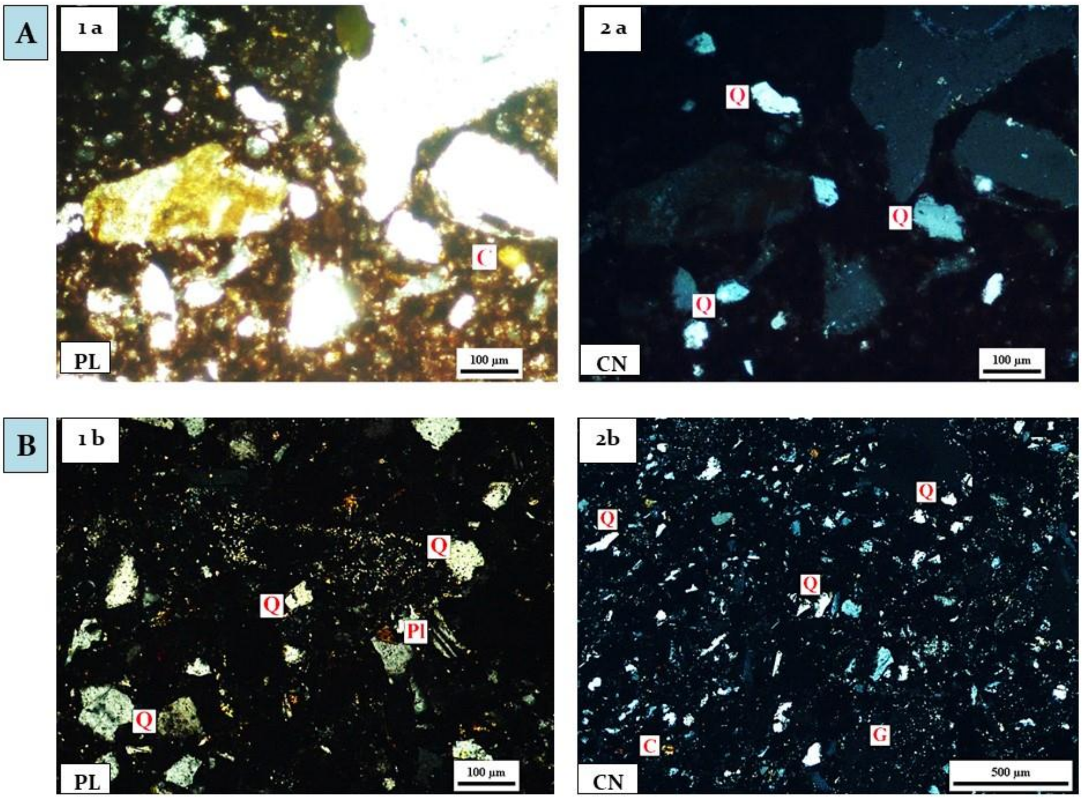


Fig.4. Photomicrographs of thin sections of red brick sample1 shows the microfabrics of grains, (A) Calcite and quartz for sample (1), (B) Quartz, calcite, and Plagioclase is inside an iron-rich fabric in sample 2.

The presence of calcite is an evidence of the addition of lime or limestone gravels as an inorganic modified and temper materials for clay [24].

On the other hand, the results of the petrographic study of pottery water pipe sample (Fig.5) (1, 3, 5) with parallel nichols (2, 4, 6) with crossed nichols, the texture of sample is rich in quartz(Q)but in different shapes and sizes. The heterogeneity of the components and the angular shape of quartz granules was observed [25]. Most pure clays consist of a large percentage of silica (SiO_2) [26]. This silica reduces the plasticity of the clay and the rate of shrinkage upon burning. The size of the particles of silica plays an important role, as the fine particles react at a low temperature, while the large particles increase the thermal resistance of the clay [27].

The detection of calcite (C) granules, which it may have been added to improve the physical properties of the pottery product, or it may be already among the components of the clay raw material [28]. Calcium compounds have many functional effects on the production of pottery, as they act as flux materials by combining with silica and alumina to form a liquid at a low melting point, thus reducing the degree of glazing and the thermal resistance of the clay. Also, the resulting phases freeze upon cooling and forming strong bonds with an impermeable hull. Calcium compounds also reduce shrinkage and facilitate drying up at low temperatures [39]. Additionally, the sample contains plagioclase (PL) and muscovite(M). These minerals may have been intentionally added by the manufacturer to improve the properties of the produced pottery, or they are natural impurities present in the used clay in the industry [25, 30].

As for grog (G) is crushed earthenware material which clearly appeared in the sample of pottery pipes. It is one of the most important inorganic materials that is intentionally added to clay by the potter to change the clay's workability or firing properties [31].

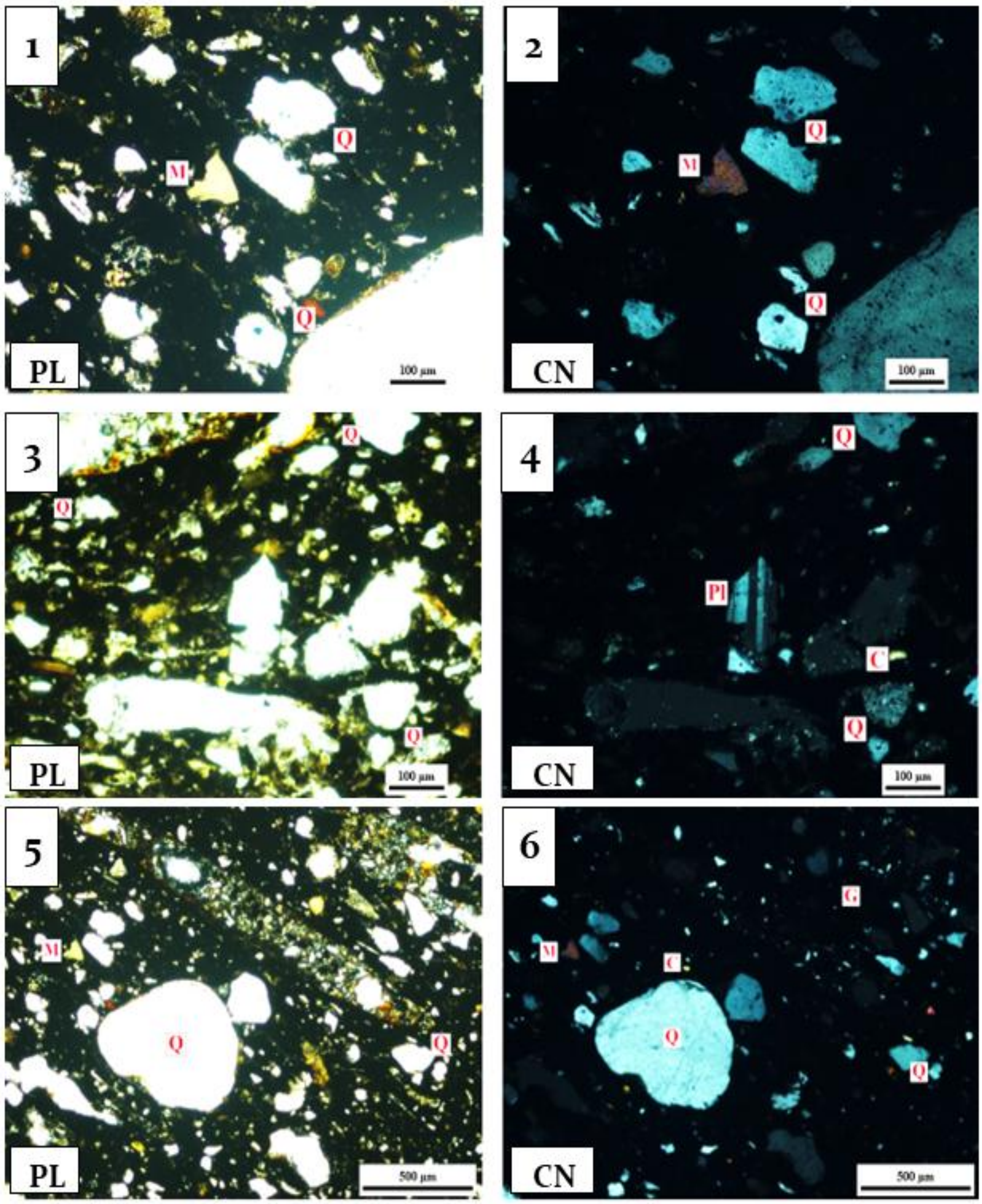


Fig. 5. Photomicrographs of thin sections of pottery water pipes show quartz(Q), calcite (C), plagioclase (PL), muscovite(M) and Grog(G).

3.3. Powder X-ray diffraction (XRD)

The semi-quantitative analysis of the mineralogical components of the red brick sample (sample 1, Fig. 6A) revealed that it consists of orthoclase (37.5%), mullite (24.6%) quartz(19.3) %, spinel (7.5%), hematite (7.2%) and halite (4.0%). As for the sample of the red brick from the north upper part of the small water tank (sample 2, Fig. 6B), it revealed the same minerals, but in different ratio as it consists of orthoclase (30.1%), mullite (25.9%), spinel (17.5%), quartz (13.7%), hematite (8.3%)and halite(4.5%).The obvious appearance of orthoclase and quartz in all samples is a result of the stability of quartz until approximately 1150°C (change from the phase α to β takes place at approximately 570 °C). K-feldspar (orthoclase a mineral of this group) is stable until 1000-1050 °C [32]. As for the minerals of mullite and spinel, they are important minerals resulting from the firing effect on clay components. At high burning temperatures above 600°C, the clay minerals undergo significant changes in the chemical and mineralogical composition. Kaolinite at more than 500 °C converts to metakaolin $Al_2O_3.2SiO_2$, which is slightly crystalline, so it does not appear when analyzed by XRD. Metakaolin decomposes at about 900°C to form spinel $2Al_2O_3.3SiO_2$. At 1050-1275°C the spinel turns into mullite $3Al_2O_3.3SiO_2$. So, the mullite could not be detected before 1100°C [25]. Moreover, the presence of hematite Fe_2O_3 is a result of iron oxides as most of the earthen clays contain a high percentage of these impurities, which is responsible for the reddish-brown color of the products after firing [10].The presence of halite, sodium chloride (NaCl) is expected as chlorides are among the most common salts in historic building. The source of these salts may be from the soil, and they move to the building materials by the underground saline water solutions [33] (Fig. 6A and B).

On the other hand, the XRD result of the pottery water pipes sample was slightly different (Fig. 6C), due to a presence of Calcite ($CaCO_3$) (8.1%). The detection of calcite may be related to the low firing temperature of water pipes, as calcite is only decomposed at temperatures above 750^0C. So, this detection of calcite indicates that the initial firing temperature of this sample did not exceed this temperature, when it is compared with the other two samples of bricks. Calcite may be attributed to a deliberate addition added as a filler to decrease the clay plasticity or it is natural impurity present in the clay raw materials [19, 34]. Also, many minerals were detected such as, orthoclase (36.5%), diopside (26.5%), quartz (15.2), hematite (10.1%) and halite (3.5%).

3.4. Scanning electron microscopy (SEM)

SEM photomicrographs showed the microstructural differences between the red brick samples and pottery water pipe sample. These differences are attributed to nature of raw materials and the effect of firing process on clay matrix. The red bricks grains homogeneity, the slight porosity, and the high amount of formed glassy phases indicate the high firing temperature used for the manufacture of bricks [29] (Fig. 7a. b).

The SEM micrographs of the pottery water pipe sample (Fig. 7 1c,3c) showed that the body contains gruff quartz with un-melted grains. The prevalence of voids and the appearance of chopped straw residues confirms the use of this organic additive for improving the properties of the clay during manufacturing [22] (Fig.7 3c). Also, this indicates that the burning process was not sufficient to get rid of the added organic materials [35].

3.5. Chemical analysis by (EDX)

The EDX analysis results of red brick samples (Fig.8 and Table 2) showed the detection of high percentage of (Si, Al) elements, as well as (Na, Mg, Cl, K, Ca and Fe) elements. These elements reflect the chemical oxides commonly found in bricks like silica (SiO_2), alumina (Al_2O_3), iron oxides (Fe_2O_3), (Fe_3O_4), potassium oxide (K_2O), sodium oxide (Na_2O), calcium oxide (CaO) and magnesium oxide (MgO). Silica and alumina constitute the base elements of clay and are usually found in the proportions of about 50% of SiO_2 and 15–20% of Al_2O_3. The detection of a very small percentage of calcium oxide CaO is due to impurities in the clay or the

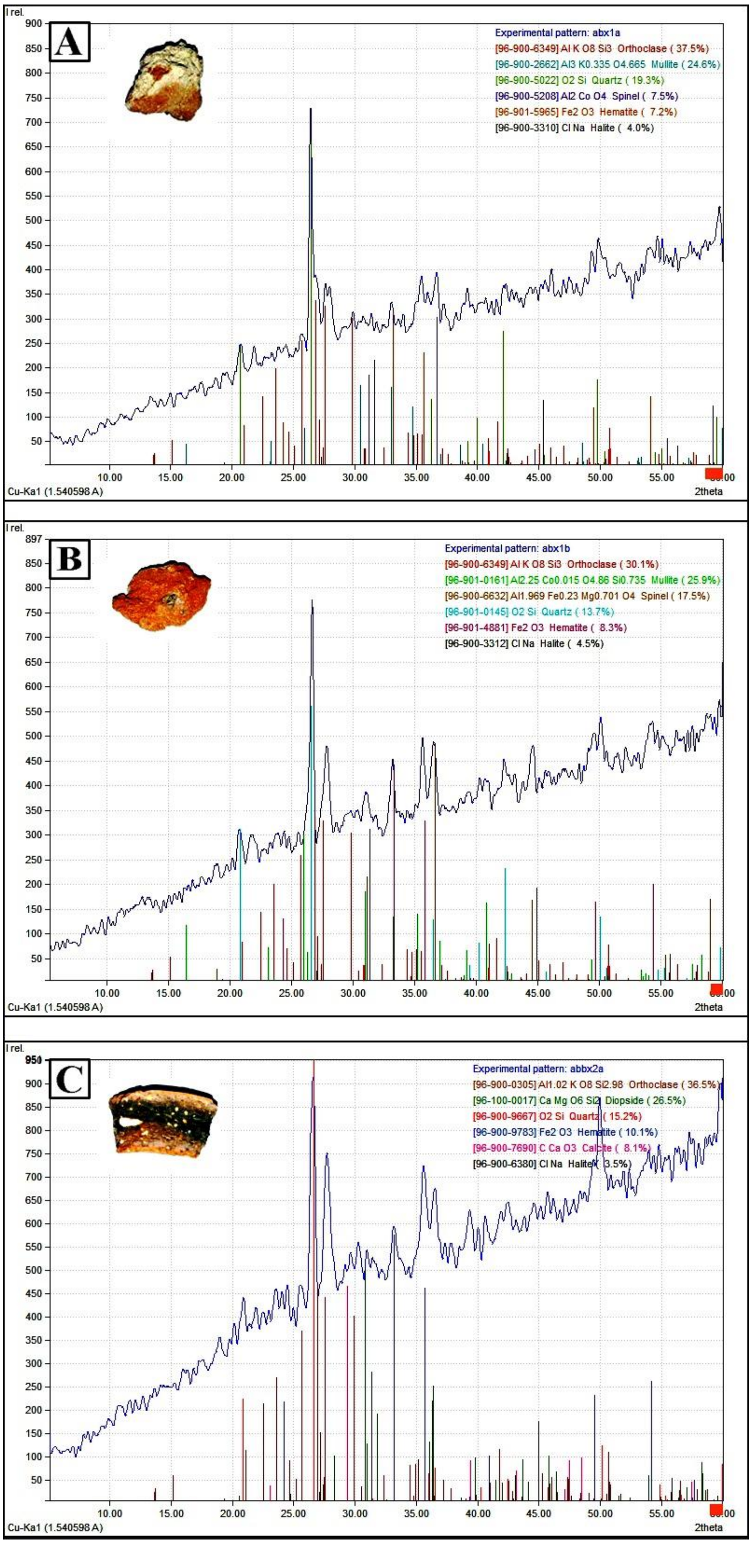


Fig.6. XRD patterns of the studied samples, (A) Red brick sample from the lower part of the south wall, (B) Red brick sample from the north upper part of the small water tank, (C) The sample of the pottery water pipes used for the transportation of water among tanks.

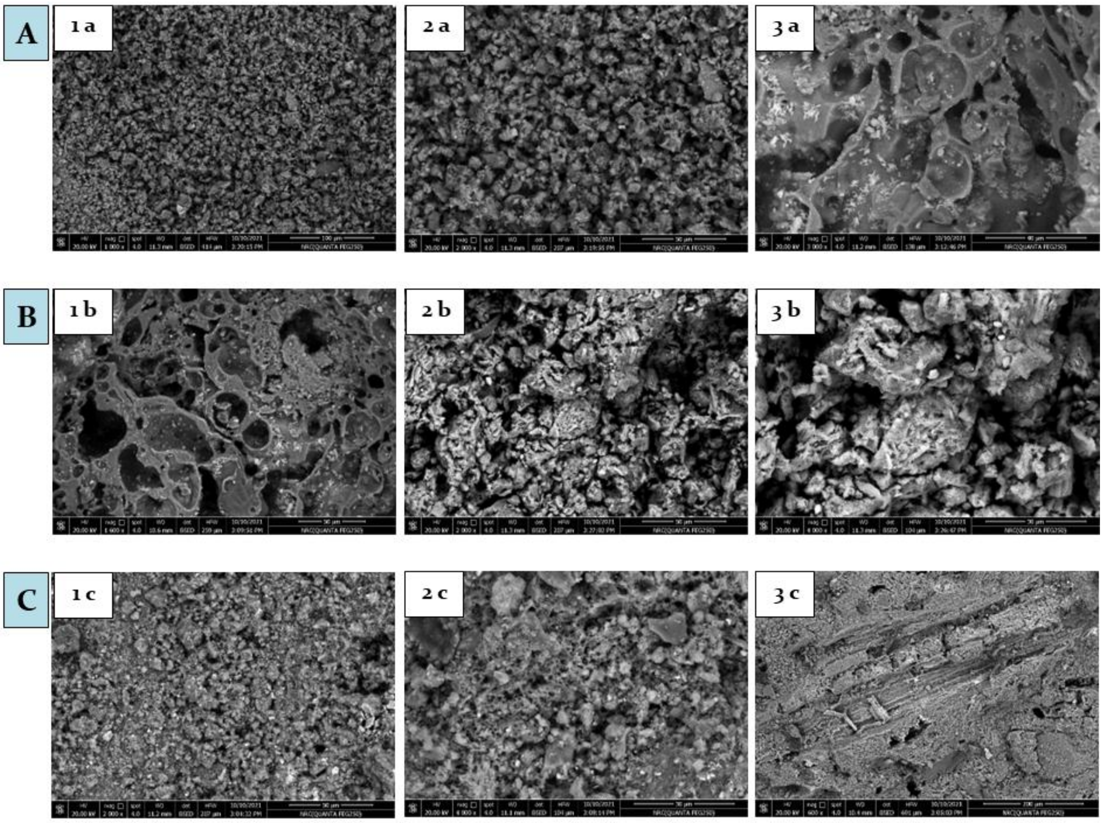


Fig. 7. SEM micrographs of the samples, (A, B) Red brick ,(C) Water pottery pipes.

contamination by lime mortars used in the building [36]. Moreover, the detection of sodium (Na) and chlorine (Cl) elements in the samples reflects the presence of halite (sodium chloride). While the detection of Fe element confirms the presence of iron compounds which are responsible for the reddish and the brownish color of the bricks after the burning process [37].

As for the elemental analysis of the pottery water pipes sample 3 it gave the same results as that of the two brick samples, but with different percentages of some elements and the appearance of titanium (Ti). The percentage increase of calcium (Ca) element confirms the presence of calcite mineral which was added to improve the mechanical properties of the clay after firing [38]. Also, it reflects the incomplete burning of pottery water pipes, especially in the inner core of the pottery pipes. The aforementioned elements indicate that the clay used in the manufacturing of bricks and pottery pipes was taken from the Nile River [39]. Moreover, results of EDX analysis confirm the previous findings.

4. Conclusion

The archaeometric study of the representative samples of red brick and pottery water pipes enriches the study with quite interesting information about their chemical composition and the raw materials used for their implementation. The digital microscope gave some valuable results about the homogeneity of red bricks, their richness in iron oxides and the additives of limestone gravels. Also, it revealed that the core of pottery pipes is not well burnt because of its black core.

The petrographic study of red brick samples revealed the presence of quartz, calcite and plagioclase. Obviously, the presence of calcite additive in pottery pipes reflects the incomplete burning of them.

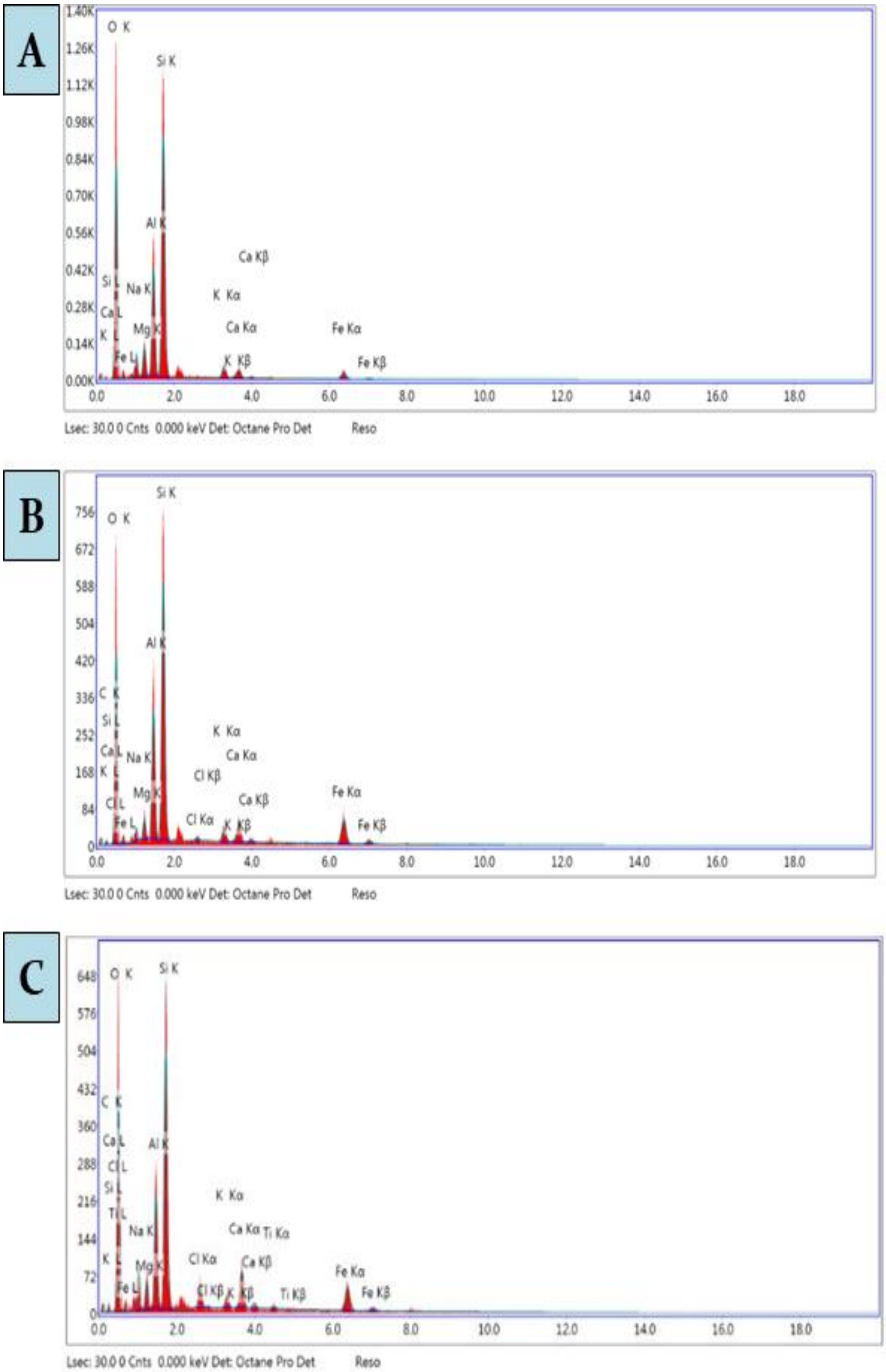


Fig. 8. EDX analysis results of the studied samples. (A, B) EDX spectra of the samples 1&2 of red bricks show that they consist of Si, Al, Na, Mg, Cl, K, Ca, C and Fe elements. (C) The sample of water pipes showed the same elements as the burnt bricks in addition to a high percentage of Ca element.

Table 2. EDX atomic weight percentage analytical results of the red bricks and pottery water pipe samples.

Atomic weight percentage of the elements in the samples											
Analyzed Sample no.	C	O	Na	Mg	Al	Si	Cl	K	Ca	Ti	Fe
1	5.44	57.45	2.17	1.56	8.62	16.51	1.21	1.88	1.57	--	3.49
2	5.76	56.47	1.57	1.79	7.97	18.23	1.36	1.77	1.37	--	3.71
3	6.81	54.32	5.24	2.04	6.25	12.26	1.21	1.7	6.4	1.15	2.64

The semi-quantitative analysis by XRD of the mineralogical components of the red brick samples and pottery pipes revealed that they consist of orthoclase, mullite, quartz, spinel, diopside, hematite and halite. Calcite is obviously detected in pottery pipes.

SEM micrographs showed the microstructure of the studied red brick, as they gave valuable information about the used raw materials, the porosity and the glassy phases which reflects the perfect burning process of most parts, moreover, they confirm the incomplete burning process of water pottery pipes because of the appearance of organic chopped straw parts and voids, as well as calcite gravels. One of the most important results, is that the EDX results are in agreement with the other results to confirm that Nile clay is the resource of clay raw materials used for the manufacturing of the studied red bricks and water pottery pipes.

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