Chemical evidence for the persistence of wine production and trade in Early Medieval Islamic Sicily

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Although wine was unquestionably one of the most important commodities traded in the Mediterranean during the Roman Empire, less is known about wine commerce after its fall and whether the trade continued in regions under Islamic control. To investigate, here we undertook systematic analysis of grapevine products in archaeological ceramics, encompassing the chemical analysis of 109 transport amphorae from the fifth to the eleventh centuries, as well as numerous control samples. By quantifying tartaric acid in relation to malic acid, we were able to distinguish grapevines from other fruit-based products with a high degree of confidence. Using these quantitative criteria, we show beyond doubt that wine continued to be traded through Sicily during the Islamic period. Wine was supplied locally within Sicily but also exported from Palermo to ports under Christian control. Such direct evidence supports the notion that Sicilian merchants continued to capitalize on profitable Mediterranean trade networks during the Islamic period, including the trade in products prohibited by the Islamic hadiths, and that the relationship between wine and the rise of Islam was far from straightforward.

transport amphorae | wine | organic residue analysis | Late Antiquity and Early Middle Ages Sicily | provenance and trade

Sicily was described by the tenth century Palestinian geographer al-Muqaddasī as "the profitable island," and new archaeological research is enhancing the evidence for its commercial prosperity, especially in the tenth to eleventh century (1–3). There is increasing evidence that trade remained active in the centuries following the fall of the Western Roman Empire, as Sicily emerged as a key commercial center. Transport amphorae produced in Sicily during the Islamic period are found throughout the central Mediterranean (e.g., refs 4–6), and a wide variety of goods were likely to have been traded with Sicilian merchants at this time, including edible commodities such as salted fish, vegetable oils, dairy products, fruits, spices, and sugar (4, 7–9). But it is not clear whether the major political and economic upheaval during the Byzantine–Islamic transition had an impact on the traded commodities themselves.

Wine was certainly one of the major high-value goods traded in the Roman and Byzantine periods (10–12). Some scholars consider that its production and trade dramatically decreased after the Islamic conquest of the island because of hadithic prohibitions (13, 14). The well-documented existence of viticulture during the Islamic period (13, 15) may instead have been oriented toward table grapes, raisins, and vinegar, which are widely used in Islamic cuisine (e.g., refs 16, 17). In contrast, the continuation of wine production in Islamic Sicily is also suggested by some sources (15), although the extent of production is hard to determine. A tax on wine is reported when the island was under the Fatimid rule (18), which suggests that it continued to be traded and of economic significance, but the volume and destination of this commerce is largely undetermined.

Indeed, the equation between the transportation of wine and the rise of Islam is likely to be far from simple and most likely fluctuated between the seventh and the thirteenth century. Perhaps our best source of evidence comes from transport amphorae which can often be provenanced by their form and composition to specific origins to reveal potential trade routes (2). In the sixth and seventh centuries, commodities carried in Late Roman type 5 through 7 amphorae, produced in the eastern Mediterranean, were reaching destinations in the Aegean, Adriatic, and Tyrrhenian seas. Some of these are thought to have carried wine (2). At the beginning of the eighth century, the Emir of the Theban region was ordering wine from Apollonopolis to supply other destinations in Egypt, including Fustat, and his cook was receiving a consignment of wine according to a document in the Christian monastery of Baouît (2). In the tenth and eleventh centuries, an important new amphora production center rose at Palermo while at the height of Islamic control, supplying commerce to North Africa and the Tyrrhenian Sea area, notably Sardinia (6). The Norman conquest of Sicily in 1061 AD is thought to mark a revival of viniculture (14), and wine is again considered a major Sicilian export after this date (4). In the twelfth and thirteenth centuries, new types of amphorae handled bulk supply in the Aegean (e.g., Calchis), but Sicily loses its primacy as an exporter and becomes a net importer, in the face of diverse and rising centers of production on the Italian peninsula (2).

Significance

As a high-value luxury commodity, wine has been transported across the Mediterranean since the Bronze Age. The wine trade was potentially disrupted during political and religious change brought about by Islamization in the Early Medieval period; wine consumption is prohibited in Islamic scripture. Utilizing a quantitative criterion based on the relative amounts of two fruit acids in transport amphorae, we show that wine was exported from Sicily beyond the arrival of Islam in the ninth century, including to Christian regions of the central Mediterranean. This finding is significant for understanding how regime change affected trade in the Middle Ages. We also outline a robust analytical approach for detecting wine in archaeological ceramics that will be useful elucidating viniculture more broadly.

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Deciphering the wine trade from the distribution of amphorae and the few documents available is, however, far from straightforward without knowing their contents. In the absence of visible residues, marks, or labels, chemical analysis of organic compounds absorbed into the walls of amphorae offers the only direct approach for assessing changes in the commodities traded during this period. Although some studies have begun to explore the contents of amphorae exchanged in Sicily in the Early Middle Ages (19–21), no large-scale investigation has been carried out to date. Furthermore, the identification of wine through chemical analysis remains controversial (e.g., ref. 22) and particularly prone to false positive identification (23). In the absence of other archaeological or historical data to confirm interpretations, application of a robust methodology including quantification of target molecules and the use of appropriate controls is essential, particularly to distinguish wine from other fruit-based products. In the context of Islamic Sicily, this is especially pertinent, as a range of fruits, their juices, and syrups, are known to have been exported (9, 15, 19). For this reason, previous reports of wine in Islamic amphorae (19–21) need to be interpreted cautiously. In one of the largest studies of its kind, here we present the analysis of more than 100 amphorae produced or imported in Sicily between the fifth and the eleventh century AD. We propose a quantitative criterion for the identification of grapevine products using the relative concentration of tartaric acid (TA) to malic acid (MA) as a proxy, validated on more than 80 control samples.

Results and Discussion

A total of 109 amphorae that were produced or imported in Sicily from the Late Roman period to the Early Middle Ages were selected from the assemblages of 17 Italian and North African sites (Table 1 and Fig. 1 and *SI Appendix*, Fig. S1). Knowledge of provenance (i.e., place of production), identified based on the typological characteristics and the petrographic composition of ceramic pastes (4, 6, 24) (*SI Appendix*, Table S1 and Fig. S1), and place of discard allowed us to distinguish the following four groups (Table 1): 1) amphorae that were found close to the center of production (local trade), 2) those produced in Sicily and exported within the island (Sicilian trade), 3) those produced in Sicily and exported outside the island (overseas export), and 4) those produced elsewhere and imported into the island (import). To facilitate comparison over time, the samples were divided into three chronological groups: the Late Roman and Byzantine periods (fifth to seventh century), the transition from the Byzantine to the Islamic period (eighth to ninth century), and the Islamic period (tenth to eleventh century). Notably, only a limited number of samples were available from the eighth to ninth centuries, reflecting the scarcity of ceramic assemblages in this period (4, 25).

Of the entire sample set, only two containers show visible residues on the inner surfaces (*SI Appendix*, Table S1) that indicate sealing with plant exudates (resin, pitch, etc.), a feature commonly used to putatively identify wine amphorae in classical antiquity (e.g., ref. 26). To facilitate the robust identification of wine, we undertook comparative analysis of control samples from similar contexts that would not have been expected to have come into contact with grapevine products, satisfying the stringent criteria outlined by Drieu et al. (23). In this case, we used cooking pots from the same contexts and, where available, wall and floor tiles and sediments (Table 1). The results were compared with control samples from replica potsherds impregnated with wine and degraded for 1 y through burial under controlled conditions and samples of archaeological pottery with known contents (*SI Appendix*, Table S2).

Criteria for the Identification of Wine. TA was yielded in 69 amphorae (63%) in varying amounts (Fig. 2 and *SI Appendix*, Fig. S2 and Table S3). Additional small organic acids were identified in most of the amphorae and controls, including malic (82% of samples), succinic (54%), fumaric (15%), maleic (10%), malonic (7%), and oxalic (5%) acids. TA was also detected in many control samples (cooking pots, sediments, and tiles) but only at low concentration ($< 0.7\ \mu g \cdot g^{-1}$) in all but two domestic cooking vessels (3.2 and 1.4 $\mu g \cdot g^{-1}$; Fig. 2 and *SI Appendix*, Fig. S2). Overall, the transport amphorae had significantly higher TA concentrations than the control sample set (Mann–Whitney *U* test: W = 5,602; $P < 0.01$), implying a difference in use (Fig. 2).

However, the detection of TA alone is insufficient to provide definitive evidence for the presence of wine, as this compound is present in many other fruits (23, 27, 28). In grapes, the proportion of TA increases with ripening while the proportion of MA decreases correspondingly (29, 30). Although the absolute amounts of both acids are dependent on the growing conditions [temperature, hydrological state, exposure to sunlight, etc. (30–32)], we are able to exploit their relative concentrations to distinguish grapevine products. A comparison of TA and MA for the identification of wine and other fruit products in an archaeological context has been noted before (33, 34), but neither quantitative data nor interpretative ranges have been reported. Consideration of authentic reference products from the literature shows that the median % tartaric acid (%TA), expressed as the amount of TA divided by the sum of TA and MA, is significantly higher in ripe grapes and grape products compared to other fruits (Mann–Whitney *U* test; W = 136,452, *P* value < 0.01), with the exception of tamarind (Fig. 3*A* and *SI Appendix*, Table S4). Fruits other than grape and tamarind have a median %TA of 7% compared to 63% for ripe grape products. The lower limit (fifth percentile) of the %TA range for ripe grape products is 35%, and over 90% of the published data for fruits and berries ($n = 163$; excluding unripe grape, pomegranate, and tamarind) have %TA below this value.

To test the robustness of this criterion, eighteenth and twentieth century Georgian qvevri, traditionally used for wine production, were analyzed. These vessels yielded %TA within the range of grapevine products (i.e., %TA > 35%; Fig. 3*C* and *SI Appendix*, Table S2). Similarly, the %TA obtained from experimental pots soaked in wine and buried for 1 y under different environmental conditions also remained within the range of grapevine products, despite some alteration in the ratio when compared to the nondegraded control (Fig. 3*B*). It cannot be excluded that degradation of fruit products, other than grapes, may lead to an increase in %TA. However, food crusts containing Viburnum berries found on the surface of Russian hunter-gatherer pottery (23, 35) show a %TA below the range for grapevine products (Fig. 3*C*), giving confidence to the use of this criterion on archaeological samples of unknown content.

Among all the transport amphorae studied, 21 show %TA > 35%, which corresponds to the range of grapevine products (Fig. 3*C*). Interestingly, all of them yielded $>0.3\ \mu g \cdot g^{-1}$ of TA, i.e., greater than all the tiles and the majority (79%) of cooking pots. The use of these amphorae to transport wine is, therefore, highly likely given the context and prior historical knowledge, although the storage or transport of vinegar, grape syrup, pomegranate, or tamarind cannot be excluded. Indeed, many of these products are mentioned in the cuisine and pharmacopoeia of the Late Antique and Early Medieval Mediterranean (e.g., refs 13, 16, 17, 36, 37) but are overwhelmingly considered less likely to be commercial commodities transported in amphorae. Hereafter, we therefore consider transport amphorae with %TA > 35% to have contained wine. It is important to note that the same rationale cannot be applied to cooking pots or amphorae produced and discarded locally (i.e., potential storage amphorae), as we cannot be sure that wine rather than other grapevine products (vinegar, grape syrup, etc.) were processed in these vessels.

Almost all of the cooking pots and 88 amphorae show %TA ≤ 35%, with varying yields of TA (Fig. 3*C*). The TA in these

Table 1. Overview of the archaeological samples examined in this study

Site	Region	Period group	Transport amphorae	Provenance group*	Control samples
Excavation Gentili (Piazza Armerina)	Sicily	Fifth to seventh	3	nd	
Valle dei Templi, Quartiere Ellenistico (Agrigento)	Sicily	Fifth to seventh	8	Imports (Tunisia) and Sicilian trade (nd)	
San Miceli (Salemi)	Sicily	Fifth to seventh	13	Imports (Tunisia)	
Mazara del Vallo	Sicily	Fifth to seventh	3	Imports (Tunisia)	
		Tenth to eleventh	22	Imports (Tunisia), Sicilian trade (Palermo), and local trade	
Rocchicella di Mineo-Paliké (Mineo)	Sicily	Eighth to ninth	3	Imports (Aegean) and local trade	
Catacombe di Siracusa	Sicily	Eighth to ninth	1	Imports (Aegean)	
Casale San Pietro (Castronovo di Sicilia)	Sicily	Eighth to ninth	2	Sicilian trade (nd)	23 cooking pots, 7 tiles, and 4 sediments
		Tenth to eleventh	7	Sicilian trade (Palermo)	
Santa Maria degli Angeli, detta della Gancia (Palermo)	Sicily	Tenth to eleventh	5	Imports (nd) and local trade	18 cooking pots
Castello San Pietro (Palermo)	Sicily	Tenth to eleventh	5	Local trade	18 cooking pots
Palazzo Bonagia (Palermo)	Sicily	Tenth to eleventh	10	Local trade	15 cooking pots
Piazza Armerina, Islamic village	Sicily	Tenth to eleventh	1	Sicilian trade (nd)	
Althiburos	Tunisia	Tenth to eleventh	1	Oversea export (Palermo)	
Castello Brina (Sarzana)	Northern Italy	Tenth to eleventh	2	Oversea export (Palermo)	
Stazione Universita''', Piazza Bovio (Naples)	Southern Italy	Tenth to eleventh	1	Oversea export (Palermo)	
Via Cavalca (Pisa)	Northern Italy	Tenth to eleventh	4	Oversea export (Palermo)	
Via Sapienza (Pisa)	Northern Italy	Tenth to eleventh	4	Oversea export (Palermo)	
Largo delle Monache Cappuccine (Sassari)	Sardinia	Tenth to eleventh	13	Oversea export (Palermo)	

*The origin of the pots, identified based on the typological characteristics and the composition of ceramic pastes, is indicated in parentheses. nd: no data, the available data do not provide information on the origin of the amphorae. More detailed information on amphora type and dates is available in *SI Appendix*, Table S1 and Fig. S1.

samples may be derived from unripe grape products or other fruits (e.g., black currants, blackberries, mulberries, raspberries, cherries, or some types of pomegranate). It is important to note that for amphorae with $\%TA \leq 35\%$, we are not able to exclude wine if it was mixed with other products containing MA (e.g., honey, other fruits, etc.) as was common in the Roman period [e.g., addition of honey to sweeten wine; (38)]. Similarly, the reuse of amphorae (e.g., for transporting wine and then other fruit juices) would reduce the %TA value leading to false negative identifications. However, subsequent reuse for transporting olive oil would not be expected to substantially alter the %TA value. The use of fruits likely explains the presence of TA and MA, sometimes in substantial amounts, in Sicilian cooking pots, in keeping with Islamic recipes available from this period (e.g., refs 17, 39). Small amounts of TA and MA (respectively around 0.1 and $1 \mu g \cdot g^{-1}$) are present in both wall and floor ceramic tiles, always with $\%TA < 25\%$ (Fig. 3*C*), most likely indicating contamination from the burial environment. Amphorae and cooking pots that yielded less TA and MA than found in these control samples, therefore, cannot reasonably be interpreted as containers of wine or fruit products.

The Sicilian Wine Trade through Time. Having established this robust criterion for the identification of wine in amphorae, we now turn to comparison of their use through time (Fig. 4*A*). First, wine was identified in all periods regardless of the political regime in power. The low number of samples available from the eighth and ninth centuries precludes identification of a specific pattern, but even during this turbulent period, it is clear that wine was also traded within Sicily. By far the most surprising result is that wine was also used in the tenth and eleventh centuries, when Sicily was under full Islamic control. A group of Sicilian-made amphorae, representing 15% of the total analyzed from this period, is clearly distinguished with a $\%TA > 35\%$ (Fig. 4*A*).

During the Islamic period, petrographic analysis shows that Palermo was the main production center for amphorae found in Sicily and Palermitan amphorae are also found throughout the central Mediterranean (e.g., refs 5, 6, 24). Five of the amphorae that contained grapevine products during the Islamic period were produced and discarded in Palermo (Fig. 4*B*). This finding is interesting since Palermo was under full Islamic control, and our results may indicate that these vessels were used for local

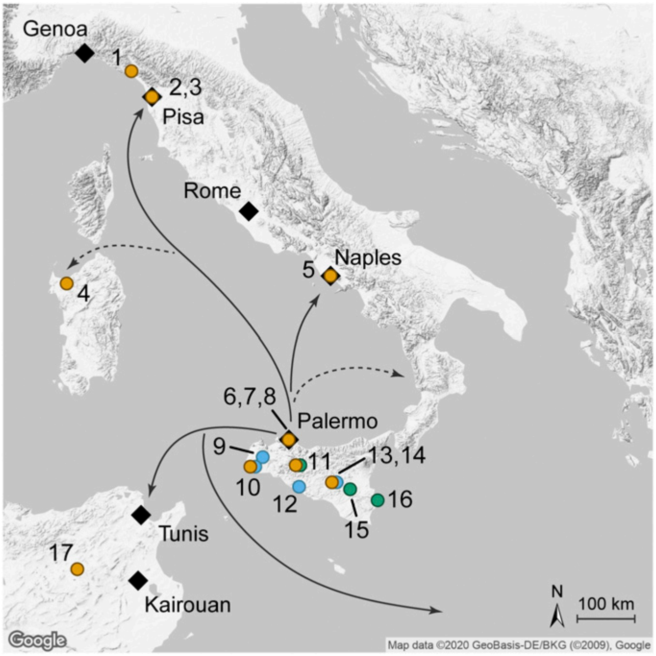

Fig. 1. Map of all the sites studied and details on the Sicilian trade routes during the Islamic period. The sites are shown by period; fifth to seventh century (blue circles); eighth to ninth century (green circles); and tenth to eleventh century (orange circles): Castello Brina (1); Via Cavalca (2); Via Sapienza (3); Largo delle Monache Cappuccine (4); Stazione Universita'', Piazza Bovio (5); Santa Maria degli Angeli, detta della Gancia (6); Castello San Pietro (7); Palazzo Bonagia (8); San Miceli (9); Mazara del Vallo (10); Casale San Pietro (11); Valle dei Templi, Quartiere Ellenistico (12); Piazza Armerina, Islamic village (13); Piazza Armerina, Excavation Gentili (14); Rocchicella di Mineo-Paliké (15); Catacombe di Siracusa (16); and Althiburos (17). Black diamonds indicate the main towns and ports in the central Mediterranean between the tenth and eleventh centuries, and the lines show the main direct (solid) and indirect (dashed) Sicilian maritime trade routes, according to the distribution of Palermo's pottery production and historical documents (6, 8).

transport or storage of wine vinegar or grape syrup rather than wine; the former was widely used in medieval Islamic cuisine, as a preservative, or for medicinal purposes (e.g., refs 17, 36, 37, 40). However, wine cannot be excluded and equally may have been produced for consumption by the Jewish and Christian communities still present in Sicily at the time (13, 41, 42) or by some members of the Muslim community, as is discernible from medieval Islamic poems (13, 41). No traces of wine were found in amphorae exported to inland Sicily, but surprisingly, grapevine products were identified in several Palermitan amphorae exported overseas to Christian mainland Italy and Sardinia (Fig. 4*B*). Therefore, by using a combination of analytical approaches aimed at provenance and use on a large corpus of amphorae, we can begin to reveal the extent of a Sicilian wine trade network that appears to encompass the city of Palermo itself and also the central Mediterranean. Of course, it is difficult to estimate the volumes of wine trade, not least as wine and grapevine products may also have been stored or transported in perishable organic containers, such as barrels or skins, which do not survive in the archaeological record (43).

It is important to note that wine was not the only product transported in the amphorae manufactured and imported into Sicily between the fifth and eleventh centuries. Degraded lipids from various fats and oils were identified in 75% of the amphorae analyzed, the majority of that also contained wine, suggesting extensive reuse of these containers, as has been previously suggested (e.g., ref. 44). Significant lipid degradation, and the potential for extensive mixing, precludes further identification in the majority of cases, with profiles dominated by saturated fatty acids. Two amphorae from the fifth to seventh century and three from the tenth to eleventh century contained more distinctive fatty acid profiles with a high relative abundance of oleic acid ($C_{18:1}$) and palmitic acid ($C_{16:0}$) compared to stearic acid ($C_{18:0}$; $C_{18:1}/C_{18:0} \geq 1.5$ and $C_{16:0}/C_{18:0} \geq 2$; *SI Appendix*, Table S1) and are broadly attributed to vegetable oils (45). We undertook individual carbon stable isotope measurements of fatty acids of all of the amphorae, and based on this evidence, we were able to exclude marine products, which have fatty acid $\delta^{13}C$ greater than −27‰ (46), in all but one amphora from the fifth century and two amphorae from the tenth to eleventh centuries (*SI Appendix*, Fig. S3 and Table S3). Therefore, fermented fish sauces and pastes, such as garum, liquamen, or salsamenta, do not seem to have been a major trade commodity during this period.

Finally, the presence of diterpenes and their degradation products derived from Pinaceae resin and pitch (47) were far less abundant in Islamic amphorae (5% of samples) compared to Late Roman and Byzantine periods (60%). Resin linings and sealants are thought to aid waterproofing or help preserve the contents and were frequently applied to Mediterranean amphorae during the Classical and Late Roman periods (e.g., refs 48–50). The presence of undetermined fats or oils in the majority of amphorae could be because of an alternative waterproofing method, as has previously been suggested for amphorae of the same period (19, 20). It is not clear whether this change in practice is unique to the Islamic period or whether it is specific to Sicilian production.

Conclusion

Using a quantitative approach for distinguishing ripe grape products from other fruits, here we provide compelling evidence that the production and trade in Sicilian wine continued into the Islamic

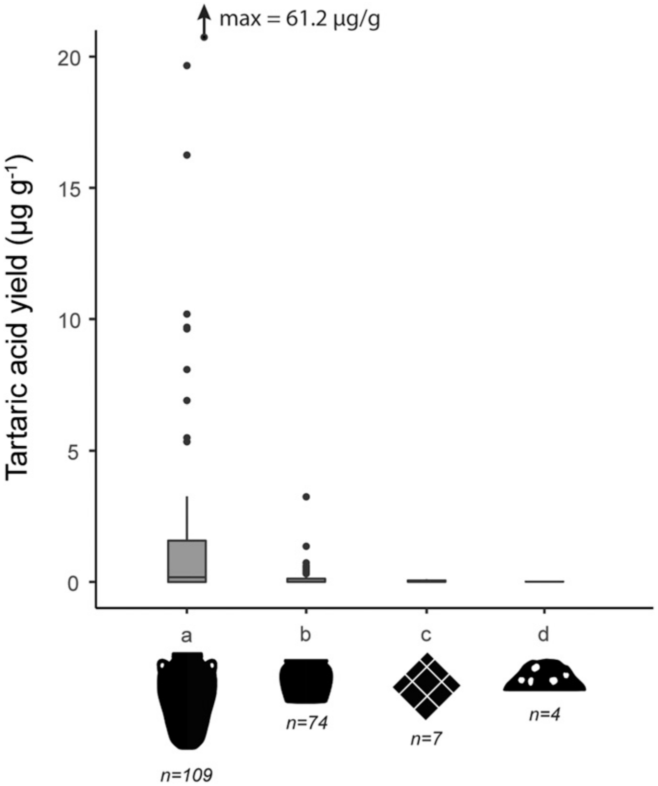

Fig. 2. Extraction yields of TA in transport amphorae and control samples. (*A*) Transport amphorae; (*B*) Cooking pots; (*C*) Tiles; and (*D*) Sediments. The number of samples analyzed is shown in italics

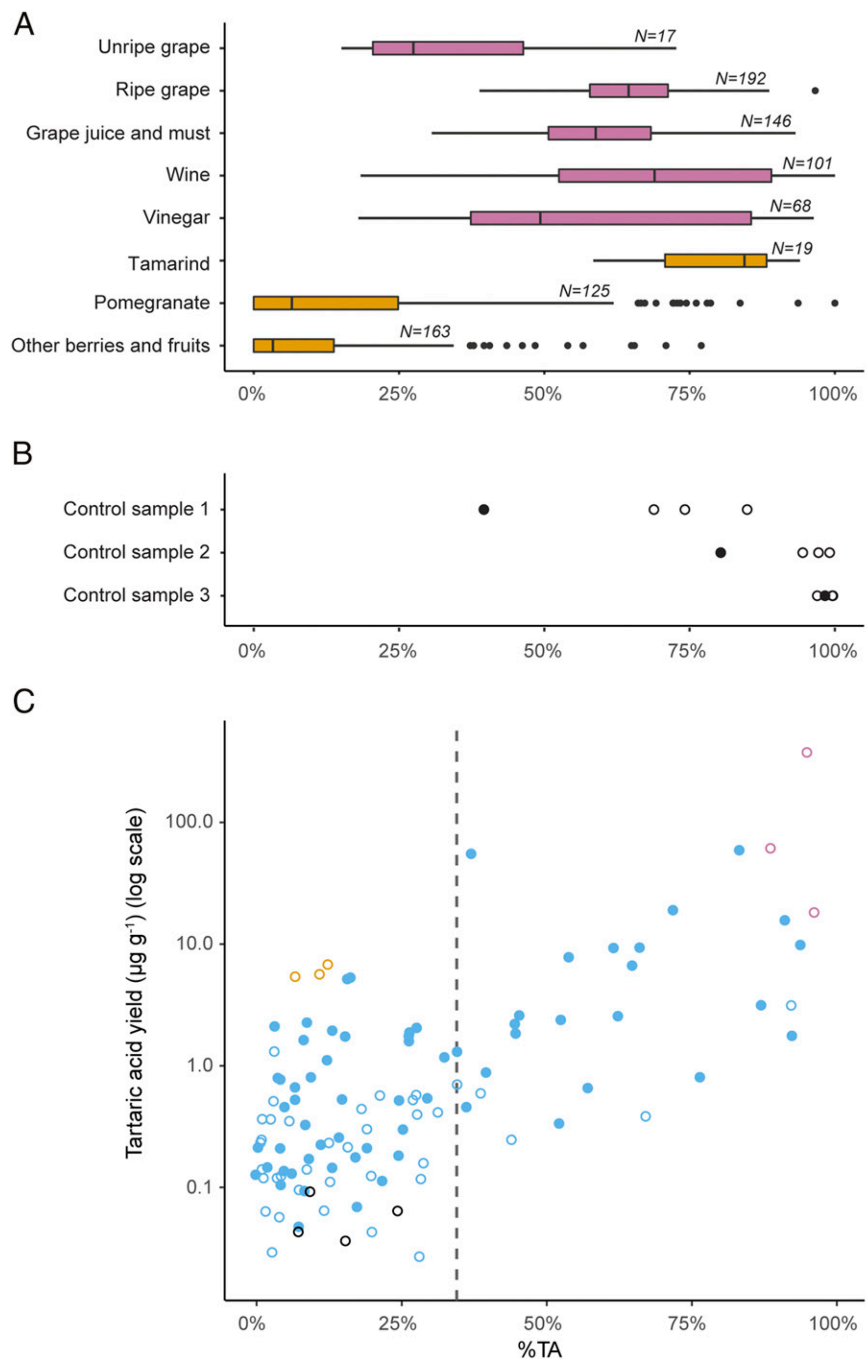

Fig. 3. Results of TA and MA analysis in Early Medieval amphorae and control samples. (*A*) Box plots of %TA, expressed as the % contribution of TA to the sum of TA and MA, in various fruits and fruit products (data from the literature, detailed in *SI Appendix*, Table S4). The number of samples considered is shown in italics. (*B*) %TA in experimental pots used to contain wine (filled circles) and degraded in different environmental contexts for 1 y (open circles). (*C*) %TA in archaeological samples, plotted versus the amount of TA extracted ($\mu g \cdot g^{-1}$, logarithmic scale) in amphorae (blue filled circles), cooking pots (blue open circles), tiles (black circles), Georgian qvevri (pink circles), and Viburnum food crusts (Zamostje, Russia; Bondetti et al., 2020; yellow circles). The vertical dashed line indicates the %TA value of 35%. Archaeological samples yielding <0.05 $\mu g \cdot g^{-1}$ TA are not shown in this figure but are reported in *SI Appendix*, Table S3.

period and, therefore, were not substantially affected by the political and religious changes in Sicily between Late Antiquity and the Early Middle Ages. These results do not necessarily imply that Islamic prohibitions (51) were not strictly observed on the island, as wine may have been produced and traded for the benefit of non-Muslim communities in Sicily and elsewhere. We found evidence that wine was exported from Palermo under Kalbid rule to the Christian regions of the Mediterranean, demonstrating continuity of the wine trade, at least since the Byzantine period when the great Sicilian estates supplied Rome with wine via the port of Palermo (52). The volumes of wine traded are difficult to discern using this approach as a range of other commodities were also transported to and from Sicily at this time in similar containers, including vegetable oils, and the organic residue analysis shows evidence of reuse. Nevertheless, there is little direct evidence to suggest that the Mediterranean wine trade decreased under Islamic control as has often been assumed,

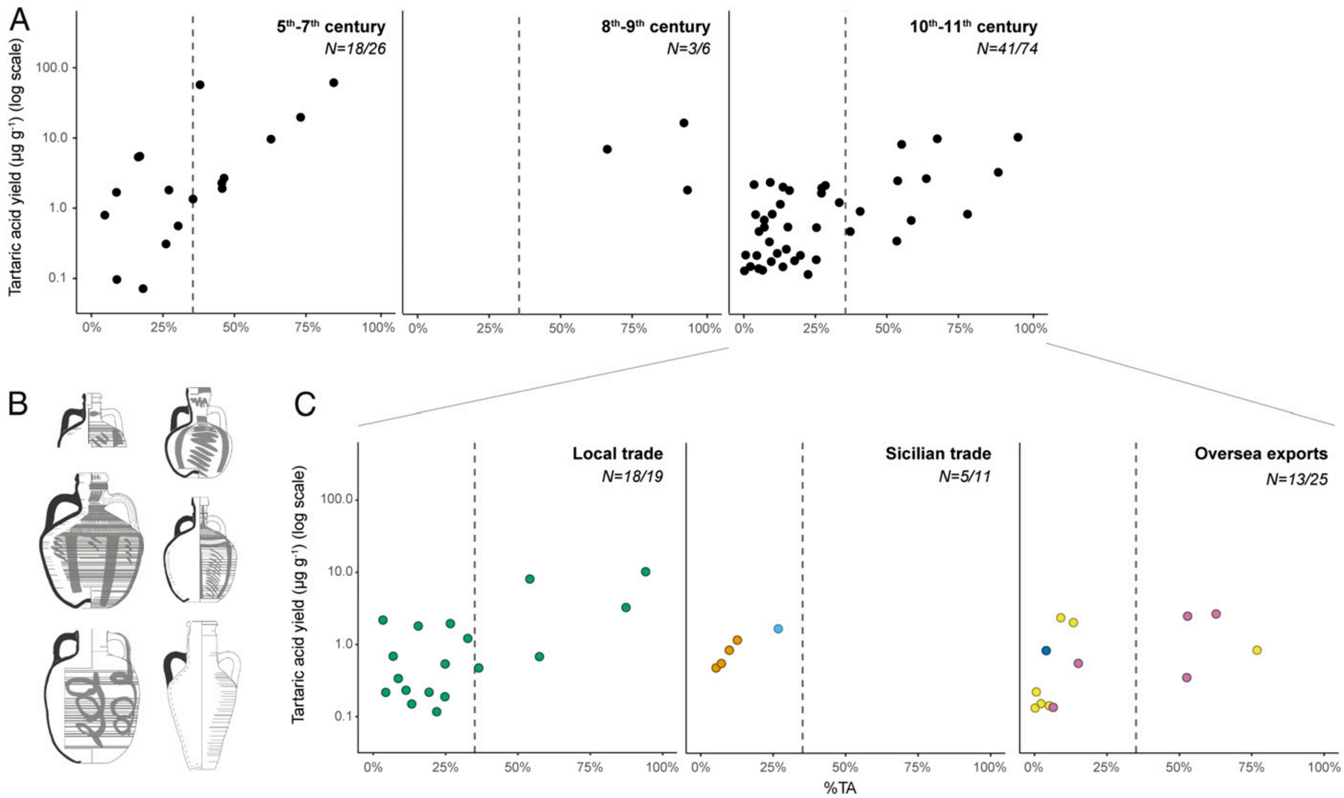

Fig. 4. Results of TA and MA analysis in amphorae by chronological period. (*A*) %TA plotted against the amount of TA extracted (µg · g^{-1}, logarithmic scale) in transport amphorae from the fifth to seventh century, eighth to ninth century, and tenth to eleventh century. (*B*) Examples of typical Palermitan amphora forms (from ref. 6). (*C*) %TA plotted against the amount of TA extracted (µg · g^{-1}, logarithmic scale) in Palermitan amphorae from the tenth to eleventh centuries found in Palermo (green), Castronovo di Sicilia (orange), Mazara (light blue), Sardinia (yellow), Tuscany (pink), and Tunisia (dark blue). The type of trade is derived from the place where the amphorae were made, the location where they were found, and their date. Samples yielding < 0.05 µg · g^{-1} TA are not shown in this figure but are reported in *SI Appendix*, Table S3. The number of samples yielding both MA and TA in relation to the total number of samples analyzed is indicated in italics. The dotted gray line indicates the %TA value of 35%.

rather Islamic merchants benefited from new markets satisfying the Christian demand for Sicilian wine, a trade that must have been approved by the Kalbid emir. Finally, we note that only by using our more robust quantitative criterion we can distinguish grapevine products and other fruits. Indeed, 69% of Sicilian amphorae and 70% of the cooking pots we tested contained TA, but only a small fraction of these could be accurately assigned to wine, avoiding false positive identifications. We recommend that this quantitative criterion should now be used to identify the presence of grapevine products in archaeological pottery, particularly in contexts in which wine production is disputed (e.g., to study the origins of viniculture).

Materials and Methods

Degradation of Authentic Wine in Pottery. Three replica pots were filled with different wine obtained from commercial producers for 2 d (*SI Appendix*, Table S2). One potsherd from each pot was directly analyzed after being emptied and dried. Other potsherds were buried for 12 mo in different environments in order to evaluate the degradation of wine molecules in different climatic conditions and soil pH: the archaeological site of Casale San Pietro in Castronovo di Sicilia (latitude 37.68, longitude 13.63; September 2018 to September 2019), a field in the south of France (Eze, Alpes–Maritimes; latitude 43.73, longitude 7.36; November 2018 to November 2019), and at the York Experimental Archaeological Research (YEAR) Centre at the University of York (United Kingdom; latitude 53.94, longitude −1.06; November 2018 to November 2019).

Experimental Approach. Following the most recent publications in terms of identification of grapevine products (23, 53), two successive extractions were used. Approximately 2 g ceramics were drilled into the inner walls of the potsherds, after removal of the outer surface (1 to 2 mm), to remove contamination from the surrounding sediments and from the handling. Then, 10 µg internal standard (n-C_{34}) was added to 1 g powder, which was then extracted three times with dichloromethane/methanol (DCM/MeOH; 2:1, vol/vol) in an ultrasonic bath. The successive extracts, that contained lipids and resin acids (terpenes), were combined and evaporated under a nitrogen flow. The powder remaining after extraction with DCM/MeOH was treated with a boron trifluoride-butanol/hexane mixture (1:2, vol/vol) for 2 h at 80 °C to extract any butylated small organic acids, in particular MA and TA. The samples were centrifuged, and the supernatants were neutralized with a saturated sodium carbonate solution. The samples were then extracted three times with DCM and washed twice with distilled water before being evaporated under a stream of nitrogen. All samples were derivatized with *N,O*-Bis(trimethylsilyl)trifluoroacetamide, 1% trimethylchlorosilane. After evaporation under nitrogen flow, 10 µg internal standard (n-C_{36}) was added, and the samples were dissolved in hexane before injection in gas chromatography-mass spectrometry (GC/MS). The untreated powder (about 1 g) was sonicated for 15 min in 4 mL methanol, before adding 80 µL sulphuric acid and heating at 70 °C for 4 h (54). The methylated lipids were extracted three times in hexane before analysis in GC/MS. Samples with sufficient lipids (>10 µg · g^{-1}) were injected in gas chromatography-combustion–isotope ratio mass spectrometry (GC-C–IRMS), to study the stable carbon isotope composition of palmitic and stearic acids and to verify the presence of marine fats.

Instrumentation. The analyses were performed on an Agilent 7890A chromatograph, equipped with a DB5-HT column (30 m × 0.25 mm internal diameter, 0.1 µm film thickness, Agilent J&W), via splitless injection. The temperature program was as follows: the oven was maintained at 50 °C for 2 min, then the temperature was raised to 325 °C at 10 °C min^{-1} and held for 15 min. The mass spectrometer used was an Agilent 5977B, used in electron ionization mode (EI, 70 eV), with mass spectra acquisition between *m/z* 50 and 1,000. The presence of TA was identified from the mass spectrum of trimethylsilylated TA dibutyl ester (*m/z* 147, 276, and 391) (53). In some samples, a peak of trimethylsilylated TA methyl butyl ester (*m/z* 147, 234, 276, and 349), resulting from the reaction with residual methanol from the DCM/MeOH extraction, was also considered for quantification. Other small acids were also identified from the mass spectrum of their trimethylsilylated dibutyl ester: malic (*m/z* 145, 161, 173, 217, and 303), succinic (*m/z* 101 and 157), fumaric and maleic (*m/z* 99, 117, 155, and 173), malonic (*m/z* 87, 105, and 143), and oxalic (*m/z* 57, 87, and 130) acids. GC-C–IRMS analyses were performed using a Hewlett Packard 7890B series gas chromatograph (Agilent Technologies) with an Isoprime GC5 interface coupled to an Isoprime 100 IRMS. The carrier gas (helium) was used at a constant flow rate of 3 mL/min. The samples were analyzed in a DB-5MS fused silica column (60m × 0.25 mm × 0.25 μm; J&W Scientific), after injection of 1 μL sample via a splitless injector at 300 °C. The eluted compounds were ionized by electronic impact (70 °C). The $^{13}C/^{12}C$ ratio of each peak was calculated from measurements of the ion intensities of *m/z* 44, 45, and 46. The calculations were carried out by comparison with measurements of a standard reference gas (CO_2), and the results are expressed compared to the international standard Vienna Pee Dee belemnite, in *m/z* (‰).

Data Availability. All study data are included in the article and/or *SI Appendix*.

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