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ANALYTICAL STUDY AND CONSERVATION PROCESSES OF TUTANKHAMEN DECORATED STICK: A CASE STUDY

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ABSTRACT

The stick studied here dates back to the new kingdom, 18^{th} dynasty, King Tutankhamen (1337-1347 B.C). More than 130 sticks and staves were founded in the tomb of Tutankhamen; many of the forms were clearly for ritual while others showed signs of use. Here we will will shed light on one of the ritual stick, where materials of decoration and conservation processes were investigated. Visual assessment, Optical Microscopy (OM), X-ray fluorescence (XRF), X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) were used in this study to elucidate the components of the stick, deterioration aspects and identify the previous conservation materials. Conservation processes entailed cleaning, removal of improper previous conservation material and filling gaps and voids.

KEYWORDS: Tutankhamen; Decorated Stick; Conservation; Wood; XRF; OM.

1. INTRODUCTION

The number of sticks and staves buried with Tutankhamen were some 130 complete and fragmentary examples, from the Antechamber, the Burial Chamber and the Annex. Many of the forms were clearly ritually; others, however, showed signs of use. (Reeves, 1990) The studied stick (Fig.1) is made of wood decorated with tree bark at the middle, while the upper and lower parts are decorated with cylindrical metals; the upper part has a knob similar to lotus flower. Metal ring decorated with *Repoussé* is existed under the metal upper part while metal cap is located at the end of the lower part; thread fibres are noticed above the lower metal part. The bark is attached to wood substrate by resinous material and coated by varnish (another resinous material). Stick dimensions are L.110 cm × Dia. Max.13.4mm.

Figure 1. The stick broken into two pieces

2. MATERIALS AND METHODS

2.1. Visual assessment

Visual assessment, by the critical eye of the team work, was applied to determine the aspects of deterioration found on the stick. This method is very effective because the causes and mechanism of deterioration may be easily identifiable. The critical eye of conservator can also determine the most effectiveness techniques of analysis, which should be applied for identifying the condition of the stick. (Abdallah, 2016; Abdel-Maksoud, 2011).

2.2. Optical Microscopy (OM)

A stereo microscopy, Zeiss Stero DV 20, equipped with Axio Cam MRC5, was used to study the stratigraphic structure of the decorated stick and assess the deterioration caused by previous conservation materials.

Compound light microscope Optika B-383PL equipped with digital camera 4083-B9 was used to for identification of wood species and thread fibres (Donaldson, 2009; Wilding, 2009) which used in previous conservation.

2.3. X-Ray Fluorescence (XRF)

In order to identify the metal parts, ground layer and Previous conservation filler found on the stick X-Ray fluorescence analysis was carried out with portable system, thermo scientific Niton XL3t analyzer includes X-ray tube with Ag anode, 50kV and 0-200µA max, metal and mining modes, spot diameter 3mm and duration of exposure 60 second.

2.4. X-ray Diffraction (XRD)

Previous conservation filler and resinous material were analyzed by X-ray diffraction using X-ray Diffractometer System PW3040–Analytical Equipment–PANalytical pro model, Cu-target tube and Ni filter at 40kV and 30MA. X'Pert-Highscore software was used for identifying.

2.5. Fourier Transformed Infrared Spectroscopy (FTIR)

Fourier transform infrared spectroscopy was done by using FTIR spectrometer (VERTEX 70, Bruker) equipped with An attenuated total reflection (ATR) technique, spectrum in the range 4000-400 cm^{-1}, range with resolution of 4cm^{-1}, sample scans16, was used to identify the resinous materials.

An FTIR spectrum of previous restoration material was measured by using Shimadzu FTIR spectrometer by the KBr pellet technique for sample preparation. Spectra were measured at a resolution of 4 cm^{-1} and 20 scans were recorded per sample by using IR-Prestige-21 –FTIR spectrometer and the IRsolution software in the transmission mode, spectrum in the range 4000–400 cm^{-1}.

3. RESULTS AND DISCUSSION

3.1. Visual assessment

The desiccation condition of wood and the presence of two relatively heavy metal parts in upper and lower parts led to carving the stick and break it finally into two pieces. It is clear that the stick is previously restored by using fillers and threads which hide the original surface. Besides, physical deterioration aspects such as friable decorated layer, separations, cracks, loss. Metal parts are corroded.

3.2. Optical Microscopy (OM)

3.2.1. *Previous conservation materials*

Examination by stereo microscope showed that the previous filler material and threads are applied directly over the original surface (Fig.2).

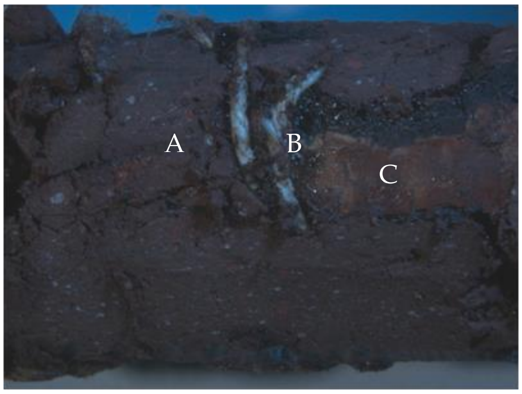

Figure 2. Previous conservation filling material obscures the original surface. A. Modern filler B. modern Threads. C. original surface. (Magnification 30x)

3.2.2. *Identification Previous conservation threads*

Morphological structure of longitudinal form of threads (Fig.3) showed that the fibres are twisted with frequent changes of direction, which indicates that the threads are cotton fibres; cotton is rather flat and looks like a ribbon when it twists (Landi, 1998; Hearle, 2007).

Figure 3. The apparent twist in these fibers is typical of cotton. A unique optical property of cotton is its lack of an extinction position. (Magnification 400x).

3.2.3. *Stratigraphic structure*

Examination of stratigraphic structure of the stick (Fig.4) shows that the stick is made of wood decorated with black resinous material, tree bark and resinous layer respectively.

3.2.4. *Wood identification*

The microscopic analysis of the xylem anatomy provided results on the wood identify as Almond (*Prunus dulcis* Mill) (Fig.5) (Crivellaro and Schweingruber, 2013). The almond wood was used in ancient Egypt for making furniture, walking sticks, spear shifts and arrows (Gale, et al., 2000).

Figure 4. stratigraphic structure shows the sequence of the stick layers (Magnification 500x)

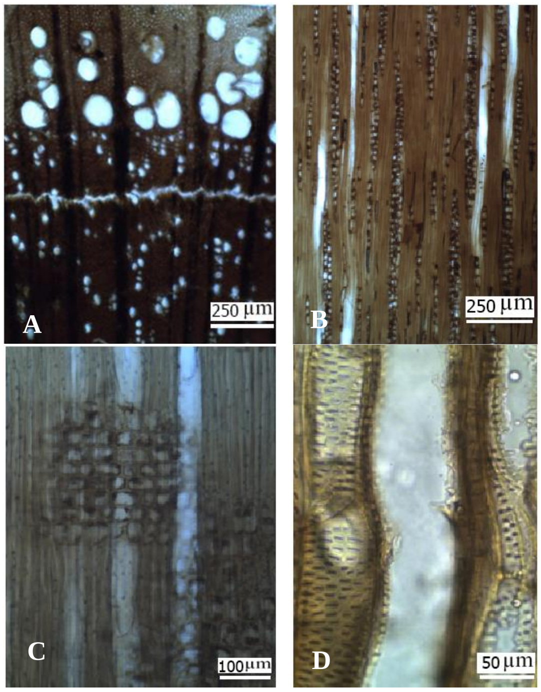

Figure 5. characteristics of wood species by (OM): (A) Transverse section (TS) shows Growth ring boundaries are distinct by the difference in vessel size between latewood and earlywood, wood semi-ring porous to ring-porous, vessels solitary and in radial multiples of 4 or more, fibers very thick-walled, axial parenchyma diffuse and scanty paratracheal (40x). (B) Tangential section (LS) shows Rays of two distinct sizes: uniseriate and 4 to 10 seriate (100x). (C, D) Radial section (R.S) shows simple perforation plates, Inter-vessel pits alternate, Vessel-ray pits rounded or angular with large apertures, Fibers with distinctly bordered pits (fiber tracheids), and Body ray cells procumbent with mostly 2-4 rows of square marginal cells (C.200x, D.400x).

3.3. X-Ray Fluorescence (XRF)

3.3.1. *Gold leaf layer, the papyrus shaped upper part and ring*

Gilding is the technique whereby thin sheets of gold are applied onto a firm support to achieve the rich appearance of solid gold (Rifai and El Hadidi, 2010). XRF microanalysis revealed that the gilded layer is made of a composition of gold (Au) as a main element and silver (Ag) a secondary element, besides Traces of Cu, (Fe), (Pb) and (Zn).

The results obtained by XRF analysis show that the upper part is made of a gold-based alloy with silver and copper, Traces of (Fe), (Cr) and (Ti) were detected. The golden ring is made from similar alloy with little changes in the amount gold; silver and copper, Traces of (Fe) and (V) were detected (Table 1).

3.3.2. *Silver lower part*

The elemental analysis by metal mode proved that the lower metal part made of silver based alloy with copper and gold, as well as traces of Si, Cr, and V. In order to identify the corrosion products mining mode was used which detects high percentage of sulphur (S) as a result of silver corrosion and chlorine (Cl) as result of copper corrosion (Table 2).

3.3.3. *Bronze cap*

The elemental analysis proved that the lower terminal metal cap is made of bronze alloy (Table 3). At the present day, ordinary bronze contains about 9 to 10 per cent of tin, but ancient bronze is more variable the proportion of tin ranging from about 2 per cent to about 16 per cent (Lucas, 1964).

Table 1. Elemental analysis of the gilded layer and golden parts using XRF.

sample	Element %								
	Au	Cu	Ag	Fe	Cr	Ti	Pb	Zn	V
Gold leaf	91.157	0.220	6.681	0.234	-	-	0.596	0.208	-
Golden upper part	87.196	6.459	5.665	0.328	0.134	0.073	-	-	-
Golden ring	90.097	2.647	6.604	0.309	-	-	-	-	0.107

Table 2. Elemental analysis of the silver part using XRF.

sample	Element %								
	Ag	Cu	Au	Si	Cr	V	S	Cl	K
Silver part (Metal mode).	90.149	4.290	3.060	0.985	0.149	0.110	-	-	-
Silver part (Mining mode).	71.795	12.977	4.005	-	-	-	7.763	1.967	0.389

Table 3. Elemental analysis of bronze cap using XRF.

sample	Element %								
	Cu	Sn	Ag	Si	S	Pb	Fe	Au	Ti
Bronze cap	92.133	2.285	1.233	1.184	1.088	0.632	0.438	0.130	0.059

3.4. X-ray Diffraction (XRD)

3.4.1. *Previous Conservation filling materials*

XRD analysis for previous conservation filling materials shows that it consists mainly of gypsum ($CaSO_4.2H_2O$) and anhydrite ($CaSO_4$), with minor amount of hematite (Fe_2O_3). The results obtained by XRD proved that the previous filler is mixture of gypsum and hematite, the gypsum is the bulk of filler paste while hematite is used to give the filler a proper colour (Fig. 6).

3.4.2. *Black resin*

Representative XRD pattern of the studied black resin sample is given in (Fig.7). The result of XRD indicates the presence of graphite which suggests that the black resin is mixture of resinous material and carbon substance.

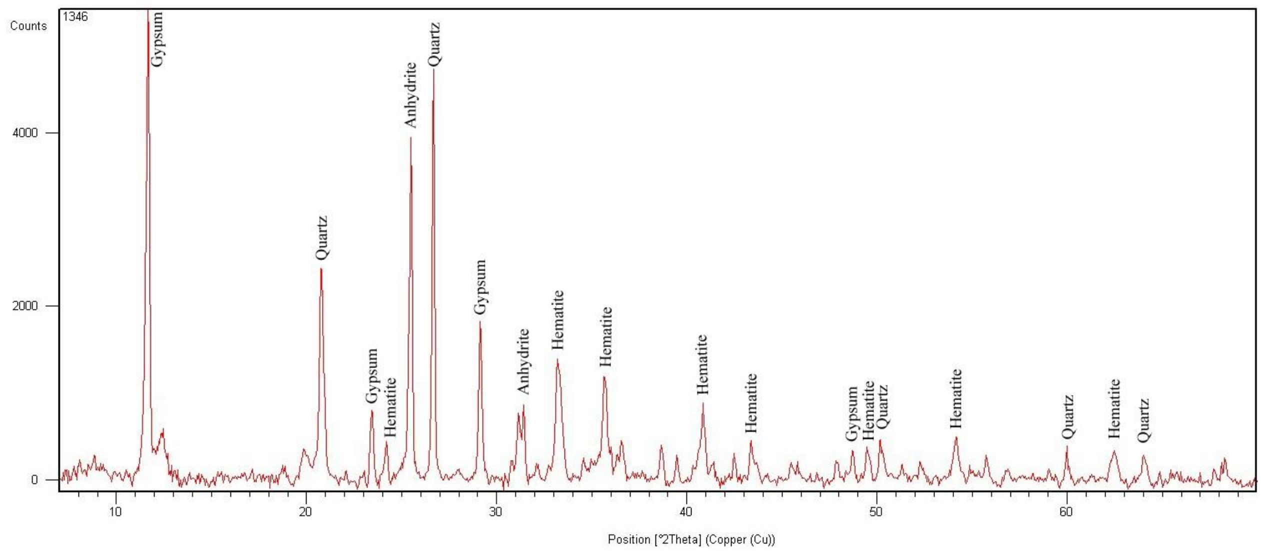

Figure 6. X-ray diffraction spectrum for previous conservation material showing the filling material is a mixture of gypsum, anhydrite and hematite

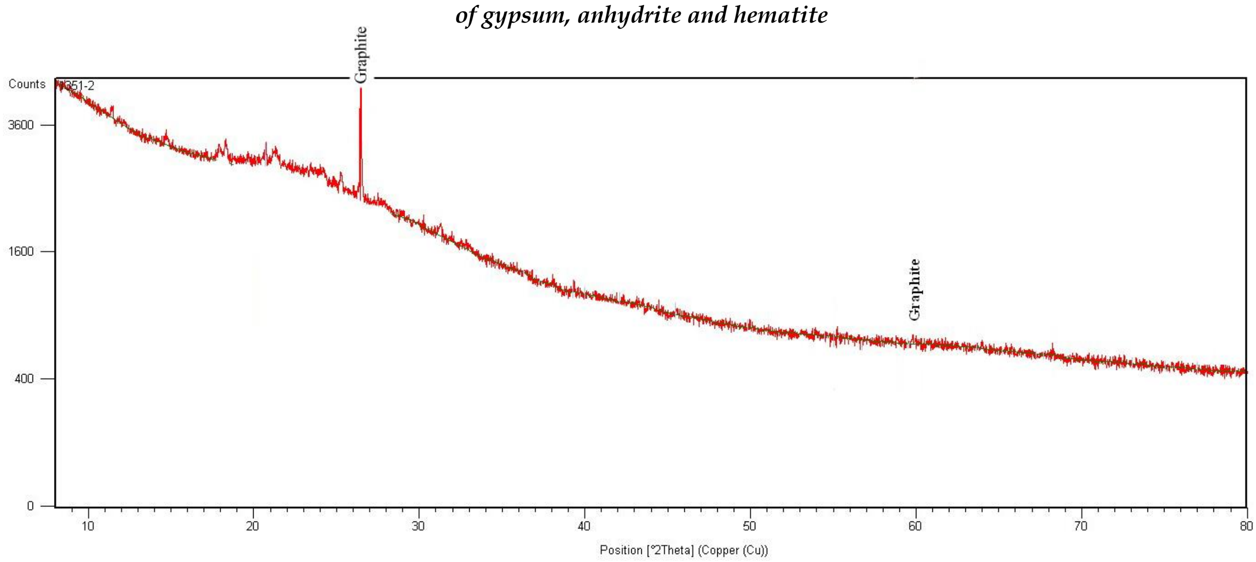

Figure 7. X-ray diffraction spectrum of previous conservation material shows the presence of graphite in the black resinous material.

3.5. Fourier Transformed Infrared Spectroscopy (FTIR).

3.5.1. Black resin

The bands at 3190 is attributed to OH stretching band, the band at 3069, 2932 cm^{-1} represent the C─H stretching band, the band at 1647(C═O stretching band) , while the bands at 1542 (C─N─H) is assigned to amide II. 1388, 1323 cm^{-1} C─H bending band, 1300-900 C-O stretching band. The spectrum of black resin suggests that the black resinous material is a mixture of mastic (*Pistacia* resin) with another nitrogenous material may be hide glue (Ikram, 2012).

3.5.2. Previous Conservation filling materials

It is clear that the band at (3547.09, 3404.36 cm^{-1} and 3489.23) represents (OH) hydroxyl stretching due to intermolecular hydrogen bonding of the hydroxyl group and NH stretch of aliphatic primary amine. The stretching vibrations of the H_2O molecules in the gypsum occur at 3547.09 and 3404.36 cm^{-1} as shown in (Fig.8) (Al Dabbas, et al., 2014), (Wei, et al. 2016). The C─H stretching vibrations occurred in the region (2958.80 and 2926.01 cm^{-1}) stretching of aliphatic groups. The band at 1622.13 (C═O stretching band) is assigned to amide I, the increasing or decreasing of C=O is dependent on the physical state of the sample. In the solid state, the frequency of the vibration is slightly decreased. While the bands at 1541.12 (C─N─H) is assigned to amide II (Abdel-Maksoud and El-Amin, 2013; Derrick, et al., 1999). The bands at 1141.86 and 1122.57cm^{-1} and the small peaks at 671.23 and 594.08 cm^{-1} are assigned to the stretching and bending modes of sulfate of gypsum spectrum (Al Dabbas, et al., 2014; Wei, et al., 2016; Abdel-Maksoud and El-Amin, 2013). The FTIR spectra indicate that the previous conservation filling paste is a mixture of gypsum as a bulk and animal used as binder while hematite which identified by XRD is used as a color.

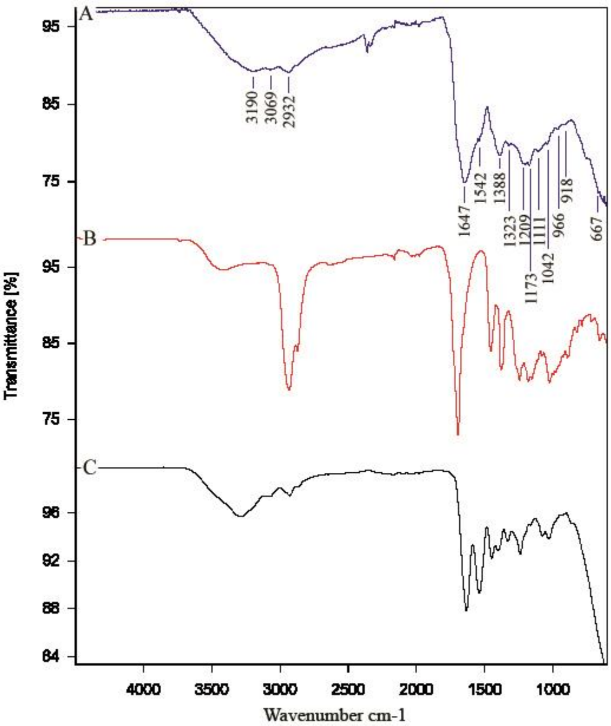

Figure 8. FTIR analysis spectra of black resinous material.

4. CONSERVATION PROCESSES

Conservation processes aim to preserve and reveal the aesthetic and historic value of the monument and is based on respect for original material and authentic documents,(ICOMOS, 2016; Abd rabou, et al., 2015) therefore, several procedures for treating and persevering the studied stick were used as soon as it was transported to the wood laboratory of the Grand Egyptian Museum-Conservation Centre (GEMCC), entailed cleaning, stabilization of the friable tree bark layer, removal of previous restorations and filling cracks and voids.

4.1. Surface cleaning

Loose dust was removed by gentle brushing. Because the stick was previously treated by paraffin wax [Griffith Institute, 2017], cotton wool buds damped with toluene were rolled over the surface to remove accumulated grime and excessive wax layer.

4.2. Stabilization and Reattaching lifting bark layers

The bark layer lost its adhesion to the resinous material on the wooden support in many places of the stick. Solution of 1% Klucel G (Hydroxypropyl cellulose) was applied by injection and then the separated bark layers were eased back into position with the help of a little gentle pressure using silicone paper. For the friable areas 0.5 - 1% w/v Klucel G dissolved in ethanol was applied on the surface using a brush (Horie, 2010; Johnson, et al., 1995; Hatchfield and Kyan 2010).

4.3. Removing of previous restorations

Knowing that the binder used for gap-filling in previous restoration was hide glue, warm distilled water was used to assess the solubility of the binding material. A pad of cotton saturated with warm water covered with polyethylene sheet was helpful in softening the gap filling materials, which were then reduced and removed by mechanical methods e.g. scalpel, dental tools, (Fig.9) etc.

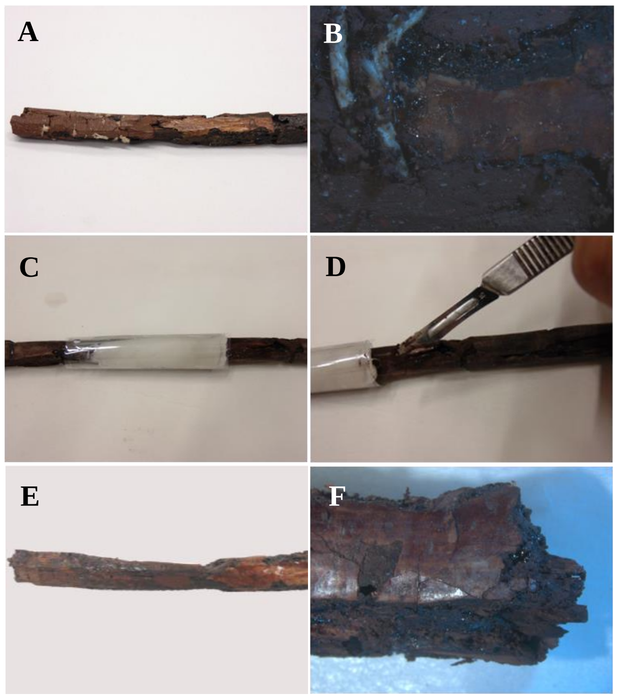

Figure 9. process of removing improper previous conservation filling material: A. previous filling paste and threads. B. the previous conservation material over the original surface. C. Softening the previous conservation paste by a pad of cotton saturated with warm water covered with polyethylene. D. removing the previous filling paste. E. after reducing the previous filling paste. F. the original surface after removing the previous filling paste.

4.4. Filling cracks and voids

Filling cracks and voids of this stick depended mainly on the gap-filling materials applied, which were required to be reversible, exhibit minimal shrinkage, easy to carve, strong yet weaker than the wood, whenever possible, exhibit good adhesive properties long-term flexibility, good light fastness, receptivity to in-painting or colouring, can be worked with a spatula and sanded (Unger, et al., 2001; Abdallah, 2009). In order to fill the gaps and voids between the bark layers and wooden support a fine putty consisting of 15% w/v Paraloid B72 in acetone, glass micro-balloons and earth pigments was applied, shaped and smoothed with a small spatula and swabs (Abdallah, 2014). In order to fill the large voids, cotton fibers were applied, injected with Paraloid B72 and then the same putty was applied, shaped and smoothed with a small spatula as final layer (Fig. 10).

Figure 10. The Stick after conservation

5. CONCLUSION

The studied ritual stick was severely damaged besides improper previous conservation, the analytical proved that the stick is made of almond wood (*Prunus dulcis* Mill) decorated by tree bark attached to wood substrate by black resin (suggested to be a mixture of *pistacia* resin and graphite besides residual of nitrogenous material may be hide glue) and coated finally with resinous material. The elemental analysis for metal parts revealed that an alloy of gold and silver have been used as a gold leaf for decorating the upper lotus shape upper part, as well as using a gold-based alloy with silver and copper for making the upper lotus shaped part and golden ring, a silver based alloy with copper and gold, and the last metal part at the lower end of the sick is made of a bronze alloy. Previous conservation filling material (a mixture of gypsum and hematite as a red pigment) was hiding the original surface, besides using threads of cotton fibres to tie the broken parts together. Procedure of conservation in wood lab included removing the improper previous conservation to clarify the original surface.

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