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ARCHAEOMETALLURGICAL INVESTIGATION, MANUFACTURING TECHNIQUE AND CONSERVATION PROCESSSES OF A COPTIC/BYZANTINE AGE HOARD OF UNSTRUCK COINS FLANS

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ABSTRACT

This work presents one of the rarest objects from the metallic heritage family, whether as archaeological finds or museum exhibits. A large collection of unstruck coins blanks (flans) and some small metallic pieces in gray color were studied. This collection was found inside a pottery jar and dates back to the Byzantine Age in Egypt (the Coptic period). The aim of this work is the characterization of corrosion products and metallic structure as well as discussing their features. Also, studying the manufacturing technique and a conservation process were performed. The study of structural and chemical composition of the metallic core was carried out by optical microscopy, X-ray fluorescence, and scanning electron microscopy with energy dispersive X-ray microanalysis while surface corrosion products were identified by X-ray diffraction. The results showed that this unstruck flans hoard was made from a lead-copper alloy and the small gray pieces consist only of lead element. The XRD results showed that the main corrosion products are copper trihydroxychlorides and cuprous oxide. The study also showed a discussion concerning the elemental and microstructural features, the formation mechanism of corrosion products and the manufacturing technique of these flans. This hoard is considered archaeological materialistic evidence (an eyewitness) and a unique opportunity to document and confirm knowledge concerning the manufacturing technology of coins and suggest the location of mints during the Byzantine Age in Egypt.

KEYWORDS: flans, coins, manufacturing technique, conservation.

1. INTRODUCTION

Archaeological excavations at Egyptian sites are still revealing rare hoards that had not been revealed before. Recently, a rare and unique hoard was excavated in Abu Rawash village, Giza city (Fig. 1(a)). Egyptian expedition in this village excavated a small pottery jar sealed with a cloth piece. This jar was filled with a rare hoard from flans (Fig. 1(b)). Flan as a term expresses the metallic piece of the coin before the striking process, and this name comes from a Latin word, flado, meaning "flat cake" (Hartmann et al., 2019). Such hoard of ancient Egyptian flans with this large number was not discovered before and there is not archaeometry or analytical study of flans, to my knowledge, while some studies were presented of coins striking tools—the anvil and punch die (Hartmann et al., 2019; Vermeule 1957; Bendall et al., 1978; Monticelli et al., 2013). Also, this hoard is considered an archaeological materialistic evidence (an eyewitness) to document and confirm knowledge concerning manufacturing technology of struck coins, as well as other evidences such as coin striking tools and ancient historians' writings, (Salem et al. 2019). The stages of making struck ancient coins are documented by four archaeological materialistic evidences (Howgego1995; Vermeule 1955) as follows:

- *Flans:* flans, blanks, and planchets terms express the piece of metal that the coin design will be stamped (Hill 1922). This flan is extremely rare as archaeological finds and museum exhibits, because the flans are converted to the struck coins at a mint.
- *Flan casting molds*: one of the flan casting methods was casting into coin-sized molds to produce flan in a coin-like shape (Hill 1922).
- *Die:* the die is the used tool in the stamping process (Monticelli et al., 2013; Bendall et al., 1978; Vermeule 1955). Most known ancient coins dies are in museum collections (though rarely on display), but some occasionally appear in major auctions, where they command high prices. One of the oldest extant dies was excavated in Egypt in 1904 and belonged to coins of Athenian types of the period 430–322 BC. This die was made of high tin-bronze and is displayed currently at the British Museum (Hill 1922). Also, dies of Athenian coins were displayed in 1910 at the British Museum. These dies also come from Egypt, having been found at Sais, and must be more or less contemporary with the one just mentioned.
- *Coins*: the coins themselves as archaeological excavations are very common, and the majority of museums contain ancient coins.

Flan casting molds, dies, and flans are extremely rare as archaeological finds. The reason is attributed to the destruction of flan casting molds and dies when they become unusable or even unused, when another governor is installed, to avoid the production of counterfeit coins Also, Flans were prepared to be coins after the striking process. So, flans only existed in mints and are not allowed to get out avoiding their use in counterfeiting purposes. Accordingly, the archaeological discovery and study of flans or striking tools is very interesting.

In contrast, archaeological coins are found in the majority of museums and have a high value among their exhibits. Also, the writings, inscriptions, and figures on the obverse and reverse side give the coins high artistic and cultural values and make them important artifacts among the metallic heritage (Salem et al. 2019). Early studies on the coins in several topics include: Coin recognition and classification (Fukumi et al., 1991; Bremananth et al., 2005; Hessa at al., 2015), Typology, metrology and chronology (Hartmann et al., 2019; Vermeule 1957; Bendall et al., 1978), Reading legends: epigraphy (Kavelar, et al. 2013), Microstructure and chemical composition characterization (Stephen, et al. 2019; Chiarantini et al. 2014; Canovaro et al. 2013; Hartmann et al. 2019), Corrosion behavior (Al-Saad et al., 2007), Conservation and preservation and Manufacturing techniques (Zwicker et al., 1993)

Recently, much literature is presented for examination and analysis of metallic structure and corrosion products of ancient coins. These studies become common because they provide a lot of information concerning the social, economic, and technological history of the mint age (Salem et al., 2019; Canovaro et al., 2013; Mezzasalma et al., 2009; Rodriguez et al., 2004). In addition, coins, like other artifacts, are a good opportunity to improve knowledge concerning long-term corrosion. This work aims to present an applied study to a pottery hoard and concerns archaeometallurgical investigation, manufacturing technique, and conservation processes of this unique hoard.

2. MATERIALS AND METHODS

2.1. Description of the studied hoard and the excavated site

This flans hoard was found inside a small pottery jar and consists of 860 copper coin flans, 11 small metallic pieces in a gray color and 5 struck copper coins. All flans are circular of a diameter ranging from 9 to 11 mm with thickness less than 1 mm. The gray pieces' diameter ranges from 1 to 2 mm and the thickness is from 2 to 4 mm. the Prehistoric copper coins and these of subsequent early decades were often bigger than these dimensions. Dimensional reduction as in this flans hoard indicates likelihood to advancement in manufacturing technology more than the indication of weak economic conditions during the mint period. Thin thickness is a usual feature of ancient gold coins. After the cleaning process, a cross appeared on the surface of two flans (Fig. 1(c)). This indicates that the studied hoard dates back to the Byzantine Age in Egypt.

The majority of these flans do not have any inscriptions, so this hoard does not sufficiently contribute to studies concerning recognition, classification, typology, metrology, epigraphy and chronology (Hartmann et al., 2019; Vermeule 1955; Bendall et al., 1978), as is the case for struck coins. Also, as a result of the absence of surface engravings, these flans do not have artistic or aesthetic values. However, it is unacceptable that this hoard of such a large number is considered of less value than its struck counterpart. The high value of this treasure attributes to the materialistic documentation of the manufacturing technique of ancient struck coins in addition to its rarity as archaeological excavations and museum exhibits.

This hoard was excavated in Abu Rawash village during the renewed excavations in 2019. Abu Rawash village is considered one of the most important regions where many artifacts were excavated.

This village is located 6 km from Giza and follows the center and city of Kerdasa. Also, many archaeological sites from different civilizations have been excavated in this village.

2.2. *Analysis and investigation methods*

2.2.1. X-ray fluorescence

The elemental analysis of metallic structure for the identification of chemical composition was carried out by energy dispersive X-ray fluorescence system (ED-XRF) (JEOL JSX 3222 model) [Vermeule 1955]. X-ray generator is 5 to 50 KV and tube current is from 0.01 to 1.0 mA. X-ray tube is end window type with Be 127 µm thick window. EDS detector has 149 eV energy resolution and from Na to U detectable elements. Four flans were put in the apparatus holder after cleaning their surfaces. Also, the gray pieces were analyzed to determine chemical composition.

2.2.2. SEM–EDS analysis

To observe and analyze the metallic core in high magnification, scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM–EDS) was used with the metallographic samples.

Two cross-section samples from flans and another one from gray pieces were prepared.

Figure 1 (a) Excavation site (Abu Rawash village), (b) the flans hoard, and (c) a cross on flans' surface.

2.2.3. Optical microscopy

The features of metallic structure were investigated by optical microscopy (OM) with the polished cross-sections before and after the etching. The used equipment was polarized light microscope model Bx5 manufactured by Olympus Company, Japan.

2.2.4. X-ray diffraction analysis

To determine the chemical nature of corrosion products, samples from corrosion powder were removed from the coins, powdered and analyzed. The corrosion samples were taken according to corrosion color. X-ray diffraction (XRD) method was used for the analysis of corrosion powder. The XRD equipment was D8 Advance model (Bruker AXS, Germany) and a CuKα source with wavelength 1.54 Å.

3. RESULTS AND DISCUSSION

3.1. *Theoretical background of Manufacturing technique of flans and striking coins*

It is well known that the manufacturing process of ancient coins is carried out by a casting or striking method. Casting is the simplest production process where coins were manufactured in one step by pouring molten metal into a pair of engraved molds. Striking is the usual method to produce ancient coins and the production process includes several stages as follows (Hill 1922; Bendall et al., 1978; Ariel 2003; Barbieri 2005; Cooper 1988; Dreni 2009; Hoover 2008; Raman 1991; Weirong 2003; Ariel 2012):

- *Casting the blank sheet:* the first stage in production was casting blanks (flans) sheet and this stage was carried out after the preparation of the alloying metals. The blank sheet should be made in the required thickness of coins. Blanks in appropriate weight and thickness were made by pouring molten metal into open or two-part closed clay molds. Pouring into a simple open mold or a flat surface can be controlled accurately to produce flans with required thickness and weight. Another method could be used to make flans using a vertical round mold; the molten metal was poured into a rod and after solidification it was divided into flans whether by cutting or sawing with shears. The flans were also cast in particular molds to produce coin-shaped discs that are more regular in shape and easier to the striking process.

- *Cutting or shearing the blanks sheet to flans* (coin-shaped discs called flans or blanks).
- *Cleaning the surface*: one further step often required before the flan could be struck, this step is the removing process of smelting slag and any impurities on a surface. Flans could have been cleaned in some organic acids or pickles.

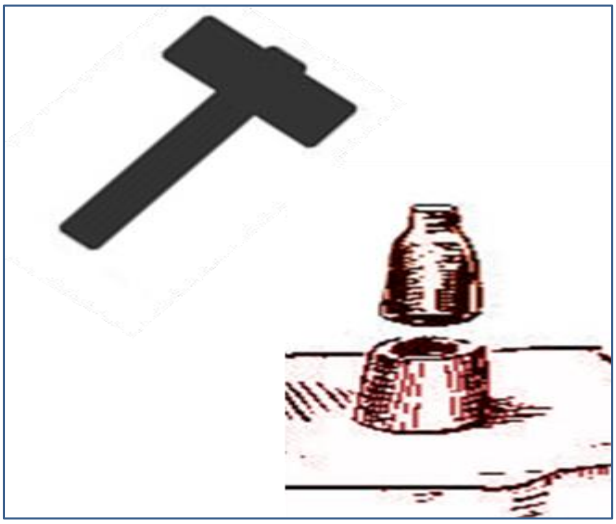

Figure 2 Schematic illustration of the process of striking or stamping flans

- *Striking or stamping flans* (Fig. 2): the used tool for stamping flans is called the die; the upper (reverse) set in a punch, and the lower (obverse) set in an anvil. A die is a hard metallic rod engraved with coin design to stamp flans. Flans were likely heated before being struck to make it easier to stamp and keep the dies from getting broken or damaged. Gold and silver flans, which are relatively soft, likely were undergone cold striking. After the striking process, the blanks become real coins and are no longer flans. A few ancient dies have been preserved. Also, many errors occurred during striking the coins.

3.2 *The chemical composition (elemental analysis) of the metallic core*

XRF results (Fig. 3) showed that the chemical composition of flans consists of copper (65.4 wt.%), lead (33.8 wt.%), and silicon (0,8 wt.%). Based on this composition, the metallic structure is lead-copper alloy and the other two elements are impurities mixed with copper and lead ores. Moreover, some literature works mentioned that the low concentration of tin, less than 2 wt.%, in copper artifacts especially from the Iron Age objects typically indicates the recycling [Ashkenazi et al., 2012; Vale'rio et al., 2010] since recycling increased significantly in this age in the ancient Mediterranean civilization. The use of tin with copper at an optimal amount of 8–10 wt. % was used to make strong weapons and hard tools. Contrarily, a soft metallic core is used for struck coins to facilitate the stamping process. Hand strikes cannot achieve the same force like machine strikes, so copper coins alloys were softened by the presence of Pb and/or by "hot striking" [Stephen et al., 2019]. Also, the addition of more than 3 wt. % Pb to copper increases the fluidity of molten metal and improves the surface finish of the solidified casting [Dilo et al., 2010; Scott 1991]. For the presence of silicon in the flans analysis (less than 1 wt.%), it was not Intentionally added to copper and bronze artifacts, and its presence in the alloy was probably a result of the pottery jar and/or as impurity combined with the copper and lead ores [Charalambous et al., 2014]. Also, XRF of the small gray pieces (Fig. 3(b)) revealed that these pieces consist of 99.6 wt.% lead. These pieces are likely the pure lead used in the casting process of flans alloy.

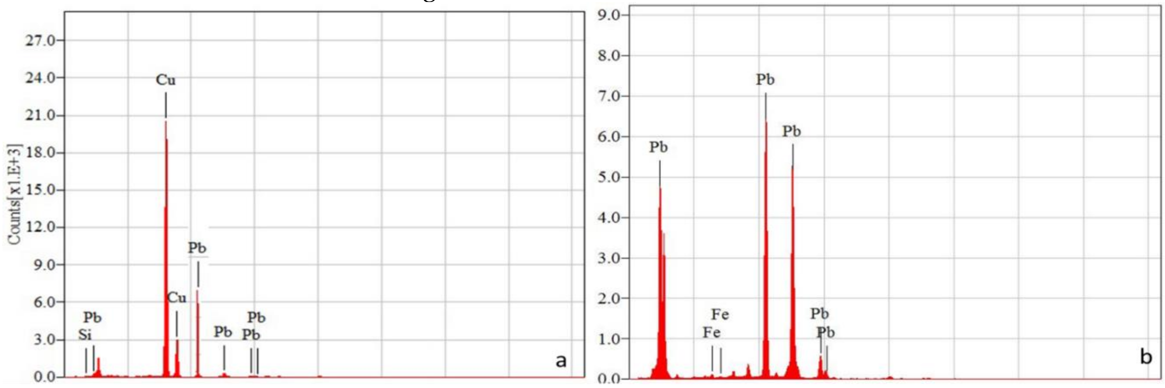

Figure 3 the elemental analysis by XRF of the copper flans (a) and lead pieces (b).

3.3. Metallography and microscopic observations

As expected, the unstruck flans did not exhibit significant metallographic features (Fig. 4), because flans do not require any flattening or annealing before the striking process [Stephen et al., 2019]. The microstructure under polarized light microscope (PLM) showed a homogenous α solid solution with α–δ eutectoid phase, with elongated shape, which is a common intermetallic phase in the ancient copper-based alloys besides the alpha phase. What is more, the microstructure showed clearly inner corrosion pits, some global inclusions, and segregated lead islands from the metallic matrix. The formation rate and globules size depend on the solidification time; slow solidification rates allow the formation of globules of large dimensions [Francesca et al. 2020; Quaranta et al., 2014; Huges et al., 2007; Di Bella at al., 2018]. In some areas, the oxide layer (cuprite) that formed over the surface penetrated the metal/corrosion interface as intergranular corrosion attacks through cracks. This phenomenon is called pseudomorphic replacements (PR) which means the formation of some pseudomorphic replacements of the alloy's microstructure with oxidation products, especially cuprite [Oudbashi et al., 2016]. Also, The SEM–EDS of two cross-sections (Fig. 5) showed that the chemical composition of the flans is approximately 60% Cu and 40% Pb. The presence of C and O attributes to internal corrosion products in metallic structure while Al, Fe, and S belong to ores impurities of copper and lead. Also, the microstructure was featured by two phases: a copper-based phase in light gray color and a lead-based phase in dark gray color (Fig. 5).

3.4. Deterioration state and surface corrosion morphology

In fact, the presence of the flans within the pottery jar does not restrict the occurrence of corrosion reactions. Clearly, even within a pottery jar, the possibility of encountering various corrosive effects on copper alloys coins buried for long-term is quite likely. As a result of the high porosity, the pottery jar does not limit the supply of various soil factors that affect flans, corrosion. Therefore, the studied flans were subjected to the usual corrosive factors in the soil environment such as oxygen, moisture, groundwater, and soluble anions and cations (HCO_3^-, CO_3^{2-}, SO_4^{2-}, NO_3^-, PO_4^{3-}, Cl^-, Na^+, K^+, Mg^{2+} Ca^{2+}) [Al-Zahrani et al., 2012; Oudbashi 2018].

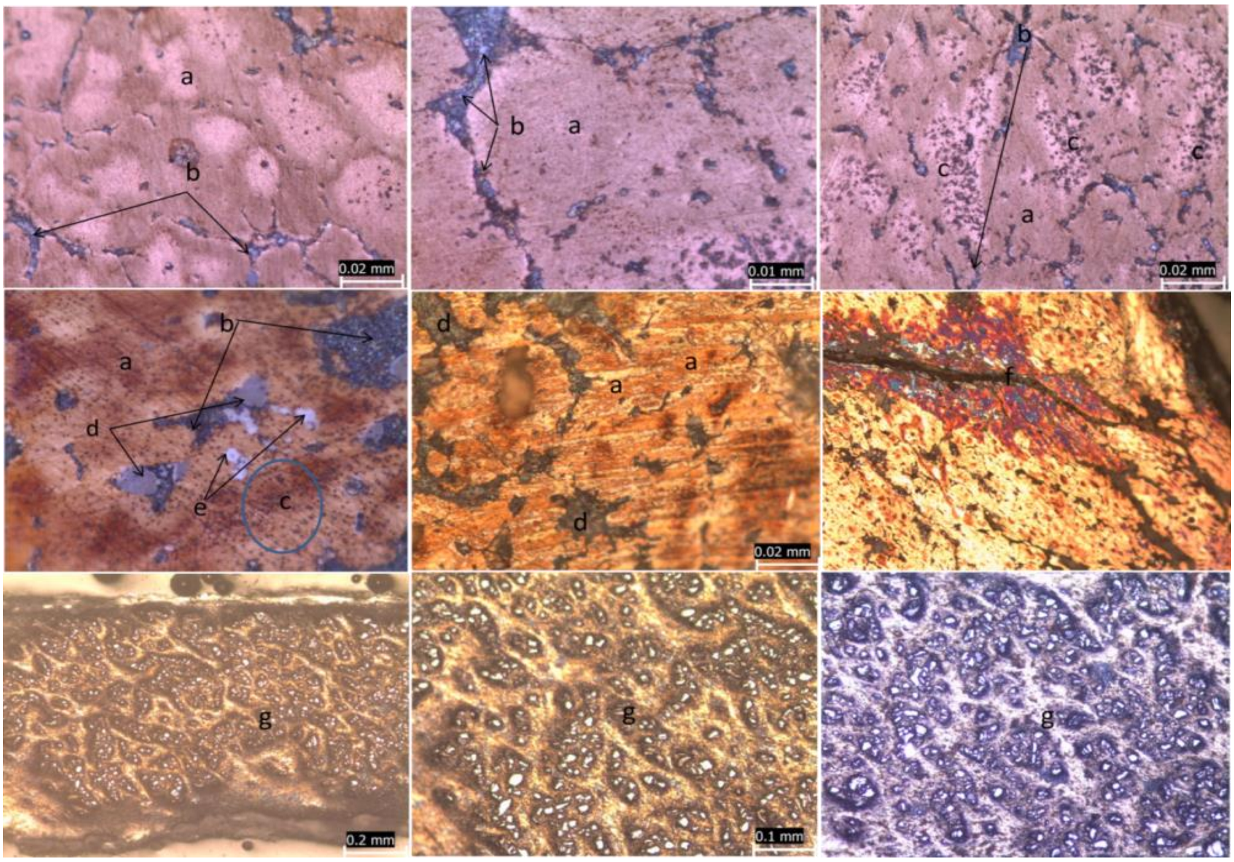

***Figure 4** The features of metallic structure of flans and lead pieces under polarized light microscope in the presence and absence of the analyzer: (a) a solid solution, (b) a–δ eutectoid phase, (c) inner corrosion pits, (d) some global inclusions, (e) segregated lead islands from metallic matrix, (f) penetrating the oxides corrosion (cuprite) of the metal/corrosion interface through cracks, and (g) the microstructure of the pure lead pieces.*

So, common corrosion morphologies and products are observed on the flans. Adding to this, the pottery jar itself can be considered as another source besides the soil for soluble anions and cations interacting with flans. Groundwater in the burial environment assists in the dissolution of water-soluble salts in the pottery jar itself. Cl^-, NO_3^-, NO_2^-, and SO_4^{2-} are anions and cations detected in the water-soluble salts effloresced on ancient pottery during long-term storage in a museum (Jin et al., 2014).

The majority of the flan hoard is partially corroded by forming an external corrosion layer in light green and green color formed above a thin noble patina in blackish-brown color (Fierascu et al., 2017). However, most flans are generally in good preservation condition and the boundaries of the original surface remain. After local grinding, the brown-red preserved metal was discovered beneath corrosion products. This simple corrosion morphology attributes probably to burial inside the pottery jar. Although this jar does not restrict the exposure to various corrosive factors of soil environment, the presence of the flans inside it gave a good preservation rate compared to direct or indirect burial in soil (Barrena et al., 2008; Graedel 1987). However, various corrosion phases appeared on the flans surface (Fig. 6, 7) such as the following:

- Weak adhesion powdery corrosion in a green color
- A thick, smooth, and lustrous corrosion crust in light green
- Uniform corrosion in blackish brown color as thin noble patina
- Blue-green (turquoise) corrosion phase
- Partial and complete mineralization
- Also, further deterioration aspects appeared such as loss and adhesion among some flans.

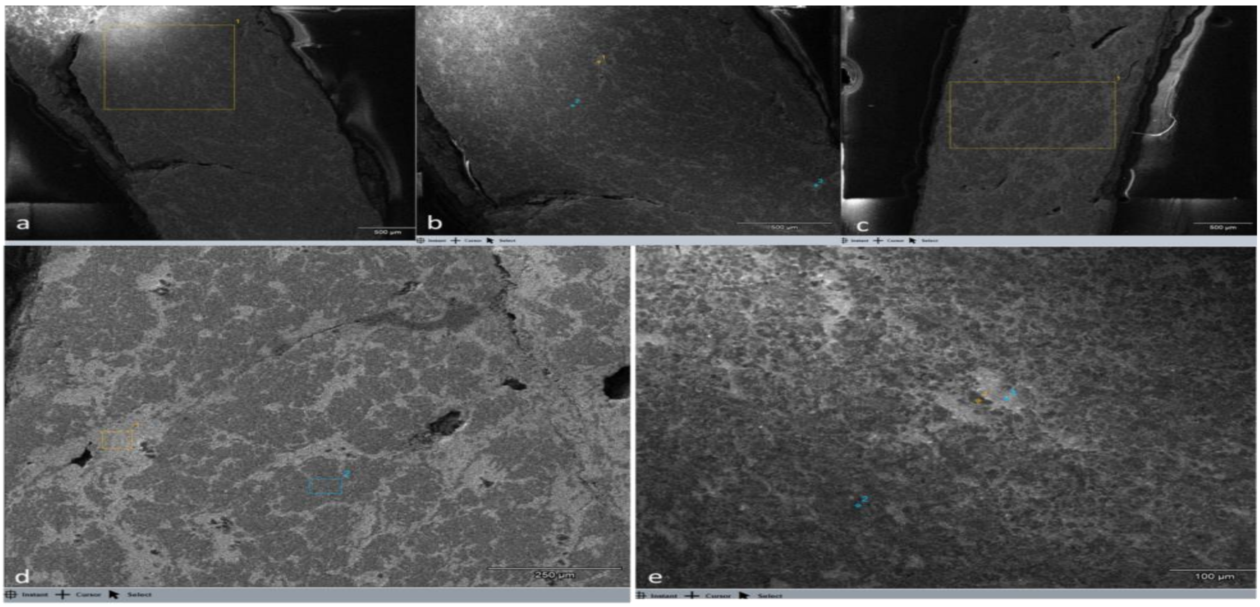

Fig.	Zone/spot	C	O	Pb	Cu	Al	Mg	Fe	S
a	zone	7.80	3.88	27.15	59.58	1.59			
b	spot 1	1.48	3.73	94.79					
	spot 2	2.72			86.35				
	spot 3	2.52	8.38	83.63		5.46			
c	zone	4.62	3.40	37.14	52.39	1.26		1.18	
d	spot 1	1.17	6.75	89.52	0.69	1.28		0.60	
	spot 2	3.60	0.09		87.22	0.65		7.71	0.74
	spot 1	11.32			86.96	1.73			
e	spot 2	9.31			88.38		2.31		
	spot 3	6.62	9.28	84.10					

Figure 5 Micrograph and elemental analysis of the metallic structure of the studied flans; the microstructure showed a copper-based phase in light gray color and a lead-based phase in dark gray color.

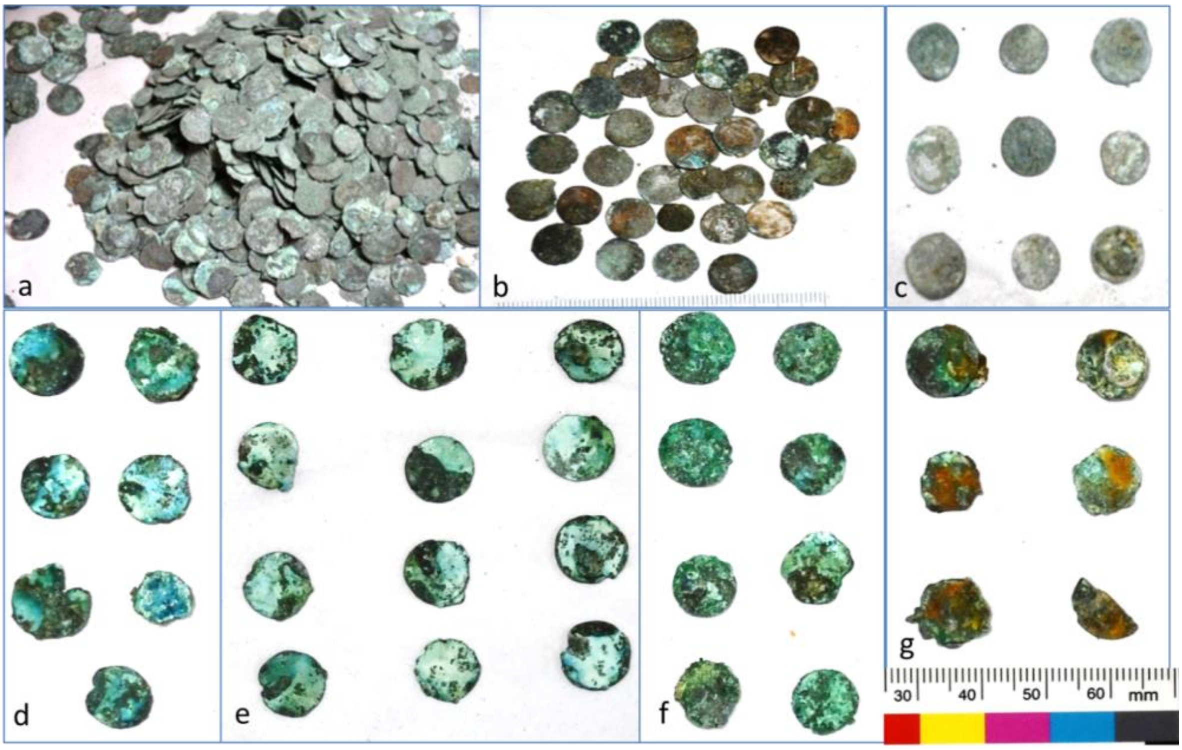

Figure 6 Forms and morphologies of corrosion products on the flans and lead pieces: (a) weak adhesion green corrosion, (b) thin noble patina in blackish brown color, (c) gray and greenish gray corrosion on lead pieces, (d) blue-green (turquoise) corrosion phase, (e) smooth and lustrous corrosion crust in light green color, (f) cohesive green corrosion, and (g) loss, adhesion among flans, and partial mineralization.

3.5. Mineralogical composition of corrosion products

The results of XRD qualitative analysis of samples from various corrosion phases are presented in Fig. 8. The results showed the presence of all corrosion products of copper trihydroxychlorides $Cu_2Cl(OH)_3$ (atacamite, paratacamite, botallackite and clinoatacamite). These products are the main corrosion products of the light green phase (thick, smooth and lustrous). These products are formed on the studied flans due to the presence of chloride ions in soil environment. Chloride ion is highly reactive toward copper-based artifacts and is found in abundance in the Egyptian soil, because it is rich in sodium chloride [Salem et al., 2019]. Botallackite and clinoatacamite as corrosion products on the copper are little compared to the other copper trihydroxychlorides because they are less stable than the other isomers [Scott 2002]. Atacamite and paratacamite are common corrosion products on copper-based artifacts excavated from soil environment. They can occur as original corrosion products or secondary from other products, especially cuprite and nantokite [Scott 2002]. Also, the XRD showed that powdery corrosion in green color is malachite $CuCO_3.Cu(OH)_2$. Some studied flans were covered by a cuprite patina only as the main layer. During the oxidation process, cuprite is the predominant oxide of copper-based artifacts and occurs over a wide range of environmental conditions [Scott 2002]. Cuprite can also form on copper artifacts as a secondary product by transformation from other products, especially cuprous chloride. Cuprite as a thin layer adjacent to the original surface assists in the protection of many flans. For lead pieces, lead has sufficient corrosion resistance in a wide range of environmental conditions so it is currently used as plating on base metal electrical contacts [Leygraf et al., 2015]. Lead was not used as a single metal to manufacture metallic objects, but it was a constituent in alloys especially copper-based alloys [Scott 2002]. Lead is susceptible to react with corrosive agents in surrounding environment [Graedel 1994]. A wide variety of lead corrosion products were obtained from pewter and bronze objects [Ian, 1991]. The studied lead pieces were carefully scraped to collect the hard crust in gray color formed on the surface. The analysis results showed that this crust consists mainly of chlorides such as laurionite Pb(OH)Cl and cotunnite PbCl2 in addition to carbonates such as cerussite $PbCO_3$ and hydrocerussite $2PbCO_3.Pb(OH)_2$. The formation of chlorides corrosion products on the flans and lead pieces as the main products indicates that the main soluble anion in soil environment is chloride Cl-. Also, there is no evidence of formation of sulfides and sulfates products among corrosion products of the flans or lead pieces.

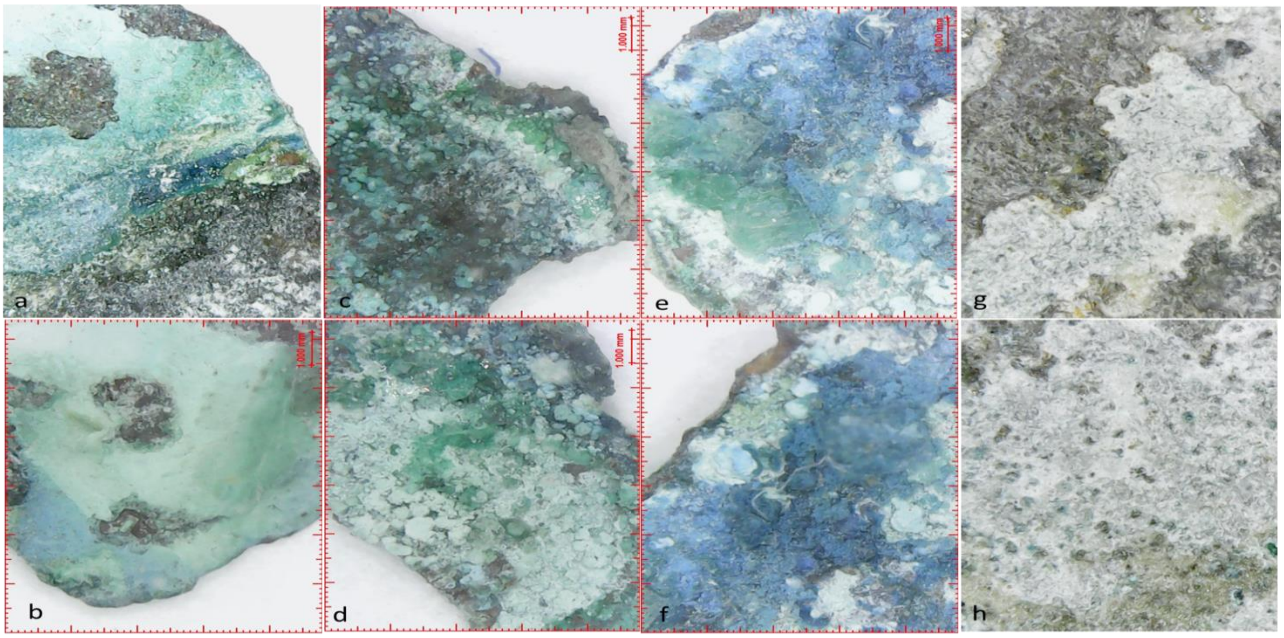

Fig. 7 Macrographs by USB microscope of the corrosion phases on the copper flans and lead pieces: (a, b) light green corrosion as a smooth and lustrous crust, (c, d) powdery green corrosion, (e, f) blue-green (turquoise) corrosion phase, and (g, h) gray phase corrosion on the lead pieces.

3.6. Treatment and Conservation

The cleaning process was necessary to ensure flans' stability, application of corrosion inhibitor, stopping active corrosion, and the possibility of detecting inscriptions on these coins (Al-Sadoun, et al., 2019). Therefore, it was necessary to clean the soiled deposits, corrosion layers, and encrustations covering these flans. Cleaning methods should be chosen based on the surface corrosion morphology and their effectiveness and safety on the surface. As usual when cleaning metallic artifacts, mechanical cleaning was selected as the first cleaning method of flans' surface. Hand mechanical cleaning tools are safer than any other cleaning method. Brushes, spatula, and scalpels were used carefully to avoid scratching the surface. Unexpectedly, the mechanical cleaning was not suitable or sufficient to clean the studied flans. Moreover, the surface was easily scratched when using a fiberglass brush—the most effective tool among mechanical cleaning tools-. Although this brush was hard enough to remove corrosion, it was not sufficient to remove corrosion layers or thin black brown patina. Therefore, it was necessary to use some chemical cleaning solution. According to the conservation rules, the cleaning process should start with the safest solution with the lowest possible concentration. Three chemical solutions were selected to remove residual corrosion after mechanical cleaning. The solutions were EDTA, Rochelle salt, and bicarbonate ammonium. The chemical substances dissolved in tap water and the concentration was 3%. According to conservation principles, it was necessary to carry out an experimental study on the selected chemicals to evaluate their efficiency and determine the best substance that can be applied in the conservation of these flans. To use less concentration with more efficiency, the flans were cleaned inside ultra-sonic wave's tank. This also contributed in reducing the cleaning time. For a more realistic experimental test, the selected solutions' efficiency was evaluated on two flans from the hoard rather than artificially corroded coupons. The experimental test flans were immersed in the selected solutions inside a bottle for 4 hours and then taken out and rinsed well with water to evaluate the efficiency of each solution. The results showed that EDTA solution gave the best results among the suggested cleaning solutions (Fig. 9). Consequently, EDTA was used to clean the blanks hoard. The flans were divided into three groups based on the remaining corrosion on the surface after mechanical cleaning. Each group was immersed in EDTA solution inside the small ultra-sonic cleaner (500 ml). Cleaning period, changing solution, and repeating the process of ultrasonic cleaner depended on the corrosion rate of each group. After finishing the cleaning process of each group inside ultra-sonic cleaner, the flans were placed in a petri dish and cleaned with a fiber brush to remove any remaining corrosion on their surfaces. Then, they were washed well with distilled water and dried.

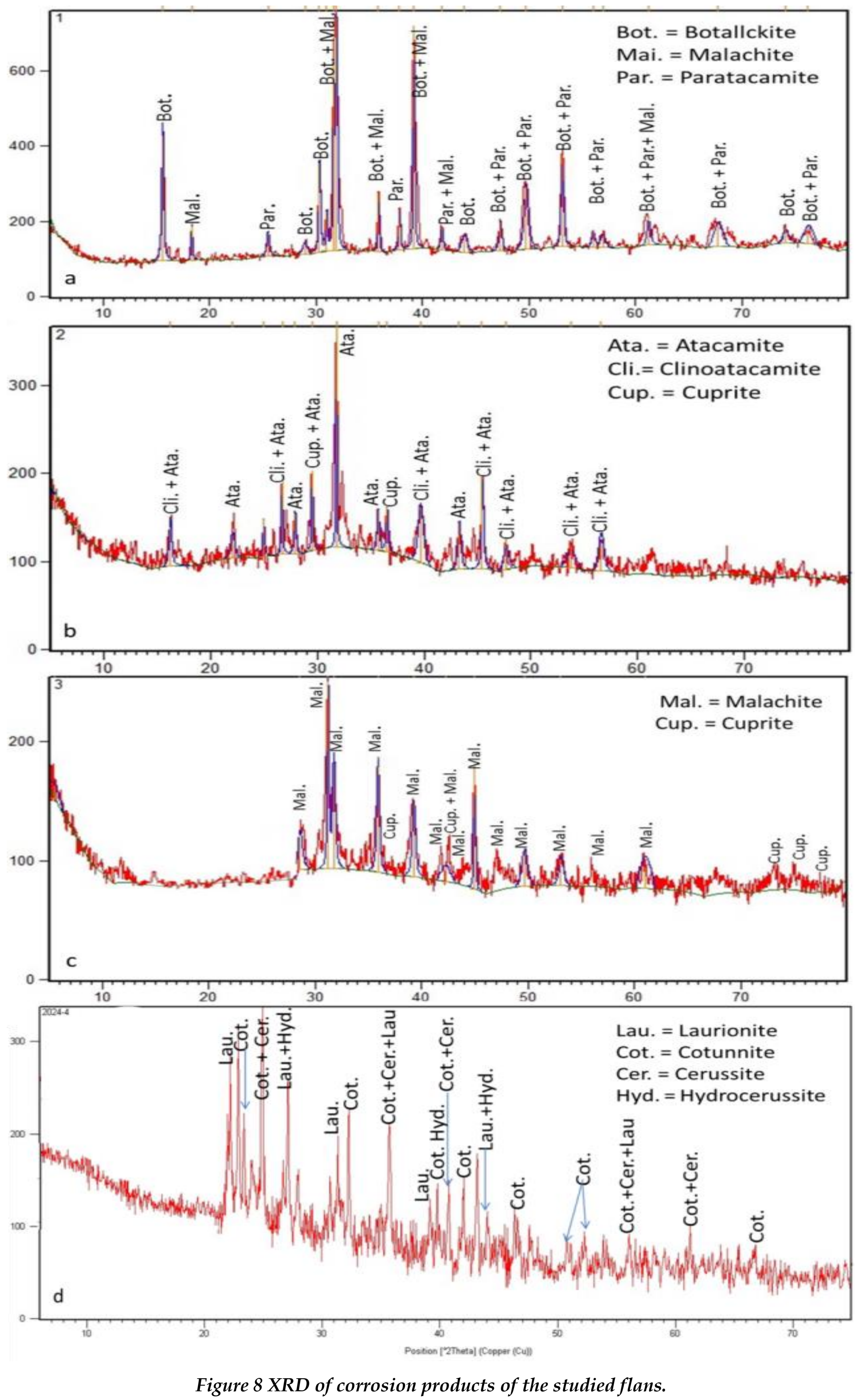

Figure 8 XRD of corrosion products of the studied flans.

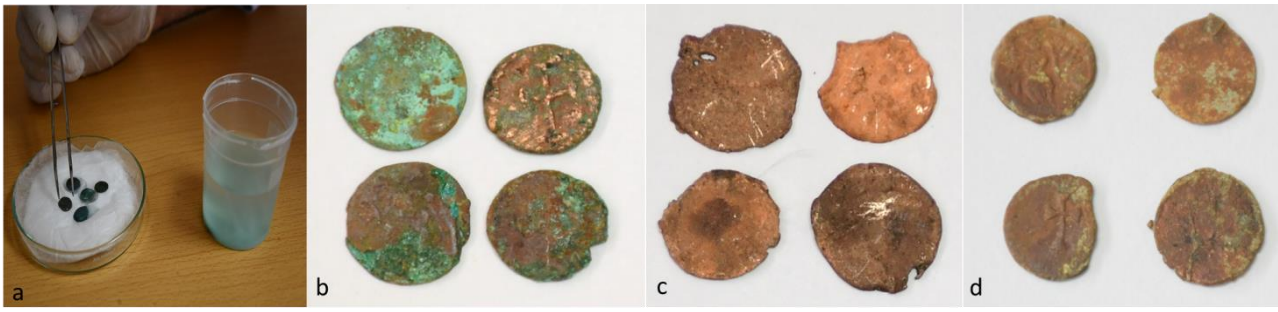

Figure 9 (a) during the mechanical cleaning, (b) after he cleaning by Rochelle salt, (c) after the cleaning by bicarbonate ammonium, (d) the cleaning by EDTA.

4. CONCLUSION

The investigated hoard is considered archaeological material evidence (an eyewitness) and a unique opportunity to document and confirm knowledge concerning the manufacturing technology of coins.

The results contribute to the knowledge documentation concerning the coins manufacturing technology during the Byzantine Age in Egypt.

The analysis results in this work have shown that the flans in this hoard were made of a lead-copper alloy. The microstructure under PM showed a homogenous α solid solution with α–δ eutectoid phase that is a common intermetallic phase in the ancient copper-based alloys besides the alpha phase.

The corrosion products identified on the flans surfaces resulted from a series of reactions between metallic constituents and soil species. The majority of the flans hoard is partially corroded by forming an external corrosion layer in light green and green color formed above a thin noble patina in blackish brown on the surface. The external corrosion layer consists of copper trihydroxychlorides (atacamite, paratacamite and clinoatacamite) and malachite while the noble patina is cuprite product.

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