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Beata Miazga[1], Sylwia Rodak[2],
Jeannette Jacqueline Lucejko[3], Erika Ribechini[4]

A UNIQUE EARLY MEDIEVAL PENDANT (*KAPTORGA*) FROM OPOLE GROSZOWICE (SILESIA, SW POLAND) IN THE LIGHT OF INTERDISCIPLINARY ARCHAEOMETRIC STUDIES

ABSTRACT

Miazga B., Rodak S., Lucejko J. J., Ribechini E. 2020. A unique early medieval pendant (*kaptorga*) from Opole Groszowice (Silesia, SW Poland) in the light of interdisciplinary archaeometric studies. *Sprawozdania Archeologiczne* 72/2, 539-554.

Finds of early medieval pendants, known as kaptorgas, are not common in Poland. For this reason, the *kaptorga* found in 1957 in Opole (Silesia), in southwest Poland, is all the more interesting. The artefact is housed in a museum, and on the occasion of its re-conservation, permission to conduct archaeometric studies was given. The kaptorga was subjected to analyses using nondestructive and minimally invasive techniques. Elemental tests with energy dispersive XRF and SEM-EDS spectrometers showed that the pendant is made of brass, not bronze sheet, as was originally thought. In its filling, there is a small fragment of plant-fiber thread (subjected to microscopic observations) and beeswax, which was identified using infrared spectroscopy and gas chromatography with a mass spectrometer.

Key words: kaptorga-pendant, early Middle Ages; Opole, Poland; non-destructive, archaeometry
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1 Institute of Archaeology, University of Wrocław, Szewska 48, 50-139 Wrocław, Poland; beata.miazga@uwr.edu.pl; https://orcid.org/0000-0003-3714-1889
2 Independent researcher, sylrod@poczta.onet.pl; https://orcid.org/0000-0003-0321-834X
3 Department of Chemistry and Industrial Chemistry, University of Pisa, via G. Moruzzi 13, 56124 Pisa, Italy; jeannette.lucejko@unipi.it; https://orcid.org/0000-0002-7717-4039
4 Department of Chemistry and Industrial Chemistry, University of Pisa, via G. Moruzzi 13, 56124 Pisa, Italy; erika.ribechini@unipi.it; https://orcid.org/0000-0002-4978-2782

INTRODUCTION

In Polish lands, West Slavic jewellery became synonymous with the economic development of the early Piast state. Along with the construction of the early Piast system, specialised craft workshops were created, producing high-quality jewellery for the needs of local elites, centered around the ruler (Kóčka-Krenz 1993, 143-157). Slavic jewellery is most often found in hoards and on sepulchral sites. Amongst the most interesting and rare early medieval neck decorations found at archaeological sites are kaptorgas. Capsule pendants can be subdivided into rectangular and trapezoidal forms. The difference between them concerns not only the structural elements, but also the manner of decoration (Kóčka-Krenz

Fig. 1. Map of Europe with the location of the site Groszowice in Opole, where the kaptorga was found (illustrated by B. Miazga)

1993, 84-87; Sztyber 2008, 283). Rectangular kaptorgas were made of one piece of sheet metal, bent in half, with one end rolled into a tubular eye. Most often they were decorated with filigree and granulation. Trapezoidal pendants have a longer bottom edge in relation to the upper edge. In contrast to rectangular kaptorgas, the trapezoidal forms were made of two pieces of sheet metal connected by soldering (front part and sides as well as back part and bottom), topped with a lid. The decorations, which also covered the outer part of the recess, were embossed, and the dominant motifs were a representation of a walking quadruped or a bird with decorative plant elements (for example tree of life), both of which refer to the iconography of the world of the East. Pendants known from Polish territory were most often made of silver and bronze, less often of gilded silver, tin and copper. They occur most often in hoards and women's graves (Kóčka-Krenz 1993, 84, 86-87; Sztyber 2010, 45-46).

Trapezoidal kaptorgas are rare finds in Poland. They are known mainly from hoards and burial grounds, and less often from settlements and strongholds (Kóčka-Krenz 1993, 86-87). These products were probably manufactured on site, as evidenced by the find of a bronze mould matrix for casting trapezoidal kaptorgas from the 11th century in a burial ground in Brześć Kujawski (Jakimowicz 1939, 379-381; Kihl-Byczko 1970, 423-424, Fig. 1). It is difficult to determine their original function. In older literature, trapezoidal kaptorgas were considered to be imports from the East, which travelled to Polish territory via Ruś (Jakimowicz 1933, 103-131; Stattler 1966, 235-236; Sztyber 2010, 48 – older literature there). Other scholars, however, sought the resemblance of the kaptorgas in the so-called portable shrines which appeared first in Western Europe in the second half of the 7th century. The current state of research on these artefacts suggests that they are containers for magical amulets (Szczepkowska-Naliwajek 2000, 27; Sztyber 2010, 48). Primarily organic substances (from Lutomiersk and Opole – Groszowice; Nadolski *et al.* 1959, 81; Miśkiewicz 1969, 288) as well as millet or foxtail millet seeds (from Poznań-Śródka) were found in kaptorgas. These seeds are assigned a symbolic meaning referring to the elements of life, which in the age of Christianity can be associated with the resurrection (Kóčka-Krenz *et al.* 1995, 285; Pawlak 1998, 259). It seems that the content of the amulet was supposed to bring luck and prosperity to the owner (Szczepkowska-Naliwajek 2000, 26-27; Sztyber 2010, 49).

THE ARTEFACT AND ITS STUDY

Since 1965 the old village of Groszowice has been an integral part of the city of Opole, which is a well-studied early medieval site (Gediga 1959, Bukowska-Gedigowa and Gediga 1986, Moździoch 1991, 2006, Gediga and Holz 2012). From the end of the 19th century, Groszowice was known for having the oldest cement plant in Europe. In 1957, an early medieval burial ground was found (dated on 10th-11th cent.; Urbańska 1958, 60) during quarrying activities in the area of the cement plant, on a limestone and sandy hill over the

Odra valley. In the same year, archaeological research was carried out, and 38 skeletal graves were discovered (Wachowski 1975, 108-109). The location of the early medieval burial ground is not random. In the region of Silesia in the early Middle Ages, as well as in other regions of Poland, sepulchral places were most often found on sandy or gravelly hills. Most frequently, the cemeteries were discovered accidentally during exploitation of the aggregate (Miśkiewicz 1969; Kufel-Dzierzgowska 1975; Wachowski 1975, 22; Jagielska 2010, 130).

At the burial ground in Opole-Groszowice, the graves were oriented along an east-west axis, in which the head was oriented to the east. In the other cemeteries, it was a general rule that the dead bodies had been buried along an east-west axis. This rule is connected with the Christian funeral rite in Early Medieval Poland (Zoll-Adamikowa 1971, 39).

During the research in Opole-Groszowice, an assortment of valuable accessories were found inside the graves. They were equipped with West Slavic jewelry (for example, temple rings, rings, glass beads, amber beads, bronze bells), as well as everyday items (*e.g.*, knives, flints, iron awls, buckets, iron buckles). The unique early medieval pendant was an integral part of a necklace with amber and glass beads found in grave no. 38. In this burial of a woman of the age of *maturus*, an amulet with a bas-relief was also found placed on her right hand (Urbańska 1958; 1959, 165-189; Miśkiewicz 1969, 288; Wachowski 1975, 108-109). It was dated to between the middle of the 10th century to the third quarter of the 11th cent. (Holz 2005, 106).

The presence of the pendant and amulet – which are considered "magical" items – in grave no. 38 in the Early Medieval burial ground at Opole-Groszowice is puzzling. The state of research on such artifacts in Silesian cemeteries generally does not allow for a broader comparison and contextualization of "magical" grave finds (Przysiężna-Pizarska 2010). However, archaeometric analyses may well help to provide a broader view of sepulcral finds of pendants in the functional and cultural context of the early Middle Ages in Poland.

The kaptorga was described as a bronze, trapezoidal pendant, 3.7 cm long and 0.8 cm wide, which was made of two pieces of thin sheet metal (Fig. 1). The upper part of the kaptorga was surrounded by a bronze strip, with a plate attached to it, probably constituting a lid. A characteristic feature of this artefact is the embossed motif of a running animal with a stylised palmette. This decoration is surrounded by a double frame. On the side walls of the kaptorga there are two holes with fragmentarily preserved string. Inside the artefact, a filling, probably organic, was discovered (Holc 2005, 106). The pendant was among the grave furnishings, next to eleven glass and six amber beads (Miśkiewicz 1969, 288). The kaptorga was cleaned immediately after its discovery, though the methods used are unfortunately unknown. More than half a century later, the artefact is not in satisfactory condition. On the surface of the pendant, one can see not only a roughness constituting a corrosive layer, but also defects in the metal sheet or traces of repair (Fig. 2). The surface is matte, without metallic gloss. The colour of the corrosion products indicates a significant share of copper in the raw material. Therefore, re-conservation of the kaptorga was

Fig. 2. State of preservation of the kaptorga before conservation and archaeometric studies. A small fragment of the kaptorga's filling is visible (photo by B. Miazga)

necessary, which was carried out simultaneously with archaeometric studies of the artefact. After separating the artefact into individual elements, the metal parts were treated with a 5% sodium carbonate solution for several weeks. The bath was supported by mechanical procedures, which were carried out using soft high-speed polymer tools (Habras discs with granulation of several micrometers). After inhibition of corrosion in 1, 2, 3-benzotriazole solution, the artefact was dehydrated, and secured with a layer of Paraloid B72 after consolidation. The remainder of the organic substance was left outside the kaptorga so as to be fully visible during the artefact's display, per the decision of its custodians.

Chemical composition studies were carried out on an energy dispersive X-ray fluorescence spectrometer (Spectro Midex) with molybdenum X-ray tube and SDD detector. The diameter of the radiation beam was 0.7 mm, which required proper preparation of the artefact's surface. The device was equipped with a visualization system in the form of a 20 × magnification CCD camera, which facilitated the precise selection of the place for examination. The device was also calibrated on certified reference materials IARM-159A (MBH Analytical Ltd, UK), BCR-691 (Institute for Reference Materials and Measurements, EU) and from the BB group (Institute of Non-Ferrous Metals in Gliwice, Poland). The semi-quantitative analysis was performed based on the Fundamental Parameters procedure and included elements with an atomic number greater than 22 (studies in air prevent the measurement of concentrations of light elements). The control examination was a chemical analysis of a small fragment of the kaptorga's sheet on an energy dispersive spectrometer attached to a scanning electron microscope, which was carried out on a Hitachi S-3400N microscope equipped with a tungsten cathode. Images with 150-3500 × magnification and

a maximum resolution of 3 nm were recorded using backscattered electrons (BSE). The content of all elements was determined by a Noran System7 analyzer with a ThermoScientific detector with 129eV resolution. Measurements were carried out at an accelerating voltage of 30 kV and a vacuum of 40Pa.

In addition to the studies conducted with the SEM, the artefact was observed using an Olympus SZX 9 (6.3-114x) light microscope equipped with an Olympus Camedia C-5060 digital camera. For larger magnifications in the 25x-1000 × range, a Nikon Eclipse LV100 metallographic microscope was used, in conjunction with the NIS Elements software, recording the image and enabling its analysis.

Chemical examination of the filling of the kaptorga was performed on a Thermo Nicolet FT 380 Fourier-transform infrared spectroscope with OMNIC software. The sample was examined using a pellet method after mixing it with spectrally pure potassium bromide. The examined spectral range covered 4000-400 cm^{-1} with 16 scans and a resolution of 4 cm^{-1}. The obtained absorption spectrum was analysed using the software search function in commercial electronic databases as well as in the IRUG Database and published literature on the subject. Gas chromatography coupled with mass spectrometry (GC-MS) was another analytical method used on the kaptorga filling. The sample was prepared by a recently published procedure (Andreotti A. *et al.* 2006), which consisted of alkaline hydrolysis (saponification) and subsequent extraction with n-hexane and diethyl ether. The aliquots of each of the two fractions were combined, dried and derivatised for GC/MS analysis, by adding the internal standards and BSTFA as a derivatising agent containing 1% trimethylchlorosilane. The sample was injected into the 6890N Network GC System (Agilent Technologies, Palo Alto, CA, USA) equipped with a PTV injector and coupled to a 5973 MS detector with quadrupole analyser (Lucejko *et al.* 2017).

RESULTS AND DISCUSSION

Metal components

The determination of the elemental composition of the artefact was carried out using XRF spot analysis (Fig. 3). Averaged results showed that the item was made of brass, with 74.7% copper content and less than 20% zinc addition. The average level of tin was 2.7% and lead 6.5% by weight. In addition, there is a trace amount of silver. The EDS spectrometer examination, performed for various areas of a few micrometers, also registered material heterogeneity (Fig. 4). The majority of the sheet metal displays a metallic phase rich in copper and zinc, amounting to 73.3% and 18.4% by weight, respectively. Small amounts of tin (2%) and lead (approximately 0.5%) (Fig. 5) were also determined to be present (analysis in points 1 and 2), Table 1. However, the examined surface area contains a different configuration of elements (Fig. 5, analysis 3-5): lead is predominantly present

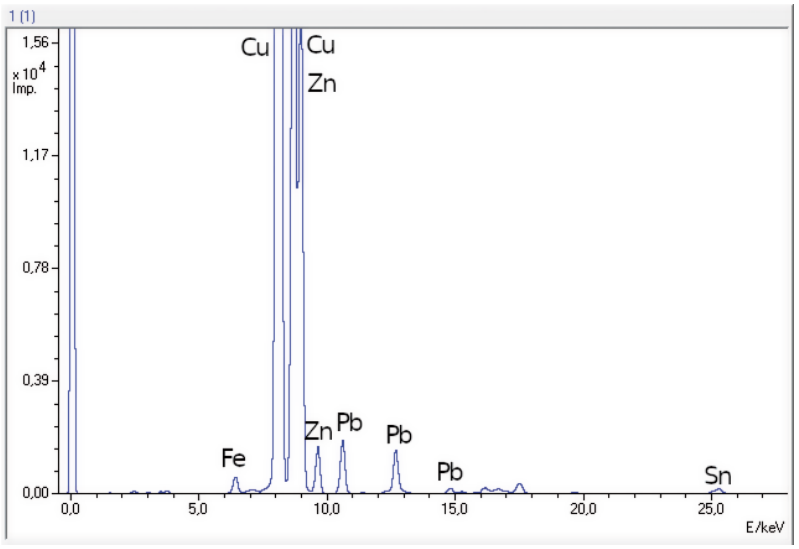

Fig. 3. ED-XRF spectrum of the kaptorga (illustrated by B. Miazga)

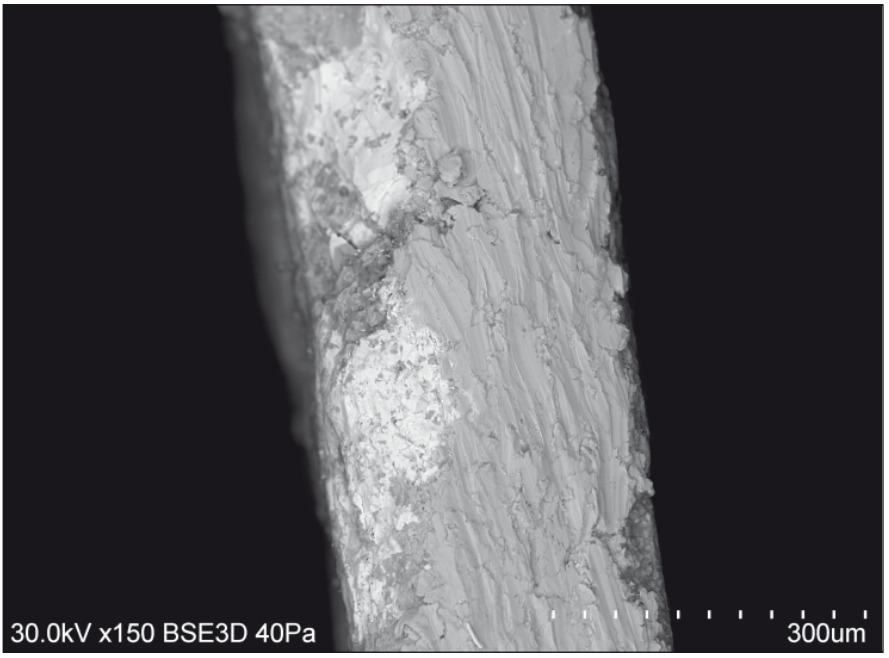

Fig. 4. Microscopic image of the cross-section of the kaptorga's metal sheet with visible heterogeneity of composition: the metallic phase containing significant amounts of copper (grey) and another phase rich in lead (white) or corrosive changes on the surface of the sheet metal (dark grey) are apparent (photo by B. Miazga)

Table 1. SEM-EDS results of elements determined on the sheet's cross section presented in Fig. 5 and in the corrosive layer visible in Fig. 6

Micro-area	Element (% wt.)							
	C	O	Ca	Fe	Cu	Zn	Sn	Pb
Cross section of the sheet (Fig. 5)								
Pt 1	3.2	2.2	-	0.2	73.4	18.4	2.1	0.4
Pt 2	3.0	2.2	-	0.2	73.4	18.4	2.0	0.8
Pt 3	6.1	24.9	-	-	5.1	1.0	0.4	62.5
Pt 4	5.2	17.3	0.2	-	7.1	1.1	0.5	68.6
Pt 5	4.1	17.6	-	-	5.2	1.3	0.5	71.3
Corrosion layer (Fig. 6)								
Pt 1	5.7	24.5	-	-	4.8	1.0	0.4	63.6
Pt 2	3.5	14.3	-	-	80.7	0.4	0.3	0.8
Pt 3	10.6	5.3	-	-	77.2	2.7	1.4	2.8
Pt 4	6.0	24.2	-	-	9.4	1.0	0.3	59.1

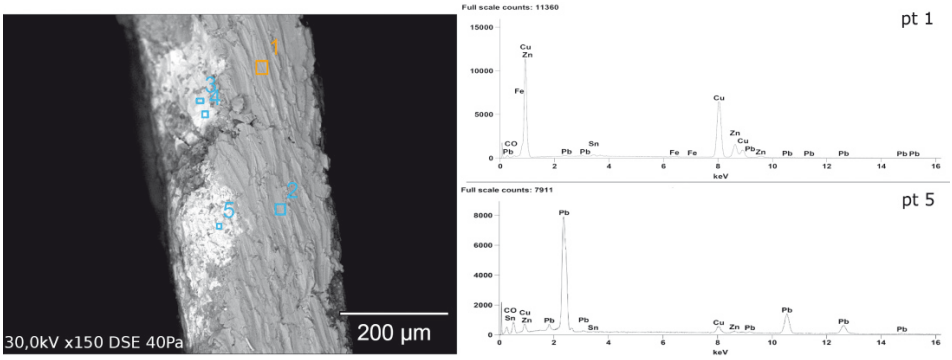

Fig. 5. Results of SEM-EDS examination of two selected microregions: point 1 – copper predominance, point 5 – lead-rich region (illustrated by B. Miazga)

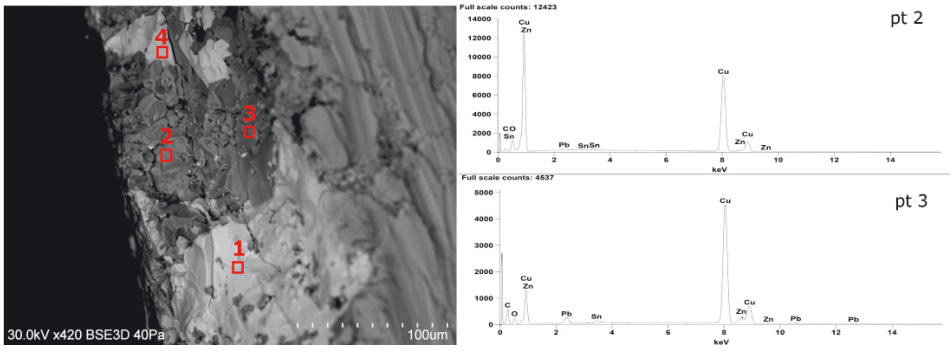

Fig. 6. Results of the SEM-EDS corrosion examination (illustrated by B. Miazga)

(concentration above 65%, Table 1), while copper and other metals are a definite minority. The explanation of this heterogeneity could include both imperfections in the preparation of brass sheet, as well as corrosion processes and the associated segregation of individual elements in the examined alloy. This hypothesis may be supported by the significant amount of oxygen and carbon found in places with a predominance of lead (Table 1), and the greater porosity of this area, indicating oxide or carbonate products of lead corrosion (Fig. 6). Another, quite likely explanation of the high lead signal is the possible presence of solder, which would have bound together the metal elements of the kaptorga. The part of the kaptorga examined on SEM-EDS comes from one of the strips joining the top and bottom parts of the artefact. This hypothesis seems to best explain the large presence of lead on the surface of the metal sheet and its much lower content in the cross section of the sheet.

Textile thread

A fragment of thread, 1 cm long and not exceeding 0.2 mm in diameter, was found on the inside of the kaptorga. The thread was composed of two single threads with a Z twist. In its current state, it is partially unravelled (Fig. 7), but it is apparent that it was made

Fig. 7. Textile thread preserved inside the kaptorga (photo by B. Miazga)

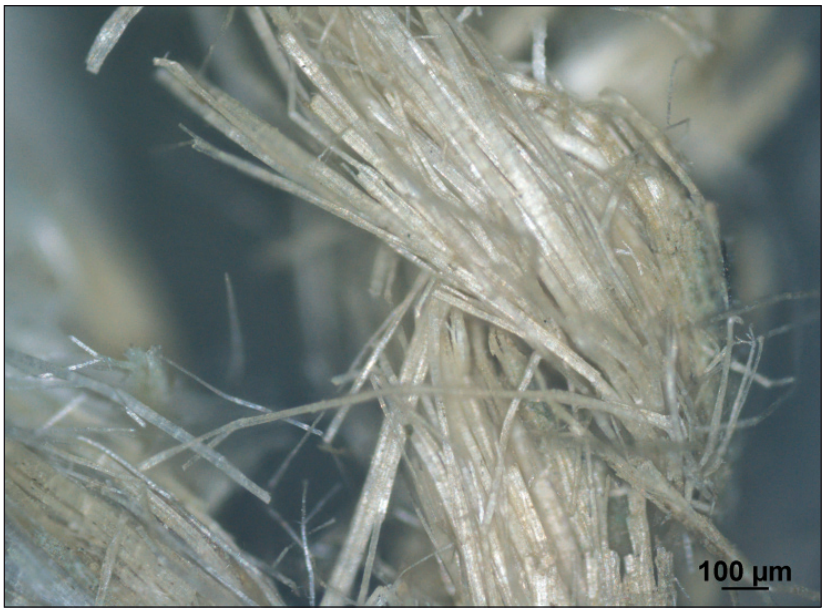

Fig. 8. Microscopic image of the thread preserved inside the kaptorga (optical microscopy, 50 × magnification) (photo by B. Miazga)

with an S twist. The structure of the thread can therefore be described as zz/S. Observation of the thread with microscopic magnification showed its structure was most probably composed of plant bast fibers – possibly flax (Fig. 8).

Examination of the wax deposit inside the *kaptorga*

The FT-IR infrared spectrum of the waxy substance (Fig. 9) indicated the organic nature of the sample. The intensive bands occurring in the region of 2900-2800, 1750, 1450, 1250 and above 700 cm^{-1} clearly indicated the presence of organic carbon compounds. Further analysis of the location and intensity of the bands showed bond vibrations occurring in hydrocarbons; 2922 and 2848 cm^{-1} to be the vibrations stretching the C-H bonds in alkanes (methyl and methylene groups). Other bands referred to as aliphatic hydrocarbon signals were identified for peaks 1473, 1463, 1376 and 730-720 cm^{-1}. Signals recorded on the spectrum in the region of 3434 cm^{-1} can be considered non-characteristic; their origin can be associated with vibrations stretching O-H bonds, occurring in various hydrocarbons. In turn, the peak located at 1736 cm^{-1} is interesting for sample identification, which should be associated with the C = O stretching vibration occurring in esters. Other signals classified as ester group signals can be recognised in the region of 1150-1250 cm^{-1}, where there is a very clear peak at 1174 cm^{-1}. Recognising the location of the aforementioned

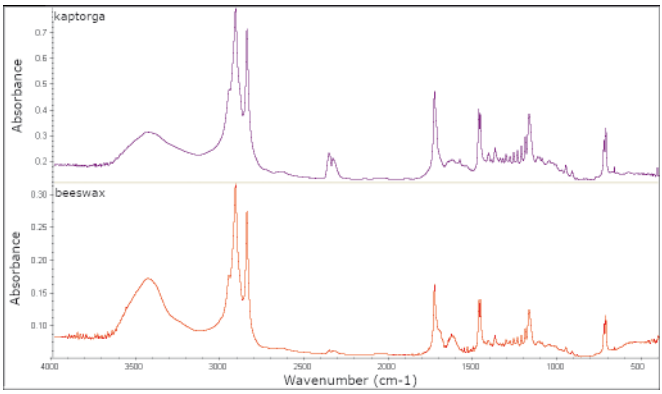

Fig. 9. Infrared spectrum of the organic sample from the inside of the kaptorga, recorded in absorbance mode, and comparison of this spectrum with the experimental spectrum of modern beeswax (illustrated by B. Miazga)

bands allows for a careful inference that the substance in question may be wax of animal origin. Data from the literature on wax studies indicate that it is a mixture of various fatty acids with an even number of carbon atoms, n-alkanes (with an odd number of carbon atoms), as well as wax esters and alcohols resulting from their hydrolysis. In addition, researchers also point to difficulties in studying wax from archaeological research. Stacey (2011) also notes the possibility of not finding signals from fatty acids in archaeological samples, which could be associated with the transformation of acids into salts as a result

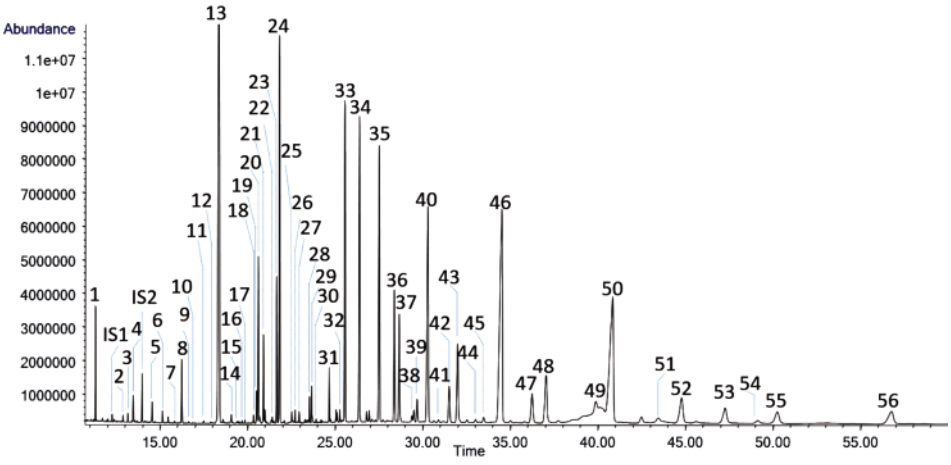

Fig. 10. Total ion current chromatogram of the resinous sample (the acidic and alcoholic species are present as TMS-derivatives). Labels refer to Table 2 (illustrated by J. J. Lucejko)

Table 2. Compounds identified in the sample (prepared by J. J. Lucejko)

No. of peak	Identified compound	No. of peak	Identified compound
1	butylated hydroxytoluene	29	15-OH- octadecanoic acid
2	dodecanoic acid (lauric acid)	30	docosanol
3	tridecanol	31	docosanoic acid
4	1,2-benzenedicarboxylic acid	32	ω-hydroxydocosanoic acid
5	azelaic acid	33	tetracosanol
6	tertadecanoic acid (miristic acid)	34	tetracosanoic acid
7	sebacic acid	35	hexacosanol
8	hexadecanoic acid methyl ester	36	1,22-dihydroxy docosane
9	pentadecanoic acid	37	hexacosanoic acid
10	hexadecano	38	1,24-dihydroxy tetracosane
11	11-hydroxy-dodecanoic acid	39	23-hydroxy tetracosanoic acid
12	palmitelaidic acid	40	octacosanol
13	palmitic acid	41	ω-hydroxy tetracosanoic acid
14	9-octadecenoic acid methyl ester	42	1,25-dihydroxy hexacosane
15	octadecanoic acid methyl ester	43	octacosanoic acid
16	heptadecanoic acid	44	1,26-dihydroxy hexacosane
17	octadecanol	45	ω-hydroxy hexacosanoic acid
18	16-hydroxy-9-octadecenoic acid methyl ester	46	triacontanol
19	15-hydroxy-hexadecanoic acid methyl ester	47	1,27-dihvydroxy octacosane
20	11-cis-octadecenoic acid	48	triacontanoic acid
21	stearic (octadecanoic) acid	49	hexacosyl hexadecanoate
22	αω -tetradecandioic acid	50	dotriacontanol
23	14-hydroxy hexadecanoic acid	51	1,29-dihydroxy triacontane
24	15-hydroxy hexadecanoic acid	52	dotriacontanoic acid
25	ω-hydroxy hexadecanoic acid	53	dotriacontil-15-idrossi-esadecanoato
26	11-eicosenoic acid	54	dotriacontaenoic acid
27	eicosanoic acid	55	tetratriacontanol
28	ω- OH-octadecanoic acid	56	tetratriacontanoic acid

of their deposition in a salty or alkaline environment, or the processes of wax aging or heating, resulting in the loss of volatile alcohols. Comparison of the spectrum of the examined sample with the experimental spectrum of modern waxes indicates that the sample contains significant amounts of beeswax (Fig. 9). The location and intensity of the bands present in beeswax, as in the medieval sample, are very similar, which further confirms its fairly good condition and rather low decomposition. However, the final confirmation of

the presence of beeswax in the sample was provided by a more specialised examination using gas chromatography coupled with mass spectrometry. Figure 10 shows the chromatogram obtained in the GC/MS analysis of the sample. Table 2 lists the identified components that are principally constituted by a series of linear carboxylic acids (ranging from C12 to C34), ω-1-hydroxy acids (ranging from C12 to C32), linear alkanes (ranging from C19 to C33, not shown in Figure 10), linear alcohols (ranging from C24 to C36) and (ω -1)-diols (ranging from C24 to C32). In detail, the main acidic components are long chain (12-34 carbon atoms) linear monocarboxylic acids. The most abundant acids are palmitic acid (hexadecanoic acid, C16:0) and 15-hydroxy hexadecanoic acid, suggesting the presence of a wax. The presence of natural wax in the sample is also confirmed by the presence of long chain alcohols. Natural waxes are indeed complex lipid mixtures mainly consisting of long chain esters (cerides) of fatty acids with long chain alcohols, free fatty acids, hydroxyacids, alcohols, diols, and alkanes (Bonaduce and Colombini 2004; Regert *et al.* 2005; Andreotti *et al.* 2008). The molecular profile varies according to the type of wax and the degree of ageing. In particular, the peaks corresponding to lignoceric acid (#34, tetracosanoic acid, C24:0) in the fatty acid profile of all samples is indicative of the presence of beeswax, which, together with 15-hydroxy hexadecanoic acid (#24) and 15-hydroxy hexadecanoic acid (#23), suggests that they originate from the saponification of cerides (long chain esters contained in natural waxes). In conclusion, the molecular composition of the sample detected using gas chromatography-mass spectrometry (GC-MS) revealed the presence of beeswax.

CONCLUSIONS

This study of a medieval kaptorga, half a century after it was discovered, enabled us to shed new light on the artefact. This was due to a non-destructive archaeometric analysis using various methods. Thanks to them, the raw material from which the kaptorga sheet was made was correctly recognised. It is brass, containing about 20% zinc – not bronze, as previously thought. The amulet 'holder' was prepared from three separate elements cut from brass sheet, which were then joined with lead solder. This is indicated by the extremely high concentrations of lead on the kaptorga surface, identified in the course of elemental studies. Re-conservation not only enabled more effective cleaning and preservation of the artefact, but also became an incredible opportunity to study the individual elements of the kaptorga. The discovery of 'new', unexplored parts of the kaptorga, which resulted from the re-conservation, have proved to be important for the better recognition of the artefact's structure. Microscopic observations made it possible to document and recognise a zz/S twisted plant thread. The organic filling of the kaptorga has been identified as beeswax by FT-IR and GC-MS examination. The early medieval pendant, as a result of widely planned archaeometric and conservation research, ceases to be an artefact, for

which the conditional mode is used. Its structure is correctly recognised, and the results of these studies enrich the state of knowledge about kaptorgas and allow for further archaeological studies on this issue.

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