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ARCHAEOMETRIC STUDY OF BLACK RESIN OF A LATE PERIOD COFFIN BY GAS CHROMATOGRAPHY-MASS SPECTROMETRY

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Abstract

This paper aims to identify black resin's composition, beginning, and uses. Black resin was a resinous material used to cover the funerary wooden artifacts in the New Kingdom. It was used for religious purposes and for covering coffins, shabti statues and boxes, stelae, human and animal statues, statue bases, and canopic chests. First, black resin was used in the era of Hatshepsut. Then, its use expanded in the era of Thutmose III. Therefore, the New Kingdom was the golden era of its use. In the New Kingdom, black resin was glossier than later times. The study utilized gas chromatography-mass spectrometry (GC-MS) attached with Thermal Separation Probe (TSP) unit was used to analyze the black resin sample. Black resin is made from natural resins, such as mastic, colophony, beeswax, bitumen, and unknown compounds. The layer of black resin has, in its complexes, a group of natural resins and other substances whose properties are anti-fungal and antibacterial. It also has insect repelling properties. Natural resins contain essential oil. Mastic resin was of high value in Ancient Egypt. The sample was taken from a coffin dating back to the Late period, in the 26th dynasty to be analyzed because identifying its composition helps choose the best material for chemical cleaning and consolidation. The coffin under investigation was covered externally with a ground layer and a painted layer, while internally it was covered with a layer of black resin.

Keywords: *Black resin; Funerary furniture; Wooden coffin; GC-MS; Thermal separation probe; Mastic; Pine; Bitumen; Beeswax*

Introduction

The Ancient Egyptians used black pigment profusely in funerary furniture from the 11th and 12th dynasties (2000-1785 B.C) onward, as shown by coffins no. CG 28030 and CG 28028 from the Middle Kingdom kept in the Egyptian Museum. Those coffins were used for theoretical study only to prove the black pigment was used before using black resin.

The New Kingdom, from the 18th to the 20th dynasties, represent the peak of the Egyptian state and its prosperity, the funerary furniture became better than before.

Black resin was used in the New Kingdom. Firstly, it was used in the era of Hatshepsut and expanded in the era of Thutmose III. Therefore, the New Kingdom was the golden era of its use. It was black when applied and not blackened over time.

A visit to the Egyptian Museum showed that most of the wooden artifacts were covered with a layer of black resin. It was observed that black resin, in the beginning, was bright black with a thick layer. However, at the end of the 18th dynasty, the layer became less in thickness and glossy.

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In this study, GC-MS is used in order to identify the organic substances and oils, the compounds in complex materials such as resins and other materials which can help determine the classification of these resins, by applying the appropriate separation (chromatographic) and identification (mass spectrometric) techniques to know the composition of the black resin and chose the best material to conservate it

According to Serpico and White [1], black resin contained unheated Pinaceae, unheated Pistacia resin, beeswax, vegetable oil or animal fats, and traces.

A sample of black resin from the Late Period's coffin from Saqqara area was analyzed and compared with the results on the database. It was found that the sample contained Pinaceae, Pistacia, beeswax, bitumen, and unknown compounds. It did not contain vegetable oil or animal fats because the essential oil was found on natural resin, and fatty acid was found on beeswax.

The sample was analyzed in order to identify the properties of its material because many substances, such as alcohol and acetone used in chemical cleaning dissolve the black resin layer and cause fading.

Materials and methods

GC-MS

The sample was taken from the falling parts of a black resin layer (Fig.1) dating back to the Late Period at Saqqara area.

Different materials can be used to make derivatization of the black resin layer and because it comprised many organic materials so when we used a solvent it can dissolved one material on its compositions. Therefore, we had to use a method without any preparation of the samples to compare this result with the new natural samples and to make a database about this work (TSP) Thermal Separation Probe unit was used to analyze the sample. It does not need any sample preparation low cost, small sample size and fast analysis of solid materials.

Fig. 1. It shows the black resin layer of the coffin from inside and the location of the sample that was analyzed: (A) The lid of the coffin; (B) The base of the coffin

Chromatographic analysis using GC–MS was performed (Agilent Technologies 7890B GC Systems combined with 5977A Mass Selective Detector). Capillary column was used (HP-5MS Capillary; 30.0m × 0.25mm ID × 0.25μm film), and the carrier gas was helium at a flow rate of 1.8mL/min with 1μL injection. The sample was analyzed with the column held initially for 3min at 40°C after injection. Then, the temperature was increased to 300°C with a 20°C/min heating ramp and a 2.0min hold. The injection was carried out in the split mode at 300°C with (10:1) ratio. MS scan range was (m/z): 50–600 atomic mass units (AMU) under electron impact (EI) ionization (70eV).

The constituents were determined by mass fragmentations with the NIST mass spectral search program for the NIST/EPA/NIH mass spectral library Version 2.2 (Jun 2014).

Results and discussion

GC-MS

Black resin is a composite material that contains many natural substances. Thus, it has some advantages, such as the property of rapid drying. The materials included in the composition of some anti-fungi, bacteria, and insect properties show their castration during the discussion of each substance separately. Figure 2 shows the Total Ion Chromatograms (TIC) of the sample analyzed, which indicates that black resin consists of some materials that have been identified with their distinctive properties.

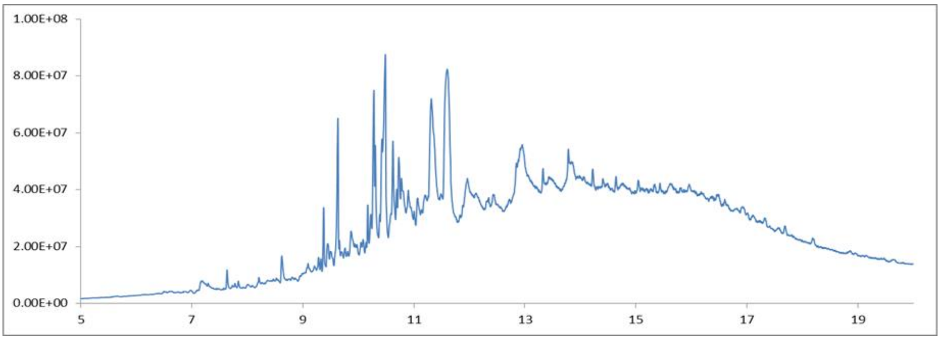


Fig. 2. Total ion chromatograms (TIC) demonstrating quantitative differences in the components of black resin from the coffin at Saqqara area

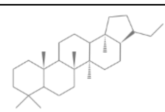
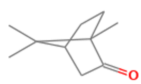
It was found that black resin consisted of Pistacia, Pinaceae, beeswax, bitumen, and unknown compounds.

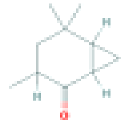
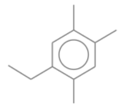
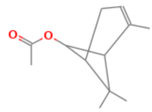
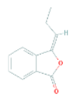
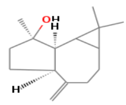
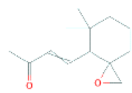
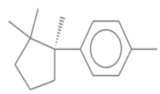
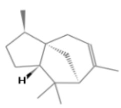
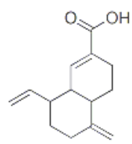
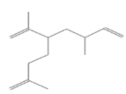
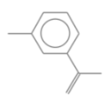
The properties and source of the materials

1- Pistacia

Mastic resin is classified under the triterpenoid [2, 3] and it can be obtained from Pistacia lentiscus [4-6].

Table 1. The compounds identified as Pistacia lentiscus (Mastic)

RT	Area Pct	Compound Name	Chemical structure
7.1908	0.2249	Benzene, 4-ethenyl-1,2-dimethyl-	
7.637	0.1161	(+)-2-Bornanone	

RT	Area Pct	Compound Name	Chemical structure
8.9964	0.1364	Bicyclo[4.1.0]heptan-2-one, 3,5,5-trimethyl-	
9.3285	0.1379	1,2,4-Metheno-1H-indene, octahydro-1,7a-dimethyl-5-(1-methylethyl)-, [1S-(1.alpha.,2.alpha.,3a.beta.,4.alpha.,5.alpha.,7a.beta.,8S*)]-	
9.7644	0.3094	Benzene, 1-ethyl-2,4,5-trimethyl-	
9.8111	0.2303	Bicyclo[3.1.1]hept-2-en-6-ol, 2,7,7-trimethyl-, acetate, [1S-(1.alpha.,5.alpha.,6.beta.)]-	
9.9408	0.2274	1(3H)-Isobenzofuranone, 3-propylidene-	
10.0498	0.3488	(-)-Spathulenol	
10.0861	0.3601	3-buten-2-one, 4-(5,5-dimethyl-1-oxaspiro[2.5]oct-4-yl)	
10.2832	1.3943	Benzene, 1-methyl-4-(1,2,2-trimethylcyclopentyl)-, (R)-	
10.7814	1.7237	1H-3a,7-Methanoazulene, 2,3,4,7,8,8a-hexahydro-3,6,8,8-tetramethyl-, [3R-(3.alpha.,3a.beta.,7.beta.,8a.alpha.)]-	
11.0045	0.5581	2-Naphthalenecarboxylic acid, 8-ethenyl-3,4,4a,5,6,7,8,8a-octahydro-5-methylene-	
11.2016	1.2957	1,8-Nonadiene, 2,7-dimethyl-5-(1-methylethenyl)-	
11.6012	6.5761	Benzene, 1-methyl-3-(1-methylethenyl)-	

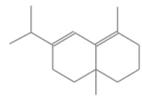
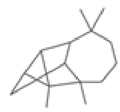
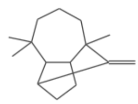
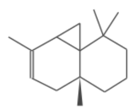
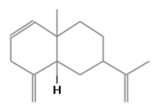
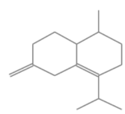
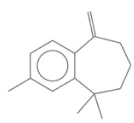
A new mastic resin sample was analyzed by GC-MS at the same condition and was compared with an archaeological sample (Table 1). The table shows that Benzene, 1-methyl-4-(1,2,2-trimethylcyclopentyl)-, (R)- (1.3943%),1H-3a,7-Methanoazulene, 2,3,4,7,8,8a-hexahydro-3,6,8,8-tetramethyl-, [3R-(3.alpha.,3a.beta.,7.beta.,8a.alpha.)]- (1.7237%), 1,8-Nonadiene, 2,7-dimethyl-5-(1-methylethenyl)- (1.2957%), Benzene, and 1-methyl-3-(1-methylethenyl)-(6.5761%) have some antifungal and antibacterial properties.

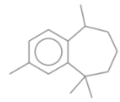
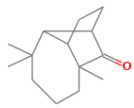
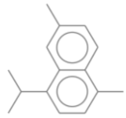
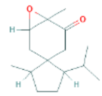
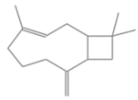
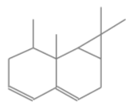
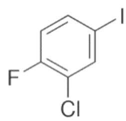
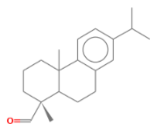
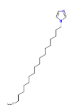
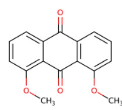
Mastic resin has a pale-yellow color [7]. It is used for some medical purposes [8] and antimicrobial agents such as Gram-positive bacteria and gram-negative bacteria. Additionally, it is used as antibacterial agent [9] and insect repellent [10]. Natural resins are non-water-soluble exudates from a wide variety of trees. Pistacia lentiscus resin comes from a small tree that grows on Chios: A smaller shrub-like that grows along the Mediterranean coasts [11-13].

2-Pinaceae resin (Colophony)

Pinaceae resin it was found in the composition of black resin (Table 2), also called Colophony [14] and rosin [15]. It was used in the ancient times [16] and an increased amount of pine was imported during the Roman Empire [17]. Currently, colophony is used in modern varnishes [18] because it becomes soft at 70-80°C [19].

Table 2. The compounds identified as Pinaceae

RT	Area Pct	Name	Chemical structure
9.287	0.228	.delta.-Selinene	
9.5049	0.4166	1,2,4-Metheno-1H-indene, octahydro-1,7a-dimethyl-5-(1-methylethyl)-, [1S-(1.alpha.,2.alpha.,3a.beta.,4.alpha.,5.alpha.,7a.beta.,8S*)]-	
9.6347	1.4776	Longifolene	
9.7073	0.3009	cis-Thujopsene	
9.9771	0.3506	(3R,4aS,8aS)-8a-Methyl-5-methylene-3-(prop-1-en-2-yl)-1,2,3,4,4a,5,6,8a-octahydronaphthalene	
10.2262	0.5504	Bicyclo[4.4.0]dec-1-ene, 2-isopropyl-5-methyl-9-methylene-	
10.3092	1.0519	.alpha.-Dehydro-ar-himachalene	

RT	Area Pct	Name	Chemical structure
10.4856	4.1459	aR-Himachalene	
10.6931	0.5717	(+)-Longicamphenylone	
10.7347	1.1202	Naphthalene, 1,6-dimethyl-4-(1-methylethyl)-	
10.9007	1.8811	Spiro[4.5]decan-7-one, 1,8-dimethyl-8,9-epoxy-4-isopropyl-	
11.1394	0.5124	4,11,11-trimethyl-8-methylenebicyclo[7.2.0]undec-3-ene	
11.321	5.5121	1,1,7,7a-Tetramethyl-1a,2,6,7,7a,7b-hexahydro-1H-cyclopropa[a]naphthalene	
13.0592	2.5522	3-Chloro-4-fluoroiodobenzene	
13.9464	0.7834	1-Phenanthrenecarboxaldehyde, 1,2,3,4,4a,9,10,10a-octahydro-1,4a-dimethyl-7-(1-methylethyl)-, [1R-(1.alpha.,4a.beta.,10a.alpha.)]-	
15.4355	1.2794	1H-Imidazole, 1-octadecyl-	
15.6275	2.9739	9,10-Anthracenedione, 1,8-dimethoxy-	

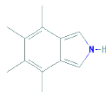
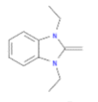
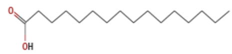
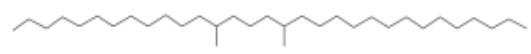
A pinaceae resin sample was analyzed. It showed the common compounds with the archaeological sample. The main compounds are Longifolene (1.4776%), alpha.-Dehydro-ar-himachalene (1.0519%), aR-Himachalene (4.1459%) and Spiro[4.5]decan-7-one, 1,8-dimethyl-8,9-epoxy-4-isopropyl- (1.8811%) were found in the essential oil.

1,1,7,7a-Tetramethyl-1a,2,6,7,7a,7b-hexahydro-1H-cyclopropa[a]naphthalene(5.5121%), 3-Chloro-4-fluoroiodobenzene (2.5522%), 1-Phenanthrenecarboxaldehyde, 1,2,3,4,4a,9,10,10a-octahydro-1,4a-dimethyl-7-(1-methylethyl)-, [1R-(1.alpha.,4a.beta.,10a.alpha.)]- (0.7834) had antifungal and bacterial properties. 9,10-Anthracenedione and 1,8-dimethoxy- (2.9739%) were found.

3-Beeswax

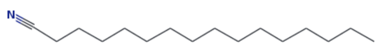
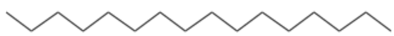
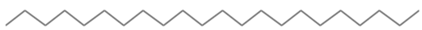
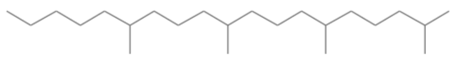
Beeswax (Table 3) it has been extensively used since ancient times [20], as a media for coloring during the Greco-Roman times [21]. In addition, it was used for medical purposes [22]. Its melting point is 64.5°C [23]. The degree of melting is useful in preparing the samples of the black resin for comparison with the original.

Table 3. The compounds identified as beeswax

RT	Area Pct	Name	Chemical structure
9.453	0.3958	Isolongifolene, 9,10-dehydro-	
10.6257	2.0329	2H-Isoindole, 4,5,6,7-tetramethyl-	
11.9644	3.9215	1H-benzimidazole, 1,3-diethyl-2,3-dihydro-2-methylene-	
12.9554	4.2373	n-Hexadecanoic acid	
15.804	0.8444	13,17-Dimethylhentriacontane	

The main constituents identified in the beeswax are Isolongifolene, 9,10-dehydro-(0.3958) Propolis anti-fungi and bacteria (Table 3), 2H-Isoindole, 4,5,6,7-tetramethyl-(2.0329%), 1H-benzimidazole, as well as 1,3-diethyl-2,3-dihydro-2-methylene-(3.9215%) found in honey, suggesting that the ancient Egyptions used beeswax from nature. N-Hexadecanoic acid (4.2373), and 13,17-Dimethylhentriacontane (0.8444%) were found. Some of the properties of these compounds are antifungal and antibacterial.

Table 4. The most common compounds identified for beeswax and bitumen

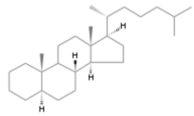
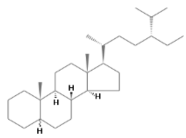
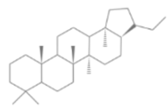
RT	Area Pct	Name	Chemical structure
12.4365	3.1768	Hexadecanenitrile	
13.329	1.9096	Hexadecane	
13.7856	3.2844	Docosane	
14.2266	1.3125	Nonadecane, 2,6,10,14-tetramethyl-	

There are similar compounds on the beeswax and bitumen (Table. 4). Hexadecanenitrile (3.1768%), Hexadecane (1.9096%), Docosane (3.2844%), as well as Nonadecane, 2,6,10,14-tetramethyl-(1.3125) have antifungal and bacterial properties.

4-Bitumen

Bitumen was found in the composition of black resin (Table 5). Bitumen is also called Asphalt [24]. It was used for different purposes over time [25]. There are many sources of bitumen [26], including the Dead Sea [27] in Palestine, other sources closer to bitumen in abodorba, and mountain oil on the banks of the Gulf of Suez in Egypt [28]. It becomes viscous at 50-60°C and resembles thick honey. At 90-100°C, it flows freely. This facilitates its use in covering mummies to protect them from microorganisms and wooden coffins [29].

Table 5. The compounds identified as bitumen

RT	Area Pct	Name	Chemical structure
14.7714	3.5717	2-Dodecen-1-yl(-)succinic anhydride	
15.0464	1.0322	1-Chloroeicosane	
16.494	3.1872	Cholestane	
17.319	0.3509	Stigmastane	
18.1855	0.0806	28-Nor-17.alpha.(H)-hopane	

The main constituents identified in the Bitumen were 2-Dodecen-1-yl(-)succinic anhydride (3.5717%), 1-Chloroeicosane (1.0322%), and Cholestane(3.1872%) that were found in crude oil. Crude oil and bitumen were found in the nature together. Their properties are antifungal, antibacterial, and insect repelling.

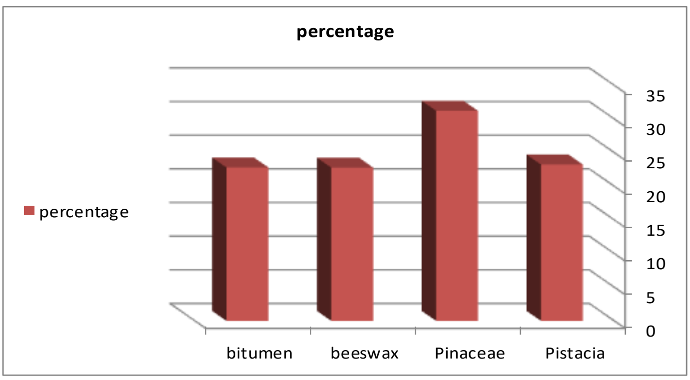


Fig. 3. The percentage of the compounds found on the black resin layer

From the previous analysis of the black resin sample, we know several materials in the black resin layer (Fig. 3). The black resin composition can be compared from this dynasty with another dynasty and another era. It can help prepare a new black resin layer to be used in experimental studies to choose the best materials for chemical cleaning and consolidation.

Conclusion

Black resin was used for the first time in the New Kingdom for religious purposes to cover the funerary wooden artifacts in the New Kingdom. A sample was taken from a coffin dating back to the Late Period to analyze its composition and to choose the best material for chemical cleaning and consolidation. Knowing the black resin components can help choose the best solvent for chemical cleaning without melting and fading the black resin and the suitable materials for consolidation. The coffin under investigation was covered with a layer of black resin from inside.

Thermal Separation Probe unit was used to analyze the sample. GC-MS revealed that it consists of mastic, colophony, beeswax, bitumen, and unknown compounds that might be impurities from natural resins or from other materials. The Ancient Egyptians knew bitumen during the New Kingdom from natural sources and it was found in crude oil. Therefore, crude oil was found in the sample.

It was discovered that the essential oil was found in natural resin and has anti-fungal and bacterial properties. The Ancient Egyptians used a soft brush to cover the coffin and funerary furniture. In addition, it was drying very fast, so it was applied while it was warm. Finally, it was found that the black resin had some anti-fungal and anti-bacterial compounds.

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