Historical and Archaeological Study of Nefer-Seshem-Ptah and *šḫntἰw* Tomb at Saqqara Necropolis

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[Abstract]

This study reports the first analytical study conducted on samples of bedrock, and plaster layers, and also painting techniques employed by the ancient craftsmen. Collected from Nefer-Seshem-Ptah and *šḫntἰw* tomb, the unique construction at Saqqara necropolis in Egypt, the samples, which comprise the microscopic particularities, were assessed by digital optical microscope, while the petrographic analysis was performed by polarized light microscope. An X-ray diffractometer was used to analyze the bedrock and plaster layers samples. Further, the molecular and vibrational attributions of some fragments and pigment grains were recognized using Fourier Transform Infrared Spectrometer and Raman Micro Spectrometer (μ-Raman). As a result, the studied bedrock indicates that calcite and quartz are the main minerals, whereas halite is revealed in other samples. In addition, assemblages were identified as clay mineral including montmorillonite and kaolinite. Further, the thin-section observation revealed a clayey fossiliferous and micrite. The observation on the plaster layers formed of coarse gypsum includes little amount of limestone powder, together with fine sand, while the second layer weas composed mainly of a clay plaster with straw plants. The cross-sectional observation on the plaster layers defined three layers, which are mainly made up of calcium carbonate (calcite), quartz and clay. Minerals were identified in varying proportions. Likely, Fourier Transforms Infrared Spectroscopy analysis of the samples disclosed that the craftsmen used animal glue as media to decorate the tomb, which means the "Tempera" Technique was used to decorate Nefer-Seshem-Ptah and *šḫntἰw* tomb.

Keywords: Saqqara; bedrock, mural painting; plaster layer; fossiliferous, animal glue

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1 Introduction

Saqqara is one of the most important and famous archaeological sites around the world (Odah, Abdallatif et al. 2005), It was capital of ancient Egypt and the religious capital of the Saqqara area including a distinct collection of fifth dynasty tombs from 2494-2345 BC (Bárta 1983, Kanawati 2013, Kuraszkiewicz 2014). The tomb of the Craftsmen is one of the typical tombs of middle-class officials in the fifth dynasty, and one of the tombs that contribute to the study of development of tombs at Saqqara because of its archaeological, historical, and social value (Hölscher and Munro 1975, Moussa, Johannes et al. 1975). The present research introduces the first analytical study conducted on the building and painting materials from the Nefer-Seshem-Ptah and *šẖntiw* tomb. Therefore, the outputs of this work will enrich our related knowledge with comprehensive data on material characterization of bed rock, bedding mortar and plaster layers. In addition, these results will help in determining the appropriate procedures and materials for future conservation intervention of the tomb.

2 A Brief History and Construction of Nefer-Seshem-Ptah and *šẖntiw* Tomb

Nefer-Seshem-Ptah and *šẖntiw* tomb dates to the period of the Old Kingdom between the end of the fifth dynasty and the beginning of the sixth dynasty (2392-2282 BC) (Kanawati 2013) (Fig. 1-4). The tomb was discovered in November of 1964 during joint excavations between the German Institute of Antiquities and the Egyptian Department of Antiquities in the southern depression 290 meters south of king Unas causeway (Moussa 1975, Moussa and Altenmüller 1977). The tomb is known as tomb of Craftsmen, and it belongs to two owners, one named (*šẖntiw*) and the other Nefer-Seshem-Ptah. *Nfr-sšm-Ptḥ* is the overseer of steal-workers, and gold smith, while *šẖntiw* was the father of Nefer-Seshem-Ptah, and he was the supervisor of the gold makers (Moussa 1975).

Fig. 1 An aerial view of Nefer-Seshem-Ptah and *šẖntiw* tomb (Google Maps)

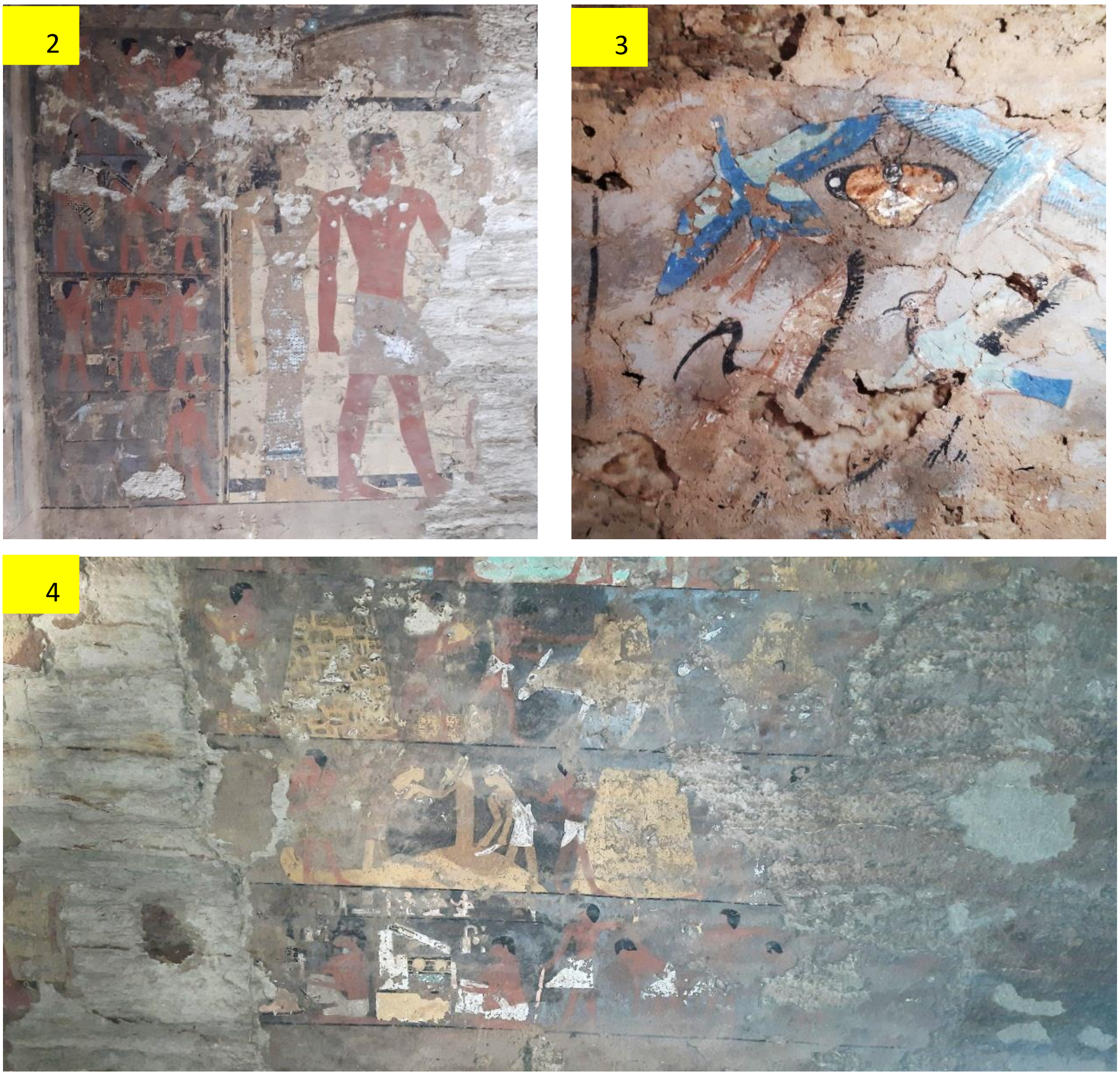


Fig. 2-4 Archaeological description of the mural paintings in Nefer-Seshem-Ptah and *šḫntỉw* tomb

The artist hewed in the bed rock to form false doors on the western wall of the tomb while applying plaster layers on the other three walls. The paintings on the eastern wall appeared well, showing a homogeneous spread of color with an interior paint. Hieroglyphics, some scenes of daily life and offerings were also painted. The mural paintings were colors of blue gray as a background.

3 Materials and Methods

3.1 Sampling and Sample Preparation

For the present study, the samples of the bedrock and mortars (gypsum-clay), their bulk samples and fractions were separated from the marl paintings (Fig. 5).

3.2 Analytical Investigation Techniques

- Optical Microscopy (OM)

The samples were observed using Optical Microscopy. The magnification varied from ×100 to ×250 depending on the size of the samples. Optical microscope was used to study the stratigraphy and topography of the mural painting including the surface characteristics of the paint, the presence of pigment mixtures, and the thickness of paint layers.

- Polarized Light Microscope (PLM)

Thin and cross sections prepared on some bedrock and plaster samples were examined with a Nikon Eclipse-E600 Polarized Light Microscope (PLM) coupled with a PixeLINK PL-A623 digital camera. The thin sections were studied at the Department of Geology of Cairo University.

- X-ray Diffraction Analysis (XRD)

X-ray Diffraction Analysis (XRD) was used to affirm the contained crystalline minerals (Duran, Perez-Rodriguez et al. 2011). The samples were analyzed with a X'Pert Pro PANalytical – Manufactured by Panalytical B.V Co., Netherlands (ISO 9001/14001 KEMA – 0.75160), model diffractometer equipped to a monochromatic Cu. The scanning speed was 2θ 4 1degree/min at operating conditions of 40 kV and 30 mA).

- Raman Spectroscopy

Raman Spectroscopy is considered one of the most powerful methods for studying mural painting. It allows rapid identification of the compounds at the micrometer scale without preparing the samples (Rosalie David, Edwards et al. 2001, Mahmoud 2013). Raman spectra of the pigments were collected using Senterra spectrometer (Bruker) coupled to a confocal Raman spectroscopy (20x - 100x objective lens).

- Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy has been a workhorse technique for materials analysis in the laboratory for over seventy years (Stuart 2007). FTIR requires a small sample about 0.5 mg and it fast analyzes the sample without need for further preparation. It characterizes different molecular groups. Although several attempts for binding media analyses are used to identify both organic and inorganic materials.

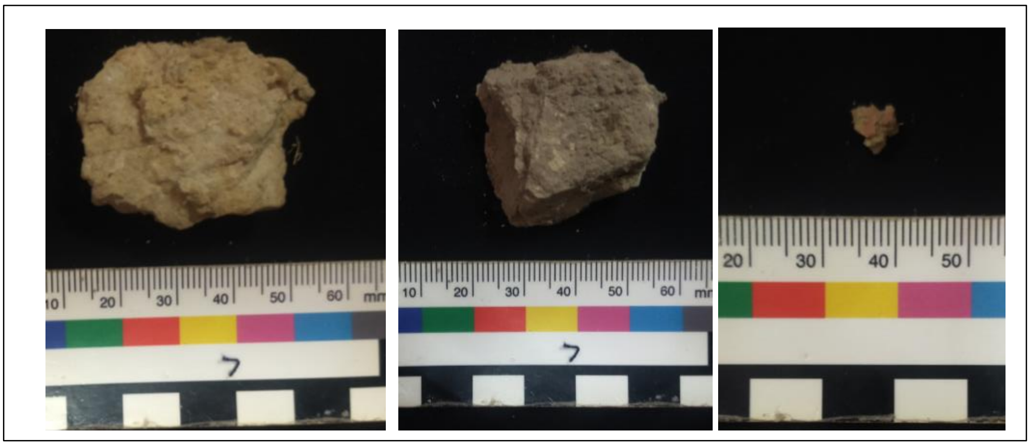


Fig. 5 Photographs showing preparation samples of the bedrock and mortars that were separated from the marl paintings

4 Results and Discussion

4.1 Petrography of the Bedrock:

Geomorphologically, Nefer-Seshem-Ptah and *šḫntiw* tomb is within the rocky layer that is geologically known as the formation of a Saqqara (Kuraszkiewicz, Trzciński et al. 2010, Scott 2016). The tomb was cut into the rock (Kanawati 2013) at the lower terrace of the valley (Edwards and Baines 1988, Strudwick and Strudwick 2011) in fragile sedimentary layer of Marl with small layers of gypsum which are irregular or lenticular shaped (Moussa, Johannes et al. 1975, Akarish 2010).

The X-ray analysis of the bedrock of wall and roof indicated that calcite ($CaCO_3$) and quartz (SiO_2) are the main minerals recorded in samples ,whereas halite ($NaCl$) is revealed in some samples, in addition to assemblages of clay mineral identified include montmorillonite ($Na_x(Al,Mg)_2Si_4O_{10}(OH)_2H_2O$), and kaolinite ($Al_2(Si_2O_5)(OH)_4$ which were found in studies of Upper Eocene sediments (Fig-6).

The thin-sections of the bedrock revealed a clayey fossiliferous micrite, micrite matrix commonly recrystallized into microsporidia, marlstone lithotype (Folk 1959, Kendall and Flood 2011) (Fig.7).

4.2 Petrography of the Plaster layers

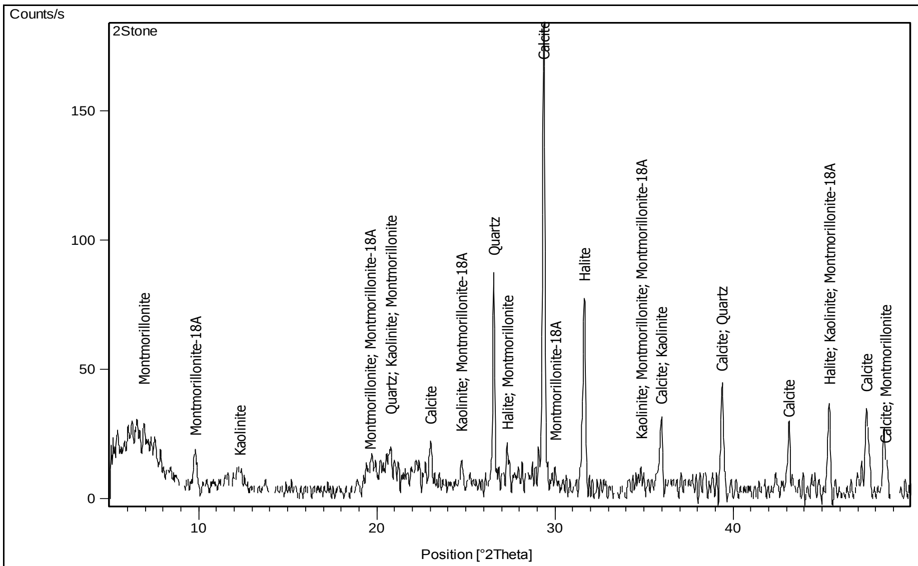
Plaster layers were applied on the bedrock walls after it had been paved (Lucas 1995). Artist applied the plaster layers to hide cracks and stone defects in the bedrock (Ambers 2004) to pave the surface (Abd El Salam 2001). The plaster was usually consisted of two layers (Ewais 2018), may reach three thin layers (Uda 2004).

The SEM micrograph of plaster layers shows hard crust, a dense coat of partly halite crystals and the prism, platy crystals of gypsum are notably scattered on. The growth of microorganism can also be seen (Fig. 8 A-B)

The thin sections of the white mortar was inspected with polarized light microscope (PLM) (The first layer, in the bottom) the coarse layer revealed a main microfacies association. So, it is thought that powder polishing of limestone from the tomb was mixed with gypsum and sand to prepare the plaster.

The cross-sections on the plaster layers presented a solid successive structure in three layers. The first layer at the bottom is coarsely heterogeneous with various components. There is sometimes a white layer and sometimes not. The second one is the intervention layer, where high content of the binder (clay minerals) can be seen, compared to the aggregates. The third layer at the top is semi fine clay coat accompanied with siliceous matters and straw plants (Fig. 8C-D).

EDX analysis shows that silicon, calcium, and aluminum are the major components, whilst sodium, sulfur, iron and chlorine are the secondary elements. In addition, there are also traces of magnesium. From the XRD, EDX and FTIR analyses, it is shown that the rendered samples contain major amounts of gypsum (dehydrate phase, $CaSO_4 \cdot {}_2H_2O$) with traces of calcite; calcium carbonate ($CaCO_3$), clay minerals such as kaolinite ($Al_2Si_2O_5(OH)_4$), quartz (SiO_2) and halite(Na Cl) (Fig. 9-10).



Compound	Chemical Formula
Calcite	Ca C O3
Quartz	Si O2
Halite	Na Cl
Kaolinite	Al2 Si2 O5 (O H)4
Montmorillonite	Nax (Al, Mg)2 Si4 O10 (O H)2 ·z H2 O

Fig. 6 XRD pattern and the main components of the bedrock, illustrating the presence of calcite and quartz, halite and clay minerals

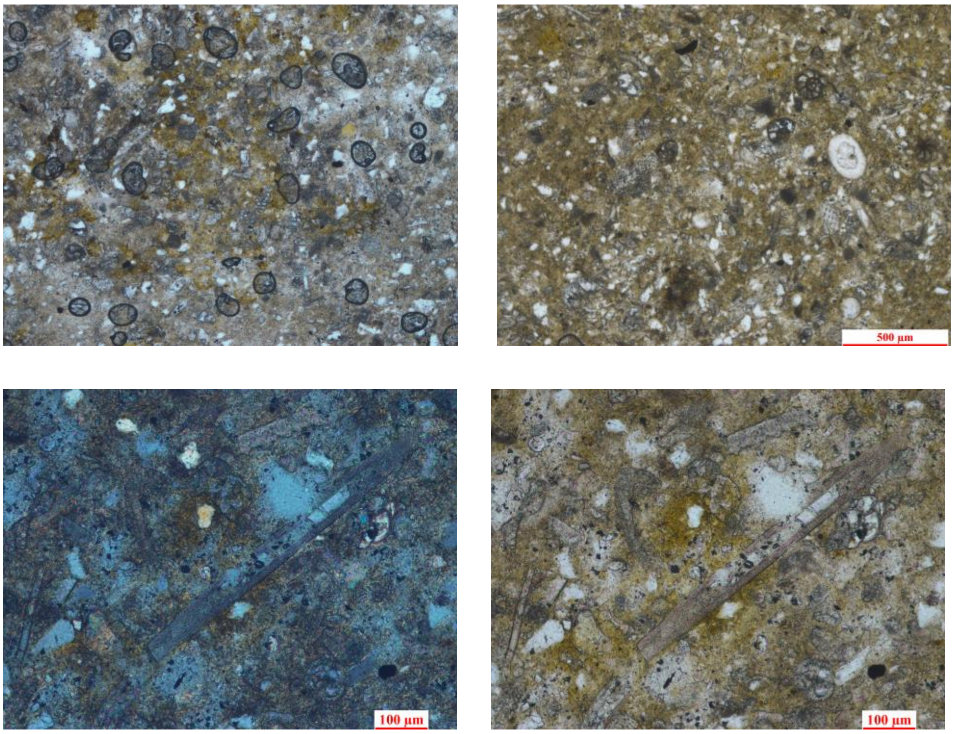


Fig. 7 Micrograpgic Photos showing the main components of bed rock

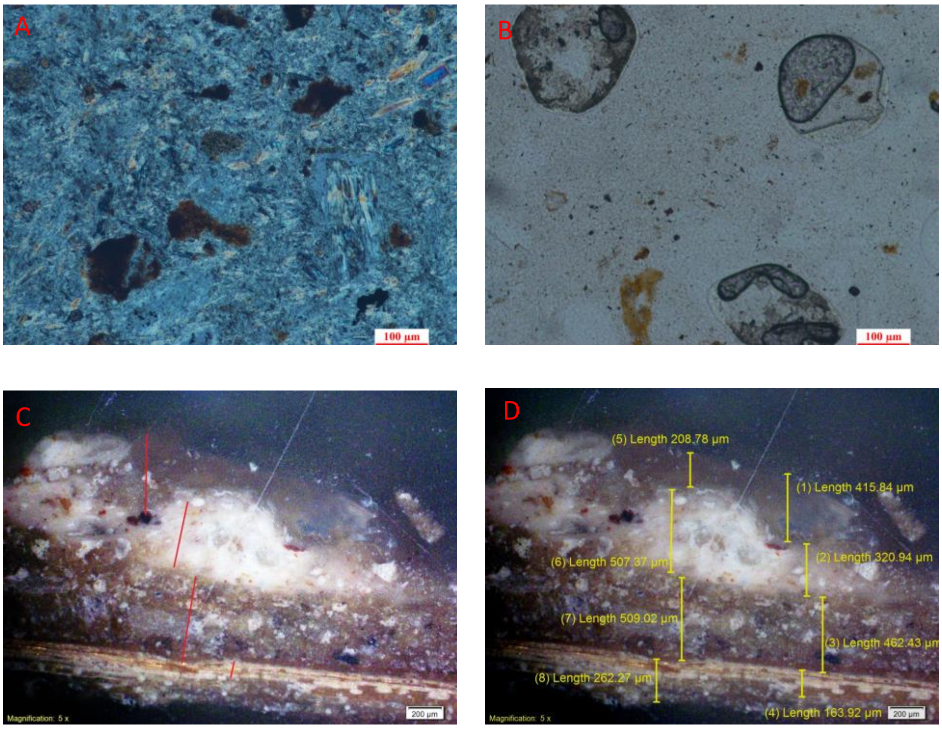
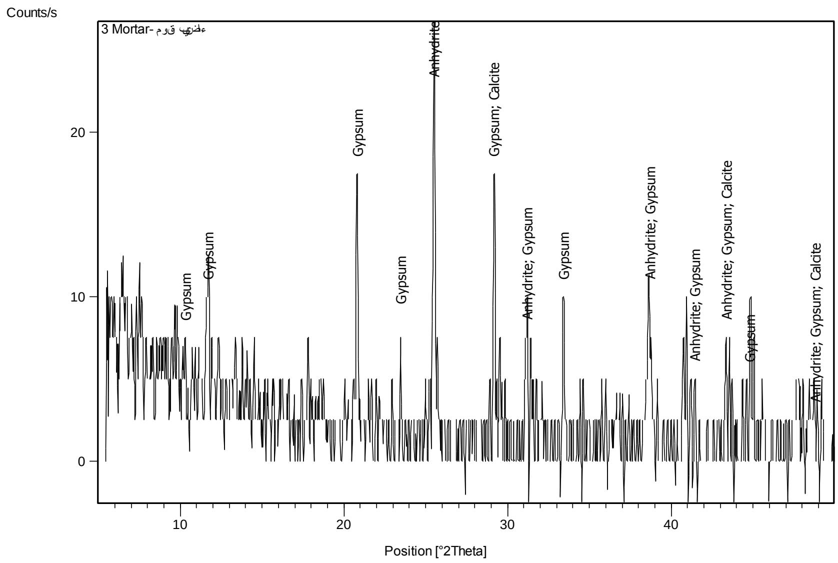
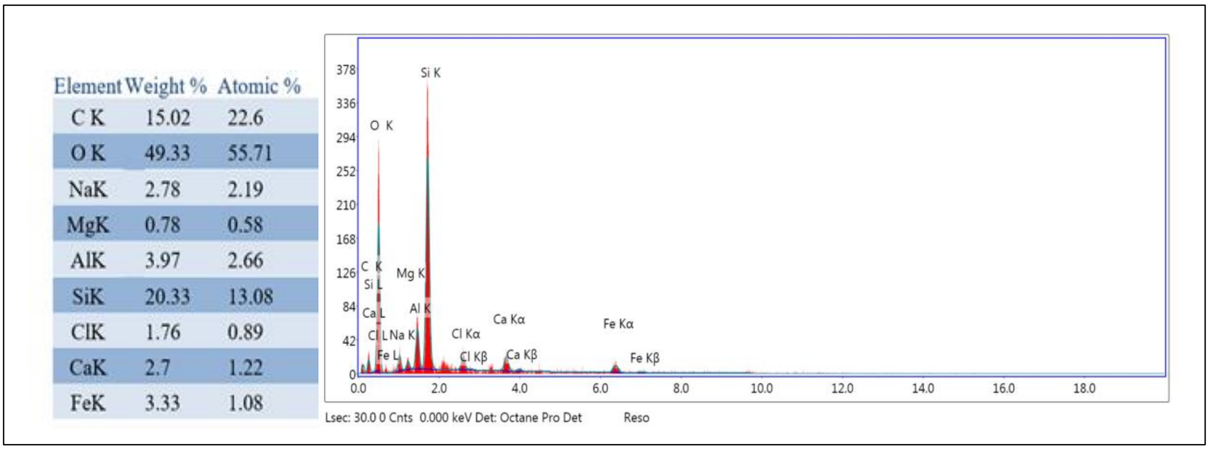


Fig. 8A-B The thin sections of the white mortar inspected with polarized light microscope (PLM) (The first layer in the bottom), the coarse layer revealing a main microfacies association and fossiliferous (Fig. 8C-D), the cross-sections on the plaster layers presenting a solid structure with differentiate three layers



Compound Name	Chemical Formula
Anhydrite	Ca S O4
Gypsum	Ca S O4 (H2 O)2
Calcite	Ca C O3

Fig. 9 the XRD pattern showing the main components of the white mortar



Element	Weight %	Atomic %
C K	15.02	22.6
O K	49.33	55.71
NaK	2.78	2.19
MgK	0.78	0.58
AlK	3.97	2.66
SiK	20.33	13.08
ClK	1.76	0.89
CaK	2.7	1.22
FeK	3.33	1.08

Fig. 10 The EDX analysis of the mortar sample showing that silicon, calcium, and aluminum are the major ions contained, while sodium, sulfur, iron and chlorine are the secondary elements, and traces of magnesium

5 Mural Painting Techniques

Craftsmen in ancient Egypt used many techniques for decorative mural paintings with the daily life scenes and offering scenes (Ewais 2018). It is known that the technique of mural paintings is distinguished according to the kind of media (Elphtah 2019). Ancient craftsmen used two main types of mural paintings techniques (Bard 2005). Tempera Technique differs according to the binding from Fresco Technique that is applied on wet lime plaster. The ancient Egyptians usually used Tempera Technique in their tombs. In Tempera Technique, binding media is usually used (Lucas 1995). Pigments were grinded and mixed with binder in order to fix pigments on the plaster (Angenot and Tiradritti 2016). The Tempera Technique differs in using the media of a prevalently proteinaceous medium based on egg (albumin- yolk) (Schadler 2017), and animal glue or polysaccharide constitutions such as Arabic gum (Aruga, Mirti et al. 1999, Ewais 2018).

Those organic matters can be identified by using different methods such as direct infrared (IR). Four samples of the plaster were chosen to analysis. The samples revealed as organic binder were obviously identified in the paint samples. These findings suggest that "Tempera" Technique, and most probably the tempera was used to decorate Saqqara tombs.

Through comparing the result of samples from the chosen pigments to the FTIR binding media standards, it was obvious that the binding media used at the mural painting was animal glue and that it goes well with the results from other places in Saqqara such as burial chamber of Ra-Shepses (Ewais 2018), the tomb of Idout (Akarish and Shoeib 2011) and the tomb of Ptah-Shepses (Elphtah 2019).

Animal glue is characterized by the existence of amid group (–NH–CO–) at 3398 - cm^{-1} which is assigned to N–H stretching. A weak band at 2991 cm^{-1} is assigned to C–H stretching (methylene groups). The existence of the band at 1636,1620cm^{-1} is also attributed to amide I groups. There is also a band at 1418 cm^{-1} indicating amide III groups. It was suggested that the use of a proteinaceous binding medium tis probably animal glue (Martin de Fonjaudran, Nevin et al. 2008, Salvant, Williams et al. 2018) (Fig. 11-13).

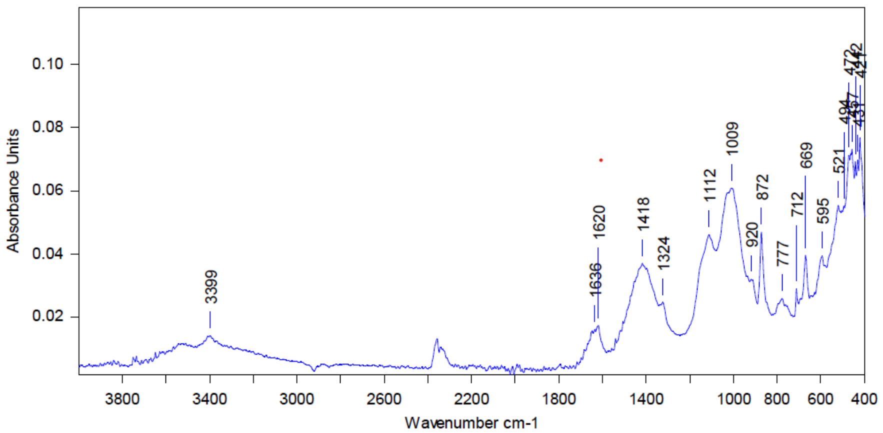


Fig. 11 FTIR spectrum (in the mid-infrared region) suggesting the use of a proteinaceous binding medium is probably animal glue

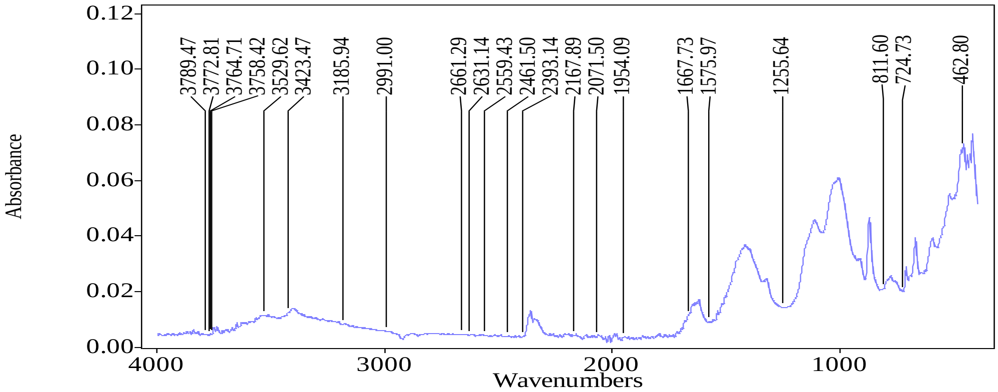


Fig. 12 FTIR spectrum (in the mid-infrared region) showing the use of animal glue as binding medium

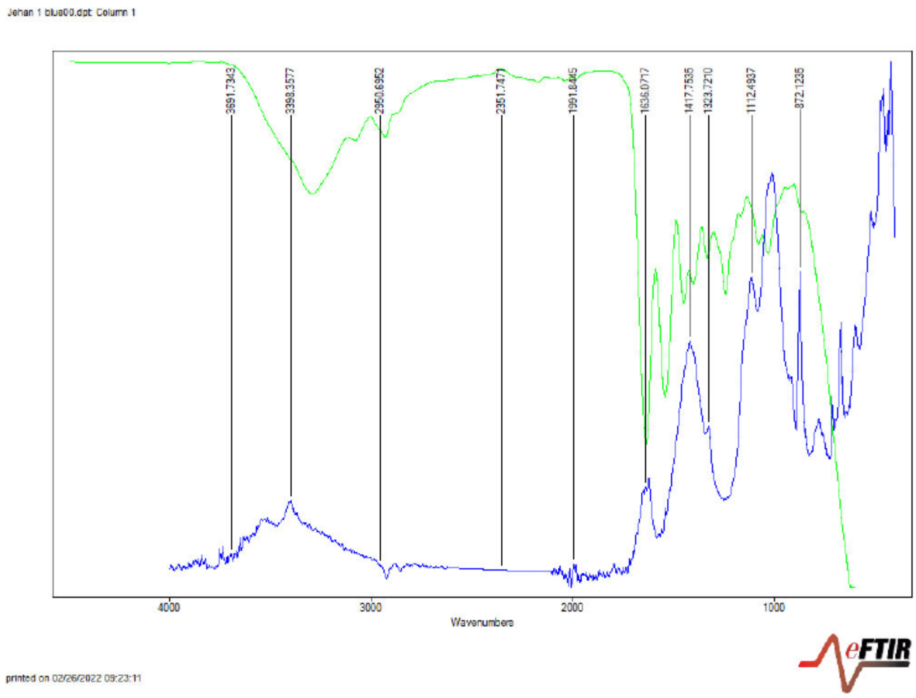


Fig. 13 FTIR spectrum (in the mid-infrared region) plaster layer, compared with a reference spectrum of animal glue

6 Conclusions

Nefer-Seshem-Ptah and *šḫntiw* tomb represented an elegant architectural design. In the present contribution, the diversified analytical methods of PLM, XRD, FTIR and μ-Raman were used to characterize the bedrock, plaster painting and painting technique. The microscopic inspection of the plaster layers gave a clear indication on their stratigraphic profile. The polarized light microscopy assisted us to acquire data concerning the petrographic distinctions of the samples. The XRD analysis carried out on the bedrock samples showed that calcite and quartz were the main minerals in the samples, whereas halite was revealed in some samples, in addition to assemblages of clay mineral including montmorillonite, and kaolinite. Further, the thin-section observation revealed a clayey fossiliferous and micrite. The observation on the plaster layers showed that it was formed of coarse gypsum, in which there is little amount of limestone powder, together with fine sand, and the second layers were composed mainly of a clay plaster with straw plants. The cross-section observation on the plaster layers defined three layers, which are mainly made up of calcium carbonate (calcite), quartz, and clay minerals. Furthermore, craftsmen used Tempera Technique in mural painting of Nefer-Seshem-Ptah and *šḫntiw* tomb. FTIR analysis has confirmed animal glue as medium.

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