



## Palaeobotanical, chemical and physical investigation of the content of an ancient wine amphora from the northern Tyrrhenian sea in Italy



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### ARTICLE INFO

#### Article history:

Received 20 April 2013

Received in revised form

20 February 2014

Accepted 25 February 2014

Available online 12 March 2014

#### Keywords:

Dressel 1B amphora

Wine residues

Archaeobotany

Minerals

Shipwreck

Stable isotopes

### ABSTRACT

Elemental, isotopic and archaeobotanical analysis were performed on the contents of an intact Dressel 1B amphora from a Roman *navis oneraria* shipwreck dating back to 100–90 BC, discovered at a depth of 42 m near Albenga (Italy). Analysis aimed at assessing the origin and nature of the samples was carried out. The chemical characterisation, in particular lead content, was consistent with an oenological product produced using ancient techniques. The organic matrix was observed and shown to have deteriorated seriously, with infiltration of sea water and migration from the clay vessel. Isotope ratio mass spectrometry (IRMS) analysis confirmed that the residue was wine, excluding the possibility of the sediment coming from the sea or the clay of the amphora. As regards palynological analysis, the pollen components were useful for diagnosing and characterising the oenological nature and geographical origin of the sample from Albenga. The extremely high percentage of grape pollen suggested that the liquid traded was must or wine subjected to little decantation. Finally, the presence of pollen from certain arboreal species widespread in central-southern Italy is in accordance with the opinion of archaeologists, who have suggested that the cargo originated in the centre-south of the Tyrrhenian area.

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### 1. Introduction

Remarkable underwater sites have been discovered in many parts of the world, the Mediterranean area being rightly recognised as one of the most fruitful locations for deep-water archaeology, due to the significant number of ancient shipwrecks discovered. Most of these are found intact, often with the contents in their original position, making it possible to understand the origin of the ship and to reconstruct the development of shipbuilding traditions (Tusa, 2009). The cargos, in particular, make key data available on trading routes and import and export centres, providing further knowledge on the heritage, culture, art, lifestyles, agricultural production, food consumption and diet.

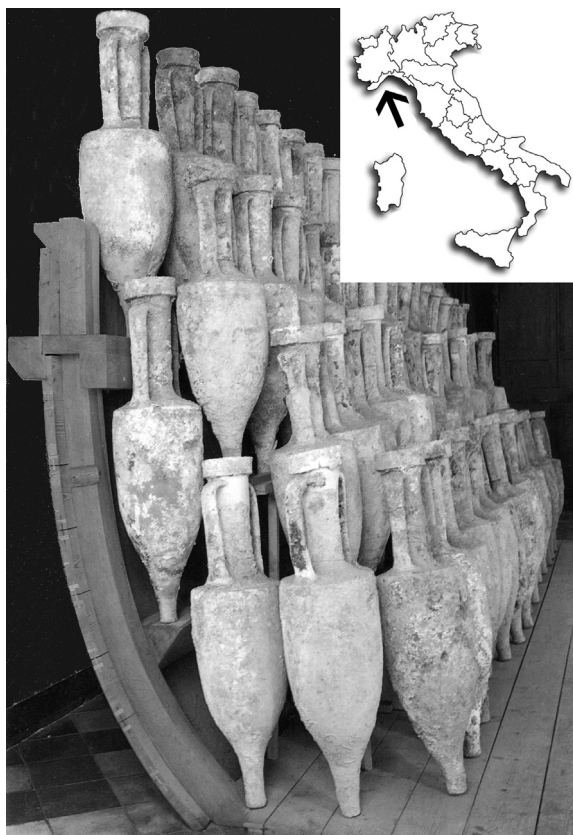
In cargo ships, amphorae are known to have been used to carry wine, olive oil, spices and fish products, as well as several other liquid or semi-liquid goods (Twede, 2002), and are extremely precious for deducing agricultural and food-related practices, on the basis – relatively infrequent – of direct archaeological evidence of residues (usually olive stones or resin linings) found inside, or – more frequently – the traces found in these relics (McGovern, 2003; Hansson and Foley, 2008).

This paper reports on research conducted on a liquid sample extracted from a Dressel 1B amphora recovered from the Albenga shipwreck archaeological site. This type of amphora is an ancient Roman container considered to have been used for the maritime transport of wine during the 2nd–1st centuries BC, and had a capacity of 26 l (Lamboglia, 1952; Pallares, 1983; Bruno, 2005). The Albenga shipwreck, one of the largest known (40 m long × 10 m wide) merchant vessels (*navis oneraria*) from the Roman Age ever to have been found in the Mediterranean basin, is still *in situ*, a mile from the coast, at a depth of 42 m, facing the ancient city of *Albingaunum*, today called Albenga, in the Liguria region in Italy (Fig. 1). On the basis of amphorae and other pottery artefacts

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**Fig. 1.** Location of the Roman shipwreck recovered from the coast of Albenga, Italy (arrow) and a replica of the cross-section of the merchant vessel with a cargo of Dressel 1B amphorae (Museo Navale in Albenga).

retrieved on board – including Campana A ware – the ship has been dated to 100–90 BC and it is assumed that it would have been travelling from a port in central-southern Italy in the Tyrrhenian region, to transport its load of over 10,000 amphorae (most of them Dressel 1B and a few Lamboglia 2) with a 450/500 metric ton net weight and other pottery to France and Spain (Lamboglia, 1952; Antika, 2012). The amphorae were still plugged with corks and sealed with lime-pozzolana paste, whilst some of them had a pinecone in the neck under the cork stopper (Fig. 2), according to a preservation strategy used in ancient wine trading (Twede, 2002).

The presence of this shipwreck has been known to fishermen since 1925 and in 1950 the archaeologist Nino Lamboglia attempted the first recovery of the precious cargo. This was followed by improved archaeological investigations in 1961. The around 700 amphorae were dragged out and stored in the Museo Navale in Albenga, Italy, making a significant contribution to the beginning of modern underwater archaeology (Pallares, 1983; Gandolfi, 2000).

In various geographical areas, amphorae and bottles still containing well-conserved or deteriorated wines have been rescued from shipwrecks dating back to the Middle Ages and the modern era (McGovern, 2003; Bertelli et al., 2004). However, the age of this material, while still impressively old, is not comparable with the ancient origin of the Albenga sample. To the best of our knowledge, this would seem to be one of the most ancient liquid samples recovered from a *navis oneraria* on the bed of the north-west Mediterranean sea. A liquid contained in a corked Lamboglia 2 amphora from a *navis oneraria* dated to the 1st century BC was found in 1967 in the sea at the port of Madrague de Giens (Var,

France) at a depth of –18 m. Chromatography testing of this sample was unsuccessful in determining whether this was wine (Formenti et al., 1978; Tchernia et al., 1978).

On the basis of this knowledge, we believe that the sample from the Albenga shipwreck is extremely precious and worth adopting for analysis of a deep-water archaeology finding. The analysis aimed to assess the nature of the sample, which on the basis of archaeological and historical evidence was expected to be wine. This paper presents the methodology used to reach this objective.

## 2. Material and methods

### 2.1. Sample origin

The Albenga sample was a 100 ml aliquot originating from a 1500 ml dark reddish brown liquid suspension (Munsell code n. 5YR2.5/2) contained in an intact Dressel 1B amphora (inv. n. 1008) sealed with lime-pozzolana paste with a cork stopper, recovered and stored at room temperature in the deposit of the Museo Navale in Albenga (Italy). To homogenise it, the amphora content was shaken and the remains were kept in a dark glass bottle.

### 2.2. Chemical and physical analysis

The Albenga sample was processed using glass and stainless steel materials washed with a 5% HNO<sub>3</sub> solution and rinsed with ultrapure water before use. The pH of the heterogeneous bulk sample was manually measured at 20 °C with a WTW pH meter (mod. 315i; Weilheim, Germany), while the sediment, separated by 15 min centrifugation at 4000 rpm (CL31 multispeed centrifuge, Thermo Scientific, USA) and washed with ultrapure water (Millipore, Bedford, MA), was observed through a stereomicroscope (SMZ 800; Nikon, Tokyo, Japan) with a colour digital camera at 150× magnification. The carbon/nitrogen ratio was measured on 100 mg of 60 °C dried sediment, prepared as above with a Vario Macro CN (Elementar, Hanau, Germania) elemental analyser. For elemental analysis, 500 mg aliquots of the bulk sample, homogenised by shaking, were directly weighed into PTFE vessels (MarsXpress, CEM, Matthews, USA), with the addition of 4 ml HNO<sub>3</sub> (69.5%, Merck, Darmstadt, Germany), 2 ml H<sub>2</sub>O<sub>2</sub> (30%, Merck), 1 ml of Ho 260 µg/l (prepared from standard solution 1 g/l; BDH, Pool, UK) was also added to the sample as internal standard. A blank sample was prepared using ultrapure water instead of the sample. The sample was digested under pressure with a MarsXpress microwave oven, (CEM) in a closed vessel, where the temperature was linearly increased up to 200 °C within 20 min and kept at that temperature for 20 min. After digestion, the solution was transferred from the vessels into a rinsed polypropylene test-tube, diluted to a final volume of 13.3 ml with Milli-Q water and stored for analysis. Li, Be, B, Na, Mg, Al, P, K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, Ge, As, Se, Rb, Sr, Y, Mo, Pd, Ag, Cd, In, Sn, Sb, Te, Cs, Ba, La, Ce, Pr, Nd, Sm, Eu, Gd, Dy, Ho, Er, Tm, Yb, Re, Ir, Pt, Au, Hg, Tl, Pb, Bi and U content was determined using an Inductively Coupled Plasma Mass Spectrometer (ICP-MS; Agilent 7500ce Agilent Technologies, Tokyo, Japan) filling the Octopole Reaction System with He gas in order to minimise polyatomic interferences on Na, Mg, V, Cr, Fe, Ni, Cu, Zn, and Eu, and with H<sub>2</sub> gas on Ca, Ga and Se. The accuracy of measurement was confirmed by using certified materials SRM 1547 (Peach Leave; NIST, Gaithersburg, MD, USA) and SRM 1547 (Wheat Flour; NIST) inside the analytical batch. Chloride, sulphate, and tartaric, malic and lactic acids were detected by isotocratic analysis with an ionic chromatograph DX-120 (Dionex Corporation, Sunnyvale, CA, USA), equipped with a 4 × 250 mm IonPac AS12A column (Dionex) using conductivity detection. The mobile phase was a Na<sub>2</sub>CO<sub>3</sub>/NaHCO<sub>3</sub> (8 mM:1 mM) solution. Amines and

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