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Original article

TECHNOLOGICAL ASPECTS, DAMAGE AND CONSERVATION OF SOME POTTERY ARTIFACTS FROM TELL RAWD ISKANDER, ISMAILIA, EGYPT

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***Abstract**:*
The archaeological pottery extracted from Tell Rawd Iskander in Ismailia is dated back to the New Kingdom. Different examinations and analyses were conducted, such as stereomicroscope examination, polarized microscope, microbiological examination, scanning electron microscope equipped with X-ray energy dispersion unit (SEM/ EDX), and X-ray diffraction. The research identified a technological process proving that the clay used in archaeological pottery was Nile clay. Tempers were sand, pottery powder, limestone powder "calcite, dolomite", and burnt straw. The forming technique was the potter wheel technique. Surface treatment is a slip layer and red wash. The burning atmosphere inside the kiln was oxidizing for the first and second pottery objects, and it was reduced for the third pottery shard. The pottery texture was fine to coarse fabric. Furthermore, the research paper proved that pottery artifacts were subjected to various damage processes resulting from different environmental factors. Most damage manifestations were surface deformation by soil sediments, iron stains, soot, and fungal growth. Pottery objects also suffered from crystallization of salts "chlorides, sulfates, carbonates, and phosphates", cracking, fracture, weakness, gaps, peeling, separation of grains, and breaking. The archaeological pottery in Tell Rawd Iskander in Ismailia was treated. Hairbrushes removed free fragile sediments. Various metal scalpels cleaned firmly attached sediments. Furthermore, clay deposits were removed by a mixture of distilled water, acetone, and ethyl alcohol in a ratio of 1:1:1. EDTA cleaned lime deposits, whereas mora poultice extracted carbonate and gypsum salts. Fungal infections were treated by nano silver oxide (0.5 %). Strengthening archaeological pottery was carried out using nano-silica (0.5%); it was applied by the spraying method. Paralloid B 82 (50%) was used in assembling pottery shards. Replacement was conducted by a mixture of micro-ballon and pottery powder.

Keywords:
Microstructure
Clay
Additives
Texture
Burning
Damage
Nano silica

1. Introduction

Tell Rawd Iskander is located about 2 km to the west of Abu Sweir city. It is located just north of the Ismailia-Zagazig Road [1]. It was known as *Bethom* in the dynastic age. The ruins of Rawd Iskandar were dated to the New kingdom. Excavations revealed mud-brick tombs, bone skeletons, burials, pottery objects, and mud-brick warehouses that matched Tell Al-Maskhouta stores [2]. Among the most important discoveries was a stone tomb of a person called "*Ken-Amun*", the scribe of royal records during the Ramses II period. The archaeological site was located within the eighth province in Lower Egypt. It was called Bir Atum city, which means" the house of god Atum" in hieroglyphic language. The site was the most important fortification in King Ramses II's period. Its current location is Rawd Iskander Village in Abu Sweir [3]. Excavations revealed many pottery artifacts, fig. (1) [4].

Figure (1) Shows the excavation site, Tell Rawd Iskandar, Ismailia.

At this excavation site, the most objects of pottery have suffered from surface deformation, soil deposits, cracking, salt crystallization, various stains, soot [5], and fracture [6]. Additionally, the pottery shards suffered from weakness and lack of durability due to various damage factors in burial soil and exposure environment [7, 8]. The cleaning materials and methods differed depending on the type of deposits attached to the surface, strength of adhesion, type of bonds, and durability or weakness of the pottery body [9,10]. The cleaning degree varied from one object to another depending on the condition of the pottery body [11]. Choosing the appropriate cleaning methods is a complex process [12] according to the condition of the pottery, damage manifestations, and multiplicity of cleaning materials [13]. Conservators use many different materials depending on the nature of pottery damage [14]. Many methods may be used in the cleaning process that can be done on a fixed table so as not to break pottery objects because of sudden mechanical shocks; it is preferable to put pottery on padding materials, such as foam, to absorb shocks [15]. Magnifying lenses can be used during cleaning [16]. One of the best materials used in the cleaning process is EDTA, a common cleaning material for removing various lime deposits or iron stains [17,18]. The cleaning process for soil deposits can utilize organic solvents, such as toluene, ethyl alcohol, or a mixture of solvents, e.g., trichloroethylene and alcohol [19,20]. Micro-emulsions are used in cleaning to avoid the effect of solvents on the pottery. They are liquid, stable, and transparent, i.e., micro-emulsions (O/W) [21]. The most important anti-fungal materials are ammonium and bromide compounds, such as methyl bromide, ethylene oxide, carbon tetrachloride, and sodium fluoride. Inert gases can be used in fungi control, but this technique suits museums [22]. Most pottery objects are characterized by a poor physical structure and lack of durability due to hygroscopic properties and breakage susceptibility [23,24]. It is the most common case of excavated pottery due to stresses [25], which requires strengthening with one of the appropriate consolidants [26]. Recently, nanocomposites have strengthened the pottery objects. Some nanomaterials are added to acrylic or silicon polymers to improve physiochemical and mechanical properties [27]. The fracture and cracking occur because of internal and external pressures [28]. Such pottery shards need an assembly process. The adhesive materials vary according to their physical and chemical nature [29]. Most extracted pottery shards from soil suffer from the phenomenon of missing some parts, which requires completing the process using one of the appropriate completion materials [30]. This research paper is characterized by originality and innovation, as no other researcher has studied the pottery at this archaeological site. Therefore, it is considered one of the first studies on the technological aspects of pottery manufacturing in the Tell. It is a key for studying the pottery of other archaeological sites in the eastern Delta, creating a local classification of pottery and its treatment methodology. The article did not only deal with technological features but also contained treatment, maintenance, and preservation of some pottery artifacts excavated from the archaeological site as an intervention treatment according to scientific and experimental studies in this field. A methodology can be used by restorers in the future.

2. Materials and Method

2.1. *Study materials*

Three pottery shards and soil samples were used in various examinations and analyses.

2.2. *Study methods*

2.2.1. Visual examination

The visual examination method is an important method that clarifies manufacturing technology and damage manifestations, whether by the naked eye or different types of lenses whose magnification ranges "4 X: 6 X"[31].

2.2.2. Stereomicroscope examination

SZ680 stereomicroscope with objectives zoom range: 0.68x - 4.7x, zoom ratio: 1-6.8, field diameter (mm): Φ23, working distance (mm): 110, stereo angle (°): 12, viewing angle (°): 35 and magnification: 3.5～22.5 was used. This examination was carried out for studying pottery technology, additives, surface flaking, salt crystallization, various gaps, and black core [32].

2.2.3. Polarized microscope examination

Olympus BX51 TF microscope attached with a digital camera under magnification 4x up to 40x) to illustrate the petrographic structure, nature of burning, texture, and damage of mineral components [33]. Thin sections of selected pottery shards were 0.03 mm for petrographic examination.

2.2.3. Microbiological examination

Biological microscope (BM-322-LED) is equipped with binocular head, total magnification: 40x - 1000x, ophthalmic lens: WF 10x and Objective lens: achromate 4x: 100x (oil) was used for investigating microbiological growth. A growth media of potato dextrose agar "PDA" was used. It consisted of 200 gm potato extract, 200 gm dextrose, 20 gm agar; 10 gm rose bengal, stiriptomycene (traces), and distilled water (750 ml^3). It was sterilized in an autoclave at 1.5 atmospheric pressure. Samples were added to PDA media. They were poured into sterile Petri dishes. Then, the dishes were incubated at a temperature of 28 °C. This was followed by the separation and purification of colonies. After that, the examination and classification of isolated fungi were carried out based on the shape, color, and density of fungal colonies. The type of fungal colonies was recognized according to Domsh classification [34].

2.2.4. Scanning electron microscope attached to EDX unit (SEM-EDX)

Scanning electron microscope (JEOL JSM-840 and SEM Quanta 200 FEG, XTE 325/ D8395" with operating conditions were "20 kV and 1×10^{-9}A) was adapted. It was used according to El-ghareb [35] to describe the morphology and damage features of archaeological pottery in our case.

2.2.5. X-ray diffraction analysis

XRD is an important method that illustrates mineralogical composition and damage of pottery artifacts [36]. The device used was Philips. The diffraction pattern used was between "4-70° - 2θ". The operating conditions were Cu-K α radiation 40 MA, 45 kV.

3. Results

3.1. *Visual examination*

The visual examination of pottery objects revealed the presence of various damage manifestations, especially surface deformation, fracture, soil deposits, various stains, soot, black core, flaking, and crystallization of salts. The shaping method was the potter wheel. Additionally, the surface treatment was a slip layer, fig. (2).

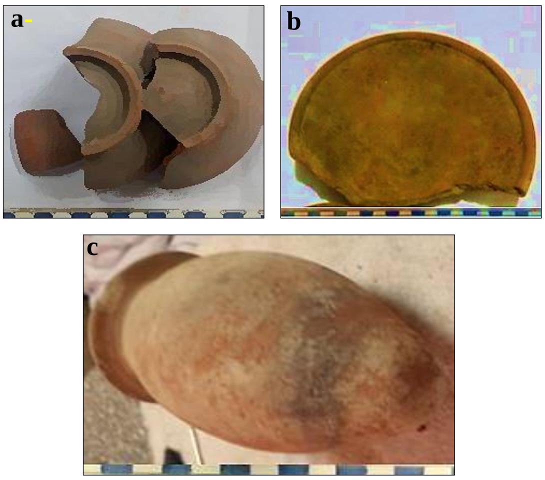

Figure (2) Shows the pottery shards excavated from Tell Rawd Iskandar, Ismailia; **a.** the first shard, **b.** the second shard, **c.** the third shard.

3.2. *Stereomicroscope*

A stereoscopic microscope examination of the first pottery shard, fig. (3-a), showed that the shaping method was the potter wheel. The surface treatment was a red wash and slip layer. It showed various damage manifestations, such as surface deformation, soil sediment, flaking, salt crystallization, gaps, erosion, iron stains, soot, calcite, and sand within the core or matrix. The examination of the second pottery shard, fig. (3-b), showed that the forming method was the potter wheel; the surface treatment was a slip layer. It also showed damage forms, such as fracture, erosion, gaps, loss of some parts, surface deformation, crystallization of salts, pitting, peeling of slip layer, and black and iron stains. The examination of the third pottery shard, fig. (3-c), showed that forming was the potter wheel method; the surface treatment was a red wash. The additions were sand, calcite, and grog. The examination showed damage forms, such as salts, erosion, peeling, crystallization of salts, and various stains.

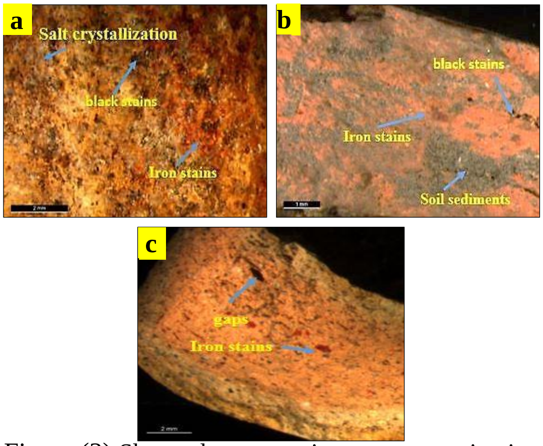

Figure (3) Shows the stereomicroscope examination **a.** crystallization of salts, black spots, and iron, **b.** soil sediments, black, and iron stains, **c.** iron stains and gaps.

3.3. *Polarized microscope*

Three samples of pottery artifacts extracted from Tell Rawd Iskandar were examined by polarized microscope where fig. (4-a) of the surface area showed the presence of fine-grained quartz, rutile, pottery powder, and calcite with a magnification of (10x - CN). Figure (4-b) of another part of the core area illustrated the presence of sharp-angled and semi-circular quartz grains, as well as muscovite, rutile, biotite, and calcite scattered inside pottery texture with a magnification of (10x - CN). The polarized microscope showed the presence of fine quartz grains, sharp-angled quartz, rutile, and burnt straw with a magnification of (10x - CN), fig. (4-c). The core area showed the presence of fine and semi-circular quartz grains, as well as rutile, biotite, masses of calcite, and plagioclase with a magnification of (10x - CN), fig. (4-d). Another part of the core area illustrated the presence of fine quartz grains, pottery powder, biotite, rutile, calcite and plagioclase with a magnification of (10x - CN). Figure. (4-e) of the surface area showed the presence of coarse, sharp-angled quartz grains, rutile, pyroxene, plagioclase, and biotite with a magnification of (10x - CN). Furthermore, Figure (4-f) of the core area showed the presence of coarse semi-circular quartz grains, some of them are sharp angles quartz grains, as well as rutile, biotite, polycrystalline quartz grains, plagioclase, muscovite, and orthoclase with a magnification of (10x - CN).

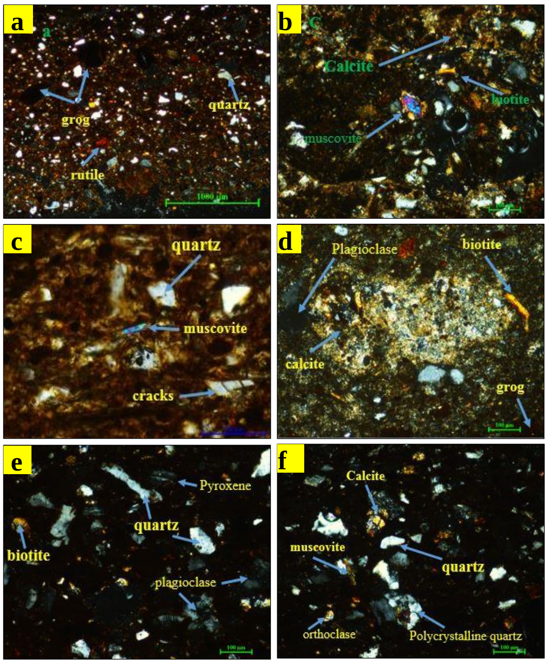

Figure (4) Shows PLM examination; **a.** surface area of quartz grains, grog, and rutile, **b.** calcite, biotite, and muscovite of the 1st sample, **c.** quartz, muscovite, and cracks, **d.** calcite, biotite, and plagioclase, of the 2nd sample **e.** quartz grains, biotite, pyroxene, and plagioclase, **f.** calcite, muscovite, orthoclase, and polycrystalline quartz of the 3rd sample.

3.4. *Microbiological examination*

After the end of the incubation period, fungi growths that appeared in dishes were taken. A purification process was carried out to obtain organisms in a pure form, which laboratory experiments could be completed. The purified organisms were defined. Microbial slides were made to know morphological characteristics compared with standard morphological characteristics found in books and scientific references. The results are presented in tab. (1) & fig. (5-a:f).

Table (1) Fungal growth colonies on pottery samples.

Pottery sample	Colony
1	*Aspergillus flavus, Aspergillus niger, and Aspergillus rhizopus sp.*
2	*Aspergillus sulphureus, Aspergillus fumigatus, and Aspergillus flavus*
3	*Aspergillus niger, Aspergillus flavus, Aspergillus fumigatus, and Penicillium sp.*

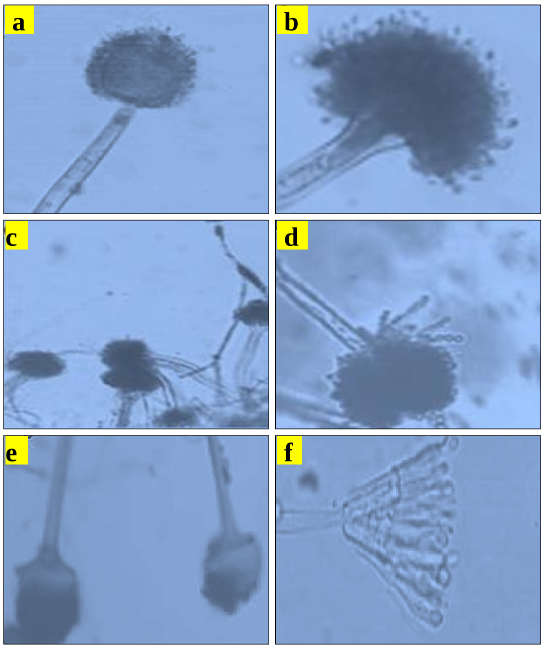

Figure (5) Shows fungal colonies: a. *As. flavus*, b. *As. niger*, c. *As. sulphureus*, d. *As. fumigatus*, e. *Rhizopus sp*, f. *Penicillium sp.*

3.5. SEM-EDX results

SEM examination of the first sample showed that the sample suffered from damage, such as slip layer peeling and some stains, in addition to soil calcifications, cracks, and gaps (200x), fig. (6-a). Figure (6-b) illustrates another part of the sample, with flaking, cracking, gaps, and salts crystallization (200x). SEM examination of the second sample of the surface area showed that the sample suffered from severe damage, e.g., gaps, cracks, and soil calcifications (1000x), fig. (6-c). Additionally, fig. (6-d) showed the examination of another part of the same sample, including cracking, gaps, and crystallization of salts of (500x). The third sample of the surface area showed that the sample suffered from damage, such as gaps, cracks, and soil calcifications with a magnification of (600x), fig. (6-e). The examination of another part of the same sample illustrated cracking, gaps, and crystallization of salts (1200x), fig. (6-f).

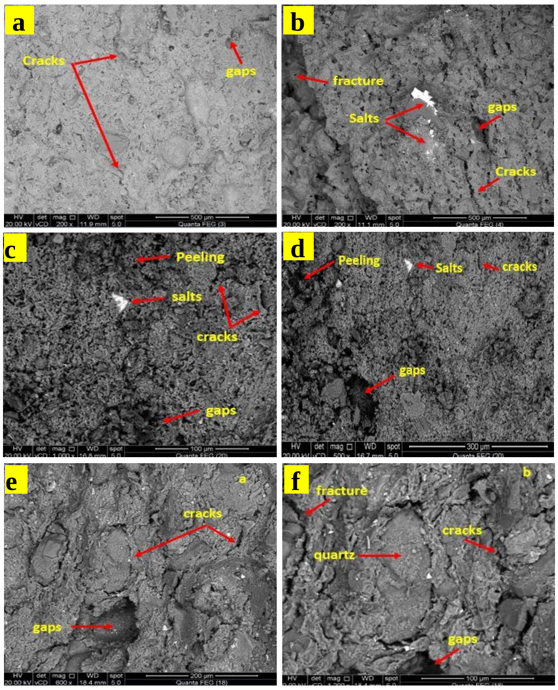

Figure (6) Shows SEM photomicrograph; **a.** & **b.** cracks, gaps, and peeling of quartz in the 1st sample, **c.** & **d.** cracks, gaps, peeling, and salts in the 2nd sample, **e.** & **f.** cracks, gaps, and fractures in the 3rd sample.

Furthermore, EDX analytical results of three samples clarified the presence of C, O, F, Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, and Fe. These results are listed in tab. (2)

Table (2) EDX analytical results of the pottery shards

Elemental Weight %	S1		S2		S3	
	Surface	*Core*	*Surface*	*Core*	*Surface*	*Core*
C	1.02	1.01	2. 02	3.85	5.68	5.63
O	19.27	23.83	27.13	27.02	22.03	22.02
F	1.20	-	-	-	-	-
Na	1.33	12.1	1.20	1.45	'.20	1.13
Mg	1.29	1.88	1.43	1.40	2.01	1.99
Al	8.13	8.11	8.22	7.35	6.23	6.25
Si	25.28	26.44	30.12	3|14	29.56	28.68
P	-	6.38	2.48	2.99	2.98	-
S	2.30	-	2.15	2.03	3.18	3.10
Cl	6.55	5.28	5.96	5.36	10.23	12.20
K	1.12	1.01	1.28	1.22	1.27	1.05
Ca	20.18	20.08	11.87	11.55	11.33	13.11
Ti	1.38	-	1.89	1.65	1.18	1.05
Fe	2. 40	4.86	4.25	3.99	3.12	3.79

3.6. XRD results

XRD pattern of the analyzed samples contained quartz (SiO_2), hematite (Fe_2O_3), albite ($NaAlSi_3O_{10}$), calcite ($CaCo_3$), dolomite, ($MgCa(Co_3)_2$, microcline ($KALSi_3O_8$), and gypsum ($CaCo_4.2H_2O$), fig. (7).

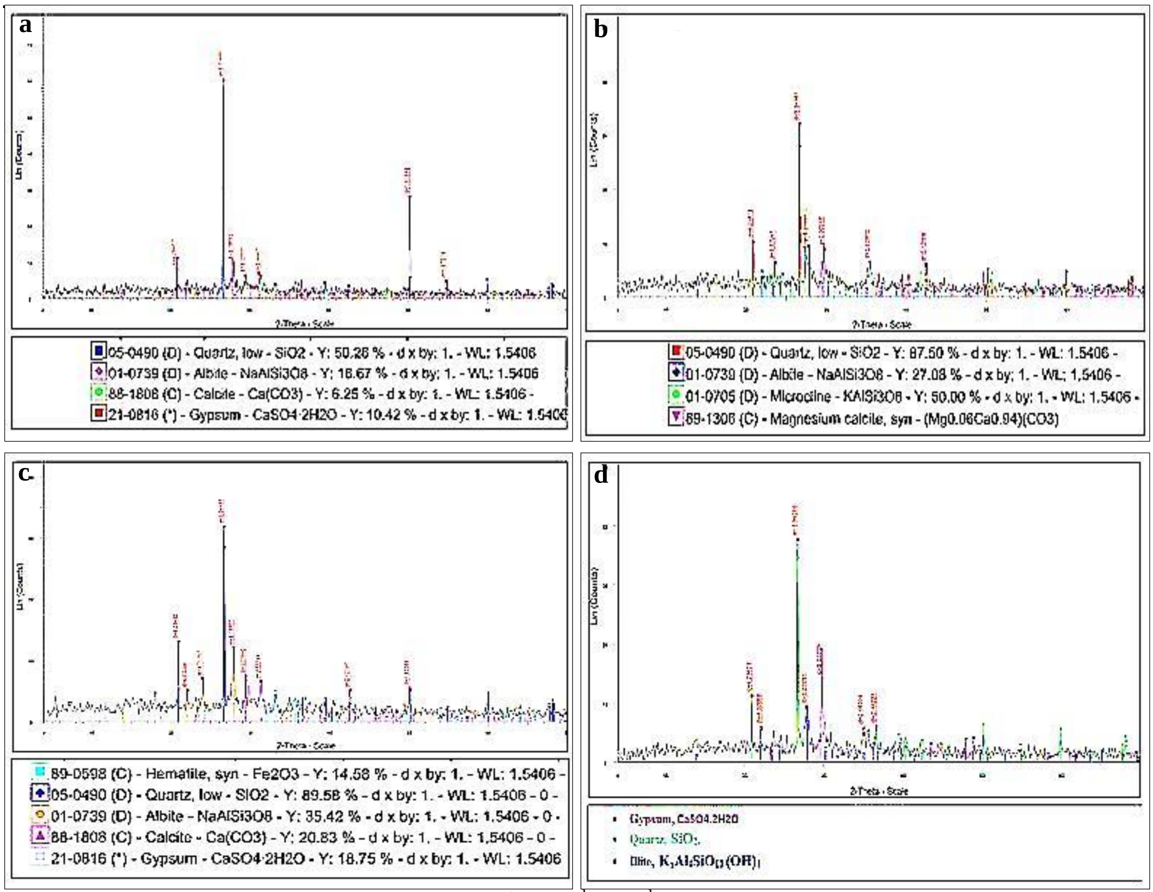

Figure (7) Shows the XRD patterns of the **a.** 1st, **b.** 2nd, **c.** 3rd pottery samples, **d.** soil sample

4. Discussion

It was clear from the visual and stereoscopic examination of the pottery objects figs. (2 & 3) that the pottery was formed by hand and the potter wheel [37]. The visual examination showed presences of sandy clay deposits due to burial in the soil [38]. In addition to salts crystals, some stains, cracking, black heart, fracture, soot, cavities, erosion, and surface deformation [39]. The polarized microscope, fig. (4-a & b) proved that the clay used in pottery manufacturing was Nile clay. It is classified as a type (**b**) due to the presence of biotite, muscovite, pyroxene, plagioclase, rutile, and orthoclase. These minerals that characterize the Nile clay in Egypt [40], PLM examination also showed the presence of some additives, such as sand, limestone powder, grog [41], and burnt straw. These tempers were the common materials used to modify clay to suit the shaping process [42,43], fig. (4-c & d). Moreover, PLM examination confirmed the presence of fine texture for the first and second shards, and a coarse texture for third pottery shard. The examination also, illustrated a mass of calcite, cracking form and quartz grains, which, is known as accidently quartz grains and polycrystalline quartz [44] fig. (4-e & f). The microbiological examination of the samples showed the presence of fungi sp. such as *Aspergillus flavus*, *Aspergillus niger*, *Aspergillus sulphureus*, *Aspergillus fumigatus*, *Rhizopus sp.* and *Penicillium sp.* as listed in tab. (1) and shown in fig. (5). They secrete organic acids that interact with the mineral components of pottery, causing physiochemical damage [45]. SEM morphological examination, fig. (6) showed that the samples have different cracks and various gaps, salts crystallization, soil sediments, erosion, fractures, surface flaking, slip layer peeling, separation of grains from each other. In addition to poor physical structure, and incoherence of the particles because of burial soil [46]. Moreover, tab. (2) showed some elements, such as Na, K, Ca, Mg, Fe, and Ti. These minerals that characterize Nile clay in Egypt [47]. SEM/EDX confirmed the burning quality of the first pottery sample for the presence of CO_2 at a low rate of 1.02% for the surface area and 1.01% for the core area. The good burning of the second sample was illustrated because carbon content reached 2.02% for the surface and 3.85% for the core area. This confirms that the firing atmosphere in the kiln was oxidizing for those samples. The percentage of carbon for the third sample was 5.68% for the surface and 5.63% for the core. This indicates a low firing degree for that shard [48]. The examination and analysis of the first pottery sample confirmed that the percentage of calcium was 20.18% for the surface and 20.08% for the core. The percentage of calcium in the second sample reached 11.87% for the surface and 11.55% for the core. Moreover, the percentage of calcium in the third sample was 11.33% for the surface and 13.11% for the core. It also confirmed the presence of halite salts, as the percentage of chlorine in the first pottery sample reached 6.55% for the surface and 5.28% for the core, while it was in the second sample 5.96% for the surface and 5.36% for the core. Furthermore, it reached in the third sample to 10.23% for the surface and 12.20% for the core. The sulfur percentage in the first sample was 2.30% for the surface, while it was in the second sample 2.15% for the surface and 2.03% for the core. The same elemental percentage in the third sample reached 3.18% for the surface and 3.10% for the core, this refers to sulfate salts. The phosphate percentage in the first pottery sample reached 6.38% for the core, while it was 2.43% for the surface and 2.99% for the core in the second sample, the same elemental percentage in the third sample reached 2.98% for the surface as XRD analysis, fig. (8) showed limestone powder (*calcite*, *dolomite*) as one of the tempers "*additive fillers*". It also revealed presence of chloride, sulfate, and carbonate salts in the pottery samples because of burial soil. It illustrated the presence of albite, quartz, and gypsum in the soil archaeological site, confirming that the soil was saline sandy clay soil [49].

5. Treatment and Conservation

Based on these results mentioned above, three pottery shards were restored and maintained according to scientific studies in the field of pottery restoration.

5.1. *Cleaning*

After documenting the objects, they were prepared to restoration steps. According to the strategies of pottery restoration, tooth and soft hairbrushes and scalpels were used due to its high efficiency for cleaning the strong adherent soil deposits. Clay sediments were locally cleaned using a mixture of (1:1:1) distilled water, acetone, and ethyl alcohol. Soot stains were also cleaned using a mixture of distilled water and ammonia topically. The calcareous deposits were cleaned using EDTA [50]. The fungal infections were removed by mechanically using soft brushes, followed by chemical cleaning using nano silver oxide 0.5%. The AT was set at 20 Ċ and the RH at 50:55% to prevent fungal infection again [51].

5.2. *Salts extraction*

Removal of salts was done mechanically by different soft metal brushes and scalpels. This step was carried out under a 6x magnification lens so as not to scratch the pottery surface. Then, soluble salts, "halite and phosphate" were cleaned using poultices. Carbonate and sulfate salts were cleaned by mora poultice [52].

5.3. *Consolidation*

The pottery artifacts were consolidated using 0.5%. Nano-silica through spraying method [53].

5.4. *Assembly*

Pottery shards were assembled using Paraloid B82 adhesive dissolved in toluene at a concentration of 50% [54].

5.5. *Replacement*

The second pottery shard was completed using a mixture of microballoon and pottery powder [55,56]. The restoration process of three pottery objects is shown in fig. (8).

Figure (9) Shows the restoration process of three pottery objects

6. Conclusion

The results, the research paper showed the microstructure and manufacturing process parameters at Tell Rawd Iskandar in Ismailia. The clay used was Nile clay (type B. The additive materials were sand, pottery powder "grog", limestone powder "calcite - dolomite", and burnt straw. The shaping technique was hand and potter wheel. The surface treatment was a red wash and slip layer. The firing atmosphere inside the kiln was oxidizing for the first and second shards and reducing for the third object. Moreover, the texture was fine for the first and second shards and coarse for the third object. The study proved that most of the pottery artifacts suffered from various damage manifestations, including soil deposits, soot, stains, crystallization of salts "halite, calcite, gypsum and phosphate". In addition severe effects resulted from fungal growths "Penicillium and Aspergillus", gaps, cracking, fracture, poor physical structure, lack of durability, and separation of granules. The pottery artifacts were treated by mechanical and chemical cleaning. A mixture of distilled water, acetone, and ethyl alcohol at a ratio of 1:1:1 was used for removing clay sediments. EDTA cleaned highly efficient lime deposits. Nano-silver oxide 0.5% cleaned fungal infections. 0.5% Nano-silica strengthened archaeological pottery by spraying method. In addition, 50% Paraloid B82 dissolved in toluene was used to assemble pottery shards. Finally, the objects were completed using a mixture of microballon and pottery powder. It should be preferable to the museum display AT 20 ^{o}C and 55:60% RH.

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