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A Multi-Analytical Study of the Archaeometric Aspects and Restoration of Some Ancient Roman Pottery Artifacts in Sinai, Egypt

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ABSTRACT

Tell El Luli is an important archaeological site in North Sinai. It holds pottery artifacts and Roman baths built of red brick dating back to the Roman period. The North Sinai excavation missions by the "Ministry of Antiquities in Egypt" discovered many pottery artifacts. Diagnostic investigations were conducted on the pottery shards samples from Tell El Luli to determine the matrix microstructure aspects and their various characteristics. Multi-analytical methods, such as stereoscopic microscope, polarized microscope, scanning electron microscope (SEM- EDX), X-ray diffraction, and X-ray fluorescence, were used. Based on the diagnostic investigations, the research demonstrated that the raw material was Nile clay, the additives were grog, added sand, dolomite powder, and burnt straw, and the surface treatment was slip layer and red wash. Additionally, it was proven that the burning atmosphere was an oxidizing and reducing atmosphere. Pottery suffered from severe damage, such as crystalline salts, cracks, gaps, soiling accumulations, calcifications, dirt accumulations, flaking, weak structure, and fragility. The pottery artifacts were restored, and the soil accumulations were cleaned mechanically and then chemically. The salts were extracted using various poultices. Thus, consolidation was conducted by nanosilica (0.5%). The assemblage process was carried out using Paraloid B72 at a (50%) concentration.

Keywords: Clay, additions, burning, matrix structure, damage, gaps, peeling, treatment

1. Introduction

Sinai is located on the continent of Asia, containing many archaeological sites from the prehistoric period to the Islamic ages. It connects Africa and Asia and covers an area of 60 thousand km^2; it is bordered by the Mediterranean Sea in the north, the Red Sea in the south, the Gulf of Aqaba in the east, and the Gulf of Suez in the west. It was known in the historical past ages as the land of turquoise, where Egyptian or foreign missions discovered some turquoise and copper mines. Sinai has had historical, religious, and military importance throughout different historical ages. It contains archaeological sites from different eras. The texts of the Deir El-Bahari temple refer to Sinai, especially Horus War Road (Nour El-Din, 2016). The ancient Egyptians extracted copper and turquoise from Sinai mines (Tallet *et al.,* 2011) and sent the army to secure Egypt's eastern borders from the Old Kingdom to the New Kingdom. Sinai had not only an economic role but a military one. One of the most important historical and archaeological evidence is Horus War Road in North Sinai. Sinai had religious importance, and many temples were built for gods. One of the most important Egyptian gods was Hathor, Lady of the Turquoise (Elghareb,2018). Serabit Al-Khadim Temple was built for Hathor. Sobed was another god in Sinai that contains a lot of Archaeometallurgical sites (Abdel-Motelib *et al.,* 2012), as in Figure (1).

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Fig. 1: Archaeological sites, Sinai, Egypt

Sinai contains many rock art sites, such as Wadi Maghara and Wadi Mukattab. Several stone rock arts in Wadi Maghara have embossed inscriptions of early rulers that document their expeditions to mine precious minerals, primarily turquoise and copper (Hemeda, & Al-ghareb,2012). Diagnostic investigations are important tools for evaluating the internal matrix microstructure of archaeological pottery (Drebushchak *et al.,* 2005), manufacturing technology, cultural and historical aspects, and civilization development throughout the ages (Decaro *et al.,* 2024). Examinations and analyses have expanded in recent times. The archaeometric aspects of pottery were identified by the analytical study, and pottery was classified using the examination results. The fabric and additions illustrated the cultural and technological progress in the ancient archaeological sites (Kostova *et al.,* 2023).

Diagnostic investigations determined the raw material used in manufacturing, whether Nile clay or desert clay (Hamdan *et al.,* 2014. Additionally, a multi-analytical study determined the burning temperature and atmosphere through the mineral (Drebushchak *et al.,* 2018). The internal pottery structure identified by glass phases (Sanjurjo *et al.,* 2018). Based on pottery, trade relations were identified between Egypt and its neighbors. Pottery has been the only tool used since prehistoric times. It was one of the vessels and amphorae used as containers for export. It contained many Egyptian products such as oils, resins, and other foodstuffs; fortunately, they documented their life details by carving on stone, clay, or papyri (Metwaly *et al.,* 2021).

Diagnostic tests determine severe physical, chemical, and biological damage (Wang& Ruiz,1990) . Restoration, treatment, and maintenance of damaged pottery depends on the results of various tests and analyses (Elghareb,2021). This study is useful and important in archaeological pottery, as it clarifies the matrix microstructure aspects, archaeometric characteristics, damage diagnosis, and restoration of pottery artifacts at Tell El Luli in Sinai.

2. Materials and Methods

2.1. Materials

Three samples were selected for diagnostic investigations in Tell El Luli in Sinai.

2.2. Methods

Naked eye examination was one of the recording and documentation tools. Different lenses were used to evaluate matrix microstructure, mineral composition, manufacturing, and damage (Nagwa,2016).

Examination by a stereoscopic microscope by ZTX-40 was used to identify matrix structure, pottery fabrics, additives, surface treatments, burning process, damage manifestations, crystallization of salts, stains, and peeling (Wawrzenczyk *et al.,* 2019). It was conducted at the Faculty of Science, Cairo University.

Petrographic microscope by MP920 was used to identify internal matrix microstructure, fabrics, additives, surface treatments, burning, and deterioration manifestations (Whitbread,1986). It was carried out at the Faculty of Science, Cairo University.

Morphological examination (JEOL JSM-840 and ESEM Quanta 200 FEG) was used to identify morphological structure internal matrix, fabrics, and deterioration manifestations (Maniatis,1982). This examination was conducted at the National Research Center in Cairo.

X-ray diffraction analysis by Philips X'PERT was one of the diagnostic examination tools for determining the mineral composition of the clay, glass phases, and various mineral changes (Kilikoglou,2007). The diffraction pattern used was between "4–80° 2θ". The operating conditions were Cu-K α radiation 40 MA, 45 kV. This analysis was carried out at the Faculty of Science, Zagazig University.

X-ray fluorescence analysis by Philips PW 1606 XRF was one of the diagnostic examination tools for determining the mineral composition, glass phases, and various mineral changes (Kawai, 2004). It was conducted at the Faculty of Science at Cairo University.

3. Results

3.1. Naked Eye Examination

Examination by the naked eye at the Tell El-Luli site proved that the ancient Egyptians used the wheel-shaping method to treat the surface with a slip layer and red wash. The excavated pottery suffered from various signs of damage, especially soil calcifications, fractures, gaps, peeling, and crystallized salts, as shown in Figure 2.

Fig. 2: Shows the Roman bath and excavation site, Tell El-Luli site, Sinai, Egypt.

3.2. Stereoscopic Microscope

The stereoscopic microscope showed matrix microstructure, pottery fabrics, additives, surface treatments, burning process, damage manifestations, crystallization of salts, peeling, and black core, as shown in Figure (3a, b).

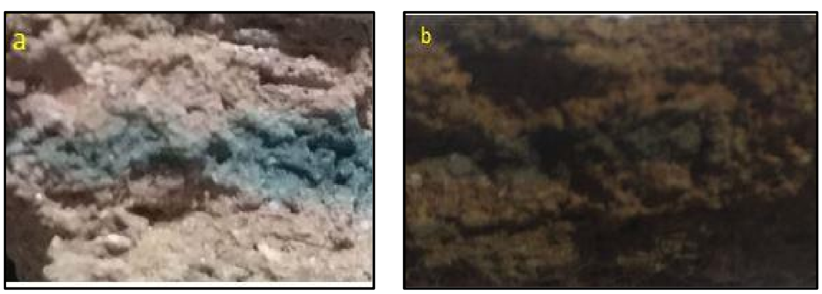

Fig. 3: Shows the stereomicroscope examination of pottery samples, a: The first sample, b: The second sample

3.3. Polarizing Microscope

The petrographic examination of the first sample was carried out. It showed the presence of quartz, grog, and iron oxide at magnification (4X - CN), as in Figure (4 a). It also revealed the presence of quartz grains, rutile, grog, calcite, and iron oxide at a magnification power of (4X - CN), as in Figure (4 b).

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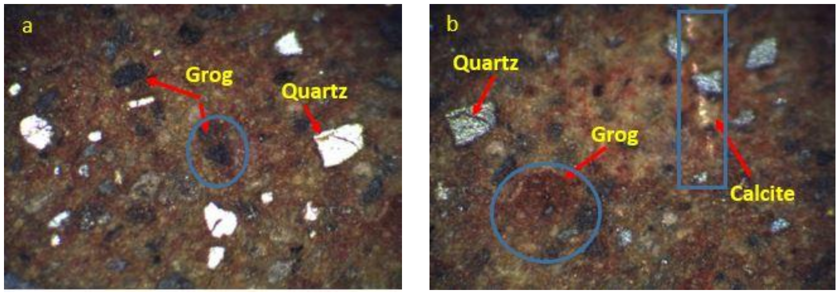

Fig. 4: Shows PLM examination: a: the first sample shows quartz, grog, and rutile, b; another part of the same sample shows quartz, calcite, grog, and iron.

The petrographic examination of the second pottery sample was carried out. It showed the presence of quartz, rutile, grog, and iron oxide at magnification (4X - CN), as in Figure (5a). It also revealed the presence of grains, rutile, grog, calcite, and iron oxide at a magnification power of (4X - CN), as in Figure (5b).

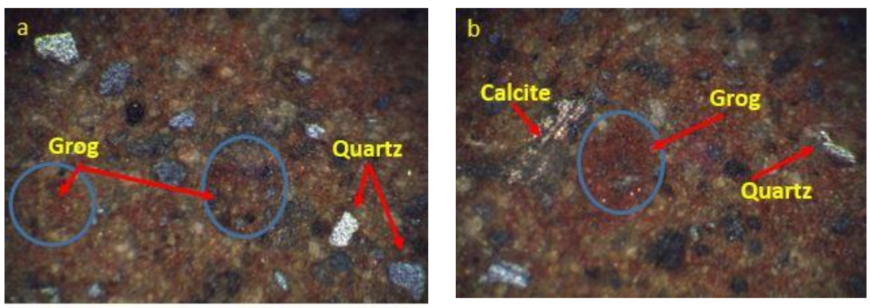

Fig. 5: Shows PLM examination, a: second pottery, b; another part of the same sample.

3.4. Examination by the Scanning Electron Microscope

Two archaeological pottery samples from the Tell El Luli site were examined by SEM-EDX. The morphological examination of the first sample showed gaps, flaking, and crystallization of salts, as in Figure (6a). In comparison, the examination of the second sample showed the presence of quartz grains, crystallization of salts, cracks, and some gaps, as in Figure (6b).

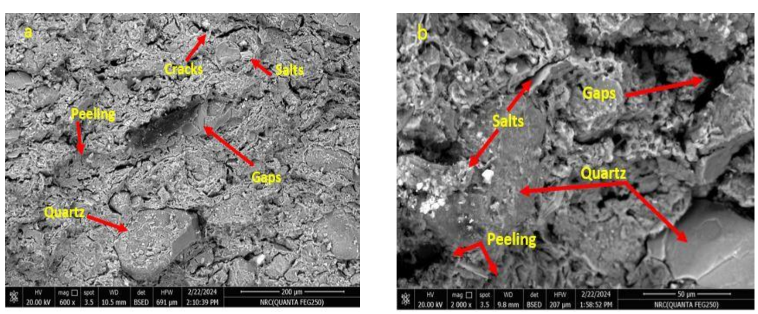

Fig. 6: Shows SEM examination of the pottery sample, a: SEM examination of the first sample with a magnification of (600 X), b: SEM examination of the second sample with a magnification of (2000 X)

Two samples from the Tell El Luli site were analyzed by EDX, which showed the presence of carbon, sodium, magnesium, aluminum, silica, phosphorus, sulfur, chlorine, potassium, calcium, and iron, as in Figures (7a & b) and table (1).

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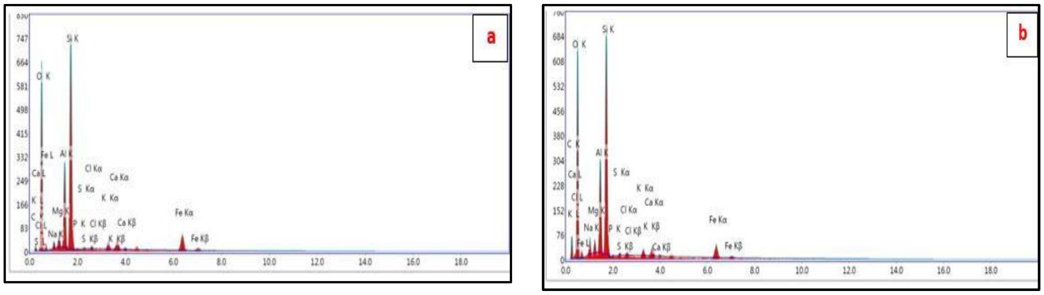

Fig. 7: Shows the EDX analysis patterns of pottery samples, a: the first sample, b: the second sample.

Table 1: EDX analysis of pottery samples, tell El Luli site

	Sample	C	O	Na	Mg	Al	Si	P	S	cl	K	Ca	Fe
Elemental Weight %	S1	8.63	40.74	2.01	2.21	8.35	12.67	2.06	3.06	2.13	0.37	5.61	1.51
	S2	16.76	41.87	3.62	1.46	6.94	14.08	1.05	1.17	4.25	0.51	4.90	2.87

3.5. X-ray Diffraction

Three samples from the Tell El Luli site were analyzed, which showed the presence of quartz, albite, magnetite, and halite, as in Figure (8a). The analysis of the second sample revealed the presence of quartz, Augite, magnetite, calcite, and rutile, as in Figure (8b). The analysis of the soil sample also revealed quartz, halite, and kaolinite, as in Figure (8 c).

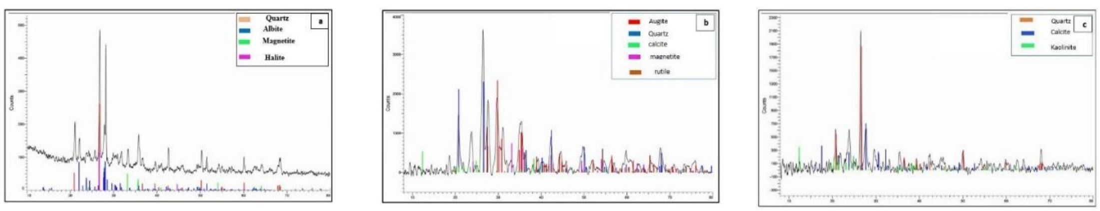

Fig. 8: Shows XRD patterns of pottery samples, a: The first pottery sample, b: The second pottery sample, c: The soil sample, tell El Luli, Sinai

Table 2: XRD analytical results

Mineralogical compositions		**Pottery**	**Samples**	
Minerals	Chemical composition	a pottery	b pottery	c soil
Quartz	SiO_2	35.2	29.3	37.4
Augite	$(Ca,Na)(Mg,Fe,Al,Ti)(Si,Al)_2O_6$	-	40.2	-
Albite	$NaAlSi_3O_{10}$	41.5	-	-
Rutile	TiO_2		8.3	
Magnetite	Fe_3O_4	5.4	7.7	-
Calcite	$CaCO_3$	-	14.5	38.8
Halite	NaCl	17.9	-	-
Kaolinite	Al2Si2O5(OH)4	-	-	23.8

3.6. X-ray Fluorescence

Two pottery samples from The Tell El Luli site were analyzed by X-ray fluorescence. The results of the analysis showed the presence of alumina, silica, phosphorus, sulfur, potassium, calcium, titanium, magnesium iron, chlorine, strontium, zircon, nybalium, molybdenum, and palladium, as in Figure (9 a, b).

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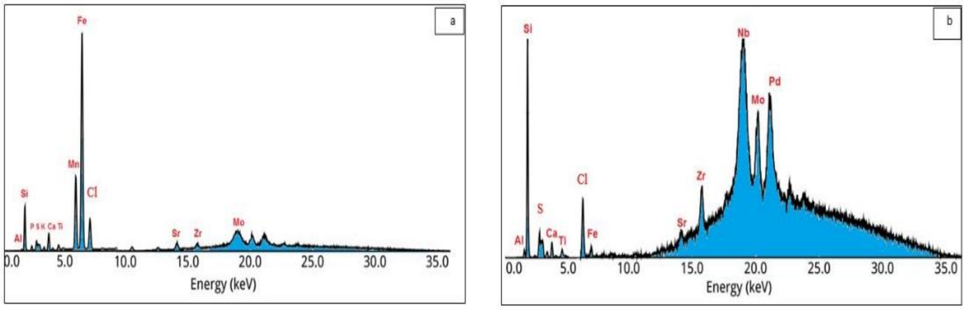

Fig. 9: Shows XRF patterns of pottery samples, a: The first pottery sample, b: The second pottery sample, Tell El Luli, Sinai

4. Discussions

Based on the results of the diagnostic investigations, the visual observations and stereomicroscope examination proved that the manufacturer used a wheel shaping technique, as shown by many researchers (Shepared,1981), and the surface treatment was a slip layer (Skibo *et al.,* 1997). The pottery suffered severe damage, such as soiling, calcifications, salts, cracks, gaps, and peeling (Sadhukhan *et al.,* 2009). These damaged appearances are shown in Figure (2). Polarized microscope examination proved that the clay was Nile clay due to the presence of muscovite, biotite, pyroxene, rutile, and iron (Elgabaly & Khadr,2006).

The petrographic examination also showed added filling materials, such as sand, grog, and limestone powder (Lakhdar *et al.,* 2021; Deckers *et al.,* 2014; Klocke,1997) . The examination revealed that the burning atmosphere was reducing; some researchers indicated that the presence of black core or magnetite suggested a reduced atmosphere (Tite, 2007). It also illustrated a rough texture in the pottery samples; some researchers indicated the presence of coarse quartz grains, referring to coarse texture (Bunge, 1995). These textures appeared in all our samples, as in Figure (4&5).

SEM examinations revealed that the clay was Nile clay due to the presence of some oxides, such as sodium, potassium, calcium, iron, titanium, and iron in the samples (Walid, 2017). The diagnostic examinations also revealed a reducing atmosphere in the samples due to the high percentage of carbon. It was 8.63% in the first sample and 16.76% in the second sample. A high percentage of carbon indicated a reducing atmosphere (Grimshaw,1971), as in Figure (7 a, b). The diagnostic examinations also showed a rough texture in the pottery samples. It also showed crystalline salts, such as halite, carbonates, and sulfates, in pottery samples, as in Figure (6&7). These damaged appearances were due to burial in the soil or climate changes after excavation (post-excavation) (El-Gohary, 2015). One of the most important manifestations of damage was crystalline salts (Steiger, 2015).

XRD showed that the clay was Nile clay due to the presence of biotite, muscovite, and pyroxene (Kamel, 2023). The analysis also proved that the additives were pottery powder, calcite, and added sand; the analysis revealed that the burning atmosphere was a reducing atmosphere. The analysis illustrated the presence of crystalline salts, such as halite carbonates and sulfates, in the pottery samples, as in Figure (8 a, b, c).

XRF showed crystalline salts, such as halite, phosphate, carbonate, and sulfate, in the pottery samples due to the presence of chlorine, sulfur, and phosphor (El-Gohary *et al.,* 2019), as in Figure (9 a, b).

5. Restoration and Treatment

Diagnostic examinations demonstrated various signs of damage. According to scientific studies and the diagnostic investigation results, the restoration processes were carried out, as follows:

Recording and Documentation

Photographic documentation recorded the pottery artifacts. The documentation process was one of the most important tools for restoring and preserving archaeological materials. One of the methods of documentation was photography (Hamilakis *et al.,* 2009).

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Cleaning

Soil accumulations were cleaned mechanically using various brushes and metal scalpels, which gave excellent results. Chemical cleaning using various organic solvents adopted this approach. A mixture of ethyl alcohol and toluene at a ratio of 1:1 was used to clean soil accumulations (Abdelmoniem *et al.,* 2024). Calcification deposits were cleaned using a poultice saturated with EDTA solution, where the pH was 11.5. The treatments designed to remove the calcareous deposits based on Na4 EDTA chelating agent applied by immersion and in gel were effective (Martínez *et al.,* 2023).

Salts Removal

The salts were extracted mechanically and then by a cotton poultice saturated with distilled water to remove soluble salts, such as halite and phosphate salts, and a tissue paper poultice saturated with EDTA solution was used to remove carbonates and sulfates (Mohamed & Khamis,2024).

Consolidation and Bonding Process

The pottery artifacts were consolidated using nanosilica at a concentration of 0.5%, and the strengthening was carried out by spraying (Elghareb, 2023). The pottery fragments were bonded using Paralloid B-72 adhesive dissolved in toluene at a concentration of 50%. After completing the restoration process, the pottery artifacts were isolated using Paraloid B72 at a concentration of 3% by brushing. Paraloid® is one of the most frequently used acrylic polymers, employed mainly for its adhesive and consolidating properties in the conservation of a wide range of materials (Vincotte *et al.,* 2019). After that, the pottery was displayed at the museum store, and the restoration process for pottery artifacts was completed, as shown in Figure (10a, b, c, d).

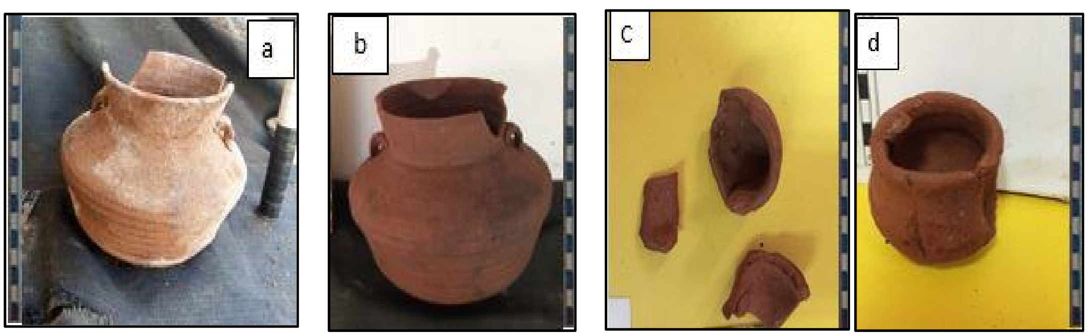

Fig. 10: Shows the restoration process: a, b: the first pottery object before and after restoration, c, d: the pottery cup before and after restoration.

6. Conclusion

The research concluded important and useful results in identifying the archaeometric aspects, damage, and treatment of pottery in the Tell El Luli site in Sinai. The clay was Nile clay. The additives were added sand, pottery powder, and limestone powder. The used forming technique was the wheel shaping technique. The surface treatment was a slip layer. The firing atmosphere was reducing. The research also proved that pottery artifacts suffered severe damage, such as soil accumulations, calcifications, fractures, gaps, peeling, salt crystallization, low durability, and poor physical structure. Using a mixture of ethyl alcohol and toluene in a ratio of 1:1 proved effective in removing soil accumulations. EDTA was used to clean calcifications. Nanosilica 0.5% was used in the strengthening process by spraying. Paraloid B72 adhesive 50 % was used in bonding. The museum display was conducted in the Qantara Sharq Museum at a temperature of 20°C, relative humidity of 60:55%, and a lighting intensity of 300 lux.

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