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Matrix Characteristics and Treatment of Ancient Egyptian Pottery at Tell Deir, Damietta, Egypt

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ABSTRACT

The research focuses on analytical investigations for identifying the archaeometric aspects, matrix microstructure, damage quantification and the treatment of pottery from Tell Deir, Damietta governorate, Egypt. The Pottery dated back to the Late Period. The used multiple diagnostic investigations are stereomicroscope, polarizing microscope (PLM), scanning electron microscope equipped with an energy dispersive X-ray unit (SEM-EDX) and X-ray diffraction analysis "XRD". The research clarified that the raw material is Nile Clay. The additives are sand, grog, limestone powder, and burnt straw. The potter's wheel is forming method in our archaeological site. The surface treatment is the slip layer. The burning process is reducing atmosphere based on high carbon percentage in matrix. The most important damage manifestations are peeling of slip layer, Soiling, cracks, weakness, fragility, soot, and crystallization of salts. The salts and soiling were treated by mechanical and chemical cleaning. Nano aluminum silicate 0.5% used in strengthening the pottery by spraying method. Paraloid B72 adhesive dissolved in toluene 50% used in assembling process. After treatment, the pottery objects are ready for museum display.

Keywords: clay, temper, burning, microstructure, matrix, damage, treatment

1. Introduction

Damietta included a group of ancient archaeological sites. These archaeological tells dated back to the late period; the most important archaeological sites in Damietta are Tell Shata, Al-Kashef, Al-Barashiya, Al-Qalaa, Al-Azam, Al-Ma'asara and Tell Deir. The Egyptian mission uncovered many archaeological pottery objects at season excavation 2020:2022 (Nour El-Din, 2016).

The pottery in Tell Deir suffered from severe damage phenomena because of the burial environment. Our burial environment caused crystallization of salts, various stains, lost parts, soiling, breaking and peeling (Mohamed & Khamis, 2024). Therefore, the research focused on multiple analytical investigations to determine the archaeometric aspects, matrix microstructure, and the various damage phenomena in Tell Deir, Damietta. Based on the diagnostic investigations, the pottery relics will be treated and restored.

2. Methodology

2.1. Study Materials

Some samples from the archaeological site were used for analytical investigations in our paper.

2.2. Study Methods

2.2.1. Visual Examination

Visual examination is one of the methods of diagnostic examinations to identify the parameters of the archaeometric aspects, various damage manifestations, the burning process and the texture. Visual observation can help diagnose the deterioration factor and choose the best technique for the conservation and restoration process (Abdelmoniem *et al.,* 2024).

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2.2.2. Examination by stereoscopic microscope

Stereoscopic microscope is one of the most important methods of examination, through which it can quantify the pottery matrix, microstructure, texture, additives, surface treatments, burning process, damage manifestations, crystallization of salts, stains, and peeling (Ahmed, 2024). The device used is ZTX-40. Magnification range 5X-240 with optional eyepieces and auxiliary objectives. Viewing Tube: Trinocular head, inclined at 45° Diopter Adjustment Eyepiece: WF10X Eyepiece with cross hair. Objective: 0.5X: 1X/3X.stand: led transmission and reflecting illumination.. Adapter: 0.5X C Mount. This examination was carried out at the Faculty of Science, Zagazig University.

2.2.3. Polarizing microscope examination

Polarized microscope examination used to identify the petrographic structure, additives, surface treatments, firing, matrix structure, and damage (Mohamed, 2022). The pottery samples were prepared in thin sections for examination using the polarizing microscope. The device is MP920, Supply high quality and contrast image with special strain free objective. Single polarization, othorgonal polarization and Conoscopic observationon. Ransmitted light and reflect light, 24V/100W long life halogen lamp. Stage with 45°located, attachable mechanical stage with large moving range, high efficiency by no interference when turning objectives. Optical System: Infinite optical system, Viewing Head: Seidentopf trinocular head inclined at 30°, Interpupilary 48-75mmEyepiece: eyepiece with cross, Plan eyepiece with gridding, Achromatic Objective: 100X/0.8/∞/0 WD 2mm.Analyzer: 360° dial rotation Minimum scale reading : 0.1°.Polarizer: Transmitted: 360° Rotatable 0° position. Illumination: reflected light: 24V/100W Halogen lamp, this examination was conducted at the Faculty of Science, Zagazig University.

2.2.4. Examination by scanning electron microscope equipped with an EDX unit

The scanning electron microscope shows the morphology of archaeological surface, the damage, the pores, cracks, gaps, the relationship of grains to each other and mineral composition (Eloriby & Mohamed, 2023). The environmental scanning electron microscope is SEM Quanta 200 FEG. The operating conditions were “20 kV and 1×10^{-9}A.” This examination was conducted at National Research Center in Cairo.

2.2.5. Analysis by X-ray diffraction

XRD is one of the important analytical methods. This analysis helps to identify the mineral components, the nature of firing process, and the damage (El-Hassan & El-Tawab,2023) .The device used is 'X'Pert Graphics' and 'Identify', by Philips, the diffraction pattern used is between "2–70° 2θ", and the operating conditions were carried out using a Cu-K α radiation lamp 40 MA, 45 kV, this analysis was conducted .

3. Results

3.1. Visual Observation

This examination of pottery at Tell Deir in Damietta Governorate showed physicochemical damage, such as soil debris, crystalline salts, cracks, breakage, and soot, as in Figure (1).

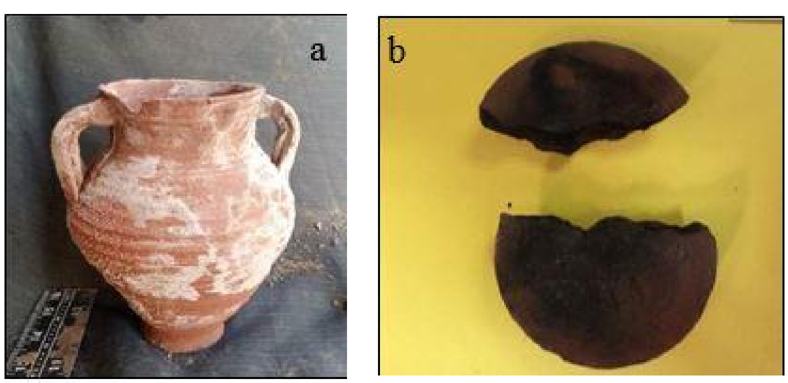

Fig. 1: Represents the pottery objects, Tell Deir, Damietta. a: Pottery vessel, b: broken Pottery plate.

3.2. Stereoscopic microscope examination

Two sample of pottery excavated from tell Deir examined by stereoscopic microscope. The examination revealed the presence of sub round quartz grains, calcite, cracks, gaps and iron oxides as in Figure (2a-b).

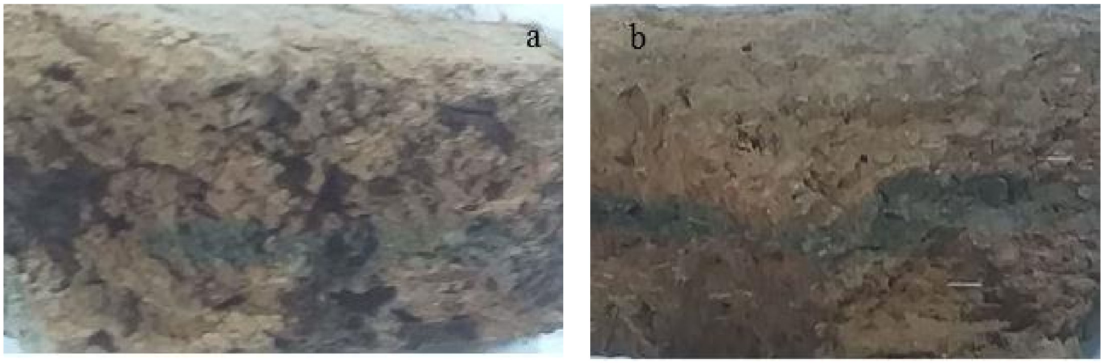

Fig. 2: Represents Stereomicroscope examination, a: first sample (quartz, gaps, cracks), b: second sample (quartz, gaps, calcite, peeling, burnt straw)

3.3. Polarizing Microscope examination

Two sample of pottery excavated from tell Deir examined by polarizing microscope. The examination revealed the presence of quartz grains, calcite, grog, plagioclases, biotite, polycrystalline quartz and iron oxides (10X - CN), as in Figure (3a). Another part of the same sample was also examined; it revealed grains of quartz, calcite, grog, pyroxene, plagioclase, biotite, as in Figure (3b),

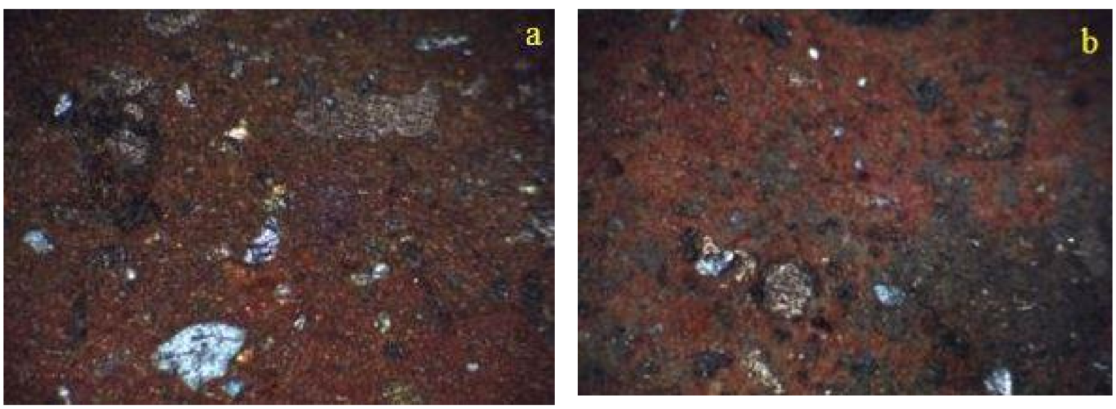

Fig. 3: PLM examination of the first pottery, a: first sample shows quartz, calcite, grog, biotite, and plagioclase, b; another part of the same sample shows quartz grains, calcite, grog, biotite, and iron.

Examination of the second pottery sample shows grains of quartz, calcite, grog and burnt straw at magnification (10X - CN), as in Figure (4a). Examination of another part of the same sample also showed quartz grains, polycrystalline quartz, limestone powder, iron, fossils and cracks at magnification (10X - CN), as in Figure (4b).

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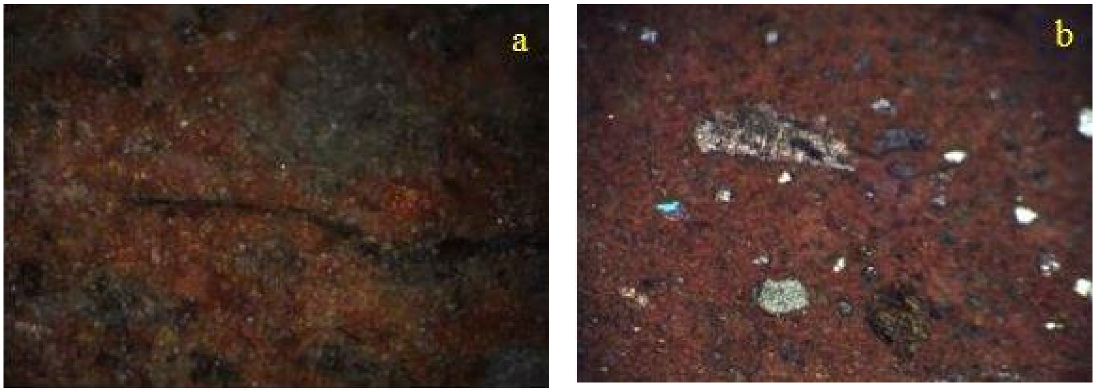

Fig. 4: PLM examination of the second pottery, a: second pottery sample shows quartz, calcite, grog, and burntsraw, b; another part of the same sample shows quartz grains, calcite, grog, plagioclase, biotite, fossil, and iron.

3.4. Scanning electron microscope examination (SEM-EDX)

SEM Examination of the first sample showed morphological aspects and microstructure as cracking, macro gaps, exfoliation of the slip layer, sub round quartz grains, detachment, and crystallization of salts at magnification (200x), as in Figure (5a). Examination of another part of the first sample also revealed cracks, gaps, detachment, and crystallization of salts at magnification (800x), as in Figure (5 b).

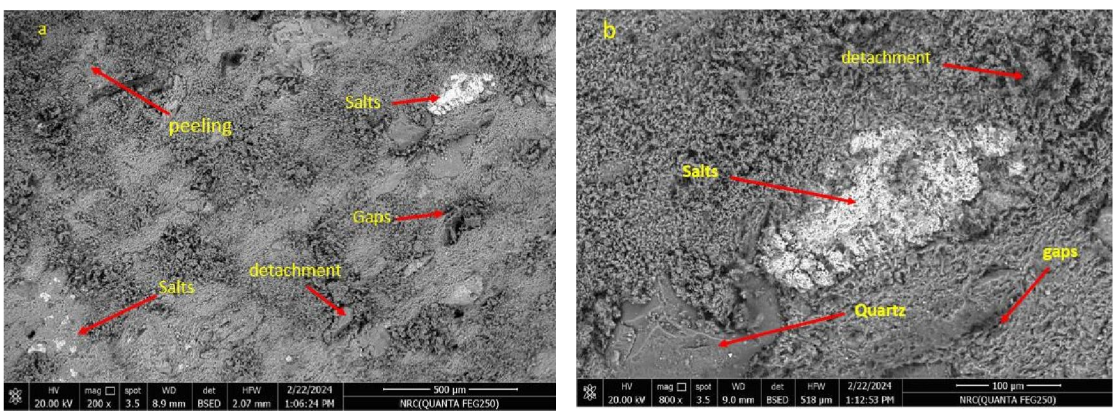

Fig.5: SEM examination of the first pottery sample, a: SEM examination of the first sample with magnification (200 X), b: SEM examination of another part of the first sample with magnification (800 X)

SEM of the second pottery sample clarified the presence of sub round quartz, gaps, cracks, exfoliation of surface, detachment, and crystallization of salts at magnification (200x), as in Figure (6a). Examination of another part of the second sample showed Various quartz grains, crystallization of salts, gaps, cracks, peeling, and separation of quartz grains at magnification (800x), as in Figure (6b).

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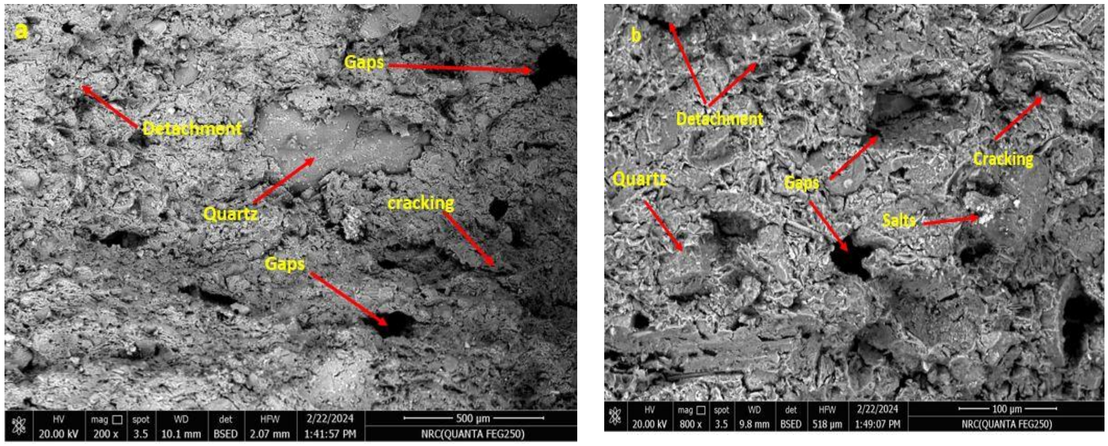

Fig. 6: SEM examination of the second pottery sample, a: SEM examination of the second sample with magnification (200 X), B: SEM examination of another part of the second sample with magnification (800 X)

EDX analysis of the first and second pottery sample showed carbon, oxygen, sodium, magnesium, alumina, silicon, phosphate, sulfur, chlorine, potassium, calcium, and iron, as in Figure (7a-b). These EDX analytical results for two sample results as shown in table (1).

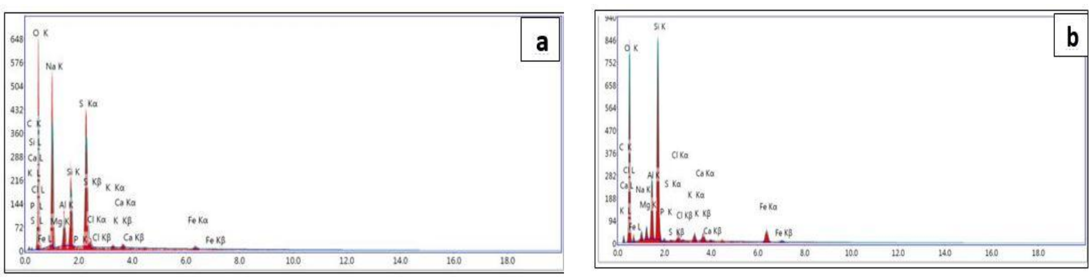

Fig.7: EDX analysis patterns of pottery samples, tell Deir, Damietta, a : first pottery object, b: Second pottery object.

Table 1: EDX analysis of pottery samples, tell Deir, Damietta

	Sample	C	O	Na	Mg	Al	Si
Elemental Weight %	S1	6.52	19.25	9.66	3.25	7.05	16.66
	S2	7.68	25.39	8.06	7.02	11.75	23.19
	Sample	**P**	**S**	**Cl**	**K**	**Ca**	**Fe**
Elemental Weight %	S1	4.04	12.28	8.05	0.22	7.40	5.62
	S2	2.28	2.07	3	0.87	3.18	5.51

3.5. Analysis by X-ray diffraction

X-ray diffraction pattern of the first sample appears the presence of calcite, quartz, muscovite, hematite, huntite, as shown in Figure (8a). Analysis of the second sample showed the presence of quartz, muscovite, magnesite, hematite, dolomite, as in Figure (8b). Analysis of the third soil sample also showed the presence of quartz, halite and kaolinite. The results of the X-ray diffraction pattern showed that the soil is sandy clay rich in saline solutions, as shown in Figure (8c). These XRD analytical results for two sample results as shown in Table (2).

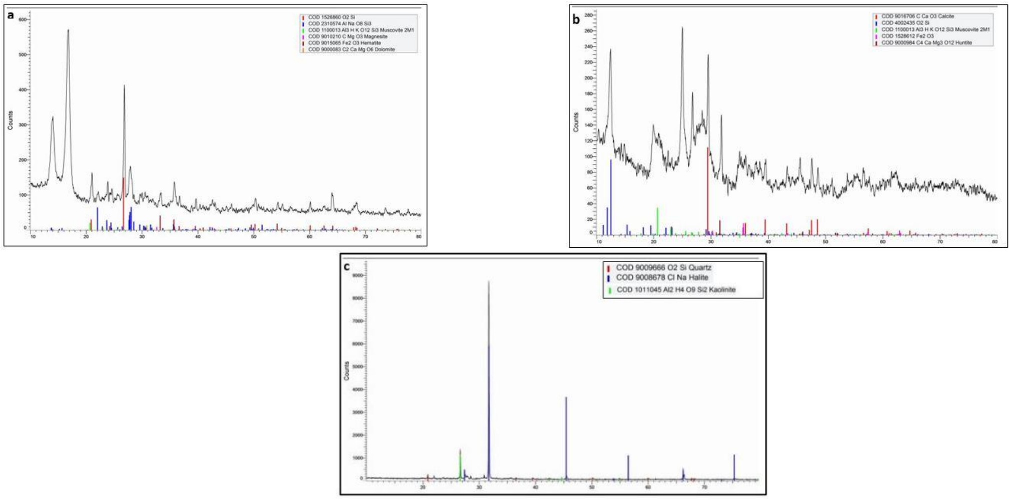

Fig. 8: XRD patterns of pottery samples, a: first pottery sample, b: second pottery sample, c: soil sample, tell Deir, Damietta

Table 2: XRD analytical results of pottery samples.

Mineralogical Compositions		**Pottery**	**Samples**	
Minerals	**Chemical composition**	**a Pottery**	**b Pottery**	**c Soil**
Quartz	**SiO_2**	27.10	24.60	19.30
Albite	**$NaAlSi_3O_{10}$**	57.20	-	-
Muscovite	**$KAl_2(Si_3Al)O_{10}(OH)_2$**	3.90	13.60	-
Magnesite	**MgCO3**	3.20	-	-
Hematite	**Fe_2O_3**	6.10	5.70	-
Dolomite	**CaMg(CO3)2**	2.60	-	-
Calcite	**$CaCO_3$**	-	45.10	-
Huntite	**Mg3Ca(CO3)4**	-	11	-
Halite	**NaCl**	-	-	58.20
Kaolinite	**Al2Si2O5(OH)4**	-	-	22.5

4. Discussion

Visual examination of the pottery objects in our site showed that the pottery is formed with a potter's wheel, the methods of shaping in Egypt is multiple, whether manual or potter's wheel (Shepared, 1981). The latter was used for finishing the clay vessels in the Naqada era. It also used for shaping from the Old Kingdom period until now. Pottery vessels in particular are one of the most abundant kinds of artifacts known from ancient Egypt (Sterling, 2004). It suffers from crystallization of salts and soiling, cracks, breaking and peeling. Most of the damage is due to the heterogeneity of the mineral composition of the pottery (Smith &Clark, 2004), the soil damage (Abd Elrahman, & Saleh, 2019), and the effect of excavation environment (Elghareb, 2019), as in Figure (1a-b). Stereomicroscope examination revealed that the pottery suffers from Soil accumulations, calcifications, salts, cracks, and tempers. Stereomicroscope Examination demonstrates pottery matrix and various aspects of damage, as in Figure (2a-b). Damage phenomenon is due to the burial environment (El-Gohary *et al.,* 2019).Petrographic examination demonstrated that the clay used in tell Deir site is Nile Clay due to the presence of quartz, mica, pyroxene, and plagioclase (Ibrahim & Mohamed,2021). The mineral components (mica) are considered one of the characteristics of the Nile clay (Elgareb,2023). The examination also appeared inorganic and organic additives as sand, limestone powder, grog and burn straw in thin sections (Szułczyńska & Konopka, 2011). The additives aim to improve the properties of clay so that the ancient Egyptian can easily form the clay. Kaolinite is not be formed without additions (Elghareb, 2007). These additives materials are dense in Egyptian pottery, Porcelain stoneware bodies consist mainly of a mixture of ball clay, feldspar and quartz- feldspathic sands, often-containing also glass-ceramic frits and pigments. (El-Fadaly, 2013), as in Figure (3-4).

The examination confirmed the surface treatment by the slip layer. Surface treatment methods include removing excess clay, red wash, slip layer and polishing "water, oils and resins". The aim of the surface treatments was to improve the physical properties according to functional purposes (Skibo *et al.,* 1997) .The examination appeared that pottery texture ranges from medium to coarse texture (Bourriau & Nicholson ,1992).The type of clay, shaping and surface treatment played an important role in specification pottery fabric and manufacture of glass and pottery (Elghareb,2018). The SEM examination demonstrated the presence of separations, cracks, and gaps, as shown in figure (5-6). The phenomenon of cracking is due to high thermal energy resulting from burning process in the kilns (Elghareb, 2021). Examination also showed crystallization of salts that caused some cracks in the pottery (Jinhyun Choo &WaiChing Sun,2018). Cracking is due to the swelling of the clay in low burning process (Elghareb, 2020). Crystallized salts in pottery are due to burial in the soil (Abd Elrahim & Mohamed, 2024) . The soil in Egypt is rich in dissolved salts (Elghareb, 2024). The SEM examination identified the nature of the burning atmosphere. The burning of the samples were low due to the presence of carbon dioxide 5.62 % for the first sample and 7.68% for the second sample. The burning was low because of high carbon percentage in our samples, as in Table (1). The percentage of carbon in the pottery analysis is an indication of low burning degree (Karaman *et al.,* 2006).EDX analysis for our samples identified the presence of chlorides, sulphates, and phosphate salts. The chlorine percentage in the first pottery sample reached 8.05% and 3% for the second pottery. The percentage of calcium was 7.40 % for the first sample and 3.18 % for the second pottery. The percentage of sulfur in the first pottery sample was 12.28% and 2.07 % for the second pottery. The percentage of phosphate in the first pottery sample was 4.04% and 2.28 % for the second pottery, as in Table (1).

Soil plays an important role in pottery damage, some researchers had attributed this reason to hygroscopic pottery material (Elghareb, 2017).It absorbs saline water from the soil ,crystallization of the salts caused pressures resulting cracks and separation (Sungyoon *et al.,* 2013).The manifestations of salt damage included cracks, surface accumulation, and quantitative estimates of both the interaction forces for the minerals and the stresses induced in the material by salts (Espinosa-Marzal & Scherer,2010).XRD analysis demonstrated additions in pottery samples. Some researchers had agreed that burnt straw, grog and limestone powder are among the most common additives in ancient Egyptian pottery (Elghareb, 2018). The analysis by XRD also confirmed the presence of carbonates and chlorides salts. According to researchers, the soil is heterogeneous containing saline groundwater because of dissolved salts (Abdelaal *et al.,* 2019). XRD analysis also revealed the presence of quartz, halite and kaolinite in the archaeological soil site, which confirms that the soil is saline clay.

5- Restoration and maintenance

Various examinations and analyzes of the pottery objects at Tell Deir in Damietta Governorate has proven the presence of soil accumulations, salts, soot and weakness. One of the pottery objects was also found in a broken state because of soil mechanical damage. Based on these results, two pottery objects were treated according to the nature of the damage and scientific studies in the field of restoration, treatment and maintenance of pottery antiquities. The pottery recorded by photographic documentation as in figure (1). It should to be the first step before treatment strategy; Photographic documentation plays a role in treatment and restoration efforts. It has been shown that photographic documentation of objects often becomes the primary source of information for damage diagnosis, restoration, and conservation efforts (Kozakova *et al.,* 2024).

Free soil accumulations were cleaned mechanically using hand tools by various brushes, the strongly attached sediments were also cleaned using metal scalpels (Abd-Allah, *et al.,* 2010).

Mechanical cleaning is carried out meticulously from top to bottom to avoid scratching the pottery. Various soil deposits were cleaned using organic solvents (Adi Darmawan *et al.,* 2024).

Chemical cleaning process was carried out locally for each piece separately. The soot removed using hydrogen peroxide 10%. The salts were removed mechanically using brushes and scalpels under magnification lenses to avoid damaging the pottery. Tissue paper poultice saturated with distilled water used to remove soluble salts. A poultice saturated with EDTA solution was applied to remove calcareous deposits (Ahmed *et al.,* 2021) .The glass and pottery artifacts were strengthened using nano silica or nano aluminum silicate 0.5% (Eloriby *et al.,* 2022).

Consolidation process is carried out by spray method (El-Hassan, 2021). The preliminary assembling process was begun to determine the locations of the breaking then, the pottery shards were assembled using Paralloid B-72 adhesive 50% (Koob, 1986). Assembling process of our pottery shards was carried out accurately. The pottery artifacts were strengthened, protected and isolated using Paraloid B82 at a concentration of 3% by brushing.

The pottery artifacts are ready for museum display after treatment process as in figure (9a, b, c, d). It is preferable to display the pottery in the museum at a temperature of 20°C, relative humidity 55:60% and light of 300 lux. It is one of the methods of preventive maintenance (Elena Lucchi, 2018).

Fig. 9: Represents treatment process, a,b: the pottery vessel before and after restoration, c,d: the pottery plate before and after restoration.

4. Conclusion

The research proved some important results in identifying the archaeometric aspects of the pottery manufacture at tell Deir site. The used clay is a Nile clay. For the additions, it turned out to be sand, burnt straw, grog, limestone powder. The shaping used in the manufacture of tell Deir pottery was wheel forming technique. The research also found that the surface treatment was slip layer. For the firing atmosphere, it was reducing atmosphere for the pottery artifacts.

It was also proven that the pottery relics suffered from physical, mechanical and chemical damage such as soil accumulations, soot, breakage, crystallization of salts" chloride, phosphate, sulphates, and carbonate", weakness and lack of durability of the pottery artifacts. The research proved Successful treatment strategies. A mixture of acetone and toluene in a ratio of 1:2 was used to remove soil accumulations. Hydrogen peroxide 20% used to remove soot. EDTA poultice was applied to clean calcareous deposits. Nano aluminum silicate 0.5% used in strengthening the pottery by spray method. Paraloid B72 adhesive 50% used in assembling pottery shards. After restoration, the treated pottery artifacts are ready for museum display.

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