



Investigation methods for evaluating the preservative organic mixtures applied on a Late Period mummy

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Received: 18 September 2017 / Accepted: 28 March 2018 / Published online: 25 April 2018
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Abstract

The resins and other biochemical substances in mixtures have an important role in achieving the preservation goal for mummification process. The identification of organic mixtures is vital in the conservation field of the unwrapped mummies, but their identification is sometimes difficult because of improper conditions, which can affect their structure and need good investigation techniques for their identification. Some endogenous or exogenous factors can cause some aspects of deterioration to organic mixtures such as cracks, color change, and chemical alteration. This study aims to evaluate the materials used for the preservation of the body in order for them to be re-used in the near future for the preservation processes on the unwrapped mummies in Egyptian museums and storehouses. Samples of organic mixtures were taken from an ancient Egyptian mummy dated to the Late Period (664–333 B.C.). A digital USB microscope and a scanning electron microscope (SEM) were used to identify the application method of the preservative mixtures used during the mummification process and to demonstrate the morphology of surface alteration on the body. Fourier transform infrared (FTIR) was used to detect the chemical changes. A portable X-ray fluorescence (XRF) was used to identify elements. The results revealed that the investigation by microscopes showed cracks in the mixtures poured inside the cranial cavity, but the application of these mixtures was by brush on the outer surface layer of the body. FTIR analysis revealed that a mixture of oil, animal fat, and resinous materials such as mastic and pine were present in the studied samples. Moreover, these samples were prone to oxidation processes through time. XRF analysis showed that the main chemical elements of the samples were calcium, potassium, phosphorus, chlorine, sulfur, and silicon and trace chemical elements such as iron, zinc, copper, and lead, which are in the chemical composition of pine and mastic resins.

Keywords Mummy · Late Period · Organic mixture · Deterioration · Microscopes · FTIR · XRF

Introduction

The mummification process can be described as a very complicated process (Klȳs et al. 1999; Ben-Yehoshua and Ondřej Hanuř 2014) which aims to preserve deceased bodies according to the belief of the ancient Egyptians that would ensure their survival in eternity (Koller et al. 2005). The Ancient Egyptian Book of the Dead indicates the importance of preservation of the body “my body is everlasting, it will not perish and it will not decay for ages” process (Klȳs et al. 1999).

According to the Greek historian Herodotus, three main types of mummification were available at the same time (David 2001). The most important elements of mummification, which were crucial in arresting the decomposition of the body, were evisceration and dehydration of the tissues. Some authors have written about the ideal technique and most expensive method of mummification, which practiced during the new kingdom and involved many stages. In the second method, cedar oil was injected into the anus, which was plugged to prevent the escape of the liquid, and the body was then treated with natron. Once this was completed, the anal plug was removed and the liquefied stomach and intestines were drained out with the oil (Abdel-Maksoud and El-Amin 2011). In the third and cheapest method, the body was purged so that the intestines came away, and the body was then treated with natron (David 2001). The mummification process was performed using many different materials (Ben-Yehoshua and Ondřej Hanuř 2014; Koller et al. 2005) such as natron salt

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and cedar wood oil, in addition to the resinous materials, which may be described as preservatives (Koller et al. 2005; Facchetti et al. 2012). Resinous materials were commonly used in the mummification process, particularly in the later periods of Egyptian history (Aufderheide et al. 2004). Many different kinds of resins were used for the mummification process in ancient Egypt (Kłys et al. 1999) such as cedar turpentine (terebinth), which was identified through the resin samples taken from some mummies dated to the Ptolemaic period (Kavkler and Frelüh 2013). Mastic resin extracted from the genus *Pistacia lentisca* and *P. terebinthus* (Colombini et al. 2000) and coniferous resin such as cedar, pine, fir, juniper, and cypress were utilized (Zesch et al. 2016). Pine resin and the wood tars such as birch bark tars were used in the prehistoric period as adhesives (Vahur et al. 2011). Melted and liquefied resins were used for different purposes and procedures in the mummification process such as filling the cranial cavity (particularly in elite class mummies) (Kłys et al. 1999), smearing the internal and external parts of the body (Brettell et al. 2015), and adhering the mummy wrappings to ensure they were tightly held together (Kavkler and Frelüh 2013). Beeswax was used extensively in preserving corpses, especially in the later periods of Egyptian history (Zesch et al. 2016). Resins are also used to coat the body for isolating it from surrounding conditions (Colombini et al. 2000), to prevent rehydration of the already desiccated tissues (Jackowski et al. 2008); it was informed that resins consisting of mastic, dammar, and traces of pine resins are used for conservation of mummies in some museums (Carminati et al. 2014). Özen et al. (2016) used advanced chemical analyses of archaeological samples to identify fats, oils, beeswax, sugar gum, petroleum bitumen, coniferous, *Pistacia*, and cedar resins in mummification materials. The resin played an important role in preventing the decomposition of archaeological mummies due to its essential preventive role against some environmental factors such as humidity in the tombs (Buckley and Evershed 2001; Abd Elhay 2013; Khairat et al. 2013). Both the archaeological and modern samples of resins have been used to identify the unknown archaeological ones and to evaluate the extent of degradation within these samples (Vahur et al. 2011; McGovern and Hall 2016). It was not an easy task to identify the ancient resins with accuracy due to the chemical alteration to which these aged resins were subjected over time compared to the fresh ones (Kłys et al. 1999). Infrared spectroscopy analysis plays an important role in the accurate identification of the ancient natural resins (Kłys et al. 1999), oils, waxes, proteins, and inorganic additives (Vahur et al. 2011). Some researchers used Fourier transform infrared (FTIR) to study resins (Smith et al. 2012) to identify an unknown vitreous black coating material from an Egyptian mummy as resin material. Moreover, McGovern and Hall (2016) identified *Pistacia* species resin in five amphoras using FTIR analysis. Unfortunately, the identification of the individual compounds

in the mixtures was not possible using FTIR due to the complexity of the ancient mixtures (Vahur et al. 2011). Visual and microscopic examinations play an important role in studying some organic residues and the resinous materials included within the organic residues (Brettell et al. 2017). A USB digital microscope can provide an easy method for imaging the surface textures of artifacts and is commonly used for the examination of artifacts (Soy and Öhrström 2013). Portable X-ray fluorescence analysis is a non-destructive technique; it can easily provide detailed information on characteristic elements of an object (Anderson and Fregni 2009). The complex composition, natural variability, and susceptibility to oxidation of some resins such as pine resin can lead to difficulties in analysis (Derrick et al. 1999). The changes encountered by the resins, preparation, and degradation over time also lead to difficulties in their study (Izzo et al. 2013). In order to explain the deterioration mechanisms, especially the chemical changes of the ancient organic residue, some criteria should be taken into consideration. Archaeological, geological, and environmental factors are all significant in addition to physical and biological activity and chemical processes such as hydrolysis and oxidation (McGovern and Hall 2016). It has been shown that some resins are particularly vulnerable to deterioration by oxidation (Derrick et al. 1999). Furthermore, the social status of the deceased person plays an important role in the final quality of the mummification process in addition to changes to the actual mummification process utilized in different periods (Derrick et al. 1999). So, this study aims to identify the types of resins used, explain the morphological and chemical deterioration aspects and mechanisms within the archaeological samples, and identify the application method of these organic mixtures used by the embalmers during the mummification process.

Materials and methods

Sample description

Reference samples of resinous materials

In Fig. 1, myrrh, mastic, and frankincense with known composition were bought from the company of Abd El Rahman M. Harraz, Agricultural Seeds, Spices and Medicinal Plants Co., Cairo, Egypt. Myrrh is a natural oleo-gum-resin in reddish-brown rounded or irregular tears (Fig. 1a) which obtained from Somaliland and southern Arabia (Lucas 1937; Abdel-Maksoud and El-Amin 2011; Shen et al. 2012; Ben-Yehoshua and Ondřej Hanuš 2014; Nigatu 2014) and obtained from the stems of the genus *Commiphora*, particularly *C. myrrha* (Jahandideh et al. 2016). Mastic is a light yellowish and semi-transparent resin (Fig. 1b) in rounded or pear-shaped tears (Abdel-Maksoud and El-Amin 2011). Mastic resin is a

Fig. 1 The reference resinous samples: **a** myrrh, **b** mastic, and **c** frankincense



viscous light-green liquid that was extracted from the bark of *Pistacia lentiscus* var. *Chia*. Frankincense (Fig. 1c) is a gum-resin that is a light yellow or light yellowish-brown tears covered with their own white dust (Lucas 1937).

Archaeological samples

Two samples of organic mixture were taken from the remains of a priest mummy under investigation at the Anthropological Laboratory-Research and Conservation of Antiquities Center, Egypt. The samples were taken from the Sawa excavation during the excavation season of 1999 and dated to the Late Period. Sawa site is in Abou Hammad Center, Sharqiyah Governorate, Egypt. This site is located just 15 km north-east of Zagazig city and about 1 km south of Saft El-henna area, and its geographical coordinates are 31°, 37° East and 30°, 33° North. One sample was taken from the cranial cavity (Fig. 2a, b) and the other from the coating over the body (Fig. 2c). The samples are of great importance because they are from a priest from Lower Egypt, but there is little information about him, and some information can be obtained from him by archaeologists in the near future.

Analytical techniques

Portable digital light microscope (USB microscope)

A portable USB digital microscope (model PZ01, made by Shenzhen Supereyes Co., Ltd., China) with the following technical specification: image sensor 0.3 megapixels, magnification factor 10–500 times, photo capture resolution 640 × 480, 320 × 240, and LED illumination light resource adjustable by control wheel was used to investigate the surface of the archaeological samples. The samples were investigated under relatively low magnifications (10–50×).

Scanning electron microscope

A scanning electron microscope (SEM) FEI Model Quanta 200 with EDAX unit was used to examine the surface morphology of the archaeological samples. This microscope is at the scanning electron microscope lab, Research and Conservation of Antiquities Center, Supreme Council of Antiquities, Egypt.

Fourier transform infrared spectroscopy analysis

Fourier transform infrared (FTIR 6100 type A, Jasco, Japan) was used for analysis at the Spectroscopic Lab., National Research Center (NRC), Egypt. The measurement range was 4000–400 cm^{-1} . FTIR analysis was carried out according to specifications described by Abdel-Maksoud and Abdel-Hady (2011). A few milligrams of reference and archaeological samples was ground up; then, each sample was mixed with KBr and placed in a DRIFT cell.

Elemental analysis by a portable X-ray fluorescence

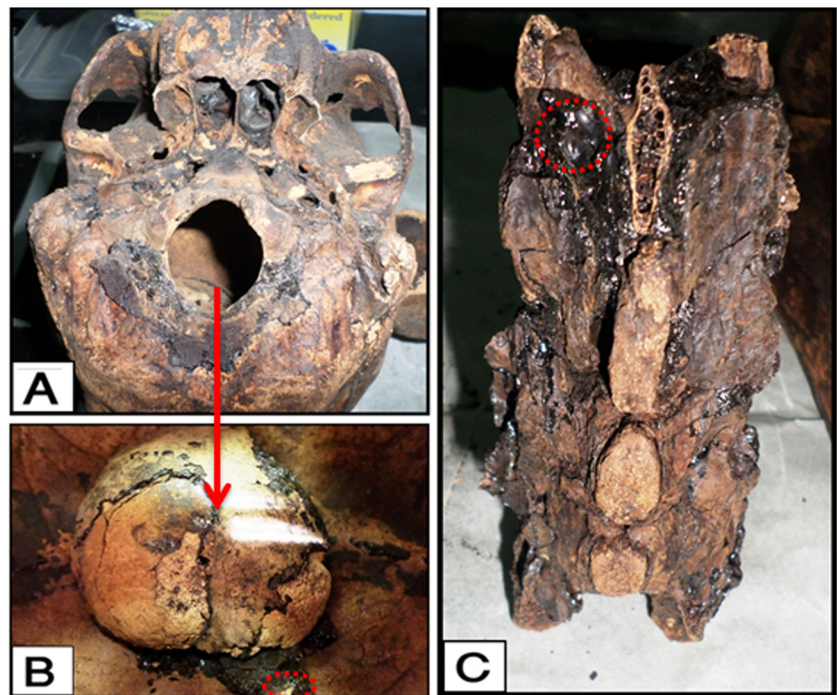
A portable X-ray fluorescence (XRF) at the Geology Department, Faculty of Science, Cairo University, Egypt, was used to analyze the elements in the reference and archaeological samples. The technical data of this tool is device S/N: 760073 (Oxford Instruments).

Results and discussion

Portable digital light microscope (USB microscope)

The microscope, especially the digital microscope, is considered as one of the most important tools available to the conservator and its images assist in the evaluation of the damage and deterioration aspects of artifacts (Anderson and Fregni 2009). Therefore, the use of microscopes may provide some details concerning the application method of the resins to the cranial cavity and over the external body of the studied mummy. From the obtained images, it was found that the sample taken from the cranial cavity had a very thin layer over the preserved organic material, which is understood to be the remains of the brain. Cracks within the thin layer were noticed (Fig. 3a). In addition to that, the preservative material beneath appeared to be shiny and smooth and with a uniform surface (Fig. 3b). This probably indicates that the application of these resinous materials was done by pouring of these melting liquid resins inside the cranial cavity by the embalmers. The pouring of molten resin inside cranial cavity after extraction of intracranial contents aimed to preserve cranial cavity from microbial deterioration due to their antimicrobial properties, and the melting of this resin occurred at high temperature which had

Fig. 2 The archaeological samples taken from remains of the priest mummy. **a** The skull base, **b** the remains of brain with black mixture in the cranial cavity and the place of taken sample (dash outline), and **c** sample from backbones and the taken sample from the dark shiny coating on the external body (dash outlines)



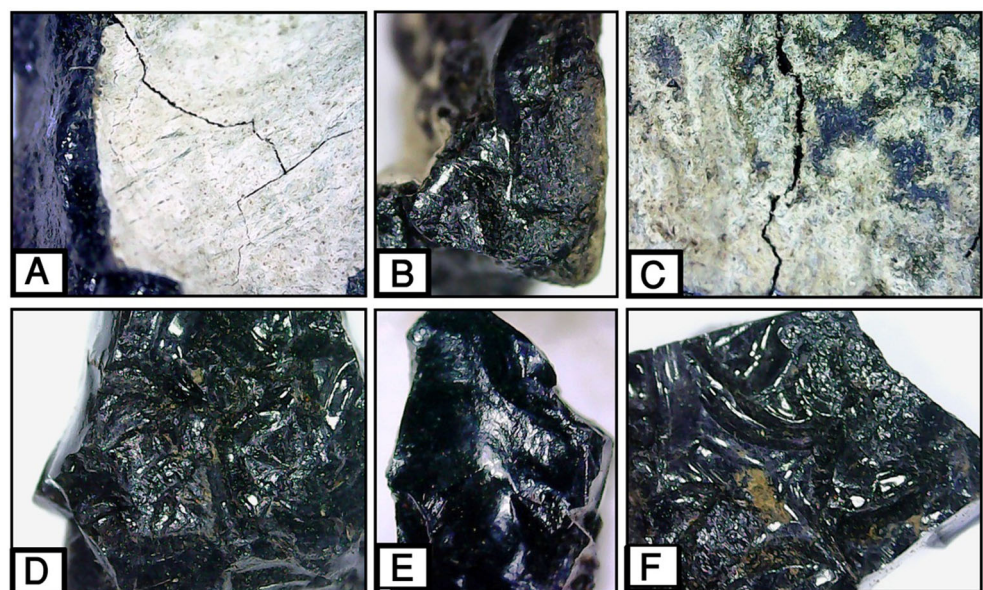
the ability to kill the microbes, especially in the case of presence brain remains. The application of the thick resinous coatings was aimed to prevent decomposition (McKnight et al. 2015). Additionally, filling of cranial cavity with resins protects the skull from decaying after extraction of the brain. It can also lead to the dryness of the brain remains and give the normal appearance for the skull and protect it from being broken. This method was observed by Boyer et al. (2003) and confirms our interpretation that the melted resin was poured into the cranial cavity after the extraction of the intra-cranial contents (brain). Moreover, a deep crack in the

lower part of the sample was observed (Fig. 3c). Regarding the other sample from the external body, the surface of the upper and lower sides of this sample was irregular in shape (Fig. 3d, f). It appeared shiny in appearance (Fig. 3e). The irregular surface may be due to the application of liquid resin by brush, which produces a non-homogeneous surface.

Scanning electron microscope

Scanning electron micrographs (Fig. 4) showed that the archaeological samples suffered from deterioration processes.

Fig. 3 **a–c** Digital microscope images: **a** cracks with the upper part of sample, **b** lateral view of the sample shows the shiny appearance, and **c** the big cracks with lower part of the sample above the skull wall. **d–f** Images of the sample from the external body: **d** the irregular appearance of the upper part of the sample, **e** shiny appearance with the lateral view of the preservative mixture, and **f** the lower part of the sample shows the brush marks used in the application of melting resins



Micro-cracks were noticed on the thin layer covering the organic mixture within the cranial cavity, which is presumed to be remains of the brain (Fig. 4a, b). Cracks were also observed on the surface within the sample collected from the exterior of the body (Fig. 4d, e). The results obtained by our analysis were consistent with those obtained by Abdel-Maksoud and El-Amin (2013), who noticed some cracks within the resinous layer on a hair sample from a gazelle mummy using SEM analysis. Moreover, SEM helped to detect some signs of the application methods of these organic mixtures used by the embalmers on this mummy. For example, some holes were shown within the sample from the cranial cavity (Fig. 4c) which are probably due to air bubbles occurring during pouring of the liquid resin into the cranial cavity. Abdel-Maksoud and El-Amin (2011) confirmed this hypothesis when they stated that the cranial cavity was filled with the melted resin after the brain was extracted. Furthermore, some brush markings were observed over the whole surface (Fig. 4e, f) of the sample from the external body. Studies carried out at the Penn Museum also confirmed this observation that melted pine resin was applied to the body with a brush. Moreover, brushes (Fig. 5) were found as one of the mummification tools at the Mummification Museum, Luxor, Egypt, confirming our observations.

Fourier transform infrared analysis

The Fourier transform infrared (FTIR) spectrum of the archaeological samples as shown in (Fig. 6) detected a number of characteristic spectral features. It was found that the samples contained oil, which was clearly seen through the hydrogen-bonded (O–H) stretching at 3413.4 and 3429.8 cm^{-1} and the stretching of aliphatic group C–H stretching at the region 2863.8–2932.2 cm^{-1} . C–H deformation of the alkaline group at the region 1457–1376 cm^{-1} in the two samples respectively and the characteristic band at 725.1 cm^{-1} in the sample from the cranial cavity were observed. This result was confirmed by Vahur et al. (2011) who stated that the C–H stretching at 2919–2849 cm^{-1} and C–H deformation of the alkaline group at the region 1456–1374 cm^{-1} indicated the high content of the compounds with long un-branched alkyl chains in the archaeological mixture sample, and finally, the band at 720–727 cm^{-1} was characteristic of compounds containing long aliphatic chains in oils. Vahur et al. (2011) revealed that the broad OH stretch band at 3000–3600 cm^{-1} from the archaeological adhesive sample, which appeared due to the phenolic compounds, alcohols, and carboxylic acids resulted from some deterioration processes such as partial hydrolysis or oxidation of the oils during the aging over time (Vahur et al. 2011). Moreover, intense carbonyl C=O stretching bands appeared at 1709.6–1710.5 cm^{-1} in the A and B samples respectively, which referred to the presence of mastic resin in the archaeological samples. The results complied with

Bruni and Guglielmi (2014) who found that the carbonyl C=O stretching bands always exceeded 1700 cm^{-1} in the case of triterpenic resins such as mastic. They also identified that the characteristic band at 1704 cm^{-1} is due to the carboxyl group in the spectra of mastic resin. This result was consistent with the characteristic band of our reference mastic resin, which appeared at 1707.66 cm^{-1} . The strongest bands in the spectra of mastic resin were the carbonyl and hydrocarbon stretching frequencies (Derrick et al. 1999). The C–H stretching at 2945.73 and 2870.52 cm^{-1} was observed in the spectra of our reference mastic resin which is consistent with the bands at 2863.8 and 2870.5 cm^{-1} in the archaeological samples. This result is consistent with the result of spectra of mastic, especially with strong C–H stretching vibrations at 2958–2930 and 2875–2865 cm^{-1} (Derrick et al. 1999). In addition, some characteristic bands of pine resin were observed in the studied sample, especially the intense carbonyl C=O stretching bands which appeared at 1709.6–1710.5 cm^{-1} in the samples. This probably referred to the presence of pine resin in the archaeological samples. The carboxylic acids (1730–1700 cm^{-1}) and ketones (1720–1690 cm^{-1}) have C=O (carbonyl) stretch bands (Vahur et al. 2011). This result was confirmed by experiments carried out by Zareva and Kuleff (2010) who did a comparative study between the archaeological resin samples from the ancient amphora and modern pine resin, and they found that there are similarities in the IR-spectroscopic characteristics within 1750–1600 cm^{-1} especially at 1707 cm^{-1} . This referred to the presence of pine resin in their archaeological samples and complied with the carbonyl bands of the samples. Based upon the previous results, it can be said that pine resin was identified within the components of the preservative mixtures. The coniferous resins, especially pine, were identified by FTIR analysis with many of archaeological samples such as those of Kłys et al. (1999) who identified this resin in the mummy of the priestess Iset-Iri-Hetes (Archeological Museum in Krakow, Poland) dated between the third and the first century B.C. The resins used in ancient Egypt were obtained from coniferous trees such as cedar, pine, fir, juniper, and cypress (Kłys et al. 1999; Abdel-Maksoud and El-Amin 2011). The characteristic aliphatic C–H stretch bands of the animal fats at 2929.3–2932.2 and 2863.8–2870.5 cm^{-1} were observed in the studied samples. This result was consistent with results obtained by Vahur et al. (2011) who discussed that the C–H stretching bands at 2920 and 2850 cm^{-1} are better separated in the spectra of the archaeological adhesive sample. This reflects the presence of the fat in the archaeological samples because this separation indicates a higher content of compounds with long un-branched alkyl chains. Moreover, Brettell et al. (2017) stated that the C–H band at 2865 cm^{-1} referred to the presence of the animal fat in their archaeological samples. From the obtained data, it was found that there are simple differences between the spectra of the

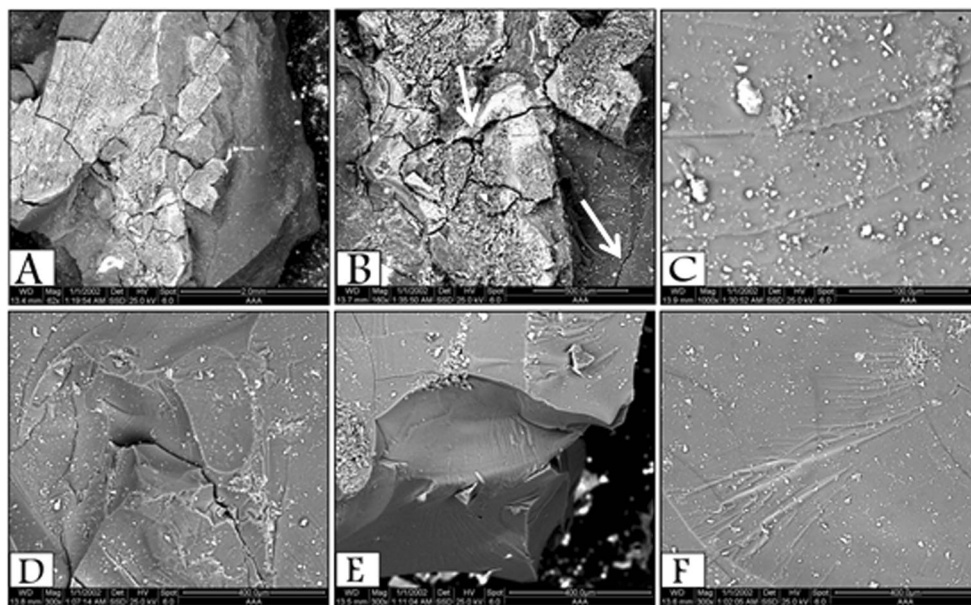


Fig. 4 SEM images of the archaeological samples showing the deterioration aspects and the indicators of application method of the melting organic mixtures: **a–c** the images of the sample from cranial cavity. **a, b** Cracks within the thin layer of brain remains and with the structure of the organic mixture (white arrow) and **c** holes within the

structure of the mixture. **d–f** The images of the sample from external body: **d** big cracks within the structure of the mixture, **e** side image of the sample shows small cracks on the surface and some lines within the structure, and **f** the indicators of the use of brush on the surface during application of the melting mixture by embalmers

archaeological samples from the cranial cavity and from the external body but the main features were the same, most likely as a result of the oxidation processes which had affected the ancient sample of the external body. This result was also consistent with Rousis et al. (2014) who indicated that the differences with the intensity (within 1750–1600- and 1380–900- cm^{-1} regions) between the internal and external archaeological resin samples from the ancient amphora was due to the oxidation processes that had affected the external archaeological sample. The natural resins were characterized with degradation resistance properties to water solubility due to their water-insoluble fraction of their composition (Brettell et al. 2015). In addition, it was clarified that the bands at 3413.4 and 3429.8 cm^{-1} in the archaeological samples A and B respectively may be assigned to hydrogen-bonded (O–H) stretching. However, in the mastic resin control sample, this band shifted to a higher position (3444.24 cm^{-1}) probably due to the oxidation processes affecting the archaeological samples. This result is consistent with results obtained by Vahur et al. (2011) who stated that the aging process led to some

differences in FTIR spectra between the archaeological adhesive and modern tar samples. In particular, the very weak broad band in the region of 3600–3000 cm^{-1} and very weak band in the region 1650–1540 cm^{-1} in the reference sample; these resulted from carboxylic acids which formed over time within the archaeological samples due to deterioration processes.

Elemental analysis by the portable X-ray fluorescence

The data obtained from the portable X-ray fluorescence of the archaeological samples analyzed (Fig. 7) detected sulfur (S 34.51, 52.92%), calcium (Ca 22.38, 3.96%), chlorine (Cl 10.51, 17.61%), silicon (Si 5.86, 8.02%), iron (Fe 2.84, 0.54%), potassium (K 0.92, 0%), titanium (Ti 0.26, 0%), phosphorus (P 0.38, 0.90%), nickel (Ni 0, 0.09%), copper



Fig. 5 The brush found in the Mummification Museum, Luxor, Egypt

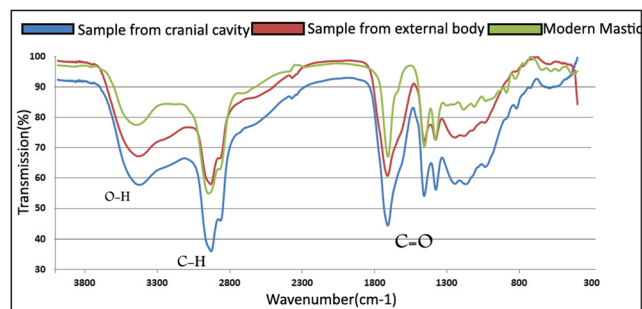


Fig. 6 FTIR spectra of the archaeological samples and reference mastic resin show that the carbonyl bands are situated at 1700 cm^{-1}

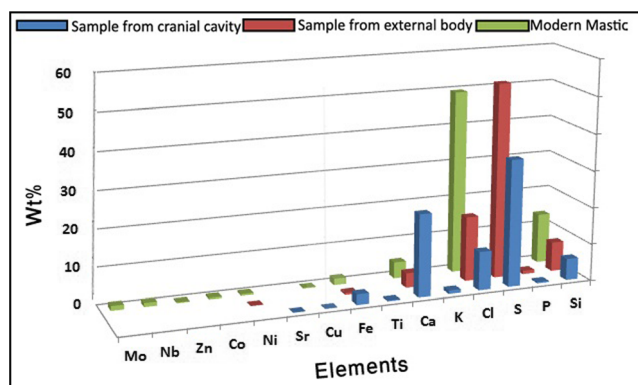


Fig. 7 The elements found by XRF in the archaeological and reference samples

(Cu 0.08, 0%), and strontium (Sr 0.05, 0%) in the samples from the cranial cavity and the external body respectively. These elements referred to the presence of pine and mastic resins in the archaeological mixture samples. The results obtained agree with the previously published work by Klys et al. (1999) who identified the main chemical elements Ca, Mg, Na, K, and P with 2.20, 0.309, 2.37, 0.868 mg/g and trace elements Fe, Zn, Cu, Pb, and Mn with 174, 24.7, 37.9, 39.5, and 7.06 $\mu\text{g/g}$ respectively in addition to Si, S, and Al from the resin sample which was taken from the Iset-Iri-Hetes mummy skull and proved that these elements referred to the pine resin. The elemental analysis of the reference mastic resin (Fig. 7) revealed also that this resin contains Cl, 49.60%; Si, 13.61%; Ca, 4.43%; Fe, 1.75%; S, 1.71%; Mo, 1.27%; Nb, 1.07%; Co, 0.49%; Ni, 0.47%; Zn, 0.24%; and Cu, 0.22%. The results of mastic resin analysis were confirmed by Rousis et al. (2014) who determined these elements as Mg, Al, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Sr, Nb, MO, Ag, Sb, Cs, and Ba in the mastic resin by using inductively coupled plasma mass spectrometry (ICP-MS). The determination of the light elements by some models of a portable XRF was difficult; however, it was useful for analyzing the heavy elements (Anderson and Fregni 2009). Mahmoudi et al. (2010) identified these elements as Cu, Fe, Zn, Mn, Ni, and Cd (27.3, 12, 12, 7, 1, and 1 $\mu\text{g/g}$) respectively in the ash of *P. lentiscus* resin by atomic absorption spectroscopy (AAS) analysis. It should also be noted that this resin contained a high ratio of Cu, Fe, and Zn elements.

Conclusion

The results obtained from the analyzed samples from the Late Period ancient Egyptian mummy give important information on the chemical composition of the materials used in ancient recipes, as well as on their state of preservation. Microscopic investigation revealed some cracks in the sample from the cranial cavity, which may be due to environmental conditions

especially fluctuations between temperature and humidity. It also indicates that pouring and brushing methods were used for the application of liquefied resin. The FTIR analysis show that our archaeological samples consisted of a complex mixture containing degraded oil (from O–H stretching), animal fat (from C–H stretching at 2919–2849 cm^{-1}), and resinous materials such as pine resin (from carbonyl C=O stretch bands at 1709.6–1710.5 cm^{-1}) and mastic resin. Comparison of the IR spectra of mastic resin between the control sample and the archaeological samples revealed there are some differences between the spectra of the reference mastic resin and those of the archaeological samples. This may be due to the aging process over time. XRF analysis revealed the presence of some chemical elements of mastic and pine resin in our archaeological samples such as S, Ca, Si, Fe, K, P, and Ti but the determination of the percentage of each material is very difficult.

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