There’s more to a vessel than meets the eye: organic residue analysis of ‘wine’ containers from shipwrecks and settlements of ancient Cyprus (4th-1st century BCE).

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# ABSTRACT

Despite growing evidence to the contrary, wine remains the assumed content of many types of ancient ceramic vessels. Vessels from the Kyrenia and Mazotos shipwrecks, and Yeronisos island presumed to have contained wine versus other commodities were subjected to three different extraction protocols to test the assumption that these vessels were used to import and serve wine. Chemical extracts reveal grapevine products but also other fruit juice, beeswax and plant oil, sometimes mixed with grapevine products due to intentional mixing or reuse. Biomarkers detected in control sediment samples from Mazotos and Yeronisos demonstrate why quantification is vital. Analyses show that even seemingly identical ceramics from the same shipwreck contained different commodities.

# INTRODUCTION

Cyprus boasts a long history of wine and olive oil production. Strabo describes this fecundity in his work *Geographica*: ‘In fertility Cyprus is not inferior to any one of the islands, for it produces both good wine and good oil, and also a sufficient supply of grain for its own use…(xiv. 6.5).’ Despite this self-sufficiency, archaeologists continue to discover imported ceramic containers demonstrating that Cyprus had a robust import economy, likely fuelled by demand from its affluent citizens for imported goods (Michaelides 1996, 139). Numerous ancient authors describe the high quality of the wine produced in Cyprus including Pliny, Strabo, and Dioscorides (Michaelides 1996, 146). Cypriot wine production has exceptional longevity: *Commandaria,* a sweet wine developed in the Medieval period is widely recognised as the oldest type of wine in continuous production (Constantinou 2004), and Cyprus was the only European wine producing region to escape the phylloxera infestation that destroyed Europe’s vineyards in the 19th century (Grigoriou et al., 2020).

Given the importance of wine in Cyprus and the wider ancient world, it is no wonder that so many types of ceramic vessels found in and around the island are commonly associated with drinking wine. From the rhyton to kalix, krater, *oinochoe* and *lagynos*, and several amphora shapes, myriad ceramic vessels are interpreted as ‘wine containers.’ To what extent these vessels held other types of products, or were reused after initially containing wine, remains unclear.

Attempts to determine the contents of transport containers used in maritime trade shows the complexity of such an endeavour. Early studies of amphorae excavated from Monte Testaccio (Dressel, 1899) and the Athenian agora (Grace, 1961, 1946, 1934) often assigned a specific cargo type to an amphora of a given shape and geographic origin, aided substantially by the study of stamped amphora handles and *tituli picti.* The contents described by *tituli picti* and stamped amphora handles, and confirmed by ancient authors (Heron and Pollard, 1988), were almost invariably one of three commodities wine, olive oil, or fish sauce. Long after Dressel and Grace, the idea that amphora shape can be safely used to establish function and past vessel contents has endured (Koehler, 1995). This can lead to false assumptions regarding the goods amphorae contained, and the volume of trade in these goods (Dallongeville et al., 2011, p. 3053).

Macro-remains from archaeological shipwrecks offer what may be the most direct evidence to challenge long-held assumptions. Amphorae found on ancient shipwrecks have contained macro-remains of butchered beef (Carlson, 2003), butchered pork (Bruni, 2000), almonds (Swiny and Katzev, 1973), whole olives, pomegranates (Haldane, 1993), and pitch (Carlson, 2003). In a similar manner to shipwreck sites, the exceptional preservation at Pompeii yielded a variety of unexpected macro-remains in amphorae, providing further empirical evidence for amphora reuse and repurposing (Peña, 2007). Such preserved contents, however, are rare. In their absence, analysis of the organic residues absorbed in the ceramic walls is the primary approach for addressing the content of ancient pottery (Evershed, 2008). It has been widely used to study transport amphorae, and although wine is among the contents often detected, a wide variety of other products have also been identified, including various plant oils and resins, animal fats, aquatic products, etc. (e.g. Beck et al., 1989; Condamin and Formenti, 1970; Drieu et al., 2021; Fujii et al., 2019; Garnier, 2015; Garnier et al., 2003, 2009, 2011; Pecci et al., 2010; 2017, 2021; Romanus et al., 2009; Stern et al., 2008; Woodworth et al., 2015). Remarkably, several products are often detected together, as a result of intentional mixing, successive reuse or surface treatments before use. Such mixing led to discussions on the permeability of coatings or their compatibility with various contents (e.g. Garnier et al., 2011; Romanus et al., 2009).

The fields of archaeology and ancient history are often intertwined, yet the difference between the two can be simply stated: the former examines the physical remains of material culture left behind by our predecessors, while the latter investigates written records from the past, be they works of literature, poetry, documentary papyrology, or *tituli picti* scrawled on the exterior of transport amphorae. The relationship between these two disciplines becomes complex when our lines of evidence disagree. Clear evidence for the reuse of amphorae is perhaps best exemplified by the case of Dressel 1 amphorae. In her study on the contents of transport amphorae, Panagou states ‘It is interesting to note that in Dressel 1 amphoras there have been found shells, resin, hazelnuts and olives, while according to *tituli picti* wine was the chief content of amphoras of this type (Panagou 2016, 319).’ Adding a further layer of complexity is the fact that there is ample molecular evidence for wine in Dressel 1 amphorae (Cau Ontiveros et al., 2018; Fujii et al., 2019; Garnier et al., 2003). The historical evidence from the *tituli picti* says one thing, often confirmed by molecular analysis of archaeological finds, while other archaeological evidence, such as preserved contents, tells a different story. Therefore, in addition to what would be considered their normal content of wine, Dressel 1 amphorae could, and did, contain other goods.

The status of wine in the ancient world as the beverage *par excellence* leads us to, perhaps rightfully, assume that domestic serving vessels such as *oinochoai*, *lagynoi*, and spouted jars were primarily used to dispense wine. However, the past contents of these vessels are likely to have shown a similar level of diversity as seen in the transport containers, since ancient authors describe a wide variety of fruit juices and honeyed beverages being consumed during this period (i.e. Pliny, *Natural History*, 14.19). Unlike transport containers, these vessels are rarely labelled with details of the contents inside. Therefore, the question remains to what extent we can safely assume these ‘wine’ serving vessels were exclusively used to serve wine. Little work has been conducted on the possible contents of Classical and Hellenistic serving vessels (Frère and Garnier, 2017), as the majority of scholarly literature on the subject deals with earlier periods (Pecci et al., 2020; Rageot et al., 2019a, b; Spiteri et al., 2020), especially in Cyprus where Bronze Age ceramics offer the opportunity to test much older material (e.g. Chovanec 2013).

Here we present exploratory research that seeks to determine whether it is possible to ascertain the presence of wine inside ceramic vessels discovered in Cyprus that are assumed to have been used to import and serve wine during the Classical and Hellenistic periods. We conducted a side-by-side comparison between the assumed contents of pottery that appear empty when discovered, and the molecular evidence for what they actually contained obtained through organic residue analysis. Understanding if these vessels contained wine can help us understand both the demand for these goods in the Eastern Mediterranean, as well as patterns of pottery use in antiquity. Three archaeological sites have been selected for their connection to supposed ‘wine’ containers: the Mazotos and Kyrenia shipwrecks, the majority of whose cargo consists of amphorae thought to have contained wine, and Yeronisos island where a disproportionate number of vessel shapes associated with serving wine were found. To this end, and in order to collect as much information as possible (presence of grapevine products, possible grape colour, possible additions of other substances), we have used an analytical protocol combining three complementary extraction methods, based on recent methodological developments (Drieu et al. 2020, 2021; Garnier and Valamoti, 2016). In order to strengthen the interpretation and the identification of grapevine products by ruling out contamination, in each context we also analysed vessels that are not supposed to have been in contact with wine, as well as environmental samples of soil and seafloor sediment.

# MATERIALS AND METHODS

## Archaeological samples

The artefacts analysed here were recovered from three archaeological sites on the island of Cyprus (Table 1). Two are ancient shipwreck sites: the Mazotos shipwreck which sank off the south coast of Cyprus around the first half of the 4th century BCE (Demesticha 2011), and the Kyrenia Ship which sank off the north coast of Cyprus around 295 BCE (Katzev and Swiny, forthcoming). The third is a terrestrial archaeological site, on the island of Yeronisos located off the southwest coast of Cyprus that dates from the twilight of the Hellenistic era (around the 1st century BCE).

The transport amphorae analysed here are named from the Greek islands on which it is thought they were produced: Rhodes, Chios and Samos. Greek amphorae typology has been much discussed in the literature (e.g. Grace 1934, 1946, 1961; Whitbread, 1995; Lawall and Lund, 2011; Lawall, 2016, Lawall, forthcoming), and their identification was provided by the principal investigator of each shipwreck site (e.g. Demesticha, 2011; Swiny and Katzev, 1973). Similarly, the identification of the domestic close-shaped vessels and the special use vessels was based on their morphology, and provided by the principal investigators of each site (e.g. Swiny and Katzev, 1973; Connelly, 2009; 2005). The pottery samples were divided into two categories: 'wine' pottery and pottery assumed to have contained ‘other commodities,’ according to the type of product they are expected to have contained (Table 1). Although not strictly ‘control’ samples, analysis of vessels assumed to have contained 'other commodities' ensures that the small and highly labile organic acids found in grapevine products are specific to certain vessels and therefore not the result of contamination (Drieu et al., 2020). In addition, where possible, soil and sediment samples were also analysed as a control in order to establish what molecules are naturally present in the deposition environment (Briggs, 2020b).

Table 1: List of samples studied and assumed contents

### Mazotos shipwreck

Mazotos is located on the southern coast of Cyprus around 20 kilometres from the ancient site of Kition, which was an important centre of trade from the Bronze Age onwards (Orphanides, 2015; Yon, 1995, Knapp, 2018). The wreck is located approximately 1.5 nautical miles from shore, at a depth of 44 metres (Demesticha, 2011). The majority of the cargo consists of amphorae produced on the Greek island of Chios (Figure 1) in the mid 4th century BCE, (Demesticha, 2011). Over 500 Chian amphorae were totally or partially visible during the initial survey (Demesticha, 2011, p. 40). It is assumed that the cargo of this ship consisted primarily of wine (Demesticha, 2011), as there is a strong association between Chian amphorae and wine (Boardman, 1967; Sarikakis, 1986). Ancient authors discuss the high quality of wine from Chios (Athenaeus, Deipnosophistae, Ath. 1.26b, 1.29e, 1.32f;), silver coins minted on Chios depict their distinctive amphora underneath a bunch of grapes, and papyri from the 3rd century BCE preserve a boasting comment that wine from an estate in Judea is indistinguishable from the celebrated wines of Chios (Koh and Cline, 2014; Skeat, 1974). However, whole olives were found in four of the over 250 Chian amphorae recovered thus far (Briggs, 2020a). Ten amphorae were sampled for analysis, including one that was found full of olive pits.

### Kyrenia Ship

Discovered off the northern coast of Cyprus near the town of Kyrenia, the site is located at a depth of 27.4 metres (Katzev, 1969). The majority of the cargo consisted of amphorae from the island of Rhodes, with several additional amphora types present in smaller numbers (Lawall, 2021, forthcoming). Widespread references to Rhodian wine by ancient Greek authors such as Athenaeus, and a corpus of stamped amphorae studied by classicists have led to a close association between Rhodian amphorae and a content of wine (Whitbread, 1995, p. 54). Accordingly, wine was the most-likely primary cargo on the Kyrenia Ship (Lawall 2011b). While exports of wine undoubtedly formed a significant part of Rhodes’ economy throughout the Classical and Hellenistic periods (Lund, 2011, p. 280), it remains a possibility that some Rhodian amphora contained other products.

Amphorae thought to have been produced on the island of Samos were also found amongst the cargo. There is both toponymic and numismatic evidence that wine was produced on Samos during this period, yet more than one ancient author speaks disparagingly of the low quality of Samian wine (Shipley, 1987, p. 16). However, the olive oil produced there was considered of good quality by ancient authors including Apuleius (Shipley, 1987, p. 16), and a papyrus document from 295 BCE mentions a shipment of oil destined for Alexandria contained within Samian jars (Grace, 1971; Whitbread, 1995, p. 122), and an olive branch was used as a device on silver coins minted in Samos during this period, suggesting olive oil production (Whitbread, 1995, p. 123). Whole almonds discovered in significant numbers in a few Samian amphorae led the excavators to deduce that these nuts constituted an element of the cargo (Katzev, 1969). The quantity of preserved almonds shows empirically that some of the Samian amphorae on board contained almonds, however the past contents of the remaining Samian amphorae were unknown. One Samian amphora was sampled for this study.

Two domestic closed-shape vessels from the Kyrenia shipwreck are also analysed here: one sherd from a pitcher, and one sherd from a krater. Kraters are drinking vessels associated with wine drinking and symposia (Clark et al., 2002). In addition to the amphorae that formed part of the cargo, these serving wares might have contained wine, and so were selected for analysis in order to determine whether wine residues could be detected

### Yeronisos island

Yeronisos island is located 200 metres off the southwestern coast of Cyprus, and 20 kilometres from Paphos. Archaeological remains dating from the Hellenistic period include finely carved stone architectural elements, fine-ware pottery, and a large circular platform that has been interpreted as part of a temple dedicated to Apollo (Connelly, 2009, p. 302). These finds suggest an elite presence on the island (Connelly, 2009, p. 302). Such an elite community is just the type of destination we would expect for imported wine, such as that contained in transport amphorae found on shipwrecks. However, an interesting facet of the Yeronisos pottery assemblage is the lack of transport amphorae, while ‘wine’ pitchers and ‘wine’ vessels abound: *lagynoi* and *oinochoai*, pitcher-like vessels thought to have contained wine, are common finds on the island (Connelly 2005; 2009). This begs the question as to how wine arrived at Yeronisos, if not in transport amphorae, and if the many *oinochoai* and *lagynoi* discovered did indeed contain wine.

The ceramic samples from Yeronisos included in this study consist of five vessels thought to have contained wine, including *oinochoai* and *lagynoi*, two spouted jars of unclear use, as well as three samples of ‘special-use’ vessels consisting of two *unguentaria*, and one chamber pot. Three *oinochoai* (singular *oinochoe*) and two *lagynoi* (singular *lagynos*), closed-shape ceramic vessels identified in the literature as wine jugs (Figure 1), were selected for analysis. Sherds from two spouted jars (Figure 1) were also analysed here. The past contents of these spouted jars are unknown, but some type of herbal infusion has been suggested as a likely past content  (Connelly, 2005, p. 168). Several *unguentaria* (singular *unguentarium*) thought to have contained unguents, perfumes, or other precious liquids, have been found at Yeronisos (Connelly, 2005; 2009). Their small size and closed shape suggest that their liquid contents were valuable. Two *unguentaria* from Yeronisos are analysed here (Figure 1).

The pottery of Yeronisos is perplexing, however, as a high proportion of fine vessels and several elegant *unguentaria* (small, piriform jars thought to have contained unguents) appear to indicate high status inhabitants, while, contrarily, widespread evidence for reuse, re-fashioning, and mending of pottery suggests that the inhabitants were slow to replace the pots and jugs they possessed (Connelly, 2005, p. 176). Several vessels preserve so many holes that have been mended with lead, it begs the question whether the cost of the lead would have exceeded the cost of replacing the pot (Connelly, 2005, p. 176). While widespread reuse of pottery was common in the ancient world (Peña, 2007), it is the combination of luxury wares with clear evidence for refashioning and mending that makes the ceramic assemblage of Yeronisos unique.

Figure 1. a) Spouted jar P.94.18/BRI\_05 (Yeronisos), b) *Oinochoe* P.13.12/BRI\_11 (Yeronisos), c) *Unguentarium* P.14.02/BRI\_28 (Yeronisos), d) Large Rhodian amphora (Kyrenia), e) Chian amphora (Mazotos),  f) Rhodian amphora sherd 468/BRI\_126 (Kyrenia), g) Chian amphora sherd P0746b/BRI\_109 (Mazotos), h) *lagynos* P.05.03/BRI\_02 (Yeronisos). Three principal amphora types analysed: 1) Samian, 2) Rhodian, 3) Chian.

## Extraction and Analysis

Ceramic samples were transported in aluminium foil. The entire extraction sequence, including the handling of the potsherds, was carried out wearing gloves and using clean glassware that was heated to 600°C for 4 hours to remove any traces of contamination. The method was designed to analyse lipids (plant oils, aquatic products) and small organic acids (plant products, in particular grapes) separately. Following Garnier and Valamoti (2016), after the solvent extraction of lipids, we performed an acid butylation of small organic acids with a boron trifluoride-butanol/hexane solution. We applied the same type of procedure by following the lipid extraction method with the small organic acid extraction method under alkaline conditions developed by Pecci et al. (2013). This double approach was implemented to assess tartaric and syringic acid respectively [(Drieu et al., 2020)](https://paperpile.com/c/8eznTR/xZxm).

In detail, approximately two grams of ceramics and sediments were sampled. The outer surface of all sherds was removed with an aluminium oxide air wick, and a sample was obtained by drilling to a depth of 2 – 5 mm with a clean drill bit. The samples were divided into two and each extracted by sonication in dichloromethane/methanol (DCM/MeOH, 2:1 v/v, 3 x 5 mL) together with an internal standard (10 µg *n*-tetratriacontane). The resulting Total Lipid Extracts (TLEs) were combined and dried under a gentle stream of nitrogen. Afterwards, one portion of ceramic was then treated with 6 mL of potassium hydroxide (KOH, 1 M) at 70° C for 90 minutes, acidified with hydrochloric acid (HCl, 2 M) until the pH of the solution reaches 6 or below, following Pecci et al., (2013). Two mL of ethyl acetate were added to the acidified solution and the two phases were allowed to separate. The ethyl acetate supernatant was collected. The ethyl acetate extraction was repeated twice and the resulting extracts were combined and dried under a stream of nitrogen. After Garnier and Valamoti (2016), the other part of the ceramic powder was treated for 2 hours at 80°C with a boron trifluoride-butanol/hexane solution (BF3-BuOH/hexane, 1:2, v/v). After centrifugation, the solution was collected, neutralised with a saturated solution of sodium carbonate and extracted three times with DCM. The extracts were combined and washed twice with distilled water and then evaporated under nitrogen. All the extracts (following solvent extraction, alkaline treatment and acid butylation) were trimethylsilylated with N,O-bis(trimethylsilyl)trifluoroacetamide (BSTFA) with 1% trimethylchlorosilane (70° C, 1h). Ten µg of internal standard (*n*-hexatriacontane) were added to each sample prior to dilution in hexane and immediate analysis by GC-MS.

The samples were analysed on an Agilent Technologies 7890A chromatograph, equipped with an apolar column (DB5-HT, 30 m × 0.25 mm i.d., 0.1 μm film thickness, Agilent J&W) and coupled with an Agilent 5977B mass spectrometer. Injection was carried out in splitless mode, and the temperature programme was as follows: the temperature was set at 50°C for 2 min, then increased at 10°C min-1 up to 325°C, before a 15 min isothermal hold.

# RESULTS AND DISCUSSION

## Identification of products

Solvent extraction yielded lipids in most samples. Most of the lipid extracts in the samples from Yeronisos (*lagynos* P.05.03/BRI\_2, *oinochoe* P.13.12/BRI\_11, *oinochoe* P.94.49/BRI\_12, *oinochoe* P.94.23/BRI\_13, *chamber pot* P.93.12/BRI\_24, *unguentarium* P.14.02/BRI\_28, and *unguentarium* P.0801/BRI\_29), however, were severely contaminated with modern products such as plastic (phthalates and benzoic acid derivatives) and cosmetic additives. Octocrylene, octinoxate and homosalate are common UV filters found in sunscreen. N,N-Diethyl-meta-toluamide (DEET) are used in insect-repellent, while tocopherol acetate and cetyl 2-ethylhexanoate are compounds frequently used in skin creams. Odd and even chain alkanes are derived from a petroleum derivative (Whelton et al., 2021). The presence of high amounts of sterols (up to 40 μg g-1), together with unusually high amounts of C18:2 and C18:1 (up to 15 μg g-1) also suggest modern contamination, maybe from cosmetics (Whelton et al., 2021). The presence of these compounds in archaeological ceramics is likely due to excavation or post-excavation handling.

The presence of some of these compounds, which have already been reported in other studies (Perruchini et al., 2018; Steele et al., 2013), including UV filters, cosmetic components and insect repellents, is in particular probably related to Mediterranean excavation conditions. Yeronisos island experiences searing heat and unrelenting sunshine in the summer, and excavators are known to frequently reapply sunscreen throughout the day. Insect repellent is also commonly used. Contamination during organic residue extraction is unlikely as the potsherds were only handled with gloves. The presence of such large amounts of modern contaminants makes the accurate interpretation of the rest of the compounds extracted from these samples very difficult, as they can originate either from the content of the archaeological ceramics or from modern contamination. Some samples from the Kyrenia shipwreck also yielded some evidence of minor plastic and insect-repellent contamination.

Sulphur was detected in five ‘wine’ vessels from the Mazotos shipwreck (Chian amphorae P0841/BRI\_104, P0784/BRI\_105, P0746a/BRI\_108, P0746b/BRI\_109 and P0395/BRI\_112) and a seafloor sediment sample from the same site (Sed 1/BRI\_120). Different hypotheses have been formulated to explain the presence of sulphur in archaeological pottery: surface treatment, wine preservative, or contamination related to bacterial activity in an anaerobic context (Garnier et al., 2011; Pecci et al., 2017; Poulain et al., 2016; Reber et al., 2018). As sulphur has only been detected here in a submerged context (the Mazotos shipwreck), and is also present in one of the sediments analysed, it is likely to be the result of bacterial activity.

Tartaric acid was detected in only one sample after alkaline treatment. Acid butylation proved to be much more efficient (15 samples yielded tartaric acid) which confirmed the results previously observed in a different archaeological context (Drieu et al., 2020). Other small organic acids such as malic and fumaric acid were detected in some samples. Although it is sometimes used as a marker for fruit fermentation, fumaric acid is also naturally present in various plants and fungi (Drieu et al., 2020), and therefore will not be used as a marker hereafter. Malic acid by itself provides little taxonomic information as it occurs in many plants. By calculating the ratio of the amount of tartaric acid to the sum of malic and tartaric acid (%TA) in various fresh plants, in archaeological containers with known contents and in experimental ceramics impregnated with wine and degraded in the soil, it was shown that a %TA > 35% indicated with good reliability the presence of grapevine products or tamarind (Drieu et al., 2021). Chemical data cannot distinguish between grapevine and tamarind products, however, no historical sources mention the consumption or trade of tamarind, which originated in tropical Africa, in Cyprus between the 4th century BCE and the 1st century CE. Tamarind was traded and consumed in the Mediterranean by the Medieval period (Goldberg 2012), but there appears to be no evidence to support widespread circulation of this fruit in the region prior to that. It cannot be completely excluded that the small organic acids detected in the Yeronisos samples are the result of cosmetic contamination. However, this risk remains minimal as the small organic acids are not systematically present in all samples with large amounts of sterols, C18:1 and C18:2 (and vice versa).

Three ‘wine’ vessels and one ‘other commodity’ vessel (*lagynos* P.15.17/BRI\_01, *unguentarium* P14.02/BRI\_28, and Chian amphorae P0746a/BRI\_108 and P0293/BRI\_110) proved to be in the range of pure grapevine products (Drieu et al., 2021): they yielded substantial amounts of tartaric acid (> 1 µg g-1) and %TA > 35% (Figure 2 and Figure 3). Six more ‘wine’ ceramic samples and one ‘other commodities’ pottery sample produced tartaric acid (> 0.4 µg g-1), but %TA < 35% (*lagynos* P.05.03/BRI\_02, *oinochoai* P.13.12/BRI\_11 and P.94.23/BRI\_13, krater P20/BRI\_100, Chian amphora P0841/BRI\_104, torpedo jar P174/BRI\_125, and Rhodian amphora 468/BRI\_126). These vessels may have been used for plant products other than grapevine products or may have contained mixtures of grapevine products with other substances rich in malic acid, either as an intentional mixture or in successive uses (Drieu et al., 2021). Fruit juices or honey, rich in malic acid (Nelson and Mottern, 1931; Walker and Famiani, 2018), are potential candidates for such mixtures. In the other ‘wine’ and ‘other commodities’ vessels, tartaric acid is present only in trace amounts or else totally absent. It cannot be excluded that these very low concentrations are due to significant degradation. However, nine of these samples have preserved malic acid (> 1 µg g-1; spouted jars P.05.07/BRI\_04 and P.94.18/BRI\_05, *oinochoe* P.94.49/BRI\_12, chamber pot P.93.12/BRI\_24, *unguentarium* P.08.01/BRI\_29, Samian amphora 551 P46/BRI\_95, Rhodian amphora 760/BRI\_96, pitcher P19/BRI\_99, Chian amphora P0426/BRI\_111; Figure 3), and experiments suggested that it is more easily degraded than tartaric acid (Pecci et al., 2013). Although such a degradation pattern needs to be verified in underwater contexts and with the BF3/BuOH extraction method that was used here, it is very likely that these samples did not contain wine, but plant, fruit, or honey-based contents. Tartaric was detected as traces (< 0.1 µg g-1) in two sediment samples (soil from Yeronisos BRI\_25 and seafloor sediment from Mazotos Sed 3/BRI\_121; Figure 2). This result, combined with the trace amounts of tartaric acid in most of the ‘other commodities’ pottery samples suggests that, when detected in very low amounts in pottery, tartaric acid is likely to be the result of environmental contamination, both in terrestrial and underwater contexts.

Figure 2: a) %TA in authentic modern plant products (adapted from Drieu et al. 2021); b) %TA plotted versus the amount of tartaric extracted from archaeological samples from Cyprus. The vertical dashed line indicates the %TA value of 35%.

In Samian amphora 551 P46/BRI\_95, the near-absence of modern contaminants and the fatty acid profile, dominated by palmitic acid (C16:0) and oleic acid (C18:1), together with the presence of C18:1 degradation products (dicarboxylic acids di-C4:0 to di-C9:0) in the butylated extracts suggests the presence of plant oil (Copley et al. 2005; Regert et al., 1998; Figure 3). Beeswax was identified in *lagynos* P.15.17/BRI\_01 based on the presence of palmitic acid wax esters (W40-W50, max W48), linear alkanes (C27-C33, max C27, including unsaturated C33) and small amounts of linear long chain alcohol [(Roffet-Salque et al. 2015)](https://paperpile.com/c/8eznTR/jgu6) (Figure 3). Although it cannot be excluded that beeswax is part of the composition of a modern cosmetic (other contaminants were identified in the sample), its detection in a single sample from this site argues in favour of the ancient use of beeswax.

Diterpenes (dehydroabietic acid, di-dehydroabietic acid, 7-oxo-dehydroabietic acid, 15-hydroxy-7-oxo-dehydroabietic acid and small amounts of abietic acid, pimaric acid, isopimaric acid) are also detected in most samples (lagynos P.15.17/BRI\_01, Rhodian amphora 760/BRI\_96 and 468/BRI\_126, pitcher P19/BRI\_99, torpedo jar P174/BRI\_125, Chian amphora P0824/BRI\_102, P0841/BRI\_104, P0784/BRI\_105, P0746a/BRI\_108, P0746b/BRI\_109, P0293/BRI\_110, and P0395/BRI\_112; Figure 3), which demonstrates the common use of conifer exudates, probably for sealing purposes. The detection of thermal transformation markers indicates the heating of resin for pitch manufacture (retene, 8-Isopropyl-1,3-dimethylphenanthrene, 10,18-bisnorabieta-8,11,13-triene), or the heating of the wood itself for tar manufacture (methyl- dehydroabietic acid, methyl-7-oxo-dehydroabietic acid) in some samples (Hjulström et al., 2006; Pollard and Heron, 2008).

Figure 3: Characteristic chromatograms. a) total lipid extract, after extraction with DCM/MeOH; b) polar molecules (fruit acids and oxidation products of unsaturated fats), after acid butylation.  *Lagynos* P.15.17/BRI\_01: beeswax and grapevine products; Chian amphora P0293/BRI\_110: conifer resin and grapevine products; Chian amphora P0824/BRI\_102: conifer tar; Chian amphora P0426/BRI\_111: fruit product; Samian amphora 551 P46/BRI\_95: plant oil. Cxx:x: fatty acids, di-Cxx:x: dicarboxylic acids, Cxx: alkanes, CxxOH: alkanols, Wxx: wax ester, MA: malic acid, TA: tartaric acid, DHA: dehydroabietic acid: Me-DHA: methyl-dehydroabietate, IS: internal standard, \*: contamination.

## Discussion of syringic acid

Syringic acid was extracted in various amounts from six samples after alkaline treatment (Table 2), three pottery samples (*lagynos* P.05.03/BRI\_02, *oinochoe* P.94.23/BRI\_13, and Chian amphora P0643/BRI\_106) and three environmental samples: a soil sample from Yeronisos (P.93.12/BRI\_25), and two seafloor sediment samples, from inside (Sed 3/BRI\_121) and outside (Sed 1/BRI\_120) the Mazotos shipwreck area. No sample yielded syringic acid after acid butylation, which confirms that this is not a suitable method for releasing syringic acid (Drieu et al., 2020). No correlation was shown between the detection of syringic acid and the presence of grapevine products (i.e. %TA > 35%; Table 2). In particular, of the samples that yielded syringic acid, half are sediment samples, including one that had no contact with products of anthropic origin, since it was taken from outside the shipwreck. In these samples, syringic acid necessarily comes from an origin other than malvidin and red grapes. As it was not extracted during solvent extraction but only after alkaline treatment, syringic acid was most likely present in the sediment in bound form, possibly to lignin, a plant polymer (Drieu et al., 2020). One Chian amphora (P0643/BRI\_106) yielded syringic acid but no tartaric acid (Table 2). Again, this suggests that syringic acid did not originate from malvidin, but from another compound or that it was strongly bound to the ceramic matrix. Woody or resinous substances related to the corking or sealing of pottery are potential candidates as parent molecules [(Woodworth et al. 2015; Stern et al. 2008)](https://paperpile.com/c/8eznTR/L20h%2BuoMO). Syringic acid was extracted from P.05.03/BRI\_02 and P.94.23/BRI\_13, which also yielded tartaric acid after acid butylation. The %TA measured in these vessels (< 35%) does not, however, confirm the presence of grapevine products. Furthermore, the previous results cast doubt on the interpretation of its presence as a red grape marker in these latter samples. Multiple scenarios can be envisaged to explain the extraction yields of tartaric and malic acids in these samples, but not one is more likely than another: a mixture of red wine and another product, a product based on fruit other than grapes and degradation of an organic stopper, etc.

Table 2: Interpretation of the detection of syringic acid in some samples

## Form and content

### Shipwreck amphorae (Kyrenia and Mazotos)

Chemical analyses on Chian amphorae from the Mazotos cargo has confirmed the initial hypothesis that some of the containers were wine carriers, as two out of ten (P0746a/BRI\_108 and P0293/BRI\_110) displayed a signal for pure grape product (%TA>35%). One Chian amphora (P0841/BRI\_104) yielded tartaric acid but did not meet this criterion, suggesting other fruit products, intentional mixing of wine with other products (e.g. fruit juice), or reuse. Four of the ten analysed Chian amphora samples (P0863/BRI\_103, P0643/BRI\_106, P0746b/BRI\_109 and P0395/BRI\_112) did not produce any molecular signals, suggesting significant degradation or contents that left no organic traces (e.g. water or solid and dry contents). In Chian amphora P0426/BRI\_111, the presence of malic acid (6 µg g-1) may be related to the presence of olive pits in the vessel. Such a hypothesis would be valid for olives that are not completely ripe (still containing malic acid; [Donaire et al. 1975](https://paperpile.com/c/8eznTR/VA2x)), preserved in an aqueous liquid (e.g. brine), favouring the migration and absorption of the malic acid into the walls. Pliny the Elder describes the best method for preserving whole olives was to harvest them ‘while green’, first preserving them in ‘strong brine’ then storing them in oil (*Natural History* 16.6). However, ancient authors also describe whole olives as being preserved in ‘wine or must’ (Celsus, *De Medicina*, 2.20).Therefore, the detection of malic acid could either relate to the presence of green (less ripe) olives, some sort of fruit juice preservative for the olives or a past content contained within this vessel at some earlier time. Archaeologically attested thin layers of pitch on their interior surface of Chian amphorae P0824/BRI\_102 and P0784/BRI\_105 were confirmed by the detection conifer exudate.

It is interesting to note that neither of the two Rhodian amphorae analysed from the Kyrenia Ship showed a pure grapevine signal, one of them even showing a clear signal of a product based on another fruit (Table 3). Pomegranate and fig are potential candidates to explain the fruit-based product signal in amphorae, as such remains have previously been found in ceramic transport containers on shipwreck sites (Haldane 1993). This finding reinforces the idea that Rhodian amphorae were not exclusively used for wine. Another interesting finding comes from the Samian amphora (551 P46/BRI\_95) which is the only sample of this corpus that unequivocally contained plant oil. This result is consistent with the textual and numismatic evidence for olive oil production and transport from the island of Samos. The presence of malic acid in this sample could be related to reuse, or to the addition of aromatics to the oily content.

Of note, all the amphorae which contained tartaric acid, i.e. grapevine products with or without other substances (Chian amphorae P0841/BRI\_104, P0746a/BRI\_108, and P0293/BRI\_110, torpedo jar P174/BRI\_125, and Rhodian amphora 468/BRI\_126), are all sealed with conifer exudates, but not the amphora that contained plant oil (Samian amphora 551 P46/BRI\_95). While recent evidence indicates at least some fish sauce amphorae were also lined, resinous linings may have been more permeable than previously thought, and re-use of pine tar lined amphorae appears to have been commonplace (Garnier et al., 2009; Romanus et al., 2009; Pecci et al., 2018, 2021), our data agrees with historical sources which mention that oil amphora are not pitched, unlike wine amphorae (Brun, 2003; Heron and Pollard, 1988). Conifer products were an important commodity in the ancient world; when Polybius describes gifts given by Antigonus to the people of Rhodes he lists ‘a thousand talents of pitch; [and] a thousand amphorae of the same unboiled’ (Polybius, *Histories,* 5.29). This suggests that both heated and unheated pine products were traded and gifted in the ancient world, shedding light on why we find molecular evidence for both heated and unheated pine products in our ceramic samples.

### Serving vessels (Yeronisos)

Only one sample of the ceramics assumed to be for wine service provided a reliable signal of pure grapevine products (*lagynos* P.15.17/BRI\_01; Table 3). With the exception of *oinochoe* P.94.18/BRI\_12, all others yielded tartaric acid, but with %TA < 35%. Historical sources inform us about the use of this type of vessel and mention the addition of various products to the wine (honey, fruit juices, herbs and spices; Bush, 2002; Stanley, 1999). Honey, mentioned by Pliny the Elder as an additive to ‘sweet wine’ (*Natural History* 14.11), contains malic acid [(Stinson et al. 1960)](https://paperpile.com/c/8eznTR/Il6N), and should cause a decrease in %TA. In *lagynos* P.15.17/BRI\_01, such an additive could explain both the presence of beeswax and the lower %TA than the amphorae where wine has been reliably identified (51%, compared to 63 and 70%). Some varieties of pomegranate are rich in malic acid, and pomegranate is mentioned by some classical authors as another additive to wine (e.g. Columella; Ward, 2003). Wine mixed with pomegranate is also mentioned in the Song of Solomon: ‘I would have you drink spiced wine, of the juice of my pomegranate’ (Song of Solomon, 8.2).

The presence of malic acid and the absence of tartaric acid in *oinochoe* P.94.49/BRI\_12 suggests that this *oinochoe* was used for a fruit product other than wine. Pliny the Elder describes a variety of drinks that he calls ‘artificial wines’ that can be produced by fermenting the juice of a number of fruits such as apples, pears, figs, dates, or mulberries (*Natural History*, 14.19). Wine made from figs on the island of Cyprus has also been described by Dioscorides, a Greek botanist from the 1st century CE (Michaelides 1996, 146, citing Diosc. Mat. Med. 5.32).

The presence of contamination in most of the samples makes it impossible to verify the presence of animal fats in combination with fruit products in serving vessels, as has been identified in contemporary *oinochoe* in Italy (Frère and Garnier, 2017).

### Unguentaria

Surprisingly, an *unguentarium* (P.14.02/BRI\_28) provided a signal of grapevine products (Table 3), with 4 µg g-1 of tartaric acid and %TA = 75%. In this sample, and *unguentarium* (P.08.01/BRI\_29), the presence of a fatty product remains difficult to interpret because the lipid extracts are highly contaminated. Grapevine products are rarely mentioned as potential contents for *unguentaria*, as these vessels were more likely used for unguents, perfumes, or other precious liquids (Connelly, 2005; 2009). However, grapevine products have also been suggested as possible contents for two *unguentaria* found in the Etruscan Hellenistic Necropolis of Banditaccia (Frère and Garnier, 2017). Comparison with the Yeronisos samples should be made with caution, however, as quantitative data for malic and tartaric acids are not provided for the Banditaccia samples. Interestingly, grapevine products (wine and lees in particular) are mentioned for various cosmetic uses in ancient Rome (dyes, hair treatment, perfumes etc.; Brun, 2000; Olson et al., 2009; Wilner, 1931). Pliny the Elder describes unguents from the island of Cyprus as being held in high esteem throughout the ancient world, and describes several recipes for unguents consisting of rose, oenanthe, or other fragrant flowers, mixed with honey and wine (*Natural History* 13.2). This could explain the presence of tartaric acid in a perfume container. Unlike the transport amphorae, the *unguentaria* analysed here did not retain any evidence for a pitch or resin lining. This may have further implications for the potential absorption of tartaric acid into the porous walls of unlined perfume vessels.

Table 3: Comparison between the expected content and the actual content of pottery, identified through ORA. \*Amphora with macroscopic content of olive pits

# CONCLUSION

From a methodological point of view, this work has confirmed the importance of cautious interpretation of low concentrations of tartaric acid (< 0.1 µg g-1) in pottery samples, which may be due to environmental contamination, as shown in other contexts (Drieu et al., 2021). Additionally, the detection of syringic acid in environmental samples from both underwater and terrestrial archaeological sites calls into question our ability to use syringic acid as a reliable biomarker for the colour of wine. These results reinforce the importance of analysing seafloor sediment and soil samples and quantifying the exact amount of fruit acids (similarly to common practices for lipid analysis in ancient ceramics) , in order to:

* identify molecules that are naturally present in the deposition environment
* rule out ceramic samples with similar or lower amounts of these molecules than the sediments
* calculate %TA, to more reliably identify grapevine products.

Using this cautious approach, our exploratory study confirmed that grape products, and probably wine, have been transported in amphorae and contained in domestic vessels found at these sites. Based on both chemical and textual evidence, we were able to show that grapevine products were largely mixed with other commodities (e.g. fruit juice and honey) and that transport amphorae were filled with mixed contents and/or were reused even when found in homogeneous cargo assemblages. In some cases, the assumed contents were confirmed by molecular analysis, as in the case of some Chian amphorae from the Mazotos shipwreck.

The use of a reliable methodology makes it possible to show that some vessels traditionally associated with wine probably did not contain grapevine products, but rather other fruit-based products (*oinochoe* P.94.49/BRI\_12) or plant oil (Samian amphora 551 P46/BRI\_95). It was also possible to reliably identify grapevine products in vessels where they were not expected (e.g. *unguentarium* P.14.02/BRI\_28). These results show that the relationship between form and content is a complex issue that should be addressed in the future with large-scale studies following for example Woodworth et al., 2015 for Keay 25 amphorae, and not on the basis of only a few ceramic specimens.

Ancient shipwreck sites offer excellent opportunities to conduct such large-scale studies. The identification of the contents through organic residue analysis, which augments the study of the macroscopic contents, confirms that transport amphorae were multipurpose containers (Knapp and Demesticha 2017), and we should not presuppose the same content in all amphorae of the same type in a single shipment. Even seemingly identical amphorae recovered from the same shipwreck site can contain a variety of goods. As discussed by Peña (2007) and Lawall (2011a), there is ample archaeological evidence for irregular and non-standard contents found inside of amphorae. While the contents of amphorae have been widely discussed, the evidence for wine in ‘wine’ pitchers, kraters, *lagynoi,* and *oinochoai* has been less explored. Molecular evidence further broadens this range of content diversity by incorporating these domestic ‘wine’ vessels as well as amphorae, and detecting plant oil, and fruit juice or honeyed beverages. This has implications for our understanding of domestic ‘wine’ vessels, which in turn can inform debates surrounding daily life, the history of food, and agricultural productivity. Moreover, as all the examined samples come from the same island, the results contribute to our knowledge about imported and consumed goods during the Classical and Hellenistic periods.

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