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MICROSTRUCTURE AND DAMAGE ASSESSMENT OF SOME PTOLEMAIC POTTERY OBJECTS AT TELL ABU YASIN IN SHARKIA, EGYPT: CASE STUDY

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ABSTRACT

Tell Abu Yasin is one of the most important sites in Sharkia governorate and dates back to Ptolemaic age. Many tests and analyses were performed to diagnose damage manifestations of some pottery pieces. The research has proved that the clay used in that pottery is Nile Clay, while tempers are sand, grog, and limestone powder, the used shaping technology is potter wheel, surface treatment is slip layer, burning atmosphere inside the kiln was reduced for the first pot and oxidized to the second and third piece. Pottery objects suffer from Physiochemical damage by soil sediments, in addition to phenomenon of fracture, scaling, cracking, weakness, fragility, and crystallization of chlorides, sulfates, carbonates and phosphates salts as a result of burial in saline clay soil. The research recommends that restoration; treatment and maintenance of these pottery pieces should base on results of examinations, analyses and damage assessment that have been proved by the researcher.

KEYWORDS: Pottery, clay, Additives, Slip layer, Firing atmosphere, Damage.
I. INTRODUCTION

Tell Abu Yasin area is located in Sharkia Governorate, it is located 4 km southeast of Abu Kabir and 40 km northeast of Zagazig city, it belongs to Ptolemaic period, where excavations were carried out by the Supreme Council of Antiquities mission revealing many different shapes and colors pottery pieces, as well as many different kilns, stoves and a cemetery including some granite sarcophagi. Tell Abu Yasin is located within district of Horbait. It was known by ti -rimo. Its name is derived from ancient Egyptian name «Hur Bit » and Farphithios in Greek age.

Its current location is village of Horbit in Sharkia, Egypt. Pottery is one of the most important types of artifacts for the regional chronology and distribution of cultures, in addition to demonstrating relationships between settlement sites due to difference and diversity of manufacturing techniques, tools, shapes and decoration patterns. Microstructure and tempers play a role in identifying technology of manufacture and classification of pottery, in which there has been clear progress in the field of examinations and analyses in recent times.

The methods of examination and analysis in field of study of archaeological materials have differed and varied according to their different objectives such as micromorphology, mineral composition, structure behavior and Thermal characteristics. Microscopic examination of pottery provides information on microcomposition and morphology of archaeological samples, while petrographic structure study plays an important role in identifying mineral components, microstructure, shape and size of grains, their relationship with each other and their distribution in the pottery specimen. Examination and analysis give a clear map of the nature of damage whether it is defects of workmanship or soil decay. The pottery pieces suffer from damage due to manufacturing defects such as fine cracks, black core and staining phenomenon as a result of burning process. Many pottery relics such as terracotta have many damaged aspects of Industrial process. Burning failure leads to rapid surface deterioration for terracotta, and high firing temperatures create glass matrix that may damage pottery product.

Pottery pieces buried in the soil suffer from physiochemical damage that deforms the surface and mineral composition. Damage speed and its rate depend on type of burial
environment, whether it is dry or wet burial environment. Pottery pieces suffer from staining phenomenon because of burning or iron deposits in soil of archaeological site. This staining phenomenon increases in porous pottery. Pottery often suffers from phenomenon of fracture due to soil mechanical damage, this phenomenon increases in marine burial environments due to intense water currents. Among the most important damage aspects of pottery buried in the soil is crystallization of salts, cracking and fracture.

The resulting damage is physiochemical deterioration, fragmentation may be done within a few minutes during excavations. This type of pottery needs restoration, treatment and maintenance process to remove soil sediments, as well as strengthening process that consolidates and links the granules and improves physical and mechanical properties especially using nano materials in consolidation in recent times.

Therefore, this study aims to determine microstructure, mineral composition and damage assessment of archaeological pottery at Tel Abu Yasin in Sharkia for its treatment and maintenance based on those results that had been proved by the researcher.

II. STUDY MATERIALS AND METHODS

1. Study Materials

Four samples were selected including three samples of pottery pieces extracted from Tel Abu Yasin in Sharkia (case study), as well as one sample of archaeological site soil, all of which were used in examination and analysis that was conducted by the researcher.

2. Study Methods

A. Visual Examination

Visual examination method is the first stage of examination process where some lenses and stereomicroscopes with different magnification are used in visual assessment process of pottery samples.

B. Petrographic Examination

Polarizing microscope is one of the important methods in study of petrographic structure, mineral changes, function use, tempers, grains size, mineral composition,

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13 FLORIAN 2000:1.
14 HODGE 1986: 147.
15 PRICE 1984: 4.
16 HAMITON 1998: 17; PEARSON 2000: 98.
17 SMITH 1999: 163.
18 CRONYN 1996: 29.
19 PLENDERLEITH 1971: 125.
20 CIENZEA,2012: 28.
21 El-GHAREB 2017: 76- 77.
22 STOLTMAN 2001: 297- 326.
surface treatment, in addition to glass phases\textsuperscript{23}. Thin section was prepared for examination using polarizing microscope; the used device was Nikon Eclipse. This examination was performed at the Geological Survey in Cairo.

C. Scanning Electron Microscopy with EDX Unit

It is one of the most important modern technologies in field of examination and analysis of pottery. Scanning electron microscope equipped with EDX unit describes morphology of pottery surface, size grains, distribution of particles, texture, surface treatment and damage forms such as gaps, cracks, crystallization of salts, peeling and mineral composition of archaeological pottery\textsuperscript{24}. The device used is model Quanta FEG250 SEM unit. This examination was performed by scanning electron microscope unit at National Research Center in Cairo.

D. XRD analysis

It gives information about nature of pottery manufacture technology especially firing process, mineral changes which were been caused by industry or burial in the soil\textsuperscript{25}. The device used is XRD device model Philips Analytical X-Ray, this analysis was done at XRD unit at Center of Microanalysis at Ain Shams University.

E. Thermal Analysis

Thermal Differential Analysis «DTA» was used to determine firing temperature of pottery due to mineral changes that occurred during firing of pottery\textsuperscript{26}. Two pottery samples were analyzed by thermal analysis device Known as Perkin Elmer STA 6000, the temperature program ranged from room temperature to 1500°C, measurement range: ± 0.2 to ± 1000 μV, heating speed: 0 to+50°C/min, temperature, mineral changes were recorded on the chart. This thermal analysis was performed at faculty of science, Cairo University.

3. Results
A. Visual Examination

Visual examination proved that the pottery pots in Tel Abu Yasin in Sharkia suffered from high percentage of clay soil sediments, crystallization of salts, various stains, missing parts, black core, peeling and separation of slip layer, it also proved that forming method was potter wheel method for the first and third piece, as well as coiling shaping method was used for the second piece as in [FIGURE 1].

\textsuperscript{23} RIEDEBER 2004: 143- 158.
\textsuperscript{24} MORO et AL 2020: 223-232.
\textsuperscript{25} CLIMENT et AL 2018: 240.
\textsuperscript{26} BAYAZIT et AL 2014: 14769- 14779.
B. Examination by polarizing microscope

Pottery samples were examined by polarized microscope as in [FIGURE 2], where surface examination with polarized light showed presence of quartz grains, most of which are fine grains, as well as some coarse quartz grains, some grains are semi-circular, others are sharp angular and biotite as shown in [FIGURE 2/A]. Another part of the same sample was also examined showing presence of quartz grains from fine grains to coarse grains, as well as limestone powder, biotite and rutile as in [FIGURE 2/B]. The examination of core showed presence of quartz grains, as well as limestone powder, biotite and rutile as in [FIGURE 2/C]. An examination of another part of core illustrated presence of quartz grains from fine grains to coarse grains, as well as grog, biotite and rutile as in [FIGURE 2/D].
FIGURE 2: Petrographic examination of the first sample

A: quartz, biotite, rutile. B: quartz, biotite, limestone powder. C: quartz, biotite, calcite & olvire
FIGURE 3: for the second pottery sample also showed presence of a delicate texture of quartz granules, in addition to remnants of calcite, pyroxene, burnt straw and rutile as in [FIGURE 3/A]. Examination of another part of the same sample showed presence of fine quartz granules, as well as calcite, biotite, plagioclase, and rutile as in (FIGURE 3/B), the examination of core illustrated presence of fine quartz grains, some accidentally quartz grains as one of additives materials, as well as calcite, biotite, rutile and burnt straw as in [FIGURE 3C], the examination «another part of core» also showed presence of fine quartz, calcite, biotite, rutile as in [FIGURE 3/D].

[FIGURE 3]: Petrographic examination of the second pottery sample. A: quartz, calcite, biotite, burnt straw & pyroxene. B: quartz, calcite biotite & pyroxene. C: quartz, biotite, calcite, burnt straw & rutile. D: quartz, calcite, biotite
For the third pottery sample, [FIGURE 4] also showed presence of fine to medium pottery texture of quartz grains, in addition to calcite, grog and rutile as in [FIGURE 4/A], another part of the same sample was examined showing presence of fine quartz granules, as well as grog, biotite, muscovite and rutile as in [FIGURE 4/B]. The examination of core illustrated presence of fine pottery texture of fine quartz granules, in addition to calcite, biotite, rutile, and burnt straw as in [FIGURE 4/C]. It also showed presence of fine pottery texture of fine quartz grains, as well as presence of calcite, biotite, rutile, and burnt straw as in [FIGURE 4/D].

[FIGURE 4]: Petrographic examination of third pottery specimen: A: quartz, biotite & rutile. B: quartz, grog biotite & muscovite. C: quartz, biotite, calcite & burnt straw. D: quartz, calcite, burnt straw, biotite, grog & plagioclase.
C. Examination and Analysis by Scanning Electron Microscope with EDX Unit.

The pottery pieces extracted from tel Abu Yasin in Shakira were examined by environmental scanning electron microscope.

**- Scanning Electron Microscope Examination**

[FIGURE 5] shows scanning electron microscope examination of the first sample (surface area), where examination showed that the sample suffers from presence of soil sediments as a result of burial in the soil for thousands of years, gaps, cracks, peeling and crystallization of salts as shown in [FIGURE 5/A]. Another part was examined for core area, where examination showed presence of some coarse quartz grains, as well as some gaps, peeling and crystallization of salts as in [FIGURE 5/B].

![FIGURE 5]: SEM examination of the first pottery specimen A: SEM examination of surface area (200X), B: SEM examination of core matrix (1600X).

The [FIGURE 6] showed scanning electron microscope examination of the second sample for surface area, where it was found that the sample suffered from many cracks, gaps and scaling as in [FIGURE 6A]. Another part of core matrix was examined, where examination showed presence of some quartz grains, as well as some cracks, peeling of slip layer and crystallized salts as in [FIGURE 6/B].

![FIGURE 6]: SEM examination of the second pottery specimen. A: SEM examination of surface area (200X), B: SEM examination of core (1600X).
The [FIGURE 7] represents scanning electron microscope examination of the third sample of surface area showing that the sample had a fine texture, as well as presence of cracks, peeling, and some parts of surface treatment as in [FIGURE 7/A]. Another part of core area was examined, where the examination showed presence of crusting of slip layer, some gaps and crystallization of salts as in [FIGURE 7/B].

![FIGURE 7]: SEM examination of the third pottery specimen. A: SEM examination of surface area (300X). B: examination by SEM of core matrix (400 X).

- Scanning Electron Microscope Analysis
  
  The results of EDX analysis of three pottery specimens for surface and core as shown in [FIGURE 8/A-F] clarified presence of carbon, oxygen, sodium, magnesium, aluminum, silica, Molybdenum, sulfur, chlorine, potassium, calcium, titanium, Vanadium and iron. These EDX analytical results for three sample results as shown in [TABLE 1].
[FIGURE 8]: A-F: EDX analysis patterns of pottery samples, AbuYasin, Sharkia. A-B: first pottery. C-D: Second pottery. E-F: Third pottery.
### TABLE 1: EDX analytical results of investigated pottery shards

| Element | S1 core | S1 surface | S2 Core | S2 Surface | S3 Core |
|---------|---------|------------|---------|------------|---------|
| C       | 5.57    | 1.66       | 1.20    | 1.06       | 2.94    |
| O       | 13.50   | 27.57      | 27.54   | 26.16      | 13.26   |
| Na      | 3.30    | 2.17       | 3.16    | 2.52       | 0.62    |
| Mg      | 1.93    | 3.04       | 2.71    | 2.82       | 0.96    |
| Al      | 2.15    | 9.87       | 9.67    | 8.60       | 6.32    |
| Si      | 4.22    | 25.85      | 20.05   | 25.68      | 17.27   |
| Mo      | 0.57    | -          | -       | -          | -       |
| P       | -       | 0.22       | -       | -          | 0.15    |
| S       | -       | 0.39       | -       | 0.42       | 0.29    |
| Cl      | 4.26    | 6.09       | 7.70    | 5.92       | 0.32    |
| K       | 0.48    | 2.23       | 2.08    | 2.07       | 6.71    |
| Ca      | 0.68    | 3.39       | 2.68    | 10.26      | -       |
| Ti      | 27.57   | 1.52       | 1.45    | 1.24       | 14.56   |
| V       | -       | -          | -       | -          | 0.26    |
| Fe      | 40.41   | 40.41      | 13.81   | 13.18      | 38.31   |

Done by the author.
X-Ray Diffraction Analysis

Three pottery samples and one soil sample from archaeological site were analyzed by XRD. The pattern of three pottery samples showed presence of quartz SiO$_2$, microcline KAlSi$_3$O$_8$, albite NaAlSi$_3$O$_10$, halite NaCl, hematite Fe$_2$O$_3$, Magnetite Fe$_3$O$_4$, anhydrite CaSO$_4$. Soil sample pattern contained quartz SiO$_2$, microcline KAlSi$_3$O$_8$, albite NaAlSi$_3$O$_10$, halite NaCl, as shown in [TABLE 2] and [FIGURE 9/A-D].

| Minerals  | Chemical composition | a   | b   | c   | d (soil) |
|-----------|----------------------|-----|-----|-----|----------|
| quartz    | SiO$_2$              | 36.6| 25.9| 25.2| 20.5     |
| microcline| KAlSi$_3$O$_8$       | 19.3| 29.6| 23.2| 30.7     |
| albite    | NaAlSi$_3$O$_10$     | 32.1| 29.5| 25.6| 43       |
| calcite   | CaCO$_3$             | 6.2 | -   | -   | -        |
| hematite  | Fe$_2$O$_3$          | -   | 7.8 | 4.4 | -        |
| Magnetite | Fe$_3$O$_4$          | 3.1 | -   | -   | -        |
| halite    | NaCl                 | 2.7 | 7.2 | 8.9 | 5.8      |
| anhydrite | CaSO$_4$             | -   | -   | 12.7| -        |

[TABLE 2]: XRD analytical results of investigated pottery shards
Done by the author.
[FIGURE 9]: XRD patterns of three pottery samples and one soil sample, Abu Yasin, Sharkia, Egypt. A: the first pot. B: the second potter plate. C: the third pottery vessel. D: soil sample.
- Differential thermal analysis

Differential thermal analysis «DTA» of the first pottery sample showed slight minerals changes in firing temperature from 94.81°C to 178.76 °C., the minerals changes increased from 406.96 °C to 705.26° C, the stability of the mineral changes from 705.26 °C to 1000 °C. The results of mineral changes indicated that the firing temperature might be about 705.26 °C, as in [FIGURE 10].

![DTA pattern of the first pottery sample, Abu Yasin, Sharkia, Egypt](image1)

Differential thermal analysis «DTA» of the second pottery sample showed slight minerals changes in firing temperature from 24.78°C to 84.88 °C., the minerals changes increased from 278.61° C to 782.99° C, the stability of the mineral changes from 782.99 °C to 1000 °C. The results of mineral changes indicated that the firing temperature might be about 782.99 °C as in [FIGURE 11].

![DTA pattern of the second pottery sample, Abu Yasin, Sharkia, Egypt](image2)
to 1000 °C. The results of mineral changes indicated that the firing temperature might be about 782.99 °C as in [FIGURE 11].

Differential thermal analysis «DTA» of the third pottery sample showed slight minerals changes in firing temperature from 50.45°C to 159.59 °C, the minerals changes increased from 379.01 °C to 773.37 °C, the stability of the mineral changes from 773.37 °C to 1000 °C. The results of mineral changes indicated that the firing temperature might be about 773.37 °C as in [FIGURE 12].

III. DISCUSSION OF RESULTS

A visual examination revealed that the pottery pieces in Tel Abu Yasin were shaped by potter wheel and coiling method, which was one of formation methods in ancient Egypt27. Visual examination also showed presence of a large proportion of soil sediments, and the severity of damage depends on texture of pottery28. Pottery suffered stains because the soil was rich in iron oxides29, and one of the four pottery pieces suffered from soot due to reduced atmosphere inside the kiln or functional use30. This pottery often suffered from presence of black core31, pottery pieces suffered from fracture due to mechanical damage of the soil. The severity of the damage depends on type of pottery, soil chemistry, soil column, plants and trees in the soil, or by

27 BOURRIAU 1981: 15.
28 BUYS & OAKLEY 1999: 24.
29 HODGE 1986: 147.
30 WELSBY 1997: 27.
31 SHEPARED 2000: 75.
environmental shocks. The visual examination also proved presence of crystallization of salts as shown in [FIGURE 1].

The petrographic study using polarized microscope proved presence of muscovite, orthoclase, pyroxene, and rutile. These minerals are characteristic of the Nile clay confirming that the clay used in pottery manufacture at Tel Abu Yasin was Nile clay. Petrographic study also demonstrated presence of sand, burnt straw «where places of burning of burnt straw have been seen as evidence of the presence of organic matter (burnt straw), but it was burned during firing of pottery inside the kiln», limestone powder and grog (fillers were added during kneading of clay to improve the properties).

Petrographic study also proved existence of surface treatment by applying slip layer, where there was a difference in size of particles of slip layer from size of granules of pottery body. Manufacturer was always assiduous to select fine clay granules in slip layer «surface treatment». It was evident in petrographic study that manufacturer had failed in surface treatment of pottery plate, it was proved that slip layer was coarse clay grains, it was larger than size of granules of pottery body itself as in [FIGURE 4].

Petrographic study proved low temperature burning for first pottery vessel; this was confirmed by absence of firing minerals such as gahlenite and diopside, which are glass phases that appear at 850 °C. It showed quality of burning of the second and third pottery piece; this was confirmed by presence of hematite.

Examination and analysis by scanning electron microscope coupled with X-ray energy dispersion unit (SEM-EDX) confirmed that clay used in manufacture of pottery at Tel Abu Yasin is Nile clay due to presence of some oxides such as sodium, potassium, calcium, iron and magnesium, which are characteristic of Egyptian Nile clay. It also illustrated presence of calcium carbonate (C, Ca) as one of the added fillers; it also showed crystallized salts such as halite, carbonates, sulfates and phosphates due to burial in saline soil as shown in [FIGURES 5-7]. Examination and analysis confirmed low burning temperature due to presence of carbon dioxide at a high rate, which reached 4.07% in some samples.

It also showed presence of sulfates salts, where the percentage of sulfur oxide reached between 2.14% to 7.65%. It also showed presence of halite salts, where percentage of chlorine in samples reached between 3.69% to 6.87% as shown in [FIGURE 8 /A-F].

Analysis by XRD revealed presence of mica and microcline confirming that clay used in manufacture was Nile clay, it also showed firing temperature of pottery samples. For the first sample, calcite was existed as a trace component up to 600-800 °C, and it decomposed completely on firing around 800-850 °C, This indicates that the burning

32 CRONYN 1996: 29.
33 NICLOLSON & SHAW 2000: 67.
34 GRIMSHAW 1971: 561.
35 EL-GOHARY et AL 2019: 17611.
36 NAGWA 2016: 22.
37 MOHAMED 2019: 232.
temperature of first sample may be between 700 °C to 800 °C. XRD also confirmed good firing for the second and third piece due to presence of hematite. It also proved that limestone powder was used as one of the additive fillers for presence of calcite in first sample as in [FIGURE 9/ A], XRD also illustrated presence of halite salts in all samples, it also showed presence of quartz, microcline, albite and halite in soil sample Which proves that source of the salts in pottery samples was soil in tell Abu Yasin in Sharkia as in [FIGURE 9/D].

Differential thermal analysis «DTA» indicated that the firing temperature degree of our samples might be about 705.26 °C for the first pottery sample; the firing temperature might be about 782.99 °C for the second sample and it might be 773.37 °C for the third pottery sample as according to minerals changes as in [FIGURES 10,11,12]38.

IV. CONCLUSION

The research has concluded some great important results in identifying technological process and damage of pottery objects at tel Abu Yasin in Sharkia. It proved that used clay was Nile clay; additions were sand, burnt straw, grog and limestone powder. Surface treatment was slip layer in samples; burning atmosphere inside the kiln was reduced for the first pot and oxidized to the second and third pieces. It also illustrated mechanical and physiochemical damage, whether from manufacture defects or effect of burial environment. It has proved the presence of soil sediments, crystallization of salts «carbonate, sulfates, chloride», cracking, peeling, stains and poor physical structure. The research recommends that it should be treated and maintained based on the results of the tests and analyses that have been proven by the researcher.

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MICROSTRUCTURE AND DAMAGE ASSESSMENT OF SOME PTOLEMAIC POTTERY OBJECTS AT TELL ABU YASININ SHARKIA, EGYPT: CASE STUDY

التركيب الدقيق وتقييم تلف بعض الآثار الفخارية البطتمية
بتل آثار أبو ياسين بالشرقية مصر: دراسة حالة

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المختصر
بتل آثار أبو ياسين من أهم مواقع محافظة الشرقية، ويقع على بعد 4 كم جنوب شرق أبو كبير و 400 كم شمال شرق الزقازيق، وهو ينتمي من الناحية الأثرية إلى العصر البطتمي حيث كشفت الحفائر التي قامت بها بعثة المجمس الأعمى للآثار عن العديد من الآثار الفخارية المختلفة الأشكال والألوان، فضلا عن العديد من مواد الأفران المختلفة وجوانب تضم بعض التوابيت الجرانيتية. وتم إجراء العديد من الفحوص والتحليلات المختلفة مثل الفحص بالميكروسكوب المستقطب (Polarized Microscope (PLM)) وتحليل طريقة حيود الأشعة السينية X-Ray Diffraction analysis (XRD) والتحليل الحراري (Thermal Analysis (DTA)) واستخدمت الفحوص والتحليلات المختلفة بغرض التعرف على تكنولوجيا الصناعة وتشخيص تلف بعض القطع الفخارية بتل أبو ياسين بالشرقية كدراسة حالة. ولقد أثبت البحث أن الطفمة المستخدمة في صناعة الفخار الأثرى بتل أبو ياسين بالشرقية جنوب الدلتا من نوع الطفمة النيلية Nile Clay، أما الإضافات Tempers للفخار الأثري بتل أبو ياسين بالشرقية بشرق الدلتا من نوع الطفمة النيلية عبارة عن الرمل والتنين المقشر limestone powder وبمسحوق الحجر الجيري pottery powder«grog» والمكشر المحترق burnt straw.

بالإضافة إلى ذلك، تتميز معظم الآثار الفخارية بتل أبو ياسين بالشرقية بسهولة الكسر breaking وتشخير cracking والتعرق peeling والضعف weakness، بالإضافة إلى ظاهرة الكروب carbonates والكبريتات sulfates والكربونات carbonates، وتتغير الأحماض مثل الكربونات carbonates والكبريتات sulfates والكربونات carbonates. وتلتزم الباحث بترميم وعلاج وصيانة تلك القطع الفخارية بناءً على نتائج الفحوص والتحليل وتقييم تلف القطع الفخارية بالموقع الأثري.

الكلمات الدالة: الفخار، الطفمة، الإضافات، المعالجة السطحية، طبقة البطانة، الجو الحرق، التشريخ، التكسر.

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