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DETERIORATION DIAGNOSIS AND TREATMENT OF SOME POTTERY OBJECTS FROM TELL ATRIB, QALYUBIA, EGYPT

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Abstract

Some pottery pots from tell Atrib in Qalyubia have been studied; it is one of the most important archaeological sites in Qalyubia dating back to the late age. Many tests and analyzes were performed such as polarized microscope (PLM), scanning electronic microscope equipped with an X-ray energy dispersion unit (EDX), X-ray diffraction (XRD) and microbiological examination. The research illustrates several important results in identifying pottery manufacture, where it was proved that the clay used in manufacture is Nile clay. Tempers are sand, burnt straw, grog powder and Calcite powder, which were used abundantly as an additive material to improve clay's properties. Shaping technique is coiling technique for the first piece and potter wheel for the second pot. Surface treatment is slip layer in all objects. The burning atmosphere was an oxidizing atmosphere for the first pottery piece and reduced for the second object. It also proved that most of the pottery pieces from tell Atrib in Qalyubia suffer from various damage manifestations such as surface deformation by soil sediments, fracture, cracking, gaps, fragmentation and crystallization of salts "chlorides, sulfates, carbonates and phosphates", damage was due to burial environment. These pottery objects were restored according to the results of tests, analyzes and damage manifestations proved by the research. Mechanical and chemical cleaning was used. Clay soil deposits were cleaned by a mixture of acetone and ethyl alcohol in a ratio of 1: 2. A solution of hydrogen peroxide (20%) was used in cleaning soot. Calcareous deposits were removed by EDTA.The pottery pots were strengthened by a mixture of nano-silica and wacker BS28 (concentration 1%). Assembling of pottery shards were done by paraloid 72% dissolved in toluene (concentration 50%).The research recommends that pottery objects in tell Atrib should be preserved after treatment intervention at a temperature 18:20 °C and Relative humidity 55: 60 % .

***Keywords**: Pottery; Damage; Soil; Salts; Thymol; Wacker; Nano Silica.*

Introduction

Tell Atrib is located 3 km northeast of city of Banha in governorate of Qalyubia. In Pharaonic era, it was known as Hat-hari-Ib meaning the central region palace, known as Atribes in the late age, but in Islamic age, it was known as Atrib [1].

It was tenth district in Delta; it is one of the most important archaeological sites in Qalyubia. Horus god represented the favorite god for Atrib [2]; there were some other local gods in Atrib such as km-wr god meaning the great lion [3]. The excavations revealed many silver and pottery artifacts dated back to the late age [4] as shown in figure 1.

Pottery objects suffer from soil damage and surface deformation such as soil deposits, stains, cracking, fracture, crystallization of salts and weaknesses [5], which requires restoration, treatment and maintenance. Cleaning process should be done according to nature of soiling [6],

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the best used materials in cleaning process are distilled water, solvents, enzymes, alkaline solutions and isolating materials, EDTA is used in cleaning of calcareous sediments or mineral stains [7]. Micro emulsions "O/W Micro emulsions" are used to avoid influence of solvents on the pottery objects; they are liquid and transparent materials [8].

Fig. 1. Excavations at the archaeological site, Tell Atrib, Qalyubia, Egypt

Nanomaterials or nanocomposites are used in cleaning and strengthening process [9]. In recent years, nanomaterials and nanocomposites have been used to strengthen archaeological materials, where some nanomaterials such as nano silica were added to acrylic and silicon polymers for improving their physiochemical and mechanical properties [10]. Pottery pieces buried in soil were broken due to weakness and Fragility of pottery, impact of internal pressures, strains and external soil loads [11]. There are many commercial materials or laboratory evaluated materials used in restoration, treatment, and maintenance process. Our presented study aims to restore and conserve the excavated pottery objects from Tell Atrib in Qalyubia based on laboratory evaluated materials and results of the examination and analysis that were proved by the research.

Materials and Methods

Materials

Two pottery samples were selected from tell Atrib in Qalyubia, as well as a sample from archaeological site soil, they were used in examinations and analyzes that were conducted by research.

Methods

Visual Examination

Using different lenses or a portable microscope is useful in identifying industrial technology and damage pottery in archaeological site [12].

Petrographic Examination

It clarifies micro-structure properties, additives materials, firing temperature and archaeological pottery damage [13]. Thin section of pottery samples "thickness 0.03 mm" was prepared for a petrographic examination by polarized microscope model: (Olympus BX51 TF japan attached with digital camera under magnification 4X up to 40X). This examination was done at Faculty of Science, Cairo University.

Scanning Electron Microscope with Energy Dispersive of X-Ray

It explains micro-structure properties, nature of firing temperature , mineral changes and archaeological pottery damage [14].Two pottery samples were examined without prior preparation using FEI Quanta 250 UK coupled with an energy dispersive X-ray spectrometer

(EDX), its Specifications: Filament Type: field emission, Detectors: SE/BSE/EDX/STEM/E-SEM, Specimen Stage: X-Y 50mm, Tilt angle = – 15° to + 75° ,Rotation = 360° (continuous),Max Specimen Size: 19×102mm (height x dia), Low vacuum capability for environmental SEM, Peltier cooling stage for low temperatures work/heating stage up to 800°C, Ideal for: high resolution imaging, And operating conditions "20kV and 1×10^{-9}A, and this examination was conducted at Electron Microscopy unit at the National Research Center in Cairo, Egypt.

X-Ray Diffraction Analysis

XRD diffraction analysis is useful in determining mineral composition of pottery pots and diagnosing archaeological pottery damage [15]. Three pottery samples were prepared for XRD analysis, the used device is Philips PW 1830 diffractometer equipped with a fine-focus tube, ICDD search/match program. It is used for qualitative and quantitative phase analysis. Operating conditions using at 40kv and 10mA, X - ray diffraction pattern was between 4:70 Ɵ, this analysis was conducted at Analytical Center at Ain Shams University.

Microbiological examination

Microbiological examination technique plays a very important role in identifying the most important fungal growths that cause archaeological pottery damage [16]. Swabs were done for infected potentially archaeological pottery pots**,** Potato Dexestrose Agar media was prepared, Operating conditions were 28°C, PH 5.5:6 for 25 minutes [17], as shown in Table 1.

Table 1. Composition of Potato Dextrose Agar growth media

Components	Weight
1- Potato Extract	200g
2- Dexestrose	20g
3- Agar	20g
4- Rose Bengal	10g
5- Stiriptomycene	Traces
6- Distilled Water	750cm^3

Then all fungi were isolated separately, then a sample was taken on a glass slide to be identified by microscope for each isolated fungus.

Results

Visual Examination

The visual examination of pottery objects of tell Atrib in Qalyubia proved that the shaping method for first pottery piece is coiling technique and potter wheel method for the second pottery pot. Surface treatment is slip layer for only outer surface. Burning is only good for the first pottery vessel. The second pottery pot suffers from black heart (core). The most important damage manifestations are surface deformation, salt crystallization, breaking and human damage by writing with ink on the pottery surface as shown in figure 2.

Examination by Polarizing microscope

Two pottery specimens from Tell Atrib in Qalyubia were examined by PLM, where figure 3 for surface of the first sample showed fine pottery fabric of sub-round and sub- angular quartz grains, in addition to added quartz grains, rutile, pyroxene and calcite (additive material) in an iron oxide-rich matrix. Figure 4 of the core also showed presence of fine quartz granules that was naturally present in the clay, as well as added quartz granules, calcite and pyroxene in an iron oxide-rich matrix.

Figure 5 for the surface of the second pottery sample showed fine pottery fabric of sub-round and sub- angular quartz grains, in addition to grog, burnt straw and rutile, in an iron oxide-rich matrix. Figure 6 of the core also showed presence of fine pottery fabric of fine quartz granules, as well as calcite, rutile and muscovite in an iron oxide-rich matrix.

Fig. 2. Damages of pottery objects excavated from tell Atrib in Qalyubia A: first pottery pot, B: second pottery pot

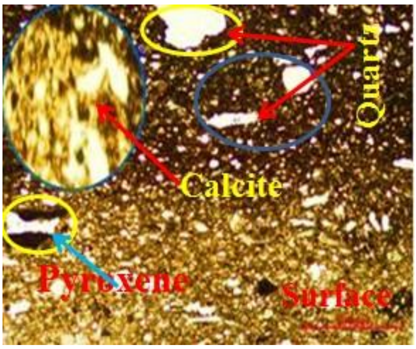


Fig. 3. Petrography micrograph of the first pottery sample shows existence of quartz, calcite, pyroxene and rutile (4X-CN)

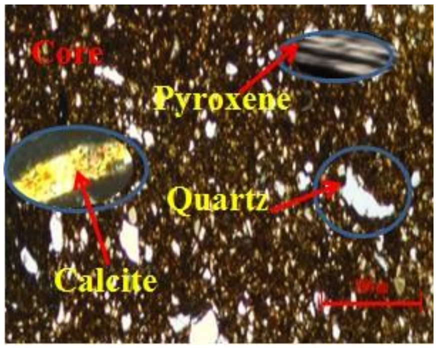


Fig. 4. Petrography micrograph of the first pottery sample shows existence of quartz, pyroxene, and calcite (10X-CN)

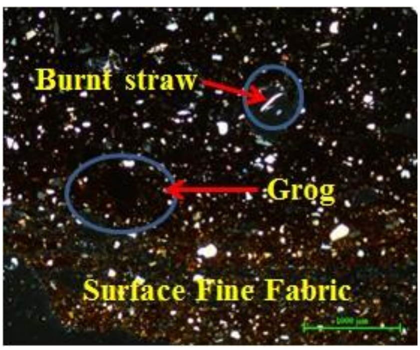


Fig. 5. Petrography micrograph of the second pottery sample shows existence of quartz, grog and burnt straw (4X-CN)

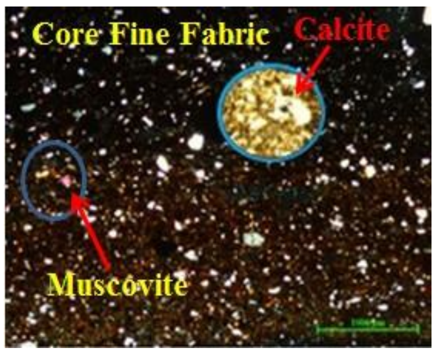


Fig. 6. Petrography micrograph of the second pottery sample shows existence of quartz, muscovite, and calcite (10X-CN)

Examination and Analysis by SEM-EDX

Figure 7 a of the first pottery sample shows that pottery sample suffers from presence of some different cracks and peeling of slip layer, as well as presence of soil deposits. Figure 7b of another part for the same first pottery sample shows cracks, gaps, crystallization of salts and granulation. Figure 8 a of the second pottery sample shows that the pottery sample suffers from presence of different cracks, flaking of slip layer, gaps and granulation, as well as presence of soil sediments. Figure 8b also shows cracks, granulation, gaps and crystallization of salts.

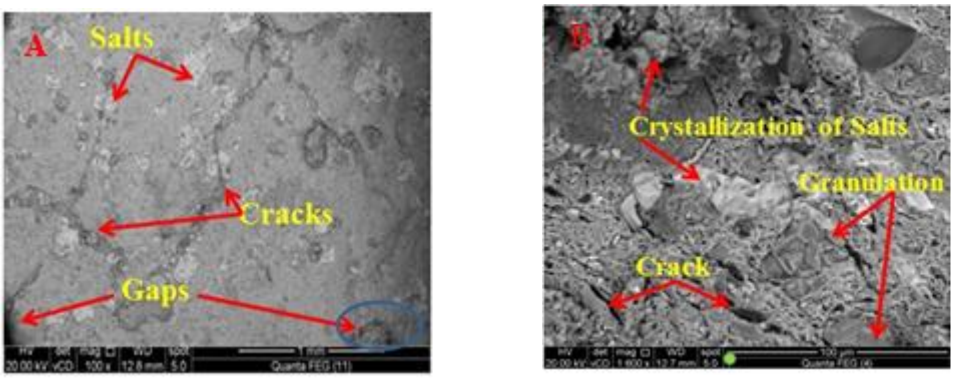


Fig. 7 a, b SEM photomicrographs of the first pottery sample

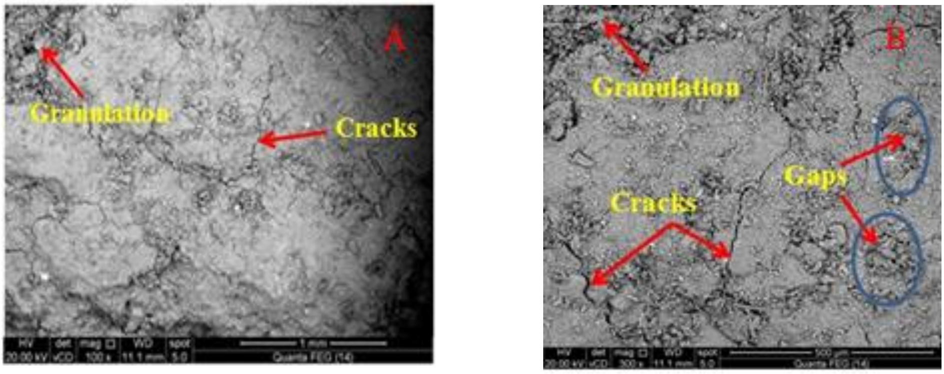
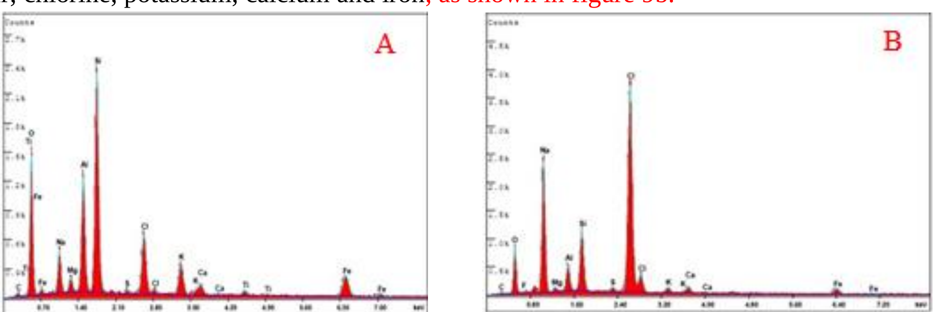


Fig. 8 a, b SEM photomicrographs of thesecond pottery sample

The results of analysis of the first archaeological pottery sample as shown in figure 9 a shows presence of carbon, sodium, magnesium, aluminum, silica, sulfur, chlorine, potassium, calcium, titanium, and iron. The results of analysis of another part of the same first pottery specimen demonstrated presence of carbon, fluorite, sodium, magnesium, aluminum, silica, sulfur, chlorine, potassium, calcium and iron, as shown in figure 9b.

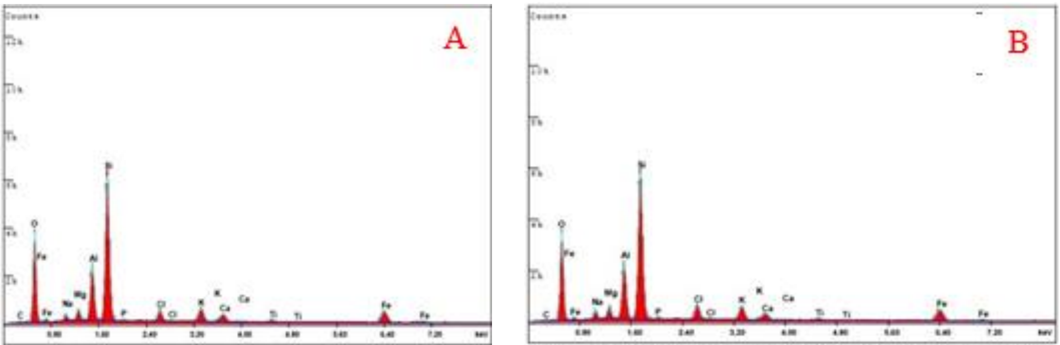


C	O	Na	Mg	Al	Si	S	Cl	K	Ca	Ti	Fe	Total
2.01	23.70	4.91	1.42	9.04	22.49	2.51	5.57	3.62	4.94	1.58	13.62	100%

C	O	F	Na	Mg	Al	Si	S	Cl	K	Ca	Fe	Total
4.42	14.44	0.28	11.90	1.70	8.90	10.71	1.88	33.33	3.01	4.39	5.04	100%

Fig. 9. EDX analysis on the surfaces of the first pottery sample

The results of analysis of the second archaeological pottery sample as in figure 10a demonstrated presence of carbon, sodium, magnesium, aluminum, silica, phosphate, chlorine, potassium, calcium, titanium, and iron. The results of analysis of another part of the same second pottery specimen showed presence of carbon, sodium, magnesium, aluminum, silica, phosphate, chlorine, potassium, calcium, titanium, and iron as shown in figure 10b.



C	O	Na	Mg	Al	Si	P	Cl	K	Ca		Ti	Fe	Total
2.54	27.61	1.46	1.95	8.11	28.92	1.42	5.06	4.71	2.95		1.33	13.94	100%

C	O	Na	Mg	Al	Si	P	Cl	K	Ca		Ti	Fe	Total
4.97	23.66	1.88	1.18	8.20	28.10	3.41	7.11	3.72	3.59		1.20	12.98	100%

Fig. 10. EDX analysis on the surfaces of the second pottery sample

X- Ray Diffraction analysis

Two pottery samples and one soil sample from the archaeological site have been analyzed by XRD. The pattern of the first pottery sample shows presence of halite NaCl, quartz SiO_2, Kaolinite $Al_2Si_2O_5(OH)_4$, microcline $KALSi_3O_8$, albite $NaAlSi_3O_{10}$, and diopside $CaSi_2O_6$ as shown in figure 11.

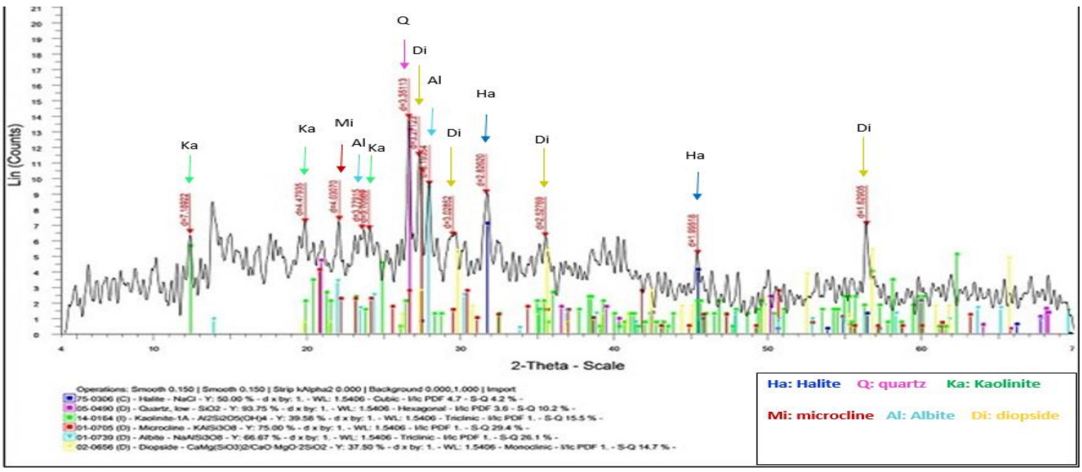


Fig. 11. X-ray diffraction pattern of the first pottery sample, Tell Atrib, Qalyubia

XRD pattern of the second pottery sample shows presence of quartz SiO_2, microcline $KALSi_3O_8$, albite $NaAlSi_3O_{10}$, calcite $CaCO_3$, magnetite Fe_3O_4 and halite NaCl as shown in Figure 12.

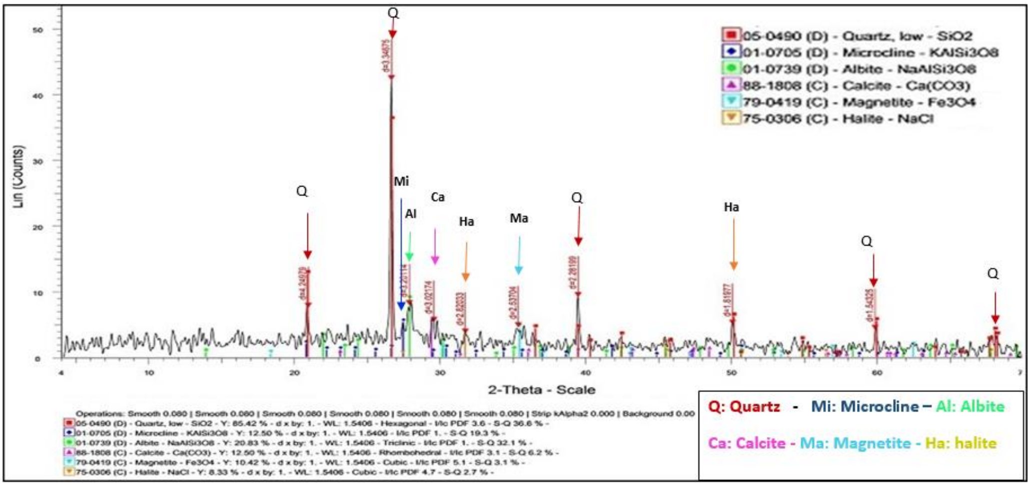


Fig. 12. X-ray diffraction pattern of the second pottery sample, Tell Atrib, Qalyubia

XRD pattern of soil sample from archaeological site shows presence of quartz SiO_2, albite $NaAlSi_3O_{10}$, calcite $CaCo_3$, halite NaCl and anhydrite $CaSO_4$ as shown in Figure 13.

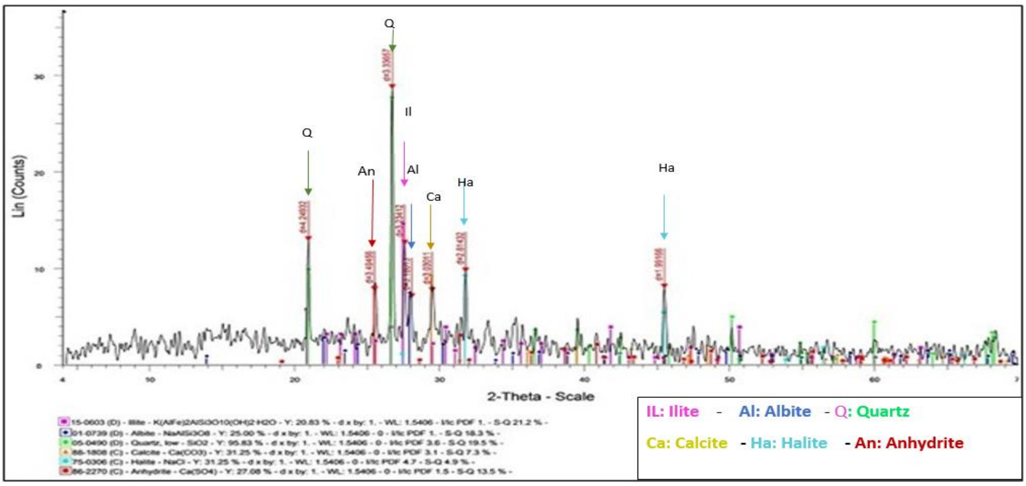


Fig. 13. X-ray diffraction pattern of soil sample, tell Atrib, Qalyubia

Microbiological Examination

The microbiological examination technique plays an important role in identifying the most important fungal growths for archaeological pottery group. The microbiological examination proved presence of fungal growths as shown in table 2 and figure 14.

Table 2. Presence of isolated fungi on samples

Pottery Samples	Identification of Fungi Isolated
S1	Aspergillus Fumigatus Penicillium Sp.
S2	Aspergillus Ochraceus Aspergillus Sulphureus

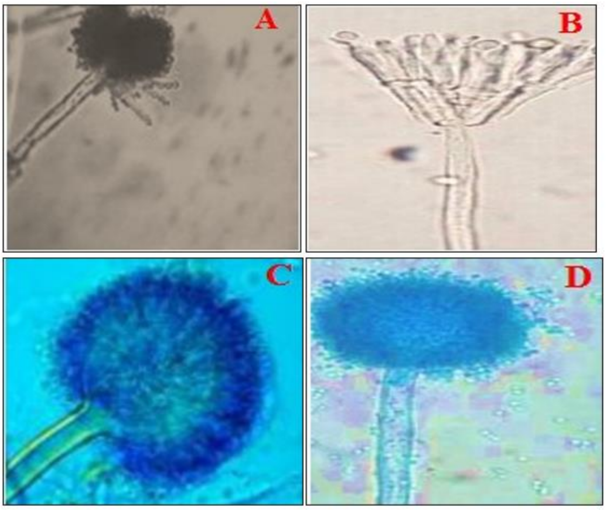


Fig. 14. Microbiological examination of archaeological pottery samples, tell Atrib, Qalyubia: A - Aspergillus Fumigatus; B - Penicillium Sp; C - Aspergillus Ochraceus; D - Aspergillus Sulphureus

Treatment Intervention

Tests and analyzes of Atrib pottery proved presence of clay sediments, as well as crystallized salts, fungal growths "penicillium and aspergillus", breaking, gaps and exfoliation. According to those results and scientific studies in the field of restoration, treatment and maintenance of pottery, our pottery was restored as the following.

Recording and Documentation

The pottery pots were recorded and documented by digital camera, photographical documentation is one of recording methods for state of pottery pieces excavated from the excavations in tells Atrib [18].

Cleaning

All loose clay deposits of pottery were cleaned by manual mechanical cleaning such as soft hair brushes [19], while all hardened and cohesive deposits of pottery pots were cleaned by scalpels and needles, cleaning was carried out from top to bottom with good care not to scratch the surface [20],mechanical cleaning process continued until it became useless [21],then chemical cleaning was used to clean sediments that were not be removed by mechanical cleaning [22], clay soil deposits were cleaned by a mixture of acetone and ethyl alcohol in a ratio of 1:2, this process was done locally, soot was cleaned by hydrogen per oxide solution (20%), calcareous deposits were removed by EDTA poultice [23], fungal stains of pottery objects were cleaned by mechanical cleaning using soft brushes [24],followed by chemical cleaning using thymol by spraying method [25].

Extraction of salts

The process of extracting salts aims to remove crystallized salts from pottery objects.it is carried out mechanically first and then chemically [26]. Through our research, salts of chlorides, sulfates and phosphates were removed mechanically first using soft hairbrushes and scalpels, this process was done under the lenses with magnification "6X" to avoid scratching pottery surface, then Japanese paper poultice saturated with distilled water was used to extract soluble halite and phosphate salts [27], Japanese paper poultice saturated with EDTA solution was used to extract calcareous deposits "carbonates-sulfates" [28].

Consolidation

Pottery pots excavated from tell Atrib were strengthened by using a mixture of nano silica and wacker BS 28 (concentration 1%) by spraying method [29, 30].

Assembling

Through our research, the pottery shards of tell Atrib suffer from breaking, pottery objects required bonding process, initial assembling process was used to find out places of shards not to be error in assembling process [31], then pottery shards were bonded using Paraloid B72 dissolved in toluene (concentration 50%) [32]. Treatment intervention of the two pottery pieces is illustrated as shown in figures 15 and 16.

Fig. 15. The treatment of the first pottery pot: A - before restoration intervention, B - during mechanical cleaning, C - before and after cleaning process and D - after bonding, consolidation and treatment

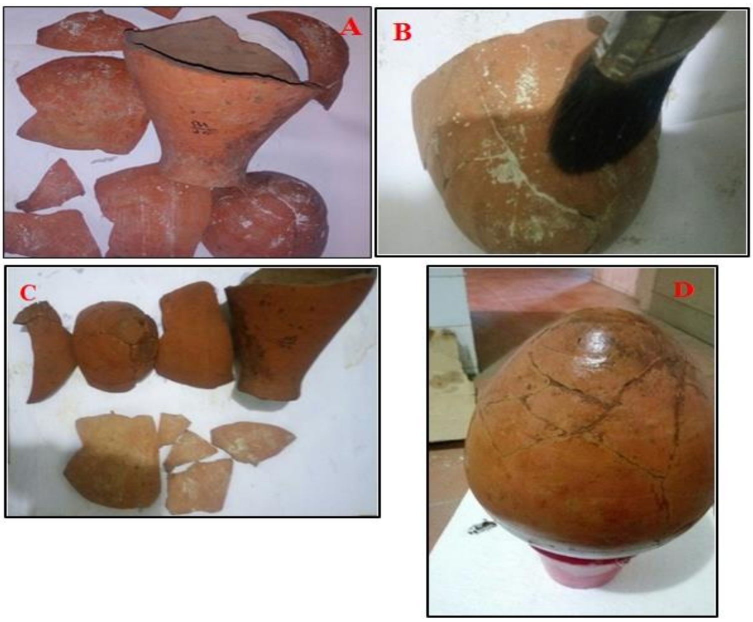


Fig. 16. The treatment of the second pottery pot: A - before restoration intervention; B - during mechanical Cleaning; C - before and after cleaning process and D - after bonding, consolidation and treatment

Discussions

It was evident through visual examination of pottery objects of tell Atrib in Qalyubia that pottery pots were formed by coiling method for the first pottery piece and potter wheel for the second pot, where signs of shaping appeared clearly on the pottery pieces, these methods are one of shaping methods in ancient Egypt as mentioned by *Shepared* [33],The visual examination also showed surface deformation by soil deposits, crystallization of salts, cracking and breaking, the deterioration was due to the soil contained most of the damage factors (burial environment) [34].The most important damage manifestations of pottery buried in the soil are phenomenon of breaking and surface deformation being pottery pots are made of mineral components characterized by heterogeneity and weak resistance to mechanical shocks [35], surface deformation by soil deposits may be highly adherent to the surface or within the gaps or cracks [36]. Damage in Atrib site is due to type of pottery, its porosity and kind of burial environment [37], formation techniques for Atrib pottery plays an important role in increasing damage rate because it is not polished [38]. Cracking was due to manufacture defects especially drying and burning process [39]. It was noted that there is a black core in the second pottery piece due to burning defect [40]. Through our research, we demonstrated existence of black core as shown in figure 2b.

Salt crystallization in pottery pots is due to absorption of aqueous and salt solutions from the soil and their evaporation by heat causing surface deformation and cracks. Salts crystallization is dependent on porosity, surface tension and temperature [41]. The degree of damage also depends on speed at which the salts crystallize and presence more than one type of salts inside the pottery body. Its danger is pressures known as hydration pressure [42]. Crystallization of salts causes weakness [43], cracking, breaking, and exfoliation are due to crystallization and recrystallization of salts [44]. Through our presented research, it was found that the pottery pieces" case study" suffer from all aspects of damage such as black core, surface deformation, clay deposits, stains, cracking, fracture, and crystallization of salts as in figure 2a and b.

Polarized microscope proved that the used clay in manufacture of pottery in tell Atrib is Nile clay because of presence of muscovite, pyroxene and rutile, these minerals are Characteristic for Nile clay [45]. Surface treatments in ancient Egypt was slip layer and red wash [46, 47]. Through our research paper, it was proved that some additives are burnt straw, sand, limestone powder and pottery powder" grog". Limestone powder is abundant in our samples. It is proved through our research that there was slip layer in samples, fine pottery fabric containing some added quartz granules as shown in figures 3-6. In ancient Egypt, Nile clay is characterized by presence of carbon, sodium, magnesium, aluminum, silica, sulfur, chlorine, potassium, calcium and iron [48]. SEM examination demonstrated presence of gaps, cracking and salt crystallization as in figures 7 and 8. EDX showed that the used clay was Nile clay because it contained above mentioned elements as in figures 9 and 10. SEM-EDX also demonstrated nature of burning process according to percentage of carbon in our samples, carbon percentage in the first sample was 2.01% and 4.97% for the second sample, which confirms that burning atmosphere was an oxidizing atmosphere for the first piece and reduced atmosphere for the second piece.

The examination and analysis confirmed presence of salts of chlorides, sulfates and phosphates as, chlorine percentage in the first sample was 8.57% and 7.11% for the second sample, sulfur percentage in the first sample reached 2.51% for the surface and 1.88% for the core, while there was no sulfur in the second sample, phosphorous percentage in the second sample was 1.42% to surface and 3.41% to core, these results confirmed presence of salts of chlorides, sulfates and phosphates due to burial environment contained dissolved salts, it caused some physiochemical damage manifestations [49]. X-ray diffraction analysis demonstrated presence of limestone powder as one of the temper additives. It also showed quality of burning for the first pottery sample that contained diopside which appears above 850 °C and low burning temperature for the second pottery sample, where absence of diopside, presence of magnetite and calcite demonstrated reducing atmosphere for the second sample [50]. The analysis also demonstrated presence of salts of chlorides and sulfates in pottery samples. XRD also proved presence of illite, microclines, albite, halite and sulfates in archaeological site soil confirming that the soil is source of salts for pottery in tell Atrib in Qalyubia, as in figures 11-13. It is known that the archaeological materials buried in the soil suffer from organic damage [51, 52], as fungi excreted organic and inorganic acids that interact with minerals components of pottery causing physiochemical damage [52, 53]. Through our presented research, the microbiological examination of pottery samples confirmed presence of fungi such as aspergillus Fumigatus, penicillium Sp., aspergillus ochraceus, and aspergillus sulphureus, as shown in figure 14.

These pottery pots were restored according to results of tests, analyzes and scientific studies in treatment of pottery, the research proved that mechanical cleaning is safer than chemical cleaning, a mixture of acetone and ethyl alcohol in a ratio of 1:2 was good to remove clay soil deposits [19], a solution of 20% hydrogen peroxide was useful in cleaning soot ,calcareous deposits were efficiently cleaned by EDTA [23], pottery pots were soundly consolidated by a mixture of nano-silica and wacker BS28 (concentration1%) using spraying method in application consolidants [29], paraloid B 72 dissolved in toluene (concentration 50%) was efficient in assembling of the pottery shards [32].

Conclusions

The research illustrates several important results in identifying pottery manufacture from tell Atrib in Qalyubia, where it was proved that the clay used in manufacture is Nile clay. Tempers are sand; burnt straw, and limestone powder, calcite was used abundantly as an additive material to improve clay's properties. Shaping technique is coiling technique for the first pottery piece and potter wheel for the second pot. Surface treatment is slip layer in all objects. The burning atmosphere was an oxidizing atmosphere for the first pottery piece and reduced for the second object. It also proved that most of pottery pieces from tell Atrib in Qalyubia suffer from various damage manifestations such as surface deformation, clay sediments, fracture, cracking, gaps, fragmentation and crystallization of salts especially salts of chlorides, sulfates, carbonates and phosphates, damage was due to burial environment.These pottery pots were restored using mechanical and chemical cleaning, a mixture of acetone and ethyl alcohol in a ratio of 1:2 was used to remove clay soil deposits. a solution of hydrogen peroxide (20%) was used in cleaning soot, calcareous deposits were cleaned by EDTA, the pottery pots were consolidated by a mixture of nano-silica and wacker BS28 (concentration 1%) using spraying method in application of consolidants, paraloid B 72 dissolved in toluene (concentration 50%) in assembling of pottery shards. The research recommends that archaeological pottery pots should be displayed in museum at a temperature 18:20°C and Relative Humidity 55: 60%.

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