

# TEMPER CHARACTERISTICS AND DAMAGE DIAGNOSIS OF SOME PRE-DYNASTIC POTTERY IN TELL HASSAN DAUD, ISMAILIA, EGYPT

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## ABSTRACT

**[AR]** الخواص التركيبية للإضافات وتشخيص تلف فخار عصر ما قبل الاسرات بتل آثار حسن داؤود بالإسماعيلية، مصر يقع تل حسن داؤود بمحافظة الإسماعيلية ويعود تاريخه الى عصر ما قبل الاسرات وتم الفحص والتحليل للعينات محل الدراسة بالميكروسكوب المجسم Stereoscopic Microscope والفحص بالميكروسكوب المستقطب polarizing Microscope والفحص بالميكروسكوب الإلكتروني الماسح Scanning Electron Microscope المزود بوحدة تشتت طاقة الأشعة السينية (EDX) والتحليل بطريقة حيود الأشعة السينية X-Ray Diffraction والتحليل الحراري Thermal Analysis. وأثبتت الفحوص والتحليل المختلفة الخواص التركيبية للإضافات المستخدمة في صناعة الفخار في عصر ما قبل الاسرات وهي التبن المقرط والرمل ومسحوق الفخار ومسحوق الحجر الجيري وتعيين نوع الطين المستخدم في تشكيل الأواني وهو الطفلة النيلية Nile Clay والمعالجات السطحية هي الغسول الأحمر. والحرق شبه جيد وجو الحرق مزيج من الجو المؤكسد والمختزل. كما اثبت البحث مظاهر تلف فخار عصر ما قبل الاسرات بالموقع الأثري حيث يعاني الفخار من تشوه سطحي بفعل رواسب التربة الرملية الطينية، فضلا عن ظاهرة تقشر السطح وطبقة البطانة وشروخ دقيقة وعميقة وظاهرة الكسر وانفصال الحبيبات الخشنة «التحبيب» وقللة المتانة والفجوات واللب الأسود والاملاح المتبلورة مثل الكلوريدات والكبريتات والكربونات والفوسفات من جراء الدفن في تربة زراعية. وبناءا على نتائج الفحوص والتحليل يحتاج الي تدخلات علاجية لترميمه وصيانته وعرضه متحفيا بتل آثار حسن داؤود بالإسماعيلية.

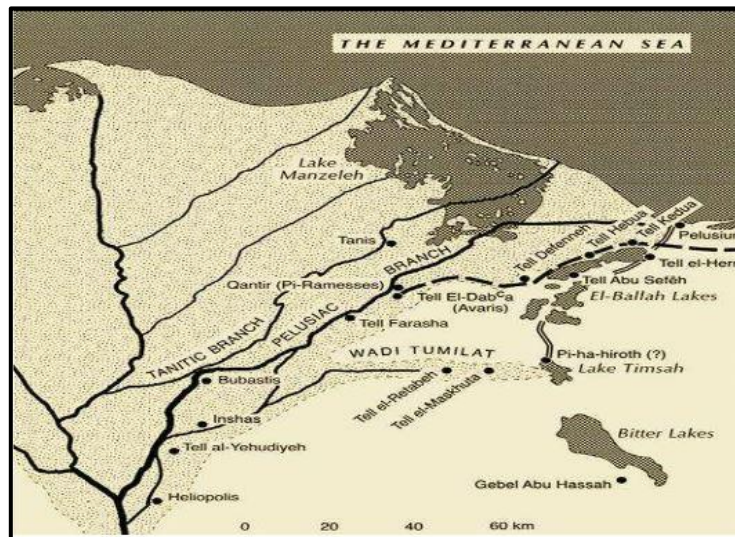
**[EN]** Tell Hassan Daoud site is located in Ismailia Governorate and dates back to the pre-dynastic period. Many microscopic examinations were used in the research such as stereoscopic microscope, polarizing microscope, scanning electron microscope equipped with an X-ray energy dispersive unit (EDX), X-ray diffraction, and thermal analysis. The various examinations and analyses proved temper characteristics in archaeological pottery, which consist of burnt straw, sand, limestone powder «calcite», and pottery powder «grog». The type of clay used in shaping pots is Nile clay. The surface treatment is red wash «hematite». The burning atmosphere is a mixture of a oxidizing and reducing. The research revealed damage to the pre-dynastic pottery at the archaeological site. The pottery exhibits superficial deformation caused by sandy clay soil sediments, surface peeling, micro cracks, macro cracks, fractures, separation of grains, granulation, poor durability, gaps, a black core, and the presence of crystalline salts such as chlorides, sulfates, carbonates, and phosphates due to burial in agricultural soil.. Based on the results of examinations and analyses, treatment interventions are required for the restoration, maintenance, and display of the pottery

**KEY WORDS:** Clay, deterioration, fabric, grog, pottery, temper, Red wash, sediments.

## I. INTRODUCTION

Tell Hassan Daoud is located about 8 km northwest of the city of al-Tal al-Kabeer. The site is far from Zagazig-Ismailia agricultural road, about 4 km away. It is 6 km west of al-Ratabi site. Its area is 400 m x 300 m. It is surrounded by agricultural lands. It is considered to be one of the most important archaeological sites in the Delta. Its oldest layers date back to the Maadi civilization period, «Butu in Lower Egypt», which is equivalent to the Naqada I period in Upper Egypt dating back to 3600 BC. The excavations at the site revealed archaeological layers dating back to the pre-dynastic and early dynastic periods<sup>1</sup>.

The first excavation of Tell Hassan Daoud was in 1989 through the mission of the Supreme Council of Antiquities. It revealed archaeological layers dating back to the pre-dynastic era. These excavations revealed some important cultural features at the archaeological site, such as a cemetery containing 620 tombs. The archaeological site was named Tell Hassan Daoud based on a small village called Hassan Daoud, which is located about one hundred and fifty meters southwest of the site. It is a very small village built on a high elevation and was most likely part of the site<sup>2</sup>, as in [FIGURE 1].



[FIGURE 1]: Represents Tell Hassan Daoud site and Wadi al-Tumailat. SALEM 1998: 82.

Egyptian and foreign excavation missions revealed thousands of different pottery pieces, many different ovens for cooking, kilns of burning pottery, and stone vessels such as schist and alabaster<sup>3</sup>. Tell Hassan Daoud site is located in the eighth district of Delta<sup>4</sup>. Pottery plays an important role in determining chronology, establishing regional geographical classification, and understanding civilizational cultural progress. These are attributed to different manufacturing techniques, burning methods, and different patterns of decoration between regions<sup>5</sup>. Advanced examinations and analytic methods contributed to accurately identifying the manufacturing parameters for archaeological pottery<sup>6</sup>.

<sup>1</sup> DIAB 2022: 3.

<sup>2</sup> SALEM 1992: 82.

<sup>3</sup> NOUREDDINE 2018: 340.

<sup>4</sup> KAMEL 1989: 116.

<sup>5</sup> TÜRKTEKI 2020: 59.

<sup>6</sup> HENDERSON et Al.2010: 1-24.

Leveraging numerous microscopic examinations in materials science serves specific objectives, providing insights into both archaeometric and technological features<sup>7</sup>. Polarizing microscopes play an important role in identifying additives, mineral composition; grain shapes their interrelationships, petrographic structure, as well as the assessment of treatments and burning<sup>8</sup>. Scanning electron microscopy shows morphological parameters<sup>9</sup>. It provides us with information about micro-structure and various manifestations of damage<sup>10</sup>. Microscopic examinations show technological aspects, mineral composition, various additives, and different kinds of damage<sup>11</sup>.

The research aims to analyze the microstructure of different temper variations, evaluating various technological aspects in the process. The research also focuses on diagnosing the damage of pre-dynastic pottery in tell Hassan Daoud in the eastern region. This involves classification, dating, and restoration, based on the results of microscopic examinations.

## II. STUDY METHODOLOGY

### Study Samples

In this study, some samples of archaeological pottery and soil samples from Tell Hassan Daoud site in Ismailia were microscopically examined.

### 1. Study Methods

#### A. Visual Examination

This is considered one of the important methods for determining artistic values<sup>12</sup>, many different lenses were used to identify the tempers, the archaeological aspects, textures, and damage<sup>13</sup>.

#### B. Stereomicroscope

This is an important method for identifying archaeometric, technological characteristics, and various types of damage<sup>14</sup>. The device used is SZ680, Objectives Zoom Range: 0.68X-4.7X, Zoom Ratio: 1: 6.8, Field Diameter (mm):  $\Phi 23$ , Working Distance (mm): 110, Stereo Angle ( $^{\circ}$ ): 12, Viewing Angle ( $^{\circ}$ ), 35, Magnification: 3.5~22.5. This examination was carried out at the Faculty of Science/ Zagazig University.

#### C. Polarizing Microscope Examination

The petrographic examination shows microstructure, type of tempers, mineral components of the pottery<sup>15</sup>, thermal changes during pottery manufacture (burning), grain size, red wash, slip layer, the nature of burning, and damage such as cracks, gaps,

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<sup>7</sup> ABDELMONIEM et Al. 2020: 7-17.

<sup>8</sup> AREZKI & OUAMAR 2020: 51-60.

<sup>9</sup> ALAWNEH et Al. 2017: 201-213.

<sup>10</sup> MABROUK 2020: 163-170.

<sup>11</sup> ELGAREB 2017: 76.

<sup>12</sup> PAWTOWSKA et Al. 2023: 99.

<sup>13</sup> SANDU et Al. 2022: 1445.

<sup>14</sup> MOUSTAFA et Al. 2022: 3-14.

<sup>15</sup> LOTOTSKA et Al. 2022: 31-42.

and flaking<sup>16</sup>, thin sections were prepared for petrographic examination. The device used is the Nikon Eclipse E200 POL. Optical system: CFI60 infinity optical system, Eyepieces: Standard set: 10x (F.O.V. 22mm), CM type with 90° crosshair and micrometer scale, Intermediate tube: Built-in focusable Bertrand lens removable from optical path; Built-in analyzer removable from optical path; Conoscopic/Orthoscopic observations switchable; With plate/compensator slot, Analyzer: 360° rotary dial; Minimum reading angle 0.1°, Coarse/fine focusing: 0.2 mm per rotation; Coarse: 37.7mm per rotation, Stage: 160mmø circular. This examination was carried out at the Faculty of Science Cairo University.

#### **D. Scanning Electron Microscope Examination**

This shows the morphological structure of the pottery samples, grain sizes, their distribution, their interrelationships, fabrics, red wash, slip layer, different manifestations of damage, and the mineral components of the pottery<sup>17</sup>. The device is a Quanta FEG250 SEM unit. This examination was carried out at the National Research Center in Cairo.

#### **E. X-Ray Diffraction Analysis (XRD).**

This is considered one of the most important analytical methods to identify mineral components, changes caused by firing, and damage resulting from burial in soil<sup>18</sup>. The device used is ADX-2500 X-ray Diffract meter. Control mode: 1kV/step, 1mA/step controlled by PC, Rated output power is 4 kW, Tube voltage: 10 ~ 60 kV, Tube current: 5~80mA, X-raytube: Cu,Fe,Co,Cr,Mo(2.4kW) Focus dimension: 1mm × 10mm<sup>2</sup> or 0.4mm × 10 mm<sup>2</sup>, Scan range of 2θ: -6° ~ 160°, Setting speed of angle: 1500°/min, This analysis was conducted at the Faculty of Science Cairo University.

#### **F. Thermogravimetric Analysis «TGA»**

This method determines the firing temperature of pottery according to loss of weight and mineral changes during the firing of samples<sup>19</sup>. Three pottery samples were analyzed by a thermal analysis device known as Perkin Elmer STA 6000. The temperature program ranged from room temperature to 1500°C, measurement range: ±0.1 to ±1000 μV, heating speed: 0 to +50°C/min. Weight loss and mineral changes were recorded on the chart.

## **2. Results**

### **A. Visual Examination**

The examination shows that the pottery samples in Tell Hassan Daoud, Ismailia in the eastern Delta suffer from sandy mud sediments, crystalline salts, cracking, fracture, flaking, breaking, and black core. The visual examination also indicates that the pots were shaped manually; the rims were made using a potter wheel, as in [FIGURE 2].

<sup>16</sup> PACHUTA et Al. 2022: 753-770.

<sup>17</sup> SANDBAKKEN et Al. 2022: 1681-1690.

<sup>18</sup> ABDELMONIEM et Al. 2022: 1087-1100.

<sup>19</sup> SEISEH et Al. 2023: 3-18.



[FIGURE 2]: The archaeological site. A. Pottery vessels; B. Pottery shards © Photo taken by the researcher

### B. Stereomicroscope

This shows archaeometric, technological characteristics, and various damage manifestations with magnification (6X) as in [FIGURE 3].

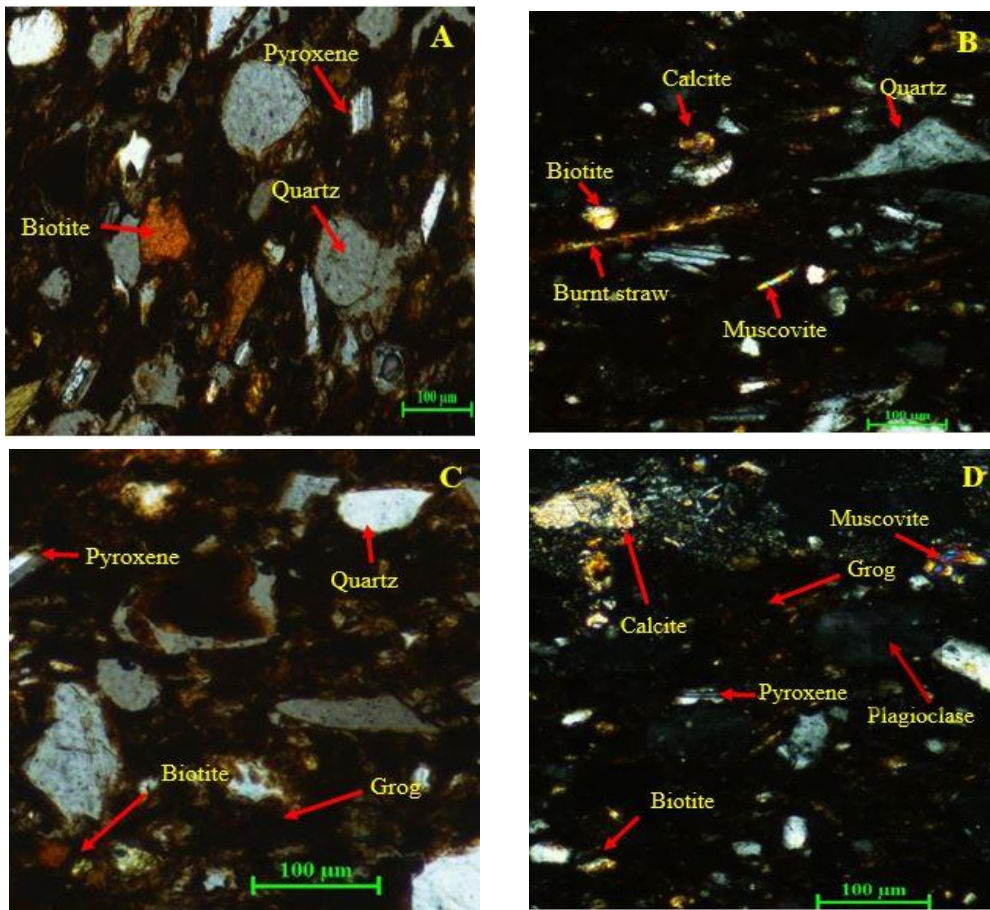


[FIGURE 3]: Stereomicroscope examination. A. First sample (quartz); B. Second sample (calcite); C. Third sample (burnt straw, gaps & cracks) © Done by the researcher

### C. Polarizing Microscope Examination

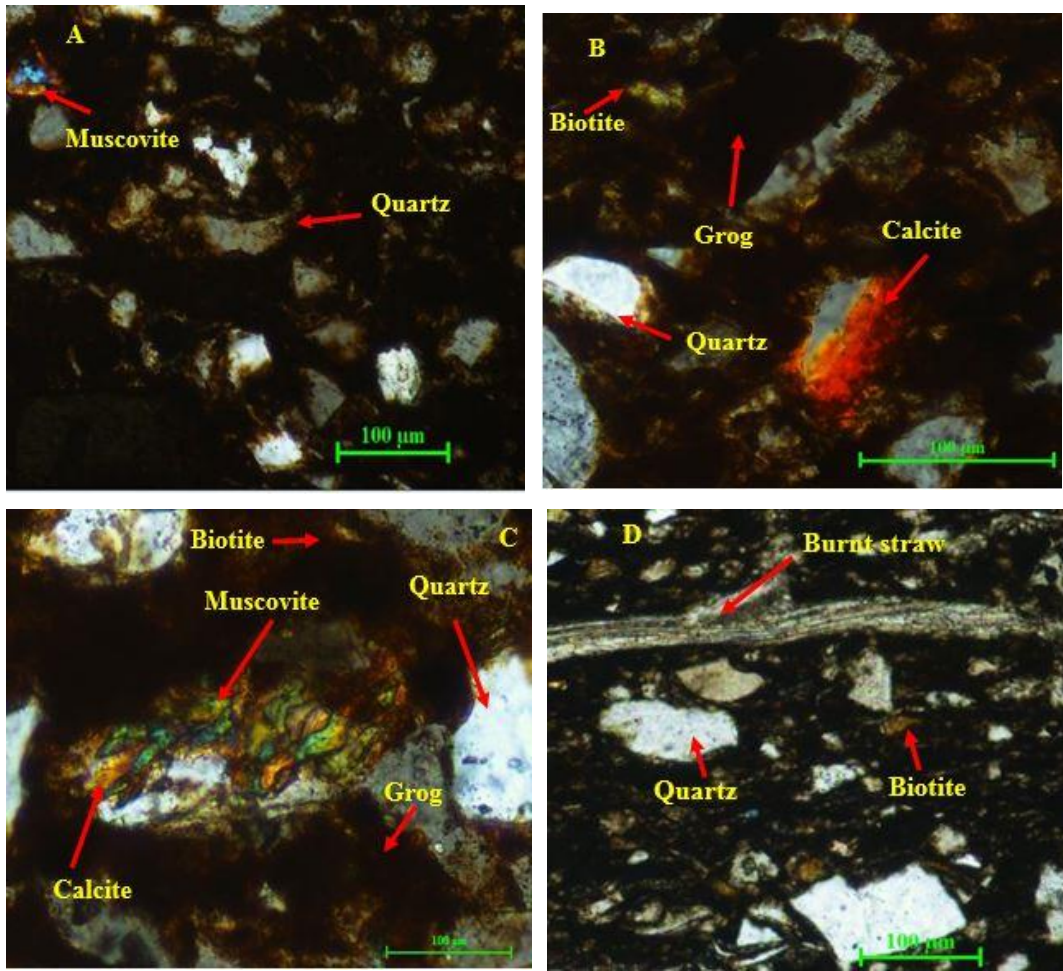
The petrographic examination of the samples is shown in [FIGURE 4], where the examination revealed coarse quartz grains. The texture is semi-round quartz granules, others are sharp angles, biotite, pyroxene, rutile and iron oxide with a magnification of (40X - CN) as in [FIGURE 4/a]. Another part of the same sample was also examined as in [FIGURE 4/b] showing coarse- quartz grains, limestone powder «calcite», biotite, muscovite, burnt straw, and iron oxide with magnification (40X – CN). Examination of the core area as seen in [FIGURE 4/c] shows the presence of coarse quartz grains, along with grog «pottery powder», mica biotite, pyroxene, and iron oxides with magnification (40X - CN). The examination of another part of the core as seen in [FIGURE 4/d] showed coarse quartz granules, as well as grog, calcite, muscovite, pyroxene, plagioclase, and iron oxide at magnification (40X - CN).





[FIGURE 4]: Petrographic examination of the first pottery sample. A. Quartz, biotite, pyroxene and rutile; B. Quartz, calcite, biotite, muscovite and burnt straw; C. Quartz, biotite, pyroxene and grog; D. Quartz, calcite, grog, muscovite, plagioclase and pyroxene © Done by the researcher

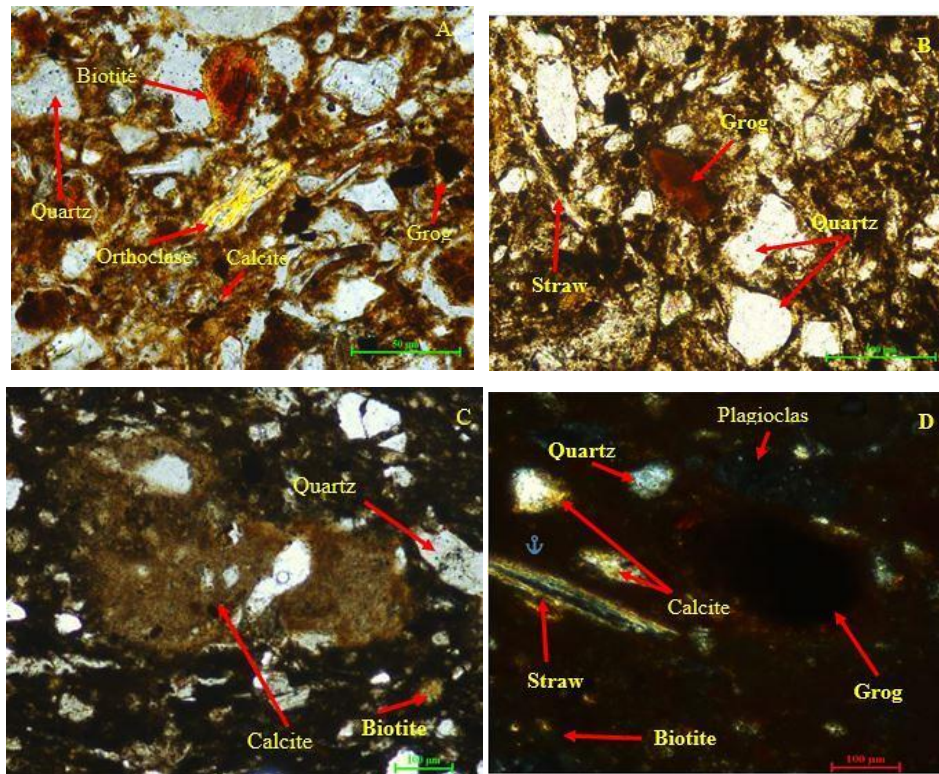
The examination of the second pottery sample also shows the presence of coarse texture of sharp-angled quartz grains, in addition to the presence of rutile, mica biotite, and iron oxide with a magnification (40X - CN) as seen in [FIGURE 5/a]. Another part of the same sample was examined; [FIGURE 5/b] shows coarse quartz grains, mica biotite, grog, calcite, and iron oxide with magnification (40X - CN). Examination of the core seen in [FIGURE 5/c] showed sharp-angled coarse quartz grains, calcite, biotite, muscovite, and iron with magnification (40X - CN). The examination of another part of the same core as seen in [FIGURE 5/d] showed the presence of coarse quartz grains, burnt straw, biotite, and Iron oxides with a magnification (40X - CN).



[FIGURE 5]: Petrographic examination of the second pottery sample. A. Quartz, biotite, iron and oxides; B. Quartz, grog, biotite and calcite; C. Quartz, biotite, muscovite, calcite and iron; D. Quartz, burnt straw and biotite © Done by the researcher

Examination of the third pottery sample showed the presence of a coarse texture of sharp-angled quartz grains, in addition to rutile, biotite, muscovite, orthoclase, and iron with a magnification (40X - CN), which is seen in [FIGURE 6/a]. Another part of the same sample was examined showing coarse quartz granules with sharp angled grains, biotite, grog, burnt straw, rutile, and iron oxide under magnification (40X - CN) [FIGURE 6/b]. The examination of the core showed coarse quartz granules, sharp angled grains, calcite, and iron under magnification (40X - CN). Another part of the same core seen in [FIGURE 6/c], showed coarse quartz grains, grog, burnt straw, biotite, calcite, plagioclase, and iron under magnification (40X - CN) [FIGURE 6/d].





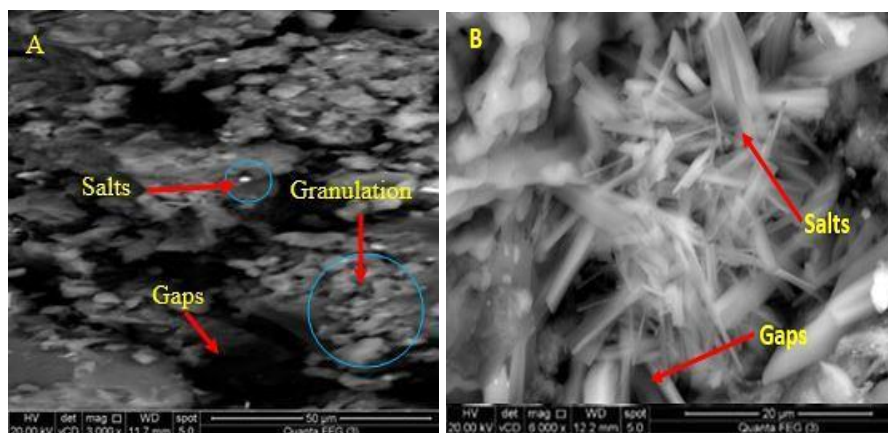
[FIGURE 6]: The petrographic examination of the third pottery sample. A. Quartz, biotite, orthoclase, grog and iron; B. Quartz, grog, burnt straw and iron; C. Quartz, calcite, biotite and iron; D. Quartz, grog, burnt straw, biotite, calcite, plagioclase and iron © Done by the researcher

### 3. Examination and Analysis by SEM-EDX Unit

The pottery samples were examined and analyzed using a scanning electron microscope.

#### A. SEM Examination

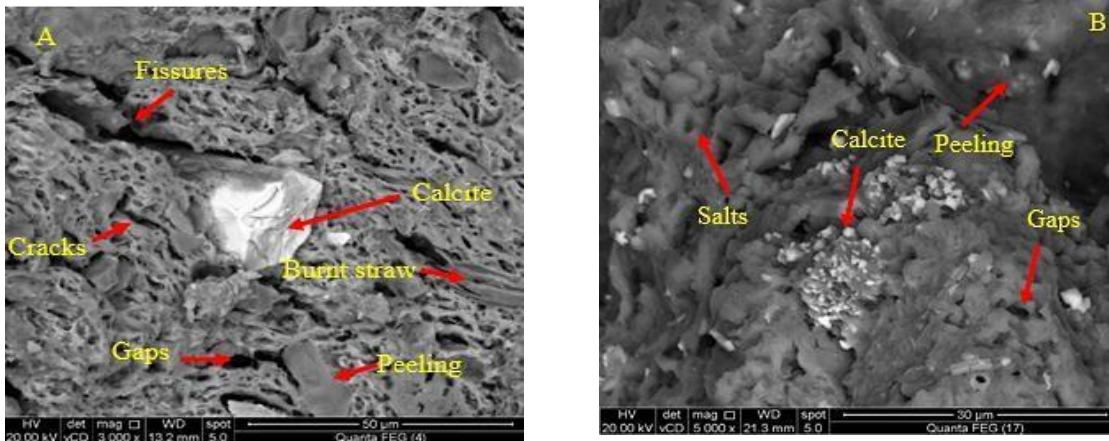
The first sample, seen in [FIGURE 7/a], shows the presence of many gaps, crystalline salts, separation of grains, peeling, and soil sediments with a magnification (3000 X). Another part was examined showing coarse texture, coarse quartz grains, gaps, flaking, crystallization of salts, and cracks with a magnification (6000X) [FIGURE 7/b].



[FIGURE 7]: SEM examination of the first pottery sample. A. SEM examination of the surface with magnification (3000 X); B. SEM examination of the core with magnification (6000 X).

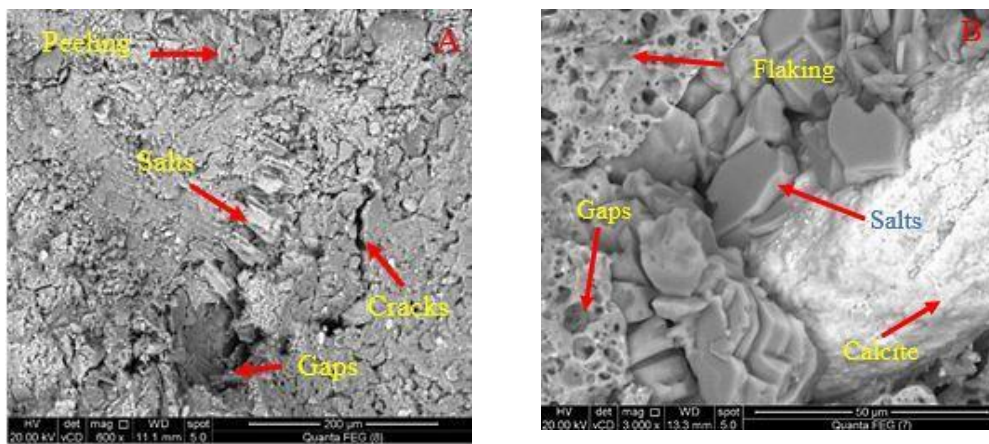


SEM examination of the second sample shows the temper «limestone, burnt straw» cracks, gaps, fissures, and flaking under magnification (3000 X), shown in [FIGURE 8/a]. Another part was examined, which showed the presence of quartz grains, crystallization of salts, gaps, peeling, and flaking under magnification (5000 X), [FIGURE 8/b].



[FIGURE 8]: SEM examination of the second pottery sample. A. SEM examination of the surface with magnification (3000 X); B. SEM examination of the core with magnification (5000 X).

Examination of the third sample shows the presence of quartz grains, crystallization of salts, flaking, voids, and cracks under magnification (600X), [FIGURE 9/a]. Another part was also examined showing a temper «limestone», gaps, flaking, salt crystallization, and granular separation under magnification (3000 X), [FIGURE 9/b].



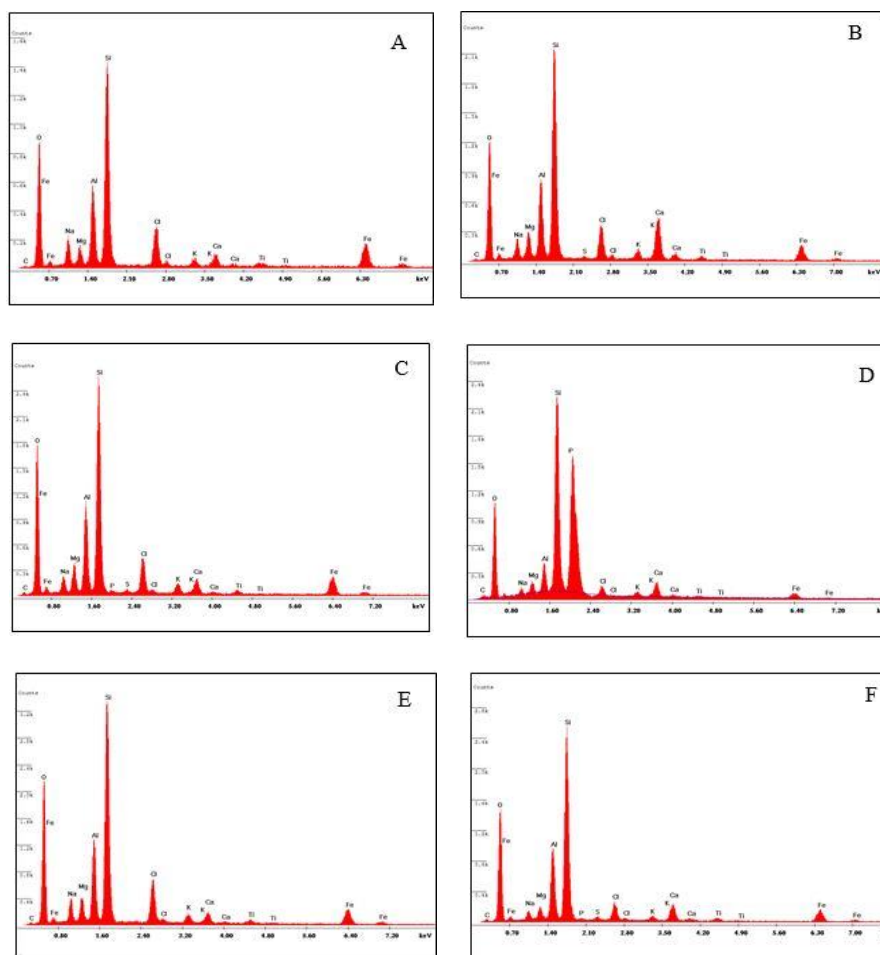
[FIGURE 9]: SEM examination of the second pottery sample. A. SEM examination of the surface with magnification (600 X); B. SEM examination of the core with magnification (3000 X).

## B. SEM-EDX Analysis

SEM-EDX analysis revealed the presence of carbon, oxygen, sodium, magnesium, aluminum, silica, phosphor, sulfur, chlorine, potassium, calcium, titanium, and iron, which is indicated in [TABLE 1 & FIGURES 10/a-f].

Elemental Wight %	S1		S2		S3	
	Surfac e a	core b	surfac e c	Core d	Surfac e e	Core f
C	3.83	3.06	4.69	4.66	3.20	4.84
O	20.58	20.16	21.19	22.59	25.54	25.81
Na	4.96	4.52	2.98	4.17	4.16	3.49
Mg	1.11	2.82	1.58	1.04	1.71	1.68
Al	7.39	6.68	3.68	6.87	7.67	6.64
Si	25.64	25.68	24.75	24.85	28.05	27.39
P	-	-	20.80	2.22	-	2.16
S	-	4.42	-	2.39	-	2.61
Cl	7.18	7.92	5.05	8.09	8.70	6.87
K	1.77	2.07	1.49	2.23	2.08	1.30
Ca	5.05	10.26	6.52	6.39	6.62	6.27
Ti	1.16	1.24	1.94	1.52	1.45	1.64
Fe	20.88	11.18	5.34	12.97	10.81	9.36

[TABLE 1]: Results of the analysis of pottery samples using SEM-EDX  
© Done by the researcher



[FIGURES 10/a-f]: EDX analysis patterns of pottery samples, Tell Hassan Daoud, Ismailia. A-B. First pottery; C-D. Second pottery; E-F. Third pottery.

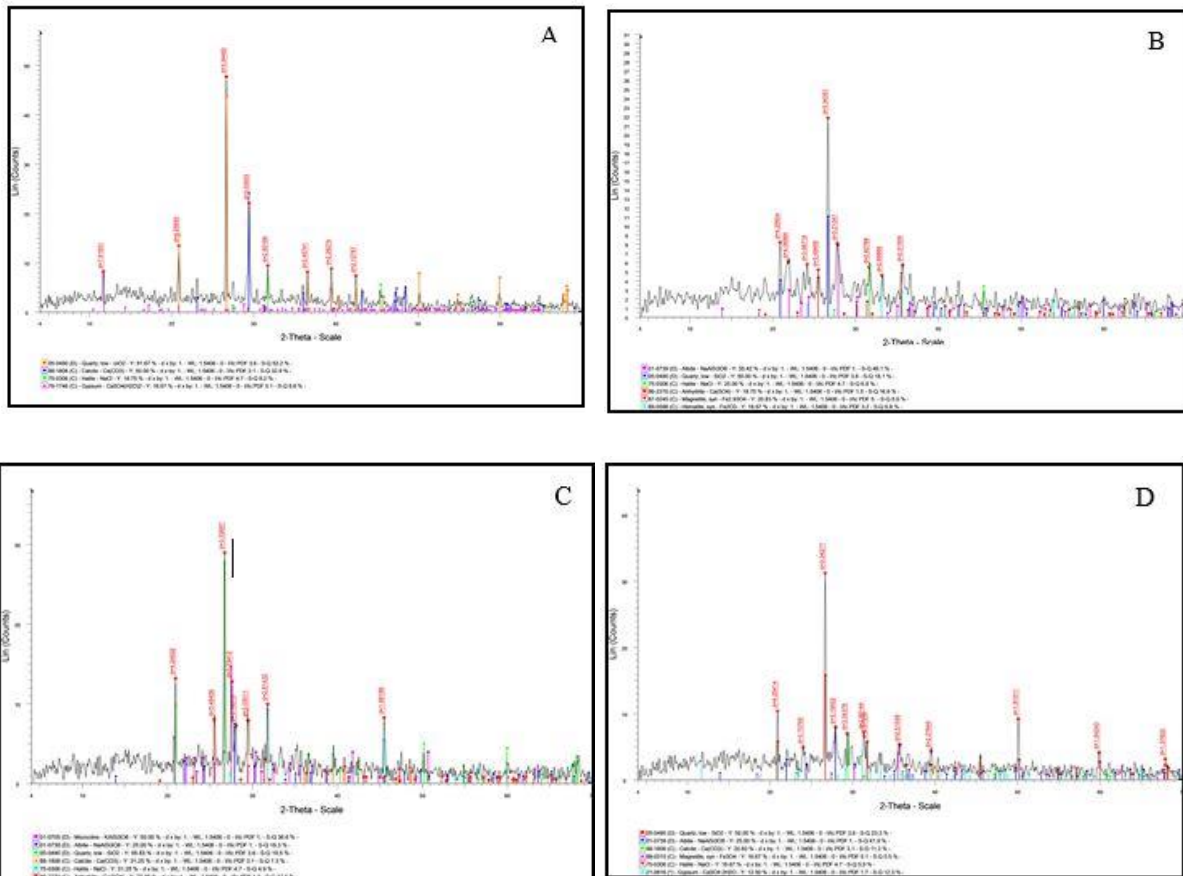
#### 4. X- Ray Diffraction Analysis

Three pottery samples and one soil sample have been analyzed by XRD. The pattern of three pottery samples shows the presence of quartz  $\text{SiO}_2$ , microcline  $\text{KAlSi}_3\text{O}_8$ , albite  $\text{NaAlSi}_3\text{O}_{10}$ , halite  $\text{NaCl}$ , hematite  $\text{Fe}_2\text{O}_3$ , Magnetite  $\text{Fe}_3\text{O}_4$ , anhydrite

CaSO<sub>4</sub>, Gypsum CaSO<sub>4</sub>.2H<sub>2</sub>O Soil sample pattern contained quartz SiO<sub>2</sub> , albite NaAlSi<sub>3</sub>O<sub>10</sub>, halite NaCl , hematite Fe<sub>2</sub>O<sub>3</sub>, anhydrite CaSO<sub>4</sub> , as shown in [TABLE 2 & FIGURES 11/a-d].

Mineralogical Compositions		Pottery	Samples		
Minerals	Chemical Composition	a	b	c	d (soil)
Quartz	SiO <sub>2</sub>	52.2	18.1	19.5	23.3
Microcline	KAlSi <sub>3</sub> O <sub>8</sub>	-	-	36.6	-
Albite	NaAlSi <sub>3</sub> O <sub>10</sub>	-	46.1	18.3	41.9
Calcite	CaCO <sub>3</sub>	32.9	-	7.3	11.2
Hematite	Fe <sub>2</sub> O <sub>3</sub>	-	6.8	-	-
Magnetite	Fe <sub>3</sub> O <sub>4</sub>	-	5.5	-	5.5
Halite	NaCl	8.2	6.9	4.9	5.9
Anhydrite	CaSO <sub>4</sub>	-	16.8	13.5	-
Gypsum	CaSO <sub>4</sub> .2H <sub>2</sub> O	6.8	-	-	12.3

[TABLE 2]: XRD analytical results of analyzed pottery samples © Done by the researcher



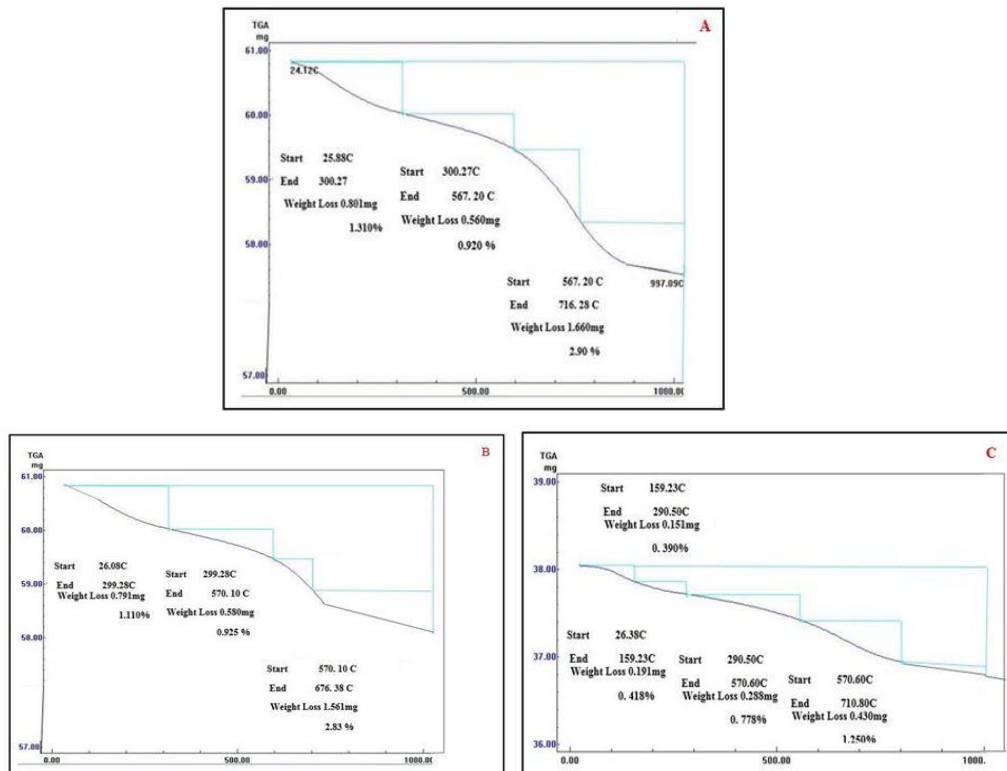
[FIGURE 11 ]: XRD patterns. A. First pottery sample; B. Second pottery sample; C. Third pottery sample; D. soil sample, tell Hassan Daoud © Done by the researcher

### 5. Thermogravimetric Analysis

Thermogravimetric Analysis «TGA» have determined the firing temperature of pottery according to weight loss during the firing of the samples at room temperature, as shown in as shown in [TABLE 3] & [FIGURES 12/a-c].

Pottery Sample	Thermal Characteristics			
	TGA			
	Start firing	End firing	Weight loss(mg)	Percentage%
a	25.88	300.27	0.801	1.310
	300.27	567.20	0.560	0.920
	567.20	716.28	1.660	2.90
b	26.08	299.28	0.791	1.110
	299.28	570.10	0.580	0.925
	570.10	676.38	1.561	2.83
c	26.38	159.23	0.191	0.418
	159.23	290.50	0.151	0.390
	290.50	570.60	0.288	0.778
	570.60	710.80	0.430	1.250

[TABLE 3]: TGA analytical results of pottery samples, tell Hassan Daoud, Ismailia © Done by the researcher



[FIGURE 12]: TGA analysis patterns of pottery samples, tell Hassan Daoud, Ismailia. A. First pottery; B. Second pottery; C. Third pottery.



### III. DISCUSSION

Visual examination shows that Tell Hassan Daoud site is located in Ismailia Governorate and dates back to the pre-dynastic period as shown in [FIGURE 1]. The pottery of Tell Hassan Daoud was shaped manually. The Potter Wheel finished the rims of vessels. These are two of the shaping methods used in the pre-dynastic period and at the beginning of the dynasties<sup>20</sup>. Fabric is coarse. Temper is limestone powder «calcite». Firing is medium to low firing. Surface treatment is applied by red wash, which is one of the common methods in the Pharaonic era from Neolithic age to the Ptolemaic period<sup>21</sup>. The examination showed deposits of sandy clay soil, and salt crystallization, which are signs of damage, and common to most of the artifacts excavated from the soil. The burial environment causes fractures, fissures, and breaking due to burial in the soil<sup>22</sup>. Damage manifestations of varied at excavation sites as a result of various damage factors that may cause cracking or fracture of the pottery body<sup>23</sup>, as shown in [FIGURES 2-3].

PLM Examination showed the presence of various tempers such as sand, burnt straw, limestone powder «calcite», and grog, as shown in [FIGURES 4 /c-d] . These tempers were used to improve the properties of clay in pottery manufacturing in the pre-dynastic period<sup>24</sup> . The petrographic examination also revealed muscovite, biotite, plagioclase, and pyroxene as shown in [FIGURES 4-5], **these minerals are characteristic of the Nile clay**<sup>25</sup>. It also showed the presence of rutile and orthoclase seen in [FIGURE 6]. The clay is classified as Nile clay according to the mineral components in the indicated petrographic examination<sup>26</sup>. PLM Examination showed the texture of the pottery samples, which have a coarse fabric of semi-circular quartz grains; some of them have sharp angled quartz grains, as shown in [FIGURES 4-6]. It is known that coarse texture resulted from non-pure selected clay or poor treatment efficiency<sup>27</sup>. One of the most important methods of surface treatment is red wash which is red ocher «hematite»<sup>28</sup> or slip layer. The latter is a mixture of micro clay grains and water<sup>29</sup>. It is noteworthy that during the pre-dynastic period to the fourth dynasty, pottery production did not involve the use of pottery powder or «grog», as indicated by the Vienna classification. Instead, commonly used additives during this period include burnt straw, sand, and calcite<sup>30</sup>.

The morphological examination using the scanning electron microscope showed that the mineral components are sodium, magnesium, aluminum, potassium, calcium, and iron, as shown in [TABLE 1]. These elements are characteristic of Egyptian clay and

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<sup>20</sup> BOURRIAU 1981: 15.

<sup>21</sup> ELGHAREB 2018: 176.

<sup>22</sup> AHMED et Al. 2020: 97.

<sup>23</sup> ELGHAREB 2023: 175-176

<sup>24</sup> ELGHAREB 2017: 74.

<sup>25</sup> ELGHAREB 2021: 170.

<sup>26</sup> SHARMIN 2020: 539.

<sup>27</sup> ZAKIN 1997: 14.

<sup>28</sup> HODGE 1964: 33.

<sup>29</sup> JOHNSON 1988: 12.

<sup>30</sup> NICHOLSON 2000: 67.

some of the heavy elements are characteristic of Nile mud in Egypt<sup>31</sup>. The examination also showed calcium carbonate (calcite) as one of the tempers. The analysis, documented in [TABLE 1] & [FIGURE 9], it played an important role in showing the nature of burning, which is a medium to low heat depending on the carbon percentage, which ranges from 3.06% to 3.83% for the first sample, from 4.64% to 4.66% for the second sample, and 3.20% to 4.84% for the third sample. EDX also showed crystalline salts such as sulfates, where the percentage of sulfur oxide ranged from 2.39% to 4.42%, halite from 5.05% to 8.09%, phosphates from 2.16 to 20.80%, and carbonates from 5.05% to 10.26%; these percentages of salts are shown in [TABLE 1] & [FIGURES 10/a-f]. Chloride salts are distinctive features found in Egyptian soil<sup>32</sup>.

Phosphate and gypsum salts are found in samples due to agricultural soil or the decomposition of organic matter in the cemetery site<sup>33</sup>. Carbonate salts in pottery samples are a result of rainwater or dissolving gases in soil water<sup>34</sup>. Many archaeological materials suffer from poor durability due to the damaging factors of the burial environment, which necessitates a strengthening process<sup>35</sup>. The post-excavation environment causes damage to pottery objects due to different environmental conditions<sup>36</sup>. X-ray diffraction analysis of pottery samples indicated the presence of sand (quartz) and calcite as one of the common tempers in ancient Egypt<sup>37</sup>, which is shown in [TABLE 2] & [FIGURES 11/a-c].

Thermal analysis «TGA» proved that the firing temperature of the first pottery sample is about 716.28 °C. The loss of physical combined water occurred in the thermal range from 25.88 °C to 300.27 °C, constituting a percentage of 1.310%. Subsequently, the weight loss persisted from 300.27°C to 567.20°C, accounting for a percentage of 0.920%. This loss is attributed to the removal of physical combined water, chemical combined water, organic residues, carbonate materials, and various iron oxides. Following that, there was a substantial weight loss in the thermal range from 567.20 °C to 716.20°C, amounting to 2.90%. This loss is attributed to the decomposition of calcium carbonate «temper» into calcium oxide and carbon dioxide. However, in the thermal range from 716.28°C to 1000 °C, the weight loss remained relatively constant in the first sample, as depicted in [TABLE 3] & [FIGURE 12/a]. TGA of the second sample proved that the firing temperature is about 676.38 °C which was interpreted because of lost physical combined water from 26.08 °C: 299.28 °C with a percentage of 1.10%. Then, the weight loss continued from 299.28°C: 570.10°C with a percentage of 0.925% due to loss of physical combined water, chemical combined water, organic residues, carbonate materials, and various iron oxides. Continuing further, there was a significant weight loss in the thermal range from 570.10 °C to 676.38°C, amounting to 2.83%. This loss is attributed to the decomposition of iron chloride and calcium carbonate «temper» into calcium oxide

<sup>31</sup> HAMDAN 2014: 987.

<sup>32</sup> OSMAN et Al. 2016: 97.

<sup>33</sup> ABDEL RAHIM 2015: 84.

<sup>34</sup> ABD-ELKAREEM et Al. 2017: 87.

<sup>35</sup> HEMEDA et Al. 2020: 485.

<sup>36</sup> ZAMORA et Al. 2020: 689.

<sup>37</sup> ELGHAREB 2021: 907.

and carbon dioxide. However, in the thermal range from 676.38°C to 1000 °C, the weight loss remained relatively constant, as illustrated in [TABLE 3] & [FIGURE 12/b].

The third sample by TGA showed that the firing temperature is about 710.80 °C. This has been illustrated because of lost physical combined water from 26.38 °C: 159.23 °C with a percentage of 0.418%. Then, the loss in weight decreased from 159.23°C: 290.50°C with a percentage of 0.390% because of lost chemical combined water, organic residues, carbonate materials, and various iron oxides. Then, the loss weight increased to a noticeable degree from 290.50 °C: 570.60°C with a percentage of 0.778% due to decomposition of iron chloride and calcium carbonate «temper» to calcium oxide and carbon dioxide. While weight loss continued from 570.60°C: 710.80°, the weight loss was almost constant from 710.80°C to 1000 °C, [TABLE 3] & [Figure 12/c]. Thus, thermal analysis is an important method for studying the thermal behavior of pottery<sup>38</sup>. It plays an important role in determining the thermal properties of material<sup>39</sup>.

#### IV.CONCLUSION

Through the results of various examinations and analyses, the research advanced our knowledge regarding tempers in archaeological pottery. Historically, it was not believed that Grog was used in the pre-dynastic period, but the research proved the potter used grog in the pre-dynastic era and the beginning of the dynasties in Tell Hassan Daoud in Ismailia. It also proved the existence of other tempers such as burnt straw, sand and limestone powder, «calcite-dolomite», at this archaeological site. The research proved that the surface treatment is red wash. The burning atmosphere is a mixture of reducing and oxidizing. The pottery texture is coarse. The burning temperature of the first sample is 716°C, the second is about 676°C, and the third is about 710°C. It also demonstrated visual damage such as sandy clay sediments, fractures, fissures, cracks, poor durability, crystalline salts, black core, flaking, separation of grains, and many gaps. Based on the results of these examinations and analyses, the pottery pieces need interventions for their treatment and maintenance before being displayed at a museum.

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<sup>38</sup> KLOUZKOVA et Al. 2016: 1311.

<sup>39</sup> STOIA et Al. 2016: 1185.

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