

THE BRITISH MUSEUM

Technical Research Bulletin



VOLUME 4 2010

Coatings and contents: investigations of residues on four fragmentary sixth-century BC vessels from Naukratis (Egypt)

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Summary The use of organic coatings on unglazed ceramic vessels, to seal surfaces and decrease permeability, has a long history extending from prehistory to modern traditional potters. A range of substances has been used, often water-insoluble natural products that tend to survive well under archaeological conditions. Identification of these coatings provides useful insights into past vessel production and properties and may also offer opportunities for interpretation of reuse and circulation of vessels. Results are presented here from the investigation of coatings on a small number of sixth-century BC vessel sherds from Naukratis (Egypt). All the deposits were found to be composed of pitch derived from conifer wood, which may be the remains of linings used to seal the vessels or residues of the vessels' contents. In one case, fig pulp and 'seeds' are associated with the pitch, which seem not only to suggest that the pitch was used as a lining, but have also offered a rare opportunity to elucidate both the sealing technology and vessel contents. The results are considered in terms of the broader interpretation of the past use of organic coated vessels and highlight the benefit of a multi-disciplinary approach to organic residue analysis.

INTRODUCTION

Porous unglazed pottery is ineffective as a container for liquids unless the surface is treated to provide a seal, the exception being where evaporation through the fabric offers a positive functional advantage as in the case of water storage jars. A considerable range of organic materials has been used for sealing by traditional potters [1]. Water-insoluble sealants such as resins, waxes and bituminous materials are relatively resistant to degradation and so can survive on archaeological ceramics, often preserved as visible surface deposits. Identification of these materials can make an important contribution to the understanding of the manufacture of vessels (both in terms of technology and location) as well as offering scope for interpretation of vessel use, reuse and circulation. This contribution presents results from the analyses of apparent surface coatings on a small group of sixth-century BC vessel sherds from Naukratis.

Naukratis, situated in the western Nile Delta, was the first permanent Greek settlement and trading post in Egypt. Established in the late seventh century BC by traders from 12 Greek cities, most of them Ionian and Dorian from East

Greece, it was the centre for economic and cultural exchange between Egypt and Greece. The site of ancient Naukratis was rediscovered by Flinders Petrie in 1883, and subsequent excavations produced thousands of finds, especially Greek pottery, a large number of which are now housed in the British Museum [2]. This collection is the focus of an ongoing British Museum research project examining the history of the site and its role in interactions between Greeks, Egyptians and other peoples.

As a part of this research, four fragmentary vessels were examined for traces of residue, see Figure 1 and Table 1. All four date to the sixth century BC and were found during Petrie's first season at Naukratis in 1884–1885. The vessel illustrated in Figure 1d was found in a well in the sanctuary of Apollo, but for the others no precise findspot is recorded. The forms and decoration of the vessels are varied and include: one shoulder fragment, with pre-firing graffiti, of what may be a fractional Samian trade amphora; one foot and base fragment of a closed vessel, perhaps an amphora or *oinochoe*, much of its outer surface covered with a black slip, with a painted mark under the base; one East Greek grey ware trefoil-mouth *oinochoe*; and one large fragment

of a (North?) Ionian *oinochoe*, probably also of trefoil-mouth shape and decorated with painted bands and wavy lines. On their interior surfaces all four vessels have brown to black, slightly sticky deposits deriving either from the contents of the vessels or from surface coatings. In one case

(GR 1886,0401.968), botanical material is preserved embedded in the residue. Microscopic examination and chemical analyses were undertaken to establish the nature and origin of the residues with a view to gaining a better understanding of the function and use of the vessels.

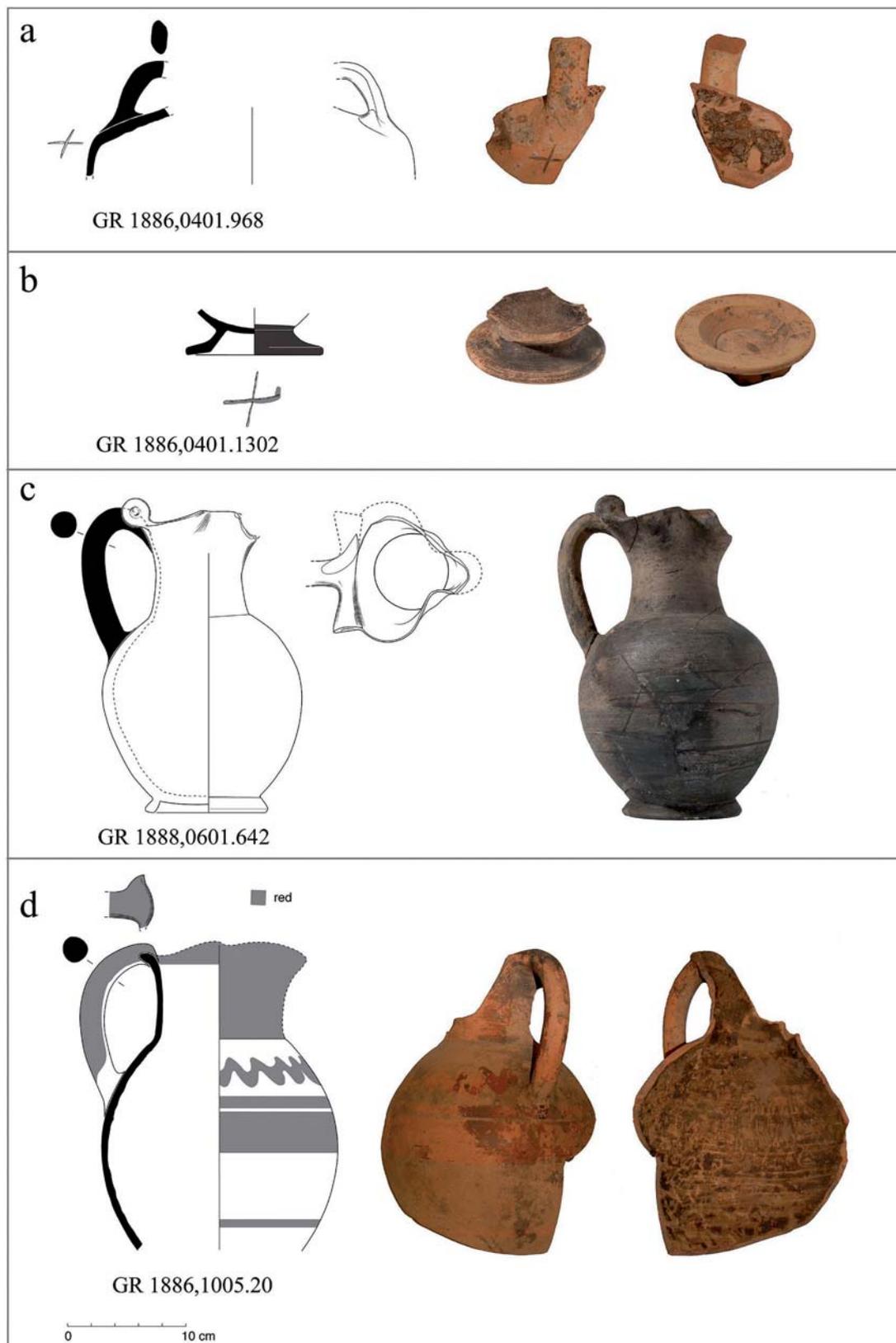


FIGURE 1. Vessels examined in this study, see Table 1 for further details

TABLE 1. Summary of vessel and sample details

Registration No.	Object details	Sample details
GR 1886,0401.968 Figure 1a	Sherd of a small (Samian?) trade amphora comprising the handle and shoulder, with a Greek pre-firing graffito, 'X'. Brown sticky deposits on interior surface with embedded fibrous material. East Greek, first half of sixth century BC	M3a: brown deposits from interior M3b: fibrous material
GR 1886,0401.1302 Figure 1b	Sherd of closed pottery vessel comprising the foot and part of the base. Black glaze on top of the foot. Black to brown residues inside. East Greek, sixth century BC	M4a: black to brown deposits from interior
GR 1888,0601.642 Figure 1c	Grey ware pottery trefoil <i>oinochoe</i> with polished surface and handle <i>rotellae</i> , reconstructed from several sherds with plaster infills. Brown residues on the interior in the neck area and inside the vessel body. East Greek, sixth century BC	M17a: black to brown deposits from interior near opening
GR 1886,1005.20 Figure 1d	Sherd of pottery <i>oinochoe</i> comprising body and rim parts with handle, decorated with bands and wavy lines. Coarse fabric with brown sticky surface deposits inside. Found in well 101 in the Apollo sanctuary. East Greek, sixth century BC	M2a: brown deposits from interior

TABLE 2. Diterpenoid compounds identified by GC-MS with details of principal mass spectral fragment ions

Peak	Compound	MW	Principal (most abundant) fragment ions [% abundance]
1	18-norabieta-8,11,13-triene	256	159[100]; 241[79]; 185[29]; 117[20]; 129[20]; 143[19]; 256[19]; 141[17]; 115[15]; 160[13]
2	19-norabieta-8,11,13-triene	256	159[100]; 241[76]; 185[34]; 117[26]; 129[24]; 128[23]; 143[23]; 256[21]
3	1,2,3,4-tetrahydroretene	238	223[100]; 238[80]; 181[51]; 165[46]; 195[33]; 167[33]; 178[24]; 179[24]; 166[23]
4	Retene	234	219[100]; 234[65]; 204[34]; 203[29]; 189[24]; 202[23]; 220[19]; 191[14]
5	Isopimaric acid TMS	374	241[100]; 73[53]; 256[21]; 91[21]; 242[19]; 105[19]; 257[16]; 359[16]; 117[16]
6	Pimaric acid TMS	374	73[100]; 121[81]; 120[39]; 91[29]; 325[26]; 75[24]; 81[24]; 257[23]; 105[23]; 93[19]
7	Methyl dehydroabietate	314	239[100]; 240[11]; 299[11]; 141[10]; 225[10]; 129[9]; 128[8]; 314[8]
8	Dehydroabietic acid TMS	372	239[100]; 240[40]; 73[34]; 357[23]; 372[21]; 173[19]; 255[15]; 143[13]; 171[13]; 117[10]; 131[10]; 129[10]; 75[10]
9	7-hydroxydehydroabietic acid TMS	460	237[100]; 73[92]; 191[69]; 445[60]; 252[31]; 75[28]; 417[27]; 155[26]; 446[23]; 253[21]
10	7-oxo-dehydroabietic acid TMS	386	253[100]; 268[79]; 73[58]; 187[35]; 269[30]; 327[24]; 75[23]; 254[19]; 386[19]; 213[16]; 178[16]

Note

Peak numbers correspond with the peak labels on the chromatogram in Figure 2; MW denotes molecular weight and 'TMS' a trimethylsilyl derivative.

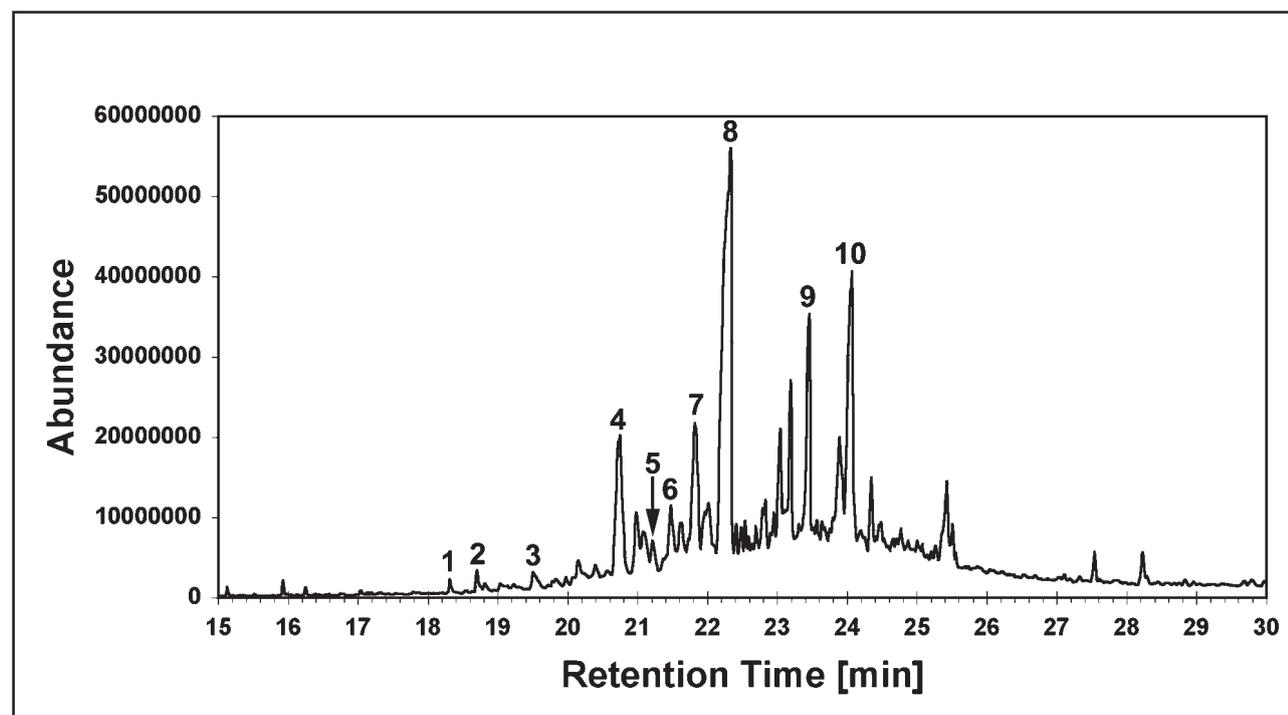


FIGURE 2. Partial total ion chromatogram obtained by GC-MS analysis of the surface deposit from vessel GR 1886,1005.20. Peak labels on the chromatogram correspond with the peak numbers in Table 2

SAMPLE PREPARATION AND ANALYSIS

Four samples (each of $c.1\text{ mm}^3$) were removed from the interior surfaces of the sherds, see Table 1. The samples were extracted with 1 mL of dichloromethane (DCM) and 50 μL aliquots were removed and dried under nitrogen. Prior to analysis these were derivatized with bis(trimethylsilyl) trifluoroacetamide (BSTFA) containing 1% trimethylchlorosilane (TMCS) to form trimethylsilyl (TMS) derivatives.

The analyses were performed on an Agilent 6890N gas chromatograph (GC) coupled to an Agilent 5973N mass spectrometer (MS). Injection was in splitless mode at 250°C and 13.05 psi (90 kPa), with a purge time of 0.8 minutes. An Agilent HP5-MS column (30 m \times 0.25 mm, 0.25 μm film thickness) fitted with a 1 m \times 0.32 mm retention gap was used. The carrier gas was helium in constant flow mode at 1.5 mL per minute. After a one minute isothermal hold at 35°C the oven was temperature programmed to increase to 300°C at 10°C per minute with the final temperature held for five minutes. The MS interface temperature was 280°C. Acquisition was in scan mode (50–600 amu per second) after a solvent delay of 7.5 minutes. Chemstation (G1701DA) software was used for system control and data collection and manipulation. Mass spectral data were interpreted manually with the aid of the NIST/EPA/NIH Mass Spectral Library version 2.0 and by comparison with published and unpublished data [3–6], see Table 2.

A sample of the ‘fibrous’ botanical material from sherd GR 1886,0401.968 was examined initially under a Leica Aristomet biological light microscope at magnifications ranging from $\times 50$ to $\times 850$. Subsequently the sample was examined in a variable pressure scanning electron microscope (Hitachi S-3700) in order to confirm the identification and document its structure by imaging.

RESULTS

All the residue samples were almost entirely soluble in dichloromethane and proved to be very consistent in composition, see Figure 2 and Table 2. The most abundant constituents are the oxidation products of resin acids, such as methyl dehydroabietate, dehydroabietic acid, 7-hydroxydehydroabietic acid and 7-oxo-dehydroabietic acid. These compounds are typical constituents of aged conifer resins, in particular those of the Pinaceae family [7]. In addition, all of the samples contain an abundance of retene. This is the stable end product formed from resin acids by strong heating, such as that required for the manufacture of pitch/tar products from wood or resin. A number of the intermediate products from the tar/pitch production process are also present, such as *nor*-abietatrienes and tetrahydrotetene [4, 8].

Unaltered resin acids can appear in pitch alongside retene and the other aromatized diterpenoids and their presence in relative abundance can indicate tars that have

been produced from the early stages of tar-kiln firing [7, 8]. Two such unaltered resin acids, isopimaric acid and pimaric acid, were seen in the Naukratis samples and in all cases both are present only at minor levels, with none found in the samples from GR 1888,0601.642. It is likely that most (or all) of the unaltered resin acid left in each sample has been oxidized over time to produce the abundance of oxidized products described above. The consistent presence of the resin ester methyl dehydroabietate in the samples suggests the use of whole wood rather than tapped resin as the raw material for the pitch/tar production [9], since the formation of resin esters requires the presence of methanol and an acid catalyst, both of which are released during the pyrolysis of wood, the former from cellulose and the latter from lignin as phenols [10].

The ‘fibrous’ botanical material from the trade amphora sherd GR 1886,0401.968 was identified as remnants of fruit pulp from the cultivated species of fig, *Ficus carica*. A scanning electron micrograph shows some of the characteristic features of the main body of the fig fruit and one of the ‘seeds’, including its peduncle, Figure 3a. The ‘seed’ is in a shrunken, flattened form instead of its usual bulbous shape, possibly as a result of the fruit having been deliberately dried for storage or having dried out from an originally moist condition. The fig fruit differs from most other fruits inasmuch as its edible structure is not matured ovary tissue but stem tissue containing both the male and female flower parts derived from a specially adapted type of inflorescence (syconium). When mature, the fig fruit interior consists of the remnants of these flower parts, including small gritty structures, often called ‘seeds’, but actually unfertilized and undeveloped ovaries. As all these features are evident in the sample (Figure 3), it is reasonable to suggest that mature fig fruits were present in this ceramic vessel, either in the form of fig syrup, dried whole figs, or fig ‘jam’. No lipids characteristic of fig fruit could be identified in the associated amorphous residue [11].

DISCUSSION

The results show that the dark brown or black sticky deposits on all four of the vessels investigated here are pitch/tar made from Pinaceae wood. A key question of interpretation is whether these deposits represent residues of the original vessel contents or are remnants of an applied sealant coating on the vessel interior. Similar questions have been considered by Porter *et al.* in relation to the interpretation of beeswax deposits on South Arabian amphorae [12], and by Stern *et al.* in the case of resin contained in Canaanite amphorae from the Uluburun shipwreck [13]. The presence of fig tissues associated with the pitch on 1886,0401.968 implies that, in this case at least, the deposit is more likely to be a lining material, although the possibility of reuse of a vessel previously used as a pitch container cannot be entirely ruled out. There is no further evidence for contents from

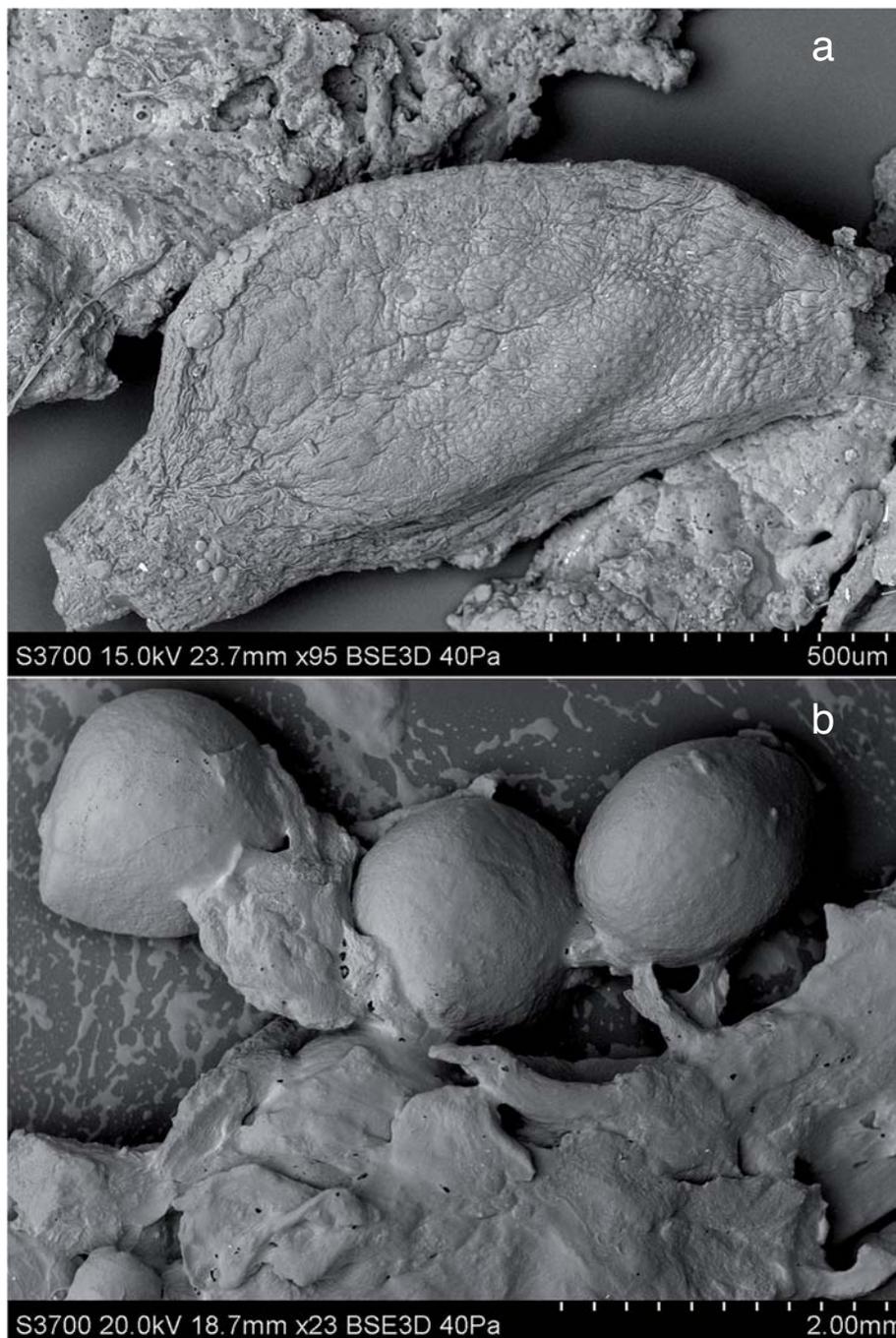


FIGURE 3. Scanning electron microscope (Hitachi S-3700N) images showing the characteristic features of: (a) mature cultivated fig (*Ficus carica*) fruit tissue including one of its 'seeds' (with peduncle) from GR 1886,0401.968; and (b) fruit tissue including 'seeds' from a reference specimen of modern soft dried cultivated Turkish fig (*Ficus carica*). Note how the seeds in the modern specimen are bulbous and much larger than those from GR 1886, 0401.968 in which the 'seed' is shrunken and flattened

the other three vessels, but since at least two of them are certainly not storage containers but jugs, i.e. pouring vessels destined to hold liquids only for short periods, it is improbable, although not necessarily impossible, that the pitch functions as a 'lining'. Perhaps more likely, however, is a scenario in which liquid pitch formed the jugs' contents (or part of their contents) at some point in their cycle of use. The use of pitch in the waterproofing of ships' timbers was common practice in antiquity [14], and jugs may have been used in

this process in the port of Naukratis or on board one of the (Greek) ships. Alternatively, they may have served as pitch jugs at Naukratis in a completely different context, perhaps for military or medicinal purposes.

The pitch must have been brought to Egypt specifically for use at Naukratis, since Egypt lacks the timber sources necessary for its manufacture [15]. While it is possible that timber could have been imported for pitch production, this would hardly have been economic and there is no archaeological

evidence for pitch production in Egypt. In contrast, the import of pitch is known and in the post-Pharaonic period pitch (πίσσα) from the Troad (north western Anatolia) and possibly Syria is documented for use in coating wine amphorae [16], an application that seems to appear in Egypt only with the arrival of the Ptolemies and Romans; in Pharaonic Egypt wine amphorae were not pitch lined [15, 17]. Vessels filled with pitch have been reported among cargoes at a number of shipwreck sites: for example, nine Mendeian amphorae filled with conifer pitch were recently found in the wreck of a small fifth-century BC cabotage ship at Tektaş Burnu off the west coast of Turkey [18]. Of course, the carriage of pitch is not necessarily always indicative of transport for trade, since a supply of pitch might be kept on board for maintenance purposes, but there is certainly literary evidence from the Hellenistic period of large-scale shipments of pitch (cf. Polybius book 5, chapter 89 [19]).

There are also many examples of pitch-lined amphorae used to transport commodities, see for example [20]. Pitch-lined vessels are often linked to wine or other liquid products, due to the role of the pitch coating in reducing the permeability of the vessel fabric. Effective sealants should preclude absorption of the original contents into the ceramic fabric, thus making it more difficult to understand the relationship between vessel treatments and use by means of residue analysis. However, recent experiments have shown that pitch linings may not provide such effective barriers as was once thought and that there is scope to identify absorbed residues from sealed vessels, in particular oil, which can penetrate the pitch lining [21]. Oils have often been thought to be incompatible with pitch linings because of the potential for interaction in this way [22], but they have nevertheless been identified in pitched amphorae from Sagalassos [21].

However, preoccupation with the sealing properties of pitch linings, and thus with liquid products, risks overlooking other commodities that may have been contained and transported in pitched amphorae. These may include more viscous products such as fish sauce [23], or solid food-stuffs such as olives [24], or grain [25]. The finding of fig pulp including seeds embedded in the pitch lining of one of the vessels examined here is particularly interesting in this respect. The western Mediterranean coastal belt of Egypt and parts of Sinai support the present-day cultivation of figs (*Ficus carica*), olives and barley [26]. The fig genus, *Ficus*, is unequivocally represented amongst the indigenous flora of Egypt in the form of *Ficus sycomorus*, the sycamore fig.¹ *Ficus sycomorus* is often shown in tomb paintings in association with the goddess Nut, a patroness of rebirth. It was used extensively in the Middle Kingdom for the construction of wooden coffins and other funerary items and its use continued into the second century AD, alongside imported timber, for some mummy portraits [27]. The species of fig identified here, *Ficus carica* (cultivated fig) has been the subject of some debate as to whether or not it is indigenous to Egypt. Egyptian botanists, such as Boulos [28], suggest that it was introduced into Egypt, cultivated and probably naturalized. The presence of *F. carica* fruits in the archaeo-

botanical record does not automatically signify [29], therefore, that *F. carica* is an indigenous taxon. Such fruits could equally well be from cultivated or naturalized trees. Alternatively, its presence at Naukratis might indicate an import commodity, given that in the rest of the Mediterranean area (Europe and the Near East) fig was one of the three principal economic mainstays and trading crops (with olive and grape) from the Neolithic onwards [25]. In East Greek culture figs were not only a staple of the diet but also played a role in the ritual of Apollo – the festival of the Thargelia in May [30]. The abundance of seeds recovered from some ancient wreck sites certainly shows that figs and other fruits were traded [31], sometimes also within amphorae [32]. The seeds in the amphora from Naukratis have a shrunken flattened appearance, which suggests that they may have been heavily processed, perhaps as a pulp, syrup or some other kind of preserve. Several Roman writers, such as Columella (*De re rustica* 12.15.1–2), describe specifically how dried figs are to be crushed and pressed into “well-pitched jars” for storage [33].

The identification of fig in this amphora underlines the need to consider pitch-lined vessels as containers for a broader range of commodities than the liquid products such as wine and oil with which they are often associated. Recent studies have achieved very specific identifications of amphora contents using DNA [34] and lipid analysis [21] but, where they survive, biological remains generally offer a speedier route to accurate characterization of vessel contents: wine, for example, has been identified from grape seeds and lees found in a sixth-century BC pitch-coated amphora from the Pabuç Burnu shipwreck [35]. The association of macro-organics with pitch linings can preserve wider information about the origin and use of the vessels; for instance, Vogt *et al.* have linked the preservation of vine material in pitch linings to on-site production of wine amphorae [36]. The survival of vulnerable fig pulp tissues in amphora GR 1886,0401.968 is probably due to association with the pitch. In this respect it is interesting to note that the preservative properties of lining materials have been recognized as a potential additional beneficial factor in their use in amphora [34]. The discovery of the remains of figs here highlights the importance of screening such apparently amorphous deposits for the presence of preserved biological tissue that can provide important insights into vessel use.

CONCLUSIONS

The black to brown sticky deposits on the four vessels examined here are all composed of pitch derived from conifer wood. The deposits may be the remains of pitch linings used to seal the vessels or residues of vessel contents. Whichever is the case, the pitch must have been imported to Naukratis, either as a raw material that was later applied (or transferred) to the vessels, or as a component of the vessels or their contents when these were imported from East

Greece. In one case a pitch lining seems more likely due to the association of fig pulp and 'seeds' with the pitch. The fig material shows evidence for processing and is also likely to be an import. The study has highlighted the benefits of looking for macro-organic remains preserved in association with residues and provides an important example of a non-liquid commodity associated with pitch-lined amphorae.

ACKNOWLEDGEMENTS

The authors thank Kate Morton for drawing the four analysed vessels, Tony Simpson for formatting the figures, and Alan Johnston and Maria Effinger for help and advice. We are grateful to colleagues who read the draft manuscript and to an anonymous reviewer for helpful comments.

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NOTE

1. The taxon *Ficus sycomorus* is rather confusing and is frequently misspelled and misused: *Ficus sycomorus* is a fig tree from the Moraceae family and is totally unrelated to the European sycamore or field maple which belongs to the genus *Acer* from the Sapindaceae family.