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Geochemical study of the luting and coating of medieval watercraft from the ship timbers discovered in the citadel of al-Balīd, Oman: Composition and origin

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ABSTRACT

Ship timbers were discovered during the excavation of the Islamic site of al-Balīd recycled in the buildings of its citadel and the Great Mosque. These fragments stripped from the hulls of Indian Ocean medieval sewn vessels form the largest collection of archaeological evidence for the ships sailing during the Middle Islamic Period (10th–15th century CE). More than one third of the timbers bears traces of a bitumen mixture used as luting and coating to seal the ships planking to waterproof and protect their hulls, a practice generally associated with sewn-plank construction.

This paper presents the result of the GC-MS, carbon and hydrogen isotope on chromatographic fractions, and X-Ray diffraction analyses on twenty-two samples of the luting extracted from fifteen timbers from al-Balīd. The nature of the constituents of the amalgam is determined showing that the luting consists of bitumen mixed with mineral matter, fat or oil and two triterpenic resins, dammar (Dipterocarpacea) and frankincense.

The chemical signature of the bitumen indicates that it was likely sourced from two seeps in southwest Iranian: Mamatain and Ain Gir-Chersch Mehrgir-Dehluran. The origin of the bitumen might suggest that the luting was applied during the construction or repairing of Indian Ocean vessels in boatyards along the coasts of the Arabian/Persian Gulf or Southern and Eastern Arabia.

1. Introduction

Bitumen was an essential material in the history of the Arabian/Persian Gulf [1]since early times. People have collected this substance from natural seeps in Mesopotamia and Iran and used it for multiple purposes such as mortar, sealant, glue and waterproof agent (Connan 1999, 2012; Connan and Deschesne 1995). Because of its versatility, this material was widely traded in the Gulf since the Neolithic, as far as Oman in the Arabian Sea (Connan and Carter 2007; Connan and Van de Velde 2010).

Bitumen also played a crucial role in the development of boat building and seafaring in Mesopotamia and the Gulf. Coastal communities used this substance to waterproof the hulls of their reed and wooden vessels, prompting both regional and international maritime trade, and enabling the flow of goods between Mesopotamia, the Gulf and Indus Valley (Cleuziou and Tosi 1994, 2000; Carter 2002). The

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practice of using natural bitumen to coat vessels persisted in southern Iraq (Ochsenschlager 2004; Thesiger 1954, 1964) and Iran until recently (Cooper et al. 2020).

Yet, our knowledge of the use of this material in boatbuilding and seafaring context during the medieval period is minimal. Due to the scarcity of medieval shipwrecks in the Indian Ocean, most of the early and middle Islamic archaeological bitumen is associated with the production of ceramic vessels,

1-Hereafter called the Gulf.

such as torpedo jars, the inside of which is sealed with a thin layer of this substance (Connan et al. 2020, 2021; Stern et al. 2008, Durand 2021, Lischi et al. 2020, Tomber et al. 2020).

Our knowledge instead relies primarily on vague historical sources mentioning a wide range of materials used to seal the hulls of watercraft in the middle Islamic period. Moreover, terms such as bitumen, tar, pitch and resin are often used indiscriminately for substances that look very similar in terms of colours and physical properties but are produced from different materials (Forbes 1955). Since it is virtually impossible to distinguish all these substances by the naked eye, references to pitch and tar might actually indicate bitumen and vice versa.

Recent excavation at the Islamic site of al-Balīd (2010–2018) (Fig. 1) brought to light several ship timbers of medieval vessels involved in the Indian Ocean maritime trade (Belfioretti and Vosmer 2010; Ghidoni 2021; Pavan et al. 2018; Vosmer 2017, 2019). These pieces of maritime material culture display the distinctive traits of the Indian Ocean sewn-plank technique, providing invaluable information about boatbuilding techniques and materials in the medieval period. A considerable number of the al-Balīd timbers retained preserved "bitumen" on their edges and surfaces used to lute the planking seams and make the hull watertight.[1]

2. The site of al-Balīd

During the medieval period, the southern coast of Oman – corresponding today to the Governorate of Dhofar - was characterized by the flourishing of several settlements, involved in the long-distance trade across the Indian Ocean. Al-Balīd, known at that time by the name Ẓafār, gained a role of leadership among all these centres, becoming the most important urban settlements along the southern coast of Oman (Newton and Zarins, 2017; Zarins and Newton, 2017). Having its flourishing period between the 13th and the 15th century, when it is mentioned in various sources (Ibn Baṭṭūṭa, 1962; Smith 1985; Yule 1871) al-Balīd was a pivotal centre for the international network developed by the Rasūlids which involved Arabia, Africa, India and, furthermore, China and East Asia (Pavan and Visconti 2020).

Ẓafār and the whole coast of Dhofar are mentioned as centres of export for local products, such as frankincense, ambergris, myrrh, dragon's blood, aloes, benzoin, liquid storax, camels and ostriches, but also as a place of distribution for goods arriving from the interior of the country or other regions of the Arabian Peninsula such as horses, or from Africa, such as rhino horns and ivory (Pavan, forthcoming).

The wealth of the ancient town finds is evinced by the numerous high-quality and expensive pottery vessels manufactured in Egypt, Iran, and China, but also in the medium-quality products, such as Yemeni, Indian, Chinese, and Far Eastern ceramics which were found in the city.

Evidence of contacts with East Africa are barely represented by pottery items, as most of the goods were perishable, but they include some samples of copal and quite a good number of coins issued by the Sultans of Kilwa, who ruled on the eastern coast of Tanzania, between the 10th century and the first half of the 16th century (see Annucci in Pavan et al. 2020). The city of al-Balīd covers a surface of 64 ha and it has roughly the shape of an irregular rectangle (Fig. 2A). The site is apparently crossed by two major roads with smaller streets encircling buildings which presumably stood rather isolated. The defensive system was integrated by four towers towards the ocean, two others along the western and eastern sides of the site. The side facing north apparently lacks gates and only a couple of semi-circular towers have been identified at the edges of the northern fortification system. Considering also the presence of a ditch, it is possible that the northern side has more of a divisionary purpose (separating the interior from the exterior) than a functional one. An internal gate, located in the north-central part of the site (Area B) was excavated by P. Costa (1979) and it was related to a possible inner wall delimiting an earlier smaller city.

The city itself could be divided into three main areas: a western one, with the most important buildings such as the citadel (*Husn*) (Fig. 2B) and the Grand Mosque located around the largest public square of the city; a central part, with private large houses and mosques, including a funerary one; and a wide, easternmost, area which was possibly used to stock, load and unload goods and animals (Fig. 2A). This sector, which is now completely flat, except for a few mounds surrounded by columns, reveals the presence of small mosques and could have hosted temporary shelters and structures built of perishable materials.

During the last four years (2016–2020) excavations have been mainly focused on the citadel (*Husn* al-Balīd). Six constructional phases have been identified so far on the basis of the data emerged from the archaeological investigations, recent studies on the materials (pottery, small finds, coins) and the results achieved during the activities carried out by the previous archaeological missions working at the site. To date only the last three phases (VI, V, and IV) have been stratigraphically investigated because the works still did not reach the earliest occupational levels. The investigated phases cover a time ranging from the end of the 13th to 18th century (Pavan et al. 2018, 2020).

3. The timbers

During several excavations at al-Balīd's citadel and Grand Mosque, archaeologists have discovered more than fifty timbers, identified as parts of hulls of sewn boats (Belfioretti and Vosmer 2010; Ghidoni 2021; Pavan et al. 2018; Pavan et al. 2020; Vosmer 2017, 2019). These ship remains form the largest collection of archaeological evidence for ships sailing in the Indian Ocean during the Middle Islamic Period. Having fulfilled their maritime purpose, the timbers were recycled in a terrestrial context, reused as structural elements within the masonry of the site's buildings. The builders of al-Balīd used the straight timbers as levelling within the walls of the citadel to help distribute the weight of the masonry (Borgese et al. 2019), and as ceiling planks, beams, shelves and lintels (Fig. 3) (Ghidoni 2022). Evidence of boat part recycling has been found on the Red Sea coast of Egypt at Berenike (Sidebotham 2008), at Quseir al-Qadim (Blue 2006; Blue et al. 2011) and the guardroom ceiling of Fort Jesus in Mombasa (Prins 1982). The vast majority of the al-Balīd timbers consist of hull planks bearing similarities with recent sewn boats, and displaying the traits of the Indian Ocean sewn-plank construction Fig. 4), such as:

- continuous sewing through holes regularly spaced along their edges;
- vegetal fibre sewing cordage recessed in rebates on the outside of the hull;
- a caulking roll (wadding) between the planking seams and the sewing inside the hull;
- frame lashings;
- oblique dowels connecting adjacent planks;
- and luting and coating material.

The timbers are different sizes and display various fastening techniques, indicating that they once belonged to different vessels. Most of the planks (86 %) have rebates on one side: small grooves or channels between the sewing holes and the edge of the plank. This evidence indicates a single-wadding technique typical of the western Indian Ocean

[1] The term "bitumen" is used here without presuming its chemical composition which could be either pure bitumen or a bituminous mixture of oil, resin and pure bitumen as illustrated in the chemical analyses below.

Fig. 1. Map showing the location of al-Balīd, Oman. (Photo: A. Ghidoni).

(Belfioretti and Vosmer 2010; Pavan et al. 2018, 2020), where these rebates connect matching holes between two planks to accommodate and protect the stitches on the outside of the hull. Inboard, the sewing is over a cushion of vegetal material (wadding) and firmly compresses it on the plank's seam. A few planks had no rebates, suggesting instead the presence of wadding under the stitching on both sides of the hull in a pattern called double-wadding (Pavan et al. 2018, 2020; Vosmer 2017), observed in the 8th-9th centuries Phanom-Surin and Belitung shipwrecks (First Regional Office of Fine Arts 2016; Flecker 2000, 2010). An illustration by Johannes Baptista van Doetechum (c.1554–1606) shows vessels sailing off Goa with a caulking roll on each side of the hull (Van Linschoten 1605) pointing to the use of this fastening system in the western Indian Ocean at the end of the 16th century. This technique was also documented in recent sewn vessels used along the east coast of India (Kentley 1985, 2003).

Botanical analyses carried out on the timbers indicate that Indian Ocean boatbuilders used a wide range of wood species to build medieval ships. The species identified include teak (*Tectona grandis*), jujube (*Ziziphus mauritiana*), *Terminalia* spp., *Anogeissus* spp., *Caesalpinia* spp., and palm (*Palmae*: species unspecified) (Belfioretti and Vosmer, 2010; Capretti et al. 2010; Ghidoni 2021; Vosmer 2019). The sewing cordage was visually identified as coir (coconut husk), a fibre that was widely employed in western Indian Ocean sewn watercraft throughout history (Chittick 1980; Edye 1834; Hornell 1942; Hourani 1963; Stanley 1869; Vosmer 1997).

The species diversity indicated by the botanical analyses also reflects a variety of characteristics, ranging from high-quality boatbuilding wood for hull planking, such as teak and some species of *Anogeissus*, to relatively poor choices, such as palm and jujube, the latter better suited for frames than planks. Moreover, the timbers distribution is wide, being native to various areas, including East Africa, India and Southern Arabia.

Radiocarbon dating analysis on sixteen samples indicates that the felling of the trees from which these timbers were fashioned occurred between the late 10th and 15th centuries CE, suggesting a similar period for the construction of the boats (Belfioretti and Vosmer 2010; Pavan et al. 2018; Vosmer 2017, 2019; Ghidoni 2021).

4. Bitumen evidence from al-Balīd

One interesting aspect of the al-Balīd timbers is the presence, in more than a third of the collection, of traces of a black substance resembling

Fig. 2. (A): Aerial view of al-Balīd with the main buildings excavated (Photo: A. Pavan). (B): general view of the citadel (*husn*) where the ship timbers were discovered (Photo: M. Massa).

bitumen (Belfioretti and Vosmer 2010; Pavan et al. 2018; Ghidoni 2021). This substance occurs on planks either with or without rebates.

A thin layer of "bitumen" is preserved on the faying edge- the surface where two planks are joined together-of seven timbers, pointing to the practice of luting, the use of a malleable material to seal the seam between two adjoining planks before their final fitting during the assemblage of the hull (Fig. 5A-B). The construction method of sewn hulls makes it impossible to make them watertight by driving caulking into their seams, in the way used in nailed ships, because the sewing cordage would be in the way. This luting activity is suggested by various ethnographic records from the western Indian Ocean (Severin 1985; Shaikh et al. 2012; Vosmer et al. 2011; Weismann et al. 2019).

In eight timbers the "bitumen" is located on the former inner surface of the plank, between the sewing holes and the edge. The best examples are BA1104065.454 and BA0604145.175, which preserved a great deal of the substance ranging in thickness between 2 and 5 mm (Fig. 5C-E). A thin dark coat of the material is visible under the wadding in BA0604128.73 and BA0604128.74, indicating that medieval boatbuilders smeared bitumen on the hull seams prior to the sewing of the planks (Fig. 5D-F).

Four planks display traces of bitumen on the outer surface. All but one, Wo37 (Fig. 6A) have a flat surface with no rebates, suggesting the presence of the wadding outboard. Traces of bitumen are located around the stitching holes of timber Wo37, and extend towards the centre of the plank, suggesting that bitumen covered the hull outboard, most likely below the waterline to increase its water tightness and address construction issues, and protect it against marine crustacea and molluscs such as barnacles and shipworms (*Teredo navalis*) (Woods Hole Oceanographic Institute 1952).

The practice of using bitumen, tar or pitch, in Indian Ocean medieval watercraft, is suggested by a number of illustrations in the *Maqāmāt* of al-Ḥarīrī (Nicolle 1989, Fig. 22; Bibliothèque Nationale, Ms Arabe 6094, fol. 68) and Persian miniatures (Eastman 1950, pl. XII; Weismann 2002, Fig. 21; Bibliothèque Nationale de France, Suppl. Persan 641f. 59) depicting black hulls. Bitumen-coated vessels have persisted in Iraq (Thesiger 1954; Ochsenschlager, 2004), where they are still in use in the marshlands in the southern region of the country (Jeffrey Rose, personal communication, February 2017). Five sewn *baggāras/āmeles* from Iran, which were recently acquired by Qatar Museums, were coated outboard with a thick layer of "bitumen" (Fig. 6B), proving that the practice of using this material to waterproof watercraft persisted in a maritime context until at least the second half of the 20th century (Cooper et al. 2020).

In the case of timbers BA0604159.263 and BA0604172.69, fragments of the same plank, a thick layer of this substance covers most of the former inner surface (Fig. 6C), showing "bitumen" was also sometimes used inboard (Cooper et al. 2020) (Fig. 6D). This would have protected the stitching against abrasion and sun exposure, preventing the breakage of the cordage, thus extending the life of the sewing.

In three timbers, "bitumen" appears to have been used to seal and smooth irregular surfaces. In the case of timber BA0301-106, it covers an uneven section near the sewing holes, to make it flush with the rest of the plank. Similarly, medieval boatbuilders smeared "bitumen" near the sewing holes of timber Wo73, to seal a crack, likely caused by the drilling of an additional hole to either repair or reinforce the sewing. On plank Wo52, "bitumen" luted a broken section of a dowel.

The site of al-Balīd also provides evidence of "bitumen" not associated with the ship timbers. Recent excavations of the citadel have discovered a lump of this substance (X35). The piece is small, measuring 60 × 100 mm, and provides no hints such as impressions of wood or cordage that could help to reveal its purpose (Fig. 7). However, it is possible that the lump was imported from the bitumen sources in Persia and Mesopotamia and stored at the site to be used in boat construction or repair. Alternatively, shipwrights at al-Balīd could have even stripped it from other vessels and stored it at the site to be reused.

Lastly, a layer of "bitumen" of irregular thickness was found lining the inside of a cylindrical metal vessel (X43) (Fig. 8). The "bitumen" is likely to be pure because it appears different from that seen on the ship planks and the lump X35, having a very smooth and almost glassy texture with no inclusions. The fact that the bitumen occurs primarily on

Fig. 3. (A): A ship beam recycled as a lintel over a small window. (B and C): Ship planks used as levelling courses in the walls of the al-Balīd citadel. (Photos: A. Ghidoni).

the sides of the metal container may point to the use of the substance to seal the inside of the container, in a practice previously observed in ceramic vessels (Connan et al. 2020). However, it is very likely that this evidence might indicate pure bitumen stored in the vessel for subsequent use. Firstly, its thickness, exceeding 5 mm, appears rather excessive if compared with that generally used to seal ceramic vessels in the region (Connan et al. 2020; Lischi et al. 2020). The material of the container also appears to strengthen this interpretation. It is indeed unlikely that a metal container would require an internal bitumen lining since, differently from unglazed ceramics, it would not have needed to be sealed. Moreover, a metal container could have been used to prepare the bituminous mixture used in the luting and coating of the vessels.

5. Experimental

5.1. Archaeological samples

We extracted 25 "bitumen" samples from 15 timbers, the lump (X35) and the seemingly pure bitumen inside the metal container (X43) (Table 1 and Fig. 7), to determine their chemical and mineralogical composition. The former helps to trace the origin of the bitumen, while the latter provides insights into the inclusions added to the bitumen to create the amalgam for maritime use. Moreover, the samples were extracted to determine whether there are differences in the "bitumen" origin and mineralogical composition which might be related to chronology or location and function of the "bitumen" on the timbers. Lastly, we intended to investigate possible similarities between the bitumen from the timbers and those from the lump and metal container, which might indicate different stages of the bitumen amalgam preparation. Overall, the comparison between the samples provides an opportunity for insights into the use and trade of bitumen during the medieval period, as well as about maritime activities, such as boat building, repair and maintenance in the western Indian Ocean and, perhaps, at the port of al-Balīd.

The sampling process was carried out with a scalpel, using gloves to avoid any contamination of the specimens. The samples, ranging between 2 and 10 mm thick, were taken from most of the timbers, particularly those with a thick layer of "bitumen":

- Eleven samples from nine planks where "bitumen" was located inboard between the sewing holes and faying edge of the plank, under the wadding;
- Three samples from three planks where the substance was on the faying edge of three planks;
- Four samples from the outboard coating of four planks;
- Three samples from the inboard coating of two timbers (BA0604172.69 and BA0604159.263), which are, in fact, fragments of the same plank;
- Two samples from two timbers, where the substance was used to fill a broken dowel hole (Wo52) and to smoothen the damaged surface of the timber, around the sewing holes outboard (BA03031-106);
- One sample from the bitumen lump X35;
- One sample from the metal container X43.

Because some of the timbers had (bore) traces of "bitumen" on either their edges and between the sewing holes and the edge (under the

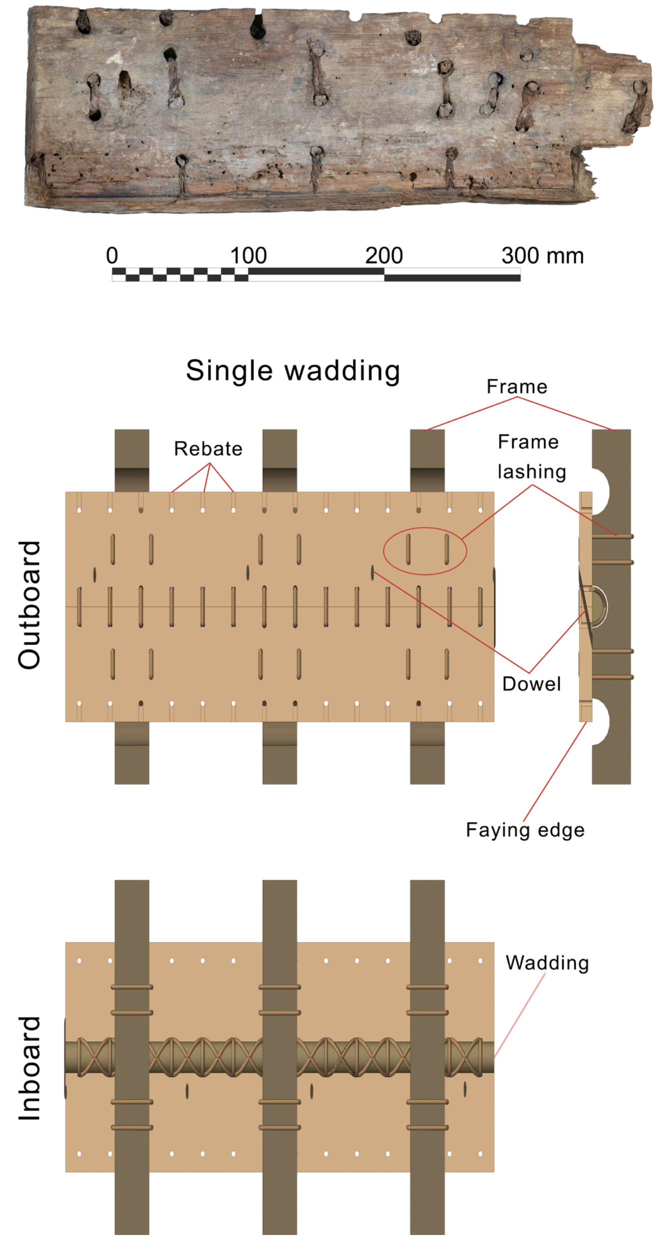

Fig. 4. Outer surface of timber BA0604145.175 (above) and the main features of western Indian Ocean sewn-plank construction technique (below).

wadding and sewing), more than one sample was extracted from those timbers.

5.2. *Oil seeps*

In order to trace the origin of the al-Balīd bitumen from the ship timbers, metal container and lump, the characteristic biomarkers and carbon and deuterium isotopes present in the ancient samples were compared against modern compositional datasets obtained from oil seeps analysed in southern Iran and Iraq (Hit, Mosul area and Kurdistan) (Fig. 9). This reference database has been consulted in previous studies (Stern et al. 2008; Connan 2011; Connan et al. 2014, Connan et al. 2020). Some complementary data were recently acquired on new oil seeps in southwest Iran given by Buyuk Ghorbani and on oils seeps from Kurdistan provided by Rzger Abdulkarim Abdula and Mohammed W. Alkhafaji which significantly increased the geographical coverage.

5.3. *Analytical procedures*

5.3.1. *Bitumen identification*

All archaeological and geological samples were subjected to the same analytical procedure at GeoMark Research Ltd. The dichloromethane extract was deasphalted using n-hexane. The desasphalted fraction was separated into saturated hydrocarbons, aromatic hydrocarbons, and resins using gravity flow column chromatography employing a 100–200 mesh silica gel support activated at 400 °C prior to use. Hexane was used to elute saturates, dichloromethane to elute the aromatic hydrocarbons and dichloromethane/methanol (50:50) to elute the resin (NSO) fraction. Following solvent evaporation, the recovered fractions were quantified gravimetrically. The C_{15+}saturated hydrocarbon fraction was subjected to molecular sieve filtration (Union Carbide S-115 powder) after the technique described by West et al. (1990). An aliquot of the total alkane fraction was not fractionated by silicalite in order to preserve access to the n-alkanes.

GC–MS of the C_{15+}branched and cyclic hydrocarbon fractions was performed using an Agilent 7890A (split injection) interfaced to an Agilent 5975C mass spectrometer. The HP-2 column (50 m × 0.2 mm, 0.11 µm film thickness) was temperature programmed from 150 °C to 325 °C at 2 °C/minute and held for 10 min. The mass spectrometer was run in the selected ion mode (SIM), monitoring ions *m/z* 177, 191, 205, 217, 218, 231 and 253 amu for branched and cyclic alkanes. The quantitative data were produced using peak surface.

To determine the absolute concentration of individual biomarkers, a deuterated internal standard (d4-$C_{29\alpha\alpha\alpha}$20Rsterane, Chiron lab, Norway) was added to the C_{15+}branched/cyclic hydrocarbon fraction. Response factors (RF) at 221 for the deuterated standard to hopane (*m/z* 191) and sterane (*m/z* 217) authentic standards were found to be 1.4 for terpanes and 1.0 for steranes. Concentration of individual biomarkers were determined using the following equation: Conc. (in ppm) = (Ht biomarker) (ng standard)/ (Ht standard) (RF) (mg B/C fraction).

The C_{15+}saturates, C_{15+}aromatics, asphaltenes and resins were analysed for their respective carbon isotope (δ^{13}C, VPDB) compositions. The asphaltenes and resins were also analyzed for their stable hydrogen isotope compositions. Details for all of the methods for stable isotope analyses are described in Connan et al. (2021).

5.3.2. *Identification of fats, oils and resins*

Analyses were carried out at the Avignon University IMBE-IRPNC laboratory. Ten mg of samples were extracted three times with 1 mL of dichloromethane, then centrifuged, finally the supernatant was derived with BSTFA/TMCS (99/1, v/v) (Sigma Aldrich) and the solution was injected into a GC–MS device. The analytical procedure was described in Mezzatesta et al. (2021a). The analyses were carried out with a ThermoFisher apparatus equipped with an 8-slot AI3000 automatic injector and a Focus GC chromatograph coupled to an ITQ 700 ion trap type mass spectrometer. The capillary column used was a GOLD TG-5MS Thermo trace (5% diphenyl and 95% dimethyl polysiloxane) with an internal diameter of 0.25 mm, a length of 30 m and a film thickness of 0.25 µm. The carrier gas used was helium with a constant flow rate of 1 mL.min^{-1}. The injector, the transfer line and the ion trap were respectively at 250, 300 and 200 °C. The electronic impact source had an ionization energy of 70 eV, an ionization time of 25,000 µs and a scan in the mass range of 40–650 *m/z*. The injection volume of each sample is 1 µL in splitless mode.

6. Results and discussion

6.1. *Gross composition of the extractable organic matter*

Gross composition data are reported in Table 2. The extractable organic matter covers a wide range, from 2% to 77.4% /weight of the sample. The average value, deduced from the treatment of the 25 samples gave 33.8 % / weight and is higher compared to the average value of

Fig. 5. (A): "Bitumen" on the edge of timber BA1104065.454 suggesting the practice of luting the seams of the planks before their fitting. (B): The luting process, in this case carried out using *dammar* resin, during the construction of the al-Hariri Boat, a reconstruction of a 13th century Indian Ocean sewn merchant vessel based on the illustrations of the *Maqamat* al-Hariri. (C-E): A thick layer of "bitumen" covers the section between the sewing holes and the plank's edge on timbers BA0604145.175 and BA1104065.454. (D-E): The boatbuilders of the *Jewel of Muscat*, a reconstruction of a 9th century sewn cargo vessel based on the Belitung shipwreck discovered in Indonesia, used dammar resin to seal the seams of the planks after their fitting and before commencing the sewing. (Photos: A. Ghidoni).

355 archaeological bituminous mixtures from the Near and Middle East, mainly Iraq, which is 23.4 % (Connan 2012). The coating of reed boats sailing in Mesopotamia and the Gulf during the Neolithic and Bronze Age (5100–1900 BCE) was found to be 17.9 % / weight. The average values for hydrocarbons (sat. + aro.) and asphaltenes of al-Balīd samples are respectively 6.0 and 73.4 % which means that the mixtures are matching the composition of archaeological bitumens (Connan 2012). At that level of interpretation, based on gross composition only, this preliminary conclusion about bitumen occurrence remains a hypothesis in regard to the real constituents of the caulking mixtures.

Plots of the gross composition data (Table 2), in two ternary diagrams (Figs. 10 and 11) differentiate the samples. Most samples cluster in the area rich in polar components, however the samples Nos. 2360 and No. 3212 are located apart (Fig. 10). The specific character of samples Nos. 2360 and 3212 is confirmed when considering the plot of hydrocarbons *vs.* resins *vs.* asphaltenes (Fig. 11). A third sample joins this group, namely No. 2362. All other samples cluster in an area with low hydrocarbons and various amounts of resins. Two samples, Nos. 3227 and 3210, are extremely devoid of saturates and aromatics and may not contain bitumen but some other components: resins, fats, oils.

Fig. 6. (A): Timber Wo37 displaying a thin layer of “bitumen”, along with a white substance, on the outer side. (B): A *baggāra/āmele* from Iran, recently acquired by Qatar Museums (QM), coated outboard with a thick layer of “bitumen”. (C): “Bitumen” The boatbuilders of the *Jewel of Muscat*, a reconstruction of a 9th century sewn cargo vessel based on the Belitung shipwreck discovered in Indonesia, used dammar resin to seal the seams of the planks after their fitting and before commencing the sewing. Covers most of the inside face of timber BA0604172.69. (D): The thick inboard bitumen coating of a *baggāra/āmele* from Qatar Museums.

Fig. 7. Bitumen lump (X-35) discovered in the recent excavations of the citadel.

Complementary detailed investigations will provide some answers. No. 3227 (Wo52) appears to be a filler over the end of a dowel driven obliquely from the outside of the plank into the faying edge of the plank above (now missing). It may have been used as a filler to smooth the surface of the plank. Gross composition of sample No. 3212 (Fig. 10) matches those of a pure bitumen with 11.1 % saturates and 14.4 % aromatics, i.e. a rather rich composition in hydrocarbons. This sample is extremely soluble in dichloromethane (61% by weight/ sample). Sample No. 3226 (Fig. 11) is a slab consisting of a characteristic archaeological bituminous mixture of mostly asphaltenes and few hydrocarbons.

Fig. 8. Metal vessel (X43) with a coat of bitumen inside.

6.2. Carbon and deuterium isotopes

Carbon and deuterium isotopes data are listed in Table 3. A plot of δ^{13}Csat (‰ / VPDB) *vs.* δ^{13}Caro (‰ / VPDB) of al-Balīd data (Fig. 12) in comparison to linear trend curves obtained from data on 32 oil seeps of Iran and 28 oil seeps of Iraq, show that some samples (Nos. 3212, 3226, 2934bis, 3211) are close to the straight lines defined by natural oil seeps while the others are located outside of these references. One may notice that the sample No. 3212, discovered in an iron container and which is obviously a pure bitumen with its shiny conchoidal fracture, and the sample No. 3226, which is a typical bitumen slab, are falling on straight lines defined by the reference oil seeps. Most of other samples are located in the upper part of the diagram. This feature suggests that these samples are composite mixtures with "recent" additives (resins, fish oils, fats, etc.) which shift the bitumen carbon values of aromatics towards enriched values.

A plot of δDasp (‰ / SMOW) *vs.* δ^{13}Casp (‰ / VPDB) in Fig. 13, exhibits a dispersed pattern on both parameters with δDasp (‰ / SMOW) values and δ^{13}Casp (‰ / VPDB) covering a wide range respectively from −180 to −60 (‰ / SMOW) for δD and −25 to −29 (‰ / VPDB) for δ^{13}C (Fig. 13). The same plot restricted to oil seeps from Iran and presented in Fig. 14 limits the δD range from −30 to −110 (‰ / SMOW). The report of this result in Fig. 13, defined two zones: one area clustering the samples with bitumen only and another zone where bitumen is likely mixed with other ingredients to be defined by specific additional analyses. The split of samples into two populations is confirmed when considering both δ^{13}Casp (‰ / SMOW) and δ^{13}Cresin (‰ / SMOW) in the plot of Fig. 15 where samples containing bitumen only are identified using deuterium data.

As a consequence, the data mean that both polar fractions are affected by the suspected additives and that their δ^{13}C data may be also partly altered. One should remember such a possibility when considering the diagram Ts/Tm *vs.* δ^{13}Casp (Fig. 20) as reference to trace the origin of bitumen.

6.3. Mineralogical composition

The mineralogical compositions, obtained by X-Ray diffraction on residue left after the dichloromethane extraction, are reported in Table 4. As a general statement, the major minerals (calcite, dolomite, quartz, feldspar, clays, gypsum, anhydrite) detected in the samples are those which were previously identified in archaeological bitumens from various sites in the Near East (Forbes 1955; Connan 1988; Connan et al. 2005; Connan 2007; Connan 2012; Badel and Kramm 2014). In addition to these conventional minerals, other specific minerals (siderite, hematite, talc, halite, etc.) have been found. These minerals reflect the available tempers incorporated to the bitumen when the caulking mixtures were prepared and therefore may be variable with the dockyard where the caulking paste was elaborated. Bitumen could have also been mixed near its source and exported ready to be used, or even recycled multiple times, its original composition changed in various harbours and boatyards. To explore such hypotheses, the samples containing clay minerals (kaolinite and illite) were identified in Fig. 16. Seven samples correspond to samples which have δ^{13}Casp (‰ / VPDB) between −27.3 and −25.5 and δ^{13}Cresin (‰ / VPDB) between −26.3 and −25.0, i.e., samples which are obviously coming from Iran and more likely from Khuzistan and Fars. All the samples with illite also have kaolinite except for sample No. 3226. The plot of gypsum occurrence in Fig. 17 leads to samples mostly with the same isotope characteristics and therefore again samples from Iran.

6.4. Biomarkers: Steranes and terpanes (Table 5)

Ratios, calculated on the basis of severely altered patterns where some molecular structures are questionable, were discarded from the record.

Characteristic fingerprints of well-preserved samples are shown in Fig. 18. Sample No. 3215 is typical of an oil seep from Iran. Terpanes do show the occurrence of 18α(H)-oleanane and a moderate Ts/Tm. Steranes are rather enriched in diasteranes 27Sdia and 27Rdia. Sample No. 3226 has a different terpane fingerprint with no oleanane and a low Ts/Tm ratio. Steranes are also different with very low diasteranes and no visible C27S and 27R diasteranes.

Sterane composition, expressed as % of C27ββR + S, %C28ββR + S, % C29ββR + S (not shown) differentiates two groups of samples. Samples Nos. 2935, 3210, 3220, 3227, 3228 (Table 5) are depleted in C27 and C28 steranes and therefore are more biodegraded than other samples which display well preserved patterns. To trace the possible origin of bitumens, two molecular parameters already used in a previous study (Connan et al 2020) were considered. A plot of 18α(H)-oleanane *vs.* δ^{13}Casp (‰ / VPDB) in Fig. 19A shows that sample No. 3215 falls within the population defined by oil seeps from Mamatain (Khuzestan) whereas some other samples, located as al-Balīd, are included in the area defined by oil seeps from Ilam-Lorestan (Ain Gir, Sarkan, Siah Kuh, Dehluran, Chersh Mehrghir, Naft Safid) but not exclusively for oil seeps of Litedan in eastern Iran (Hormozgan Province) and Dalaki in Bushehr are also matching. Four samples (Nos. 2382, 3209–3208, 2935 and 2935bis) gave unexpected 18α(H)-oleanane values in regard to their corresponding δ^{13}Casp (‰ / VPDB) data. These samples have already been defined as mixed samples where bitumen is not alone. The addition of other organic ingredients may be responsible for the significant shift in the δ^{13}Casp (‰ / VPDB).

The comparison of results obtained on al-Balīd samples to samples of oil seeps from Iraq (Mosul area, Hit and Kurdistan, Fig. 19B), indicates

Table 1
Basic information about the al-Balīd samples analyzed.

archaeological reference	lab number	date range	type of sample	comment
BA 0604128.74A	**2360**	1420–1440 CE	on plank seam	brown powder which shows black to orange lumps. Pitch according to touch and odor?
BA 0604128.74B	**2361**	1420–1440 CE	substance on stitching to hold the hull planks together (probably coconut fibre rope)	brown powder with numerous minerals. Microscopic aspect: yellow-orange powder. Resins?
BA 0604172.69A	**2362**	1000–1400 CE	coating on plank	pitch aspect
BA120621-1	**2933**	1320–1350 CE	luting sample from boat plank discovered in the Husn (Fortress) at al-Baleed-gate timber-21/06/2012	bituminous mixture with very fine minerals, occurrence of wood fiber. Very hard texture
	2933 bis	1320–1350 CE		big black lump, rather homogenous, extremely hard, vegetal debris visible in the freshly brocken mass, bituminous aspect
BA 1104065.454.1	**2934**	1020–1160 CE	luting sample from boat plank discovered in the Husn (Fortress) at al-Baleed-gate timber-21/06/2012	very hard and black sample with fine minerals
	2934 bis			numerous small black grains with vegetal debris visible in the mass
BA 1104065.450–1	**2935**	1020–1170 CE	luting sample from boat plank	
	2935 bis			composite sample with vegetal fibers + bituminous mixture. Bituminous mixture selected for analysis
BA1104065.454	**3208**	1020–1160 CE	dark substance on the inside of the plank between the stitching and the edge	black plate with traces of wood on one side
BA0604159.263	**3209**	1280–1440 CE*	dark substance on the inside of the plank	black plate with big lumps of wood on one side. On the other side, smooth whitish surface in which minerals are not clearly visible
WO37	**3210**			numerous grains with some traces of wood. The finest grains are yellow (resin aspect?)
BA0604145.175	**3211**	1190–1240 CE*	well preserved plank with bitumen on the inner surface	typical bituminous mixture with numerous mineral grains clearly visible in the mass. Vegetal debris probably present
fragmentary container in iron with cylindrical shape	**3212**	17th century CE	solidified material inside container which appears as a pure bitumen	no residue after extraction, rare cristallized minerals
BA0301-106	**3215**	10-15th CE	traces of thick and dark substance on the outside of the plank around the sewing holes	black sample with traces of vegetals on the surface and in the mass
BA0604172.69	**3218**	1280–1440 CE*	dark substances on most of the inside plank	numerous grains which some minerals (quartz?). Some wood imprints
Wo 73	**3220**	10–15 th CE	substance found near the edge of the plank between and inside the stitching holes	sample with rope and imprints of wood
Wo 98B	**3221**	1380–1418 CE*	substance found in the inner face of the plank, near the edge and above the seam	black plate whose texture seems homogenous. If there are minerals present, they are extremely fine
Wo 98B	**3222**	1380–1418 CE*	thick layer on the edge of the plank	black lumps, rather homogenous. If there are minerals present, they are extremely fine. Wood imprints at surface
Wo 98A	**3223**	1384–1422 CE*	substance on the outer surface of the plank, between the stitching holes and the edge.	very black sample with wood debris at surface. Very fine minerals in the mass
Wo 98D	**3225**	14-15th CE	Substance found on the inside of the plank, near the edge	very black plate with extremely fine minerals in the mass.
Husn 17.107 X35	**3226**	16-17th CE	lump of bitumen discovered in the citadel (Husn) of al-Balid	very black bituminous mixture with very fine minerals
Wo 52	**3227**	14-15th CE	bitumen inside a dowel on the surface of the plank	Faily pure bitumen or resin because brown-yellow appearance on small pieces
BA01.11.01	**3228**	1044–1098 CE*	small fragments of bitumen (or resin?) in a bag next to the plank	bitumen aspect with very fine minerals. Wood imprint with wood debris in the mass.
BA0604128.74	**3229**	1420–1440 CE*	dark substance under the stitching on the inner surface of the plank	brown powder with some minerals (quartz). A piece of seashell and some pieces of wood

that few samples in Iraq (Mishraq and Qaiara in the Mosul area and Sangaw in Kurdistan) match the data of the al-Balīd samples. Three samples fall in the area defined by Mishraq-Qayara in the Mosul area and Sangaw in Kurdistan and three samples (Nos. 3209, 3218 and 2362) match the Zakho oil seeps located in the far north of Iraq, close to Turkey.

As a consequence, the molecular and isotopic data tend to favour an origin of the bitumen in two major areas: Mamatain (Khuzestan) and Dehluran-Ain Gir-Chersh Mehrgirh (Illam). Some seeps of Iraq cannot be ruled out for both their molecular and isotopic characteristics are similar to those recorded in oil seeps from Iran however they are further north and rather unlikely as more proximate Iranian sources are available.

The plot of Ts/Tm *vs.* δ^{13}Casp (‰ / VPDB), also currently used for correlation studies, confirm the diversity of the samples from al-Balīd (Fig. 20). The comparison of these data to those of oil seeps from Iraq (Fig. 21) and Iran (Fig. 22) brings some additional information about sources. Some samples from Iraq (Mishraq and Qaiara in the Mosul area, Sangaw and Pungala in south Kurdistan, and Zhako in northern Iraq) fall within the zone defined by al-Balīd samples but most samples do not match the al-Balīd archaeological samples. The same diagram, built with oil seeps from Iran (Fig. 22), does show that 10 samples from al-Balīd match the oil seeps from Mamatain on one side and Dehluran-Ain Gir-Siah Kuh- Chersch Mehrghir on the other side.

The source of some samples remains unknown, but one may remember that the carbon isotopes on asphaltene are not fully reliable and ascribed to bitumen for they are disturbed by the other organic substances present as suggested by the 18α(H)-oleanane data which are unexpected in regard to their corresponding carbon isotope values. In addition, one may not exclude the use of mixtures of oil seeps from different sources to prepare the medieval caulking material as determined elsewhere (Connan et al. 2020; Connan et al. 2018). Oils from different origins (J.Connan, unpublished) have been identified on the

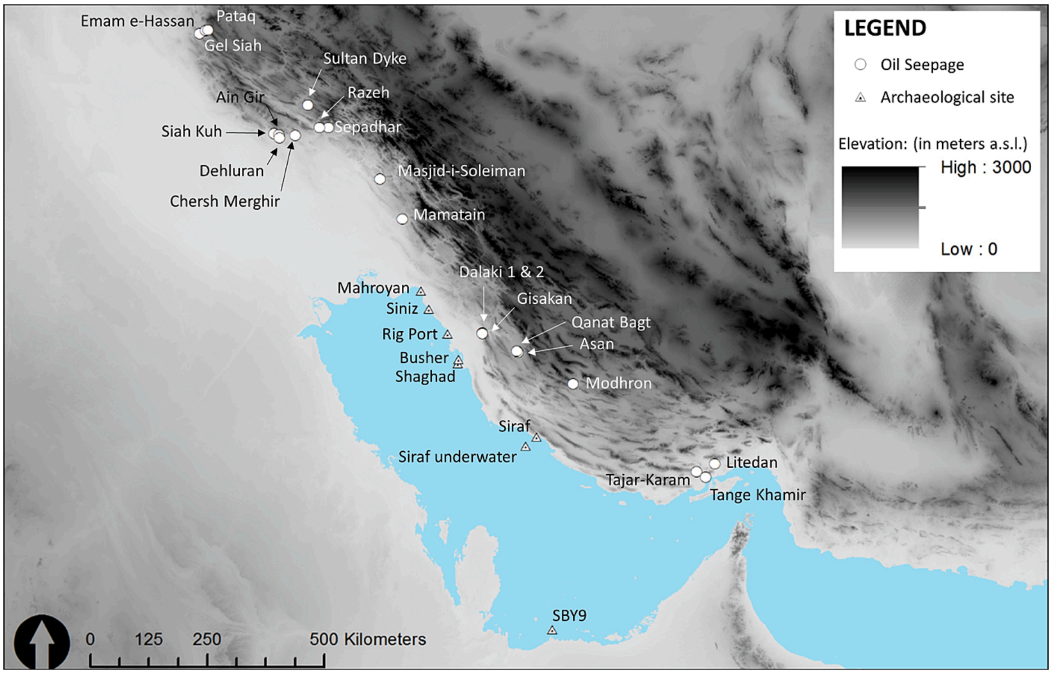

Fig. 9. Map showing the reference oil seeps analyzed in Iran (after Connan et al. 2020).

hull of a *shahoof*, a small vessel from Dubai, formerly part of the EISCA collection at the Eyemouth Maritime Centre, Scotland, and recently acquired by the Oman Across the Ages Museum (OAAM) in Manah, Oman.

6.4.1. Composition of the organic mixtures: bitumen, fats and resins

In this study, 11 samples were analysed (samples Nos. 3210, 3215, 3218, 3221, 3222, 3223, 3225, 3226, 3227, 3228 and 3229) in order to determine the presence of organic matter.

The samples Nos. 3210 and 3227 demonstrated the presence of fatty acids which are characteristic of fatty matter. It is here difficult to determine the nature of this fatty matter because of the absence of specific fatty acids and/or sterols. In sample No. 3210, the proportion of fatty acids such as palmitic, stearic and oleic acids indicated a well-preserved oil while in sample No. 3227, the occurrence of a large proportion of dicarboxylic fatty acids such as adipic, pimelic, suberic and azelaic acids indicated the presence of an oil that is naturally degraded with time (Mezzatesta et al. 2021b). The chromatographic analyses of the samples No. 3221, No. 3222 and No. 3226 showed only molecules of hopanes and steranes interpretable as the presence of bitumen. For the other six samples referred as Nos. 3215, 3218, 3223, 3225, 3228 and 3229, ursane and oleanane molecules were detected. These molecules are markers indicating the presence of a triterpenic resin but the nature of this resin is unknown considering the lack of specific biomarkers detected and/or identified.

Taking into account these new results, Fig. 19 has been updated in Fig. 23 which shows that the preliminary evaluation of sample compositions, carried out on the basis of carbon and deuterium isotopes of asphaltenes was relatively accurate. Complementary investigations, carried out by Philippe Schaeffer and Pierre Adam (Strasbourg Institut of Chemistry, UMR 7177, Strasbourg University) on eleven archaeological samples and three copal samples, excavated near tower 10, in the external part of the city wall of al-Balīd, have shown that copal is not part of the ingredients used in the bituminous mixtures analyzed on planks. Two triterpenic resins have been identified as mixtures: dammar (Dipterocarpacea) and frankincense. The presence of fats and bitumen has been confirmed by this additional study.

7. Discussion

7.1. Bitumen

One important outcome of the analysis on the luting and coating of the al-Balīd timbers is that the substance used is a bitumen-based amalgam. This aspect is particularly interesting because historical and archaeological evidence indicate that Indian Ocean boatbuilders used a variety of different materials to seal the planking of sewn boats. In the 10th century Abū Zayd al-Sīrāfī remarks that the seams and sewing holes of the boats of Siraf were caulked with oil mixed with other substances (Al-Sīrāfī and Ibn Faḍlān 2014). Portuguese sources describe the use of pitch, bitumen and resin, in the vessels they saw along the coasts of East Africa and western India in the 16th century (Agius 2007; Moreland 1939; Prins 1986; Stanley 1869). A dark substance on the surfaces and edges of the Quseir al-Qadim sewn-ship timbers has been tentatively interpreted as bitumen, but no scientific analysis has been carried out to confirm the identification (Blue et al. 2011). The builders of the 9th-century Belitung shipwreck mostly used a lime-based putty to seal the hull planking, posts and through-beams (Flecker 2000), though bitumen was found encrusted on one of its through-beams (Burger et al. 2010).

The apparent homogeneity in luting material in the al-Balīd timbers clearly contrasts with the heterogeneity indicated by the textual sources and other ship remains of the Indian Ocean. It may also suggest that the timbers could have belonged to vessels from a particular region of the Indian Ocean, which privileged the use of this substance (see below). Considering that the date and thickness of the timbers (Ghidoni and Pavan 2022) point to vessels of different sizes and functions sailing between the late 10th and the mid-15th centuries, it also suggests that bitumen amalgams were commonly used for luting and coating in the Indian Ocean for at least four centuries.

This apparently common use of bitumen in the medieval Indian Ocean is also important because this material is predominantly associated with Neolithic and Early Bronze Age boatbuilding in the region, and more generally to reed-built vessels. It is assumed that from the Iron Age, bitumen was replaced by other substances, at least for coating watercraft, and thought to have gradually disappeared in the Islamic era (Vosmer 2007). However, the evidence from al-Balīd indicates that the use of this material in the Indian Ocean boatbuilding context persisted

Table 2
Gross composition data on the al-Balīd samples.

date range	type of sample	lab number	EO (% weight/ sample)	sat, (% / EO	aro, (%/EO)	sat,+aro, (%/EO)	res, (%/EO)	asp, (%/EO)	polars = res,+asp (%/EO)
1420–1440 CE	On plank seam	**2360**	38.5	21.8	12.1	33.9	36.6	25.7	62.3
1000–1400 CE	coating on plank	**2362**	37.4	4.2	3.4	7.6	48.1	42	90.1
1320–1350 CE	luting sample from boat plank discovered in the Husn (Fortress) at al-Baleed-gate timber-21/06/2012	**2933**	27.1	7	6	13	15.4	71.6	87
1320–1350 CE		**2933 bis**	31.7	5.7	4.2	9.9	19.3	70.8	90.1
1020–1160 CE	luting sample from boat plank discovered in the Husn (Fortress) at al-Baleed-gate timber-21/06/2012	**2934**	24.4	1	1.6	2.6	11	86.4	97.4
		2934 bis	25.4	1.3	1.8	3.1	16.7	80.2	96.9
1020–1170 CE	luting sample from boat plank	**2935**	24.3	0.8	0.7	1.5	6.3	92.1	98.4
		2935 bis	57.5	1	1.6	2.6	22.8	74.6	97.4
1020–1160 CE	dark substance on the inside of the plank between the stitching and the edge	**3208**	14.4	0.7	0.7	1.4	20.1	78.5	98.6
1280–1440 CE*	dark substance on the inside of the plank	**3209**	31.8	0.3	0.6	0.9	19	80.1	99.1
		3210	35.5	0.3	0.3	0.6	11.5	88	99.5
1190–1240 CE*	well preserved plank with bitumen on the inner surface	**3211**	29.7	1.2	1.6	2.8	29.9	67.2	97.1
17th century CE	solidified material inside container which appears as pure bitumen	**3212**	68.3	11.1	14.4	25.5	22.1	52.3	74.4
10-15th c CE	traces of thick and dark substance on the outside of the plank around the sewing holes	**3215**	25.7	2.1	1.4	3.5	18	78.5	96.5
1280–1440 CE*	dark substances on most of the inside plank	**3218**	46.5	0.4	0.7	1.1	16.3	85.6	101.9
10-15th c CE	Substance found near the edge of the plank between and inside the stitching holes	**3220**	38.2	0.5	1.3	1.8	19	79.2	98.2
1380–1418 CE*	substance found in the inner face of the plank, near the edge and above the seam	**3221**	30	1	3.4	4.4	15.6	77	92.6
1380–1418 CE*	Thick layer on the edge of the plank	**3222**	31.2	3.5	2.6	6.1	17.8	76.1	93.9
1384–1422 CE*	Substance on the outer surface of the plank, between the stitching holes and the edge,	**3223**	31.3	2.2	1.8	4	20.1	75.9	96
14-15th c CE	Substane found on the inside of the plank, near the edge	**3225**	29	2.5	2	4.5	22.9	72.6	95.5
16-17th c CE	Lump of bitumen discovered in the citadel (Husn) of al-Balīd	**3226**	27.4	2.6	2.9	5.5	18.6	75.9	94.5
14-15th c CE	Bitumen inside a dowel on the surface of the plank	**3227**	77.4	0.1	0.1	0.2	6.1	93.7	99.8
1044–1098 CE*	Small fragments of bitumen (or resin?) in a bag next to the plank	**3228**	2.1	3.5	1.2	4.7	28.2	67.1	95.3
1420–1440 CE*	dark substance under the stitching on the inner surface of the plank	**3229**	27.6	1.1	1.4	2.5	24.4	73.1	97.5

well into the medieval period.

7.2. Bitumen in 19-20th-century Indian Ocean sewn boats

The common use of a bitumen-based compound in medieval boatbuilding as suggested by the al-Balīd timbers also contrasts with the evidence from more recent sewn vessels of the Indian Ocean, which indicates various materials. Resin appears to have been the most common material for luting in ethnographic records of the Indian Ocean (Hornell 1942; Hill 1958; Severin 1985; Agius 2002; Vosmer et al. 2011). Omani boatbuilders used it to impregnate the wadding of the *beden seyad*, a sewn fishing boat documented in Muscat in 1839 (Pâris 1843). In East Africa, a paste made with mangrove bark was the preferred luting for the *mtepe* planking (Lydekker 1919). In fact, bitumen-based luting and coating are seldom attested in sewn boats in the region with only a few cases from the Gulf and Oman. The sewn *baggāras/āmeles* of Hormuzgan, southern Iran, in the collection of Qatar Museums, have their hulls covered inboard and outboard by a black substance only provisionally identified as bitumen (Cooper et al. 2020). However, although no chemical analysis was carried out to scientifically identify the substance, interviews with Iranian boatbuilders confirmed that this type of boat was generally coated with bitumen (Kalantar et al. forthcoming). Chemical analysis carried out by the authors of this paper identified bitumen as the luting of an Omani *kambārī,* a sewn vessel on display in the Museum of the Frankincense Land, Salalah. However, this boat is a 1980s reconstruction (Weismann et al. 2019), built for display only, and the bitumen used in the luting could be a product recently used by the builders and might not reflect past practices and materials. Other evidence from Indian Ocean sewn boats might point to the use of bitumen luting. The "pitch" sealing the sewn *beden* planking recorded by Chittick (1980) in Hafun, Somalia, could also have been bitumen, since no analysis was carried out to confirm the nature of the material. Similarly, Shaikh (2012) mentions coal-tar being used, but it is not clear whether the substance was identified scientifically.

7.3. Bitumen mixture

The other important outcome of the geochemical analysis is that the

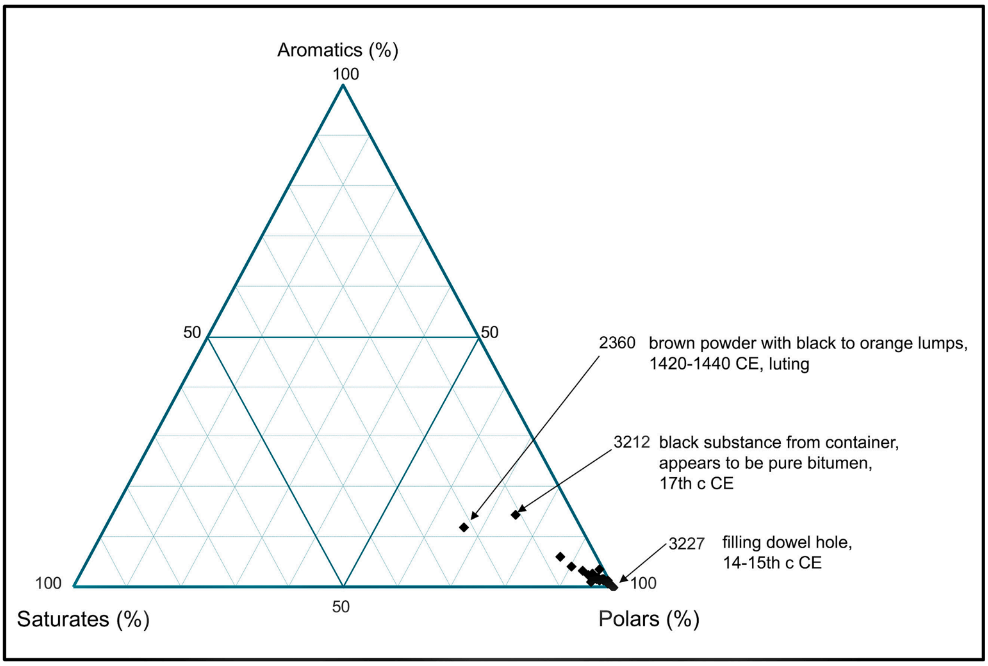

Fig. 10. Gross composition data in a ternary diagram: Saturates (%) *vs.* Aromatics (%) *vs.* Polars (%).

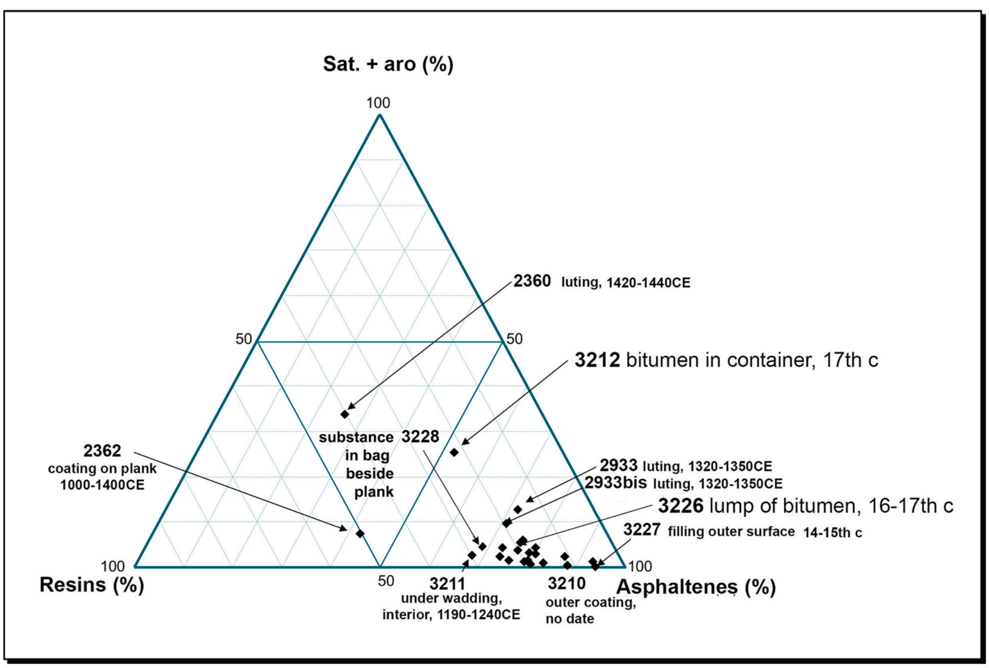

Fig. 11. Gross composition data in a ternary diagram: Saturates + Aromatics (%) *vs.* Resins (%) *vs.* Asphaltenes (%).

al-Balīd luting samples are not made exclusively of pure bitumen. Instead, medieval boatbuilders added different matter to the bitumen to create a compound, where bitumen is only one of the many ingredients. This is certainly not a surprise, but rather a confirmation of a practice used in boatbuilding—as well as in other terrestrial purposes—in Mesopotamia, the Gulf, and perhaps Arabian Sea, in the Neolithic and Bronze Age (Cleuziou and Tosi 1994; Connan et al. 2005; Connan and Van de Velde 2010; Potts 1997: 100; Vosmer, 2003a; 2003b).

The reason for adding modifiers of different material to the bitumen is to change its physical properties and obtain a compound suitable for the use in a boatbuilding context. To be used as luting, and more generally as a sealing agent, this amalgam has to be waterproof; maintain a certain flexibility or plasticity which prevent it from cracking; avoid melting or becoming too fluid when subjected to heat and sunlight; and maintain adherence to the timber under a variety of conditions, particularly when wet (Vosmer 2003a).

Tests carried out during the construction of a hypothetical 3rd millennium BC bitumen-coated reed-built vessel (Vosmer, 2000; 2001; 2003a; 2003b; 2007), indicate that the use of mineral additives improves the working properties of bitumen. The use of calcium hydroxide – $Ca(OH)_2$ or in particular, calcite (calcium carbonate - $CaCO_3$) which is the predominant mineral additive in the samples from al-Balīd, increases the strength and durability of the bitumen without compromising its flexibility (Vosmer 2003b; Vosmer 2007). Mineralogical analysis shows

Table 3
Isotopic data on the al-Balīd samples.

location	date range	type of sample	lab number	δ13Csat (‰ / VPDB)	δ13Caro (‰ / VPDB)	δ13Cres (‰ / VPDB)	δ13Casp (‰ / VPDB)	δDres (‰ / SMOW)	δDrasp (‰ / SMOW)
Al-Balīd	1000–1400 CE	On plank seam	**2360**			−25.75	−26		
	1000–1400 CE	coating on plank	**2362**			−26.5	−27.2		
	1320–1350 CE	luting sample from boat plank discovered in the Husn (Fortress) at al-Baleed-gate timber-21/06/2012	**2933**	−27.1	−26.2	−26.5	−26.4		
	1320–1350 CE		**2933 bis**	−27.2	−26	−26.5	−26.4	−70.6	−74.5
	1020–1160 CE	luting sample from boat plank discovered in the Husn (Fortress) at al-Baleed-gate timber-21/06/2012	**2934**	−28.3	−27.6	−26.5	−26.4		
			2934 bis	−27.8	−27.6	−26.6	−26.9	−123.9	−110
	1020–1170 CE	luting sample from boat plank	**2935**	−28.7	−26.2	−27.3	−28.4		
			2935 bis	−27.3	−26.5	−27.1	−28.2	−139.4	−118.5
	1020–1160 CE	dark substance on the inside of the plank between the stitching and the edge	**3208**	−26.7	−27.5	−27.5	−27.2	−139.8	−138.3
	1280–1440 CE*	dark substance on the inside of the plank	**3209**	−26.8	−26.1	−26.1	−27.2	−165.8	−157.9
			3210	−26.6	−26.4	−26.2	−27.6	−194.9	−177.6
	1190–1240 CE*	well preserved plank with bitumen on the inner surface	**3211**	−26.2	−25.6	−25.9	−25.9	−137.8	−136.7
	17 th century CE	solidified material inside container whic appears as pure bitumen	**3212**	−27.3	−26.9	−26.9	−27	−109	−104.4
	10-15th AD	traces of thick and dark substance on the outside of the plank around the sewing holes	**3215**	−27	−26	−25.1	−25.5	−134.8	−102.1
	1280–1440 CE*	dark substanceson most of the inside plank	**3218**	−26.7	−25.9	−26	−27	−159.3	−137.9
	10–15 th AD	Substance found near the edge of the plank between and inside the stitching holes	**3220**		−26.7	−26.8	−27.8	−167.7	−160.1
	1380–1418 CE*	substance found in the inner face of the plank, near the edge and above the seam	**3221**	−27.2	−26	−26.3	−26.3	−78.4	−87.3
	1380–1418 CE*	Thick layer on the edge of the plank	**3222**	−27.2	−26.1	−26.1	−26.6	−77.9	−69.4
	1384–1422 CE*	Substance on the outer surface of the plank, between the stitching holes and the edge.	**3223**	−27.4	−26.3	−25.7	−26.7	−120.4	−120.5
	14-15th AD	Substane found on the inside of the plank, near the edge	**3225**	−27	−25.7	−25.9	−26.3	−103.8	−107.6
	16-17th AD	Lump of bitumen discovered in the citadel (Husn) of al-Balid	**3226**	−28.4	−28.1	−28.3	−27.9	−72.2	−72.4
	14-15th AD	Bitumen inside a dowel on the surface of the plank	**3227**		−26.9	−26.9	−28.4	−141.7	−152.4
	1044–1098 CE*	Small fragments of bitumen (or resin?) in a bag next to the plank	**3228**		−27.7	−27.9	−28.9	−117.7	−123.2
	1420–1440 CE*	dark substance under the stitching on the inner surface of the plank	**3229**	−27.8	−26.9	−26	−26	−124	−116.9

that boatbuilders added a large quantity of calcite in the bitumen used to coat reed vessels in the Gulf and Mesopotamia during the 3rd millennium BC (Connan et al. 2005). This indicates not only that this practice lasted several millennia, but that during this period the bitumen mixture has not changed much and that similar amalgams were used for different purposes in both reed and wooden vessels.

The mineral matter in the bitumen amalgam also makes the bitumen resistant to heat exposure. This would have been a major issue in Mesopotamia and the Gulf, with extremely high temperatures in summer, causing pure bitumen to 'creep' easily, again reducing its sealing properties. Even today, calcium carbonate is added to various hydrocarbon polymers to improve their impact resistance and working properties, as well as being used in bituminous cements and bituminous paving (ASTM 2019).

Lastly, being a considerable part of the total compound's weight, this mineral matter increases the volume of the bitumen. This would have been an important factor for ancient boatbuilders, enabling them to reduce the amount of bitumen used thus saving on such a valuable and expensive material.

The presence of triglycerides (natural fats and oils from animal and plants) in the amalgam increases the adhesive properties of bitumen (Cleuziou and Tosi 1994; Vosmer 2003b). Bitumen is in fact hydrophobic and does not adhere easily with wood, which is hydrophilic. Natural fats and oils in small percentage (2.5–3%) act as "antistripping" agents and help the bond between different materials (Vosmer 2007). Their use is further necessary after the addition of the mineral (inorganic) matter which reduces the adhesion of the bitumen. Another important function of triglycerides is to increase the plasticity of the amalgam, when the addition of minerals might make it hard and brittle, and prone to crack.

The results of the bitumen analyses do not show any correlations between their geochemical signature and their location within the timbers. It does not look as though medieval boatbuilders used different amalgams whether they used the bitumen on the faying edge of plank or

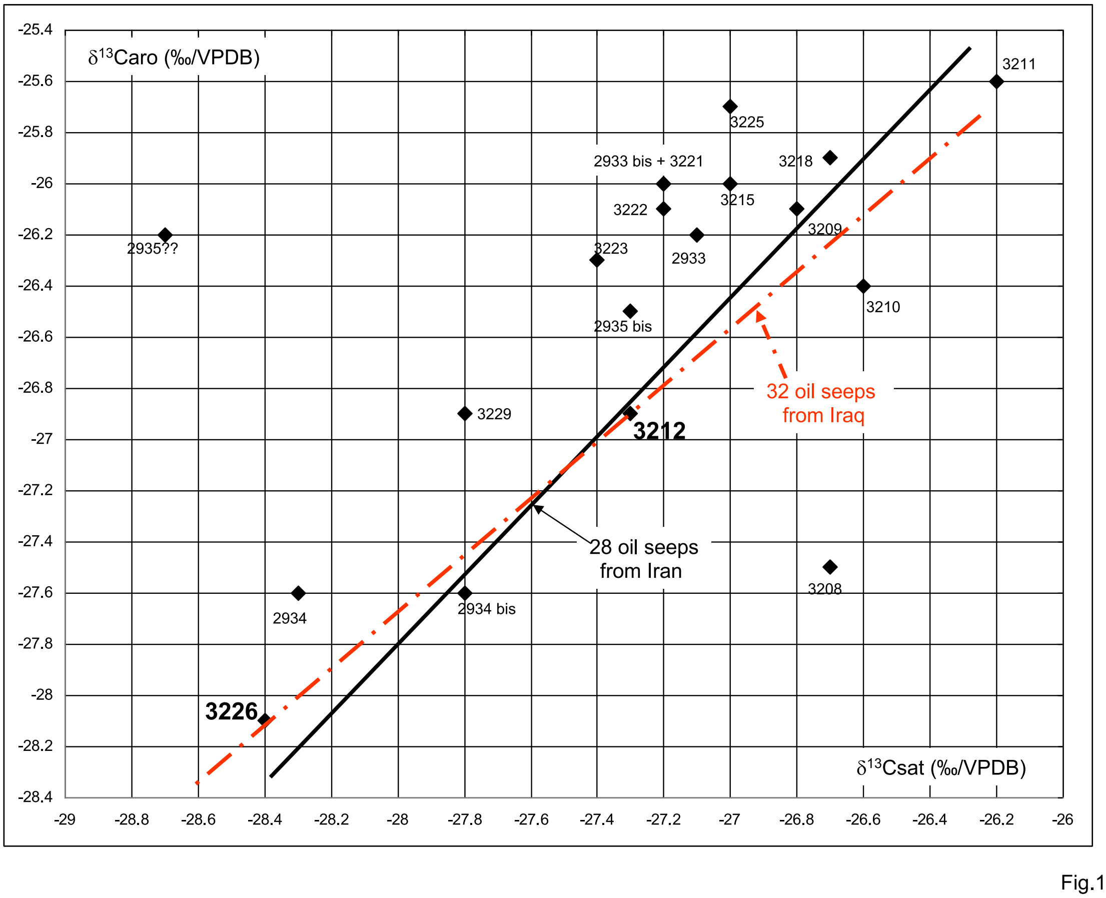

Fig. 12. δ13Caro (‰ /VPDB) *vs.* δ13Csat (‰ /VPDB): comparison of al-Balīd samples to reference curves from 32 oil seeps of Iraq and 28 oil seeps of Iran.

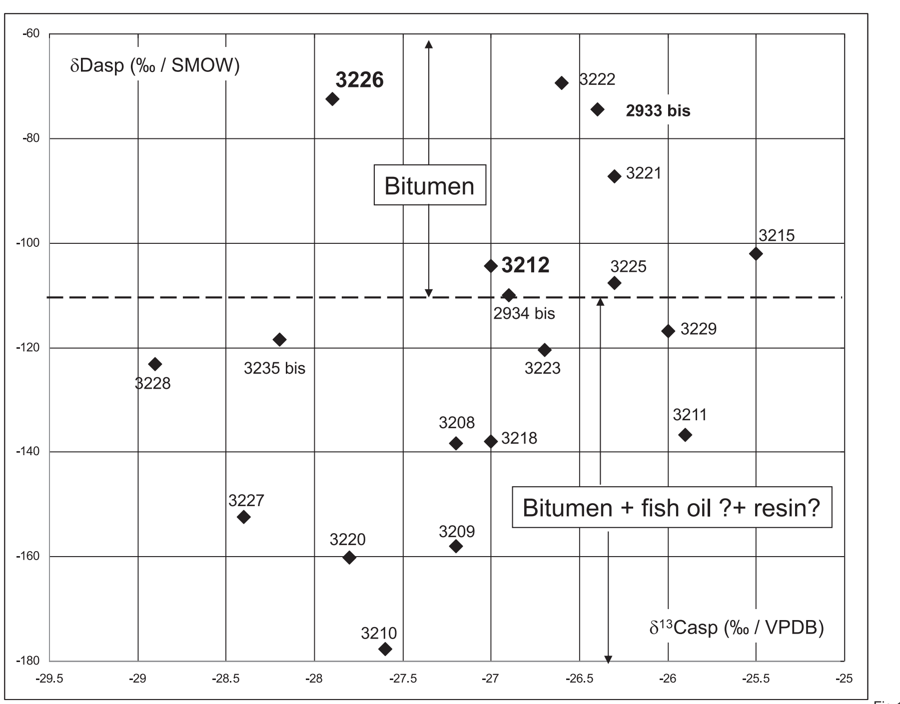

Fig. 13. δDasp (‰ / SMOW) *vs.* δ13Casp (‰ /VPDB): expected composition of al-Balīd samples.

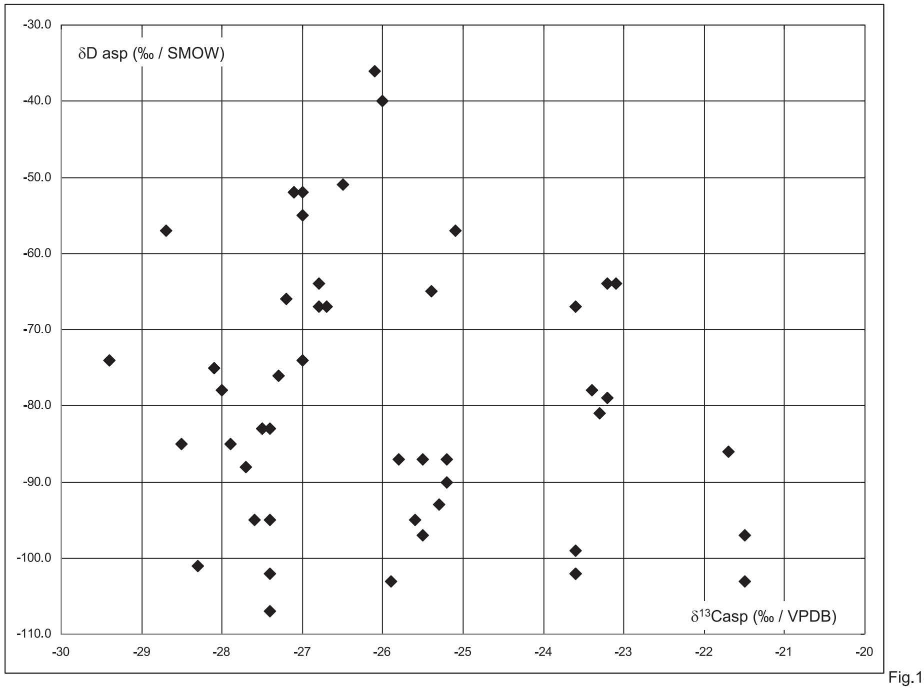

Fig. 14. δDasp (‰ / SMOW) *vs.* δ^{13}Casp (‰ /VPDB) for oil seeps of Iran.

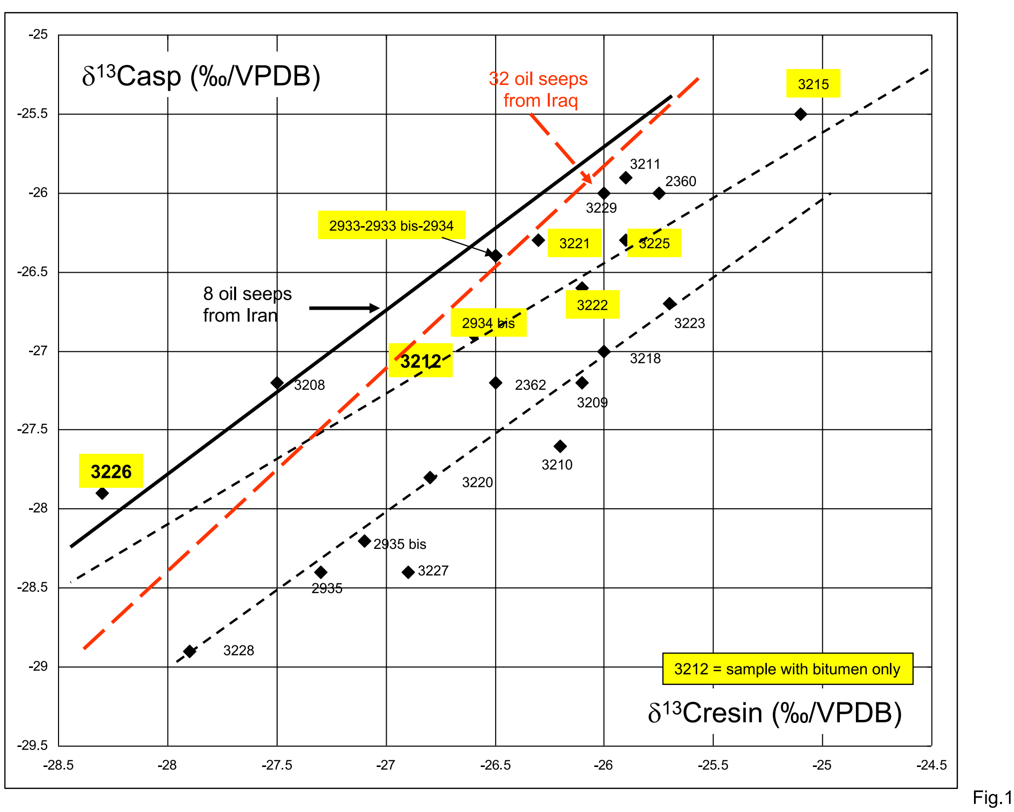

Fig. 15. δ^{13}Casp (‰ /VPDB) *vs.* δ^{13}Cresin (‰ /VPDB): comparison of al-Balīd samples to the reference curves of 8 oil seeps from Iran and 32 oil seeps from Iraq. Identification of al-Balīd samples referred as containing bitumen only.

on the plank under the wadding. This evidence might point to the presence of a “maritime” bitumen amalgam, used for the luting, caulking and coating of Indian Ocean vessels, its differences in chemical composition depending to where it was prepared. This product intended for boatbuilding could have been mixed near the sources and exported to maritime centres in the Indian Ocean as a ready-made product. Alternatively, Indian Ocean boatyards could have imported pure bitumen from the Iranian sources and that boatbuilders mixed it *in situ* during the construction of the boats, using the same compound regardless of its location and function.

Table 4
Results of the mineralogical analysis of some samples of al-Balīd.

n° échantillon	site	date range	Quartz	Calcite CaCO3	Dolomite CaMg(CO3)2	Albite NaAlSi3O8	Gypsum (CaSO4, 2H2O	Anhydrite (CaSO4)	Bassanite (CaSO4 0.5H2O	Muscovite-2M#1	Kaolinite	Illite	Clinoenstatite MgSiO3	Microcline KAlSi3O8	Halloysite Al2O3.2SiO 2.2H2O	Talc Mg3SiO4O10(OH)2	Hematite (Fe2O3)	Siderite (FeCO3)	Halite NaCl
3208	Al-Baleed	1020-1160 AD	xxxx	xx	xx	x				x									
3209		1280-1440 AD	xxxx	xx		x	x			x	x						xx		
3211		1160-1260 AD	xxx	xxxx		x	x		x	x	x						x		
2934bis		1020-1160 AD	xxxx	xx	x	x	x	x		x									
2935		1020-1170 AD	xxxx	xxx	x	x	x	x		x									
3228		1028-1184 AD	xx	xxxx															
2933bis		1320-1350 AD	xx	xxxx	x	x	x	x		x									
3218		1260-1400 AD	xxxx	xx	x	x				x				x	x		x		
3221		1300-1369 AD	xx	xxxx		x	x				x					x			
3222		1300-1369 AD	xxx	xxxx	x	x					x	x		x	x			x	
3223		1304-1364 AD	xxxx	xxxx	xx	x				x	x	x	x		x		x		
3215		10-15th AD	xxx	xxxx	xx	x				x	x	x	x	x	x		x		
3225		14-15th AD	xx	xxxx	x	x	x				x	x			x				
3226		16-17th AD	xxxx	xxxx	x	x	x			x		x			x				
3229		1400-1460 AD	xx	xxx		x	x	x		x				x					
3232	Al Baleed-Chunam	14-15th AD	x	xxxx		x	x												x

calcite (yellow) — magnesian calcite (orange) (Mg.064Ca.936)(CO3)

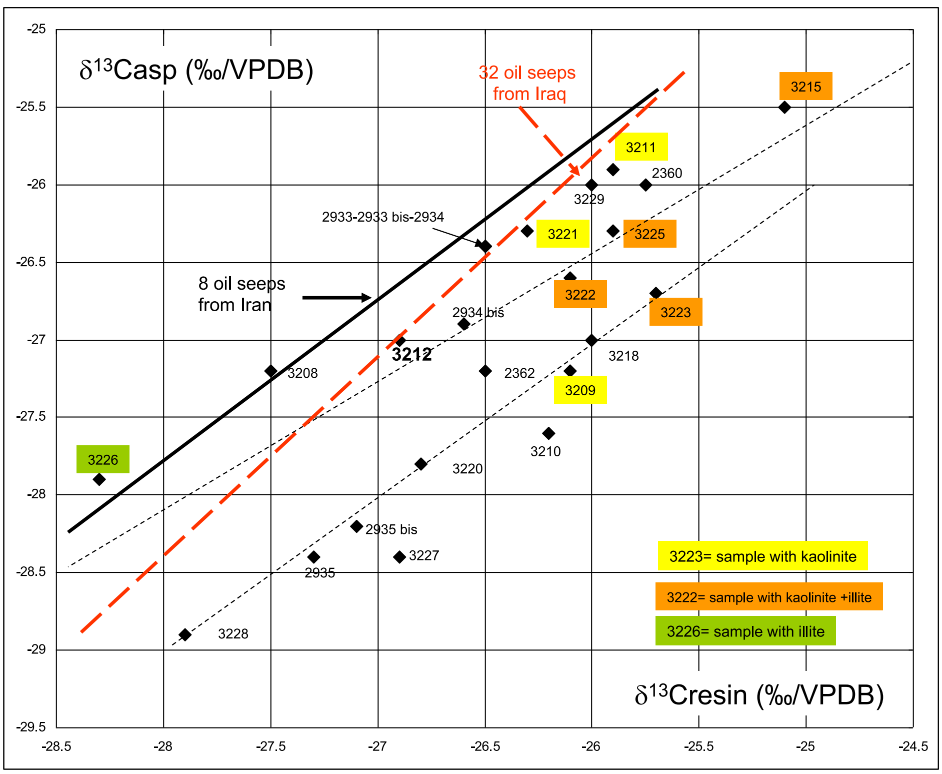

Fig.16

Fig. 16. δ[13]Casp (‰ /VPDB) *vs.* δ[13]Cresin (‰ /VPDB) of al-Balīd samples with the identification of their clay minerals: kaolinite, kaolinite + illite, illite.

The bitumen geochemical analysis does not suggest any particular trend or change of the mixture in time. Although variations occur within the chemical and mineralogical composition of the samples examined, these appear too random to suggest intentional modifications or changes of the amalgam by Indian Ocean boatbuilders during the period indicated by the date of the timbers. It is also important to note that bitumen is a material that can be recycled multiple times, and some, if not all, of the bitumen in the al-Balīd timbers could have been stripped from old vessels and reused in the building and repairing of other boats.

Because of the scarce evidence of bitumen products associated with boats from this period it is not possible to compare the amalgams from al-Balīd with other archaeological 'bitumens' from the region. However, the al-Balīd mixtures appears rather unique for these are diversified amalgams ranging from pure bitumen with minerals to complex mixtures with bitumen, oil, resin and minerals which have been rarely observed for this period.

7.4. Mixture contamination (materials unintentionally added to the amalgam)

One should be aware that the presence of calcite, resin, and, possibly, oil and fat, in the bitumen, could also be explained by the fact that these materials are generally associated to boatbuilding in the Indian Ocean, and could perhaps not have been part of the original mixture. For example, fish oil was widely used to preserve and maintain the planking of both sewn and nailed wooden vessels in the Indian Ocean until recently (Agius 2002; Al-Hijji 2001; Bowen 1952; Donaldson 1979; Hornell 1942, 1970; Johnstone and Muir 1962; Lorimer 1915; Miles 1919; Vosmer 1997). Various medieval sources mention the use of fish and whale oil to coat the hulls of ships sailing in the Indian Ocean (Agius 2007), a practice that appears to have been in use in the Gulf since at least the 3rd millennium (Cleuziou and Tosi 1994). Moreover, oil was also used on the stitching to protect it and keep it moist, extending the life of the ship (Severin 1982), while experiments with dry, wet and oiled sewing fibres of *Arenga pinnata* and coir (coconut husk fibre)

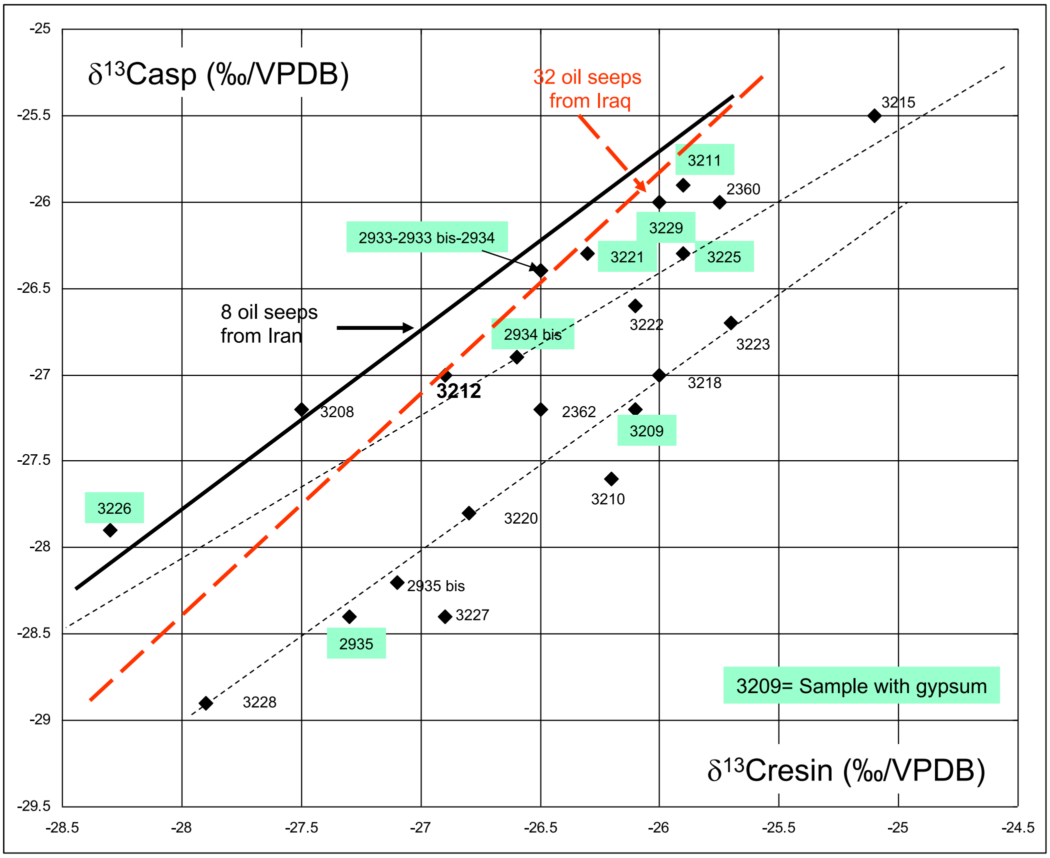

Fig.17

Fig. 17. δ^{13}Casp (‰ /VPDB) *vs.* δ^{13}Cresin (‰ /VPDB) of al-Balīd samples with the identification of samples containing gypsum.

demonstrated that soaking in oil increased the strength of the fibres (Tyrrell 2019). By smearing oil on the hull planking, medieval boatbuilders would have applied it on the luting, unintentionally mixing it with the bitumen amalgam.

Quicklime (CaO), obtained from burnt shells and corals, and goat fat are the main ingredients of the traditional antifouling (Arabic: *shaham*; Malay/Tamil/Chinese: *chunam*) of Indian Ocean watercraft (Agius 2002; Yule and Burnell 1886). The hulls of vessels were periodically coated with this material outboard below the waterline to protect them against ship worms (*Teredo navalis*) and barnacles (Agius 2002; Lorimer 1915; Vosmer 2007, 2017; Weismann et al. 2014). References to this practice date back to the medieval period in Arab, Indian and Chinese ships, as mentioned by al-Masʿūdī (1861) and Marco Polo (Yule 1871). The use of a lime-based substance is also indicated by archaeological evidence from medieval shipwrecks from China and Southeast Asia (Flecker 2001; 2007; Li 1989). Timber Wo37 from al-Balīd bears traces of a white substance on its outer surface which resembles *chunam*. The amount of substance was too little to allow scientific analyses to determine its chemical composition and the identification of the material. However, its colour and texture match those of the Indian Ocean antifouling, corroborating information provided by historical sources. As in the case of oil, *chunam* would have coated the planking seams and luting, perhaps leaving some traces in the bitumen below.

Seven samples reveal the presence of resin in the bitumen mixture. Apart from a 16th century Portuguese source reporting vessels in East Africa caulked with black "pitch" mixed with resin and incense (Prins 1986), resin is not usually associated with bitumen in boatbuilding. A lump of bitumen discovered in Akkaz, Kuwait, and dated to the Partho-Sasanian Period is the only other archaeological evidence for the use of resin in a bituminous compound (Connan 2011). The function of this mixture is still not clear, but it appears very similar to substances used for mummification in Pharaonic Egypt (Connan 2011), and it might not have used in a maritime context. As we have previously seen, resin was widely used with the same purpose of bitumen in other sewn boats of the Indian Ocean and often mentioned in the historical sources. Perhaps the bitumen luting in the al-Balīd timber was applied when the vessel was resewn and smeared over a previous luting made of resin, or vice versa, causing the two substances to mix together.

Overall, the evidence mentioned above raised the possibility that some of the materials in the bitumen amalgam on the al-Balīd timbers were not originally added to the mixture by the boatbuilders. The fact that it is likely that some of these materials occurred on the hull of medieval vessels, might suggest that their presence in the amalgam could have been instead the result of an accidental mixing at a later stage, when these elements and the bituminous mixture came into contact.

Chemical analysis provides insights into the sources of the natural bitumen used in the al-Balīd timbers, suggesting southwest Iran seeps for most of the samples. A possible Iranian origin corroborates the only documentary source on the topic by the Dominican missionary Jordanus (1863), who, in the early 14th century, reports that "in Persia are springs, from which flows a kind of pitch". A similar origin for the bitumen samples is significant, because it points to this geographical area as the main source of bitumen in the Indian Ocean during the medieval period. It also appears to confirm a trend noted for earlier periods based on the study of bitumen lining torpedo jars, which indicated a shift from Iraqi to Iranian sources between the 1st and the 10th century CE.

Very little is known about the use and trade of bitumen in the medieval period, but this material would have probably been transported from the natural seeps of the interior to the coastal centres in the southern Iranian coast, such as Siraf, Qays (Kish) and Hormuz.

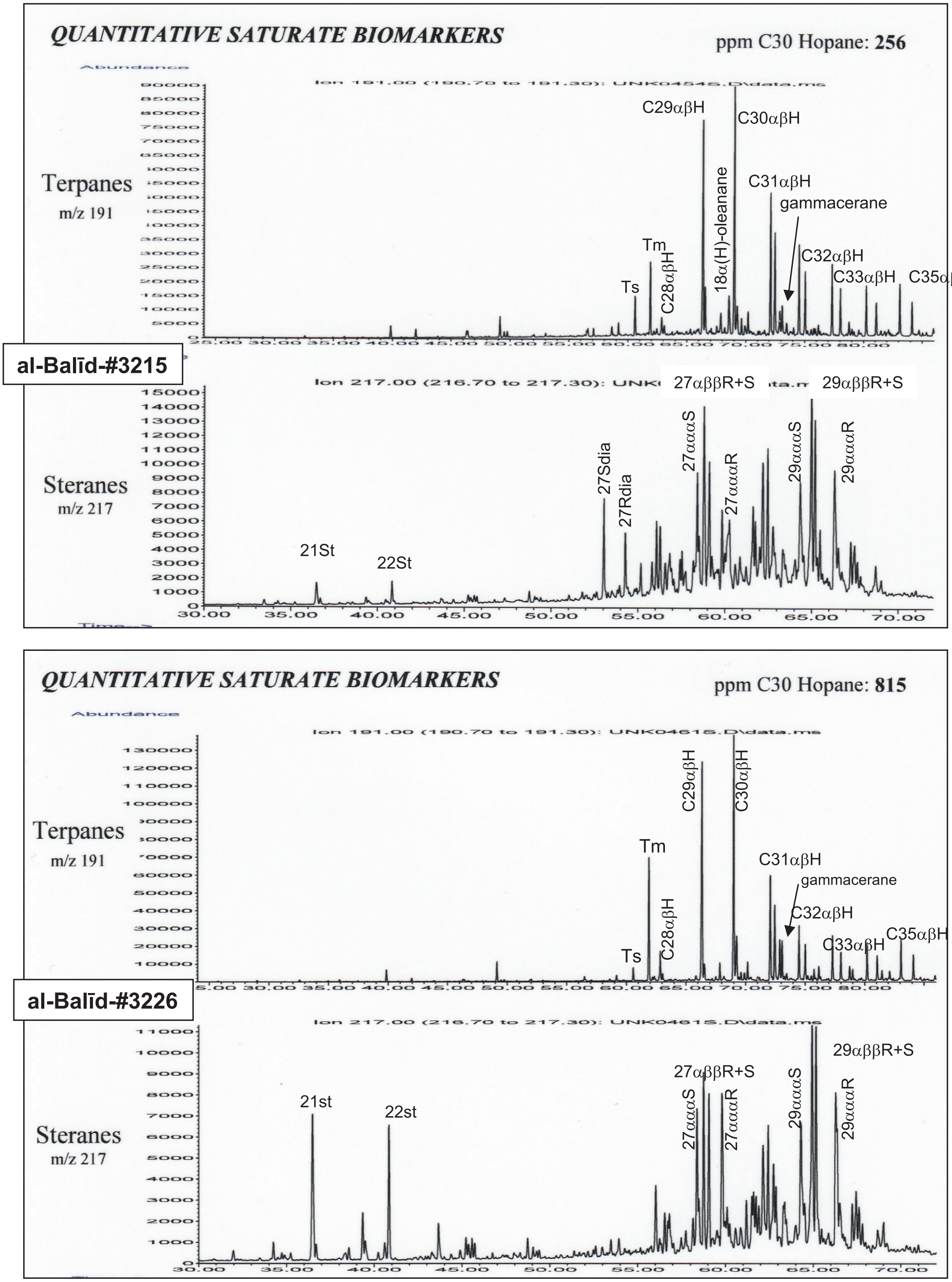

Fig.18

Fig. 18. Mass fragmentograms of steranes (*m/z* 217) and terpanes (*m/z* 191) of two samples of al-Balīd: No. 3215 and No. 3226. Significance of abbreviations: 21St = pregnane, 22St = 20-methylpregnane, 27αααS = 5α,14α,17α-cholestane 20S, 29αααR = 5α,14α,17α-stigmastane 20R, 29αααR = 5α,14α,17α-stigmastane 20R, 27Sdia = 13β,17α-diacholestane (20S), 27Rdia = 13β,17α-diacholestane (20R), Ts = 18α,22,29,30-trisnorneohopane, Tm = 17α,22,29,30-trisnorhopane, C29αβ = 17α,21β-30-norhopane, C31αβH = 17α,21α-hopane, C31αβHS = 17α,21β-30-homohopane(22S).

Table 5
Molecular data on the al-Balīd samples.

Lab number	C30hopane ppm	Ts/ Tm	Olean/ Hop	GA/ C31HR	ster/ terp	Rear/ Reg	C27ββR + S	C28ββR + S	C29ββR + S	C29ββS/ C29ααR	steranes	terpanes
2360		0.43	0	0.32							perserved	preserved
2362		0.62	0.91	0.25							altered	preserved
2933	116	0.43	0.03	0.44	0.16	0.17	26.7	33.4	39.9		slighly altered?	preserved
2933 bis	93	0.39	0.02	0.52	0.22	0.29	27.8	31.4	40.8	0.98	slightly altered	well preserved
2934	760	0.11	0.01	0.56	0.11	0.12	24.7	25.5	49.8		preserved	preserved
2934 bis	439	0.11	0.01	0.55	0.14	0.12	25.1	24.5	50.4	1.77	preserved	preserved
2936	116	0.33	0.02	0.51	0.09	0.08	16.7	35.1	48.3		slightly altered	preserved
2935 bis	124	0.22	0.2	0.43	0.32	0.15	14	9.8	76.3		altered	slightly altered?
3208	681	0.11	0.01	0.53	0.13	0.13	24	23.4	52.6	1.62	preserved-siighly altered	preserved
3209	242	0.63	0.65	0.22	0.55	0.31	22.5	22.6	54.9	0.66	altered	slightly altered?
3210	5	0.2		0.06	8.16	0.04	4.7	17.9	77.4	0.26	severely altered	severely altered
3211	116	0.61		0.2	0.42	0.63	29.9	27.5	42.6	30.2	altered	altered
3212	48	0.38	0.01	0.18	0.24	0.26	30.3	24.6	45.1	1.19	preserved	preserved
3215	256	0.54	0.15	0.22	0.35	1.27	28.6	31.2	40.2	1.36	well preserved	well preserved
3218	175	0.66	0.66	0.22	0.54	0.32	23.3	23.7	52.9	0.75	altered	partially altered
3220	15	2.48		0.14	3	0.04	4	14.8	81.2	0.31	severely altered	severely altered
3221	255	0.36	0.01	0.56	0.19	0.27	27.6	32.6	39.8	0.95	partly altered	preserved
3222	183	0.37	0.01	0.54	0.19	0.25	27.5	29.4	43.2	1.11	slighly altered	preserved
3223	284	0.27	0.02	0.49	0.2	0.21	29.5	26.5	44	1.32	slightly altered	preserved
3225	141	0.49	0.07	0.46	0.21	0.4	23.1	31.3	45.6	1.29	slighly altered	preserved
3226	815	0.11	0	0.53	0.18	0.06	31.5	23.6	44.9	1.43	preserved	preserved
3227	2		1.13	0.77	4.8	0.1	6.8	12.6	80.6	0.13	severely altered	severely altered
3228	22		0.18	0.4	0.3	0.77	19	13.8	67.2	0.71	severely altered	severely altered
3229	694	0.12	0.03	0.52	0.17	0.09	28.3	21.9	49.8	1.3	preserved	preserved

Unfortunately, the geochemical analysis cannot tell us whether the bitumen would have reached coastal areas already mixed and ready to use, or the amalgam was prepared at the boatyards before being used.

One important question raises from the identification of the bitumen sources: can the origin of the bitumen help us to determine the places where the boats luted with it were built? It is difficult to answer this question because material origin in the Indian Ocean does not necessarily match with the places where these materials were used.

It is plausible that at least some of the al-Balīd timbers belonged to ships built in city ports in southern Iran. Siraf is probably the closest to the bitumen sources and had strong links with southern Arabia and particularly East Africa, the latter being the region where many of the timber species from the al-Balīd timbers are native. Historical sources also inform that the city imported exotic wood from India and East Africa (Agius 2007; Chittick 1977), while the pottery type discovered during the excavations also reveals a link with East Africa (Priestman 2021).

Ships from Siraf were so common in southern Arabia and Red Sea, that the Arabian Sea until Yemen was also known as *Bahr Fāris*, "the sea of Fars", where most of the "shipbuilders and seafaring men are Persians" (Hourani 1963). Hourani (1963) also states that it is probable that in the 10th century ships from Siraf reached East Africa by sailing along the coast to Aden and round the Somali coast, instead of choosing a direct route to Cape Guardafui, to avoid Indian pirates from Socotra. Diaspora from Siraf helped the growth of ports like Jedda and Aden and contributed to the development of East African centres like Mogadishu, Kilwa and Malindi (Agius 2007).

However, the city of Siraf declines in the 10th century, a date earlier than the date range of the al-Balīd timbers (late 10th-15th centuries CE). The island of Qays (Kish), which flourished in the 11-12th centuries, and where some of the merchants of Siraf moved after the city's demise, might be a better candidate as a boatbuilding place for the "al-Balīd ships". Historian Yāqūt claims that the ruling house of Quays had Southern Arabian origin and controlled the Arabian Sea and was in fact the ruler of Oman (Goitein and Friedman 2007).

New Hormuz would have also been an important boatbuilding place. It was an important city port, actively involved in the Indian Ocean trade, with a seaborne empire controlling coastal centres in Oman and in the Gulf (Agius 2007).

Unfortunately, none of these Iranian coastal sites have provided direct evidence for boatbuilding facilities, let alone the use of bitumen in a maritime context, which might link the al-Balīd timbers to ships from southern Iran. In fact, the probable Iranian origin of the al-Balīd bitumen does not necessarily imply that boats were built near the material source. In a world like the Indian Ocean, with intertwined material trade networks connecting various centres along its coasts, it is likely that bitumen was one of the materials traded. As in the case of timber for shipbuilding, lacking in places like the Arabian Peninsula, Red Sea and the Gulf, luting material such as bitumen had to be imported for the construction of vessels in these regions.

However, the use of bitumen as opposed to other substances, might hint towards some regions of the western Indian Ocean, or exclude others, as possible places where the "al-Balīd" boats were built or repaired. For example, ethnographic records indicate that the preferred material to lute Indian sewn boats from the Lakshadweep, Kerala and Goa was a mixture made of resin (Shaikh et al. 2012; Varadarajan 1993; Severin 1985, Vosmer et al. 2011), which is largely available in those areas, and of good quality (Edye 1835; Regert et al. 2008). In East Africa, boatbuilders of the sewn *mtepe* smeared a thick paste of local mangrove bark to lute its hull (Hornell 1941). Although these are relatively recent evidence, one might consider it unlikely that these areas would have needed to import bitumen from Iran, when they could use locally available and cheaper material. The bitumen luting of the al-Balīd timbers, therefore, might point to boatbuilding places in the Gulf,

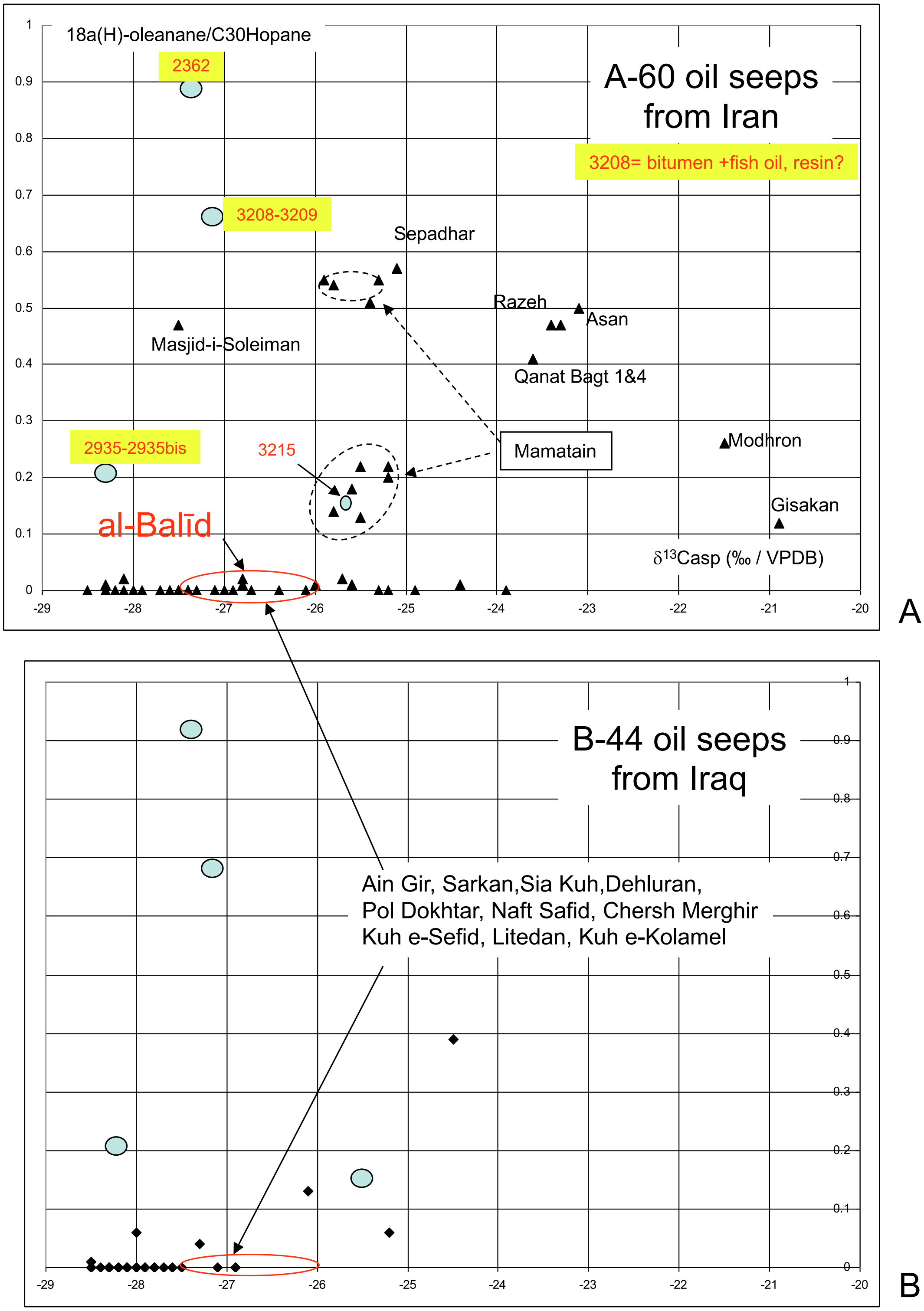

Fig. 19. Plot of 18α(H)-oleanane *vs.* δ^{13}Casp (‰ /VPDB). Comparison of al-Balīd samples with (A) –60 oil seeps from Iran, (B) 44 oil seeps from Iraq.

Arabian Peninsula, and Red Sea, which have no luting material such as resin and are relatively close to the Iranian bitumen sources.

In the Gulf, Bahrain is one possible origin for some of the "al-Balīd boats", because of its long maritime history and proximity to Iran. Archaeological excavations at Qalʿat al-Bahrain indicate that the site imported bitumen for millennia until the 16th century, the sources of which were exclusively Iranian during the medieval period (Connan et al. 1998).

Some of the al-Balīd timbers could have belonged to vessels built in al-Balīd. A city so deeply involved in maritime trade would have certainly had shipbuilding facilities. Newton and Zarins (2017), who directed the excavation at al-Balīd between 2005 and 2012, have suggested that a large complex located in the southeast limit of the site could be interpreted as a drydock. The large number of ship timbers recycled in the citadel of the site also strongly point to the building and repairing of watercraft (Ghidoni and Pavan 2022). Occurrence of frankincense in the bituminous mixtures suggests at least repairs of boats at al-Balīd. Moreover, the pure bitumen in the metal container

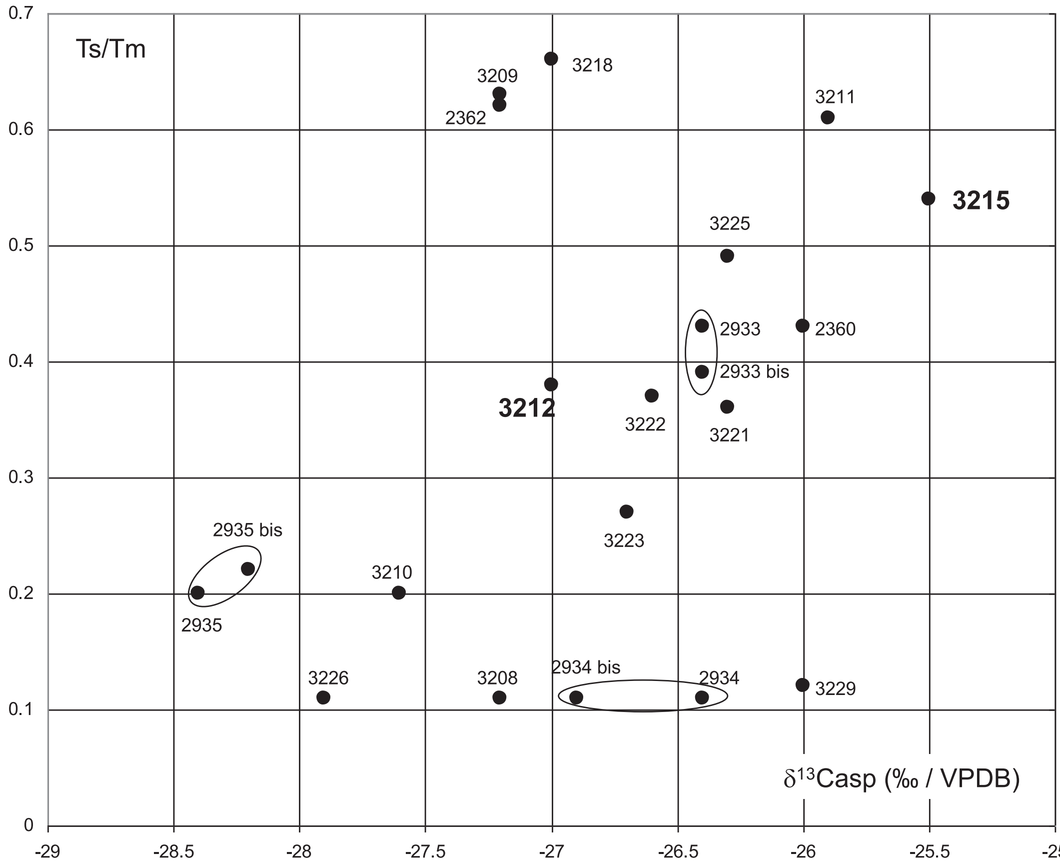

Fig. 20. Plot of Ts/Tm *vs.* δ^{13}Casp (‰ /VPDB) for the al-Balīd samples.

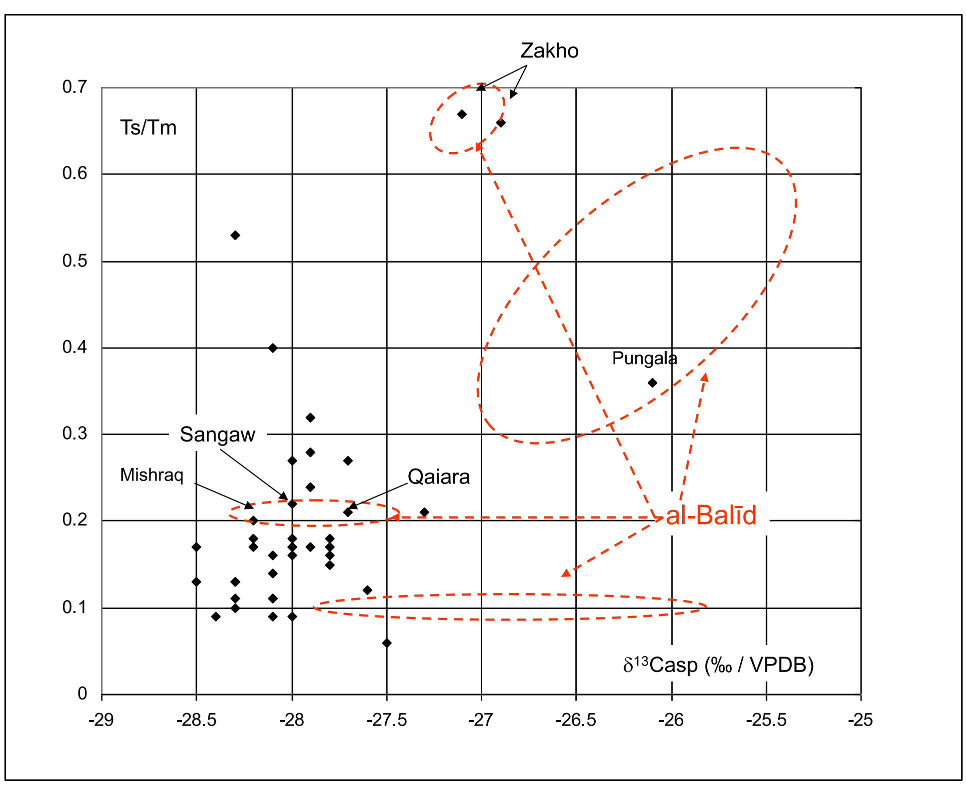

Fig. 21. Plot of Ts/Tm *vs.* δ^{13}Casp (‰ /VPDB) for the oil seeps of Iraq: comparison to the al-Balīd samples.

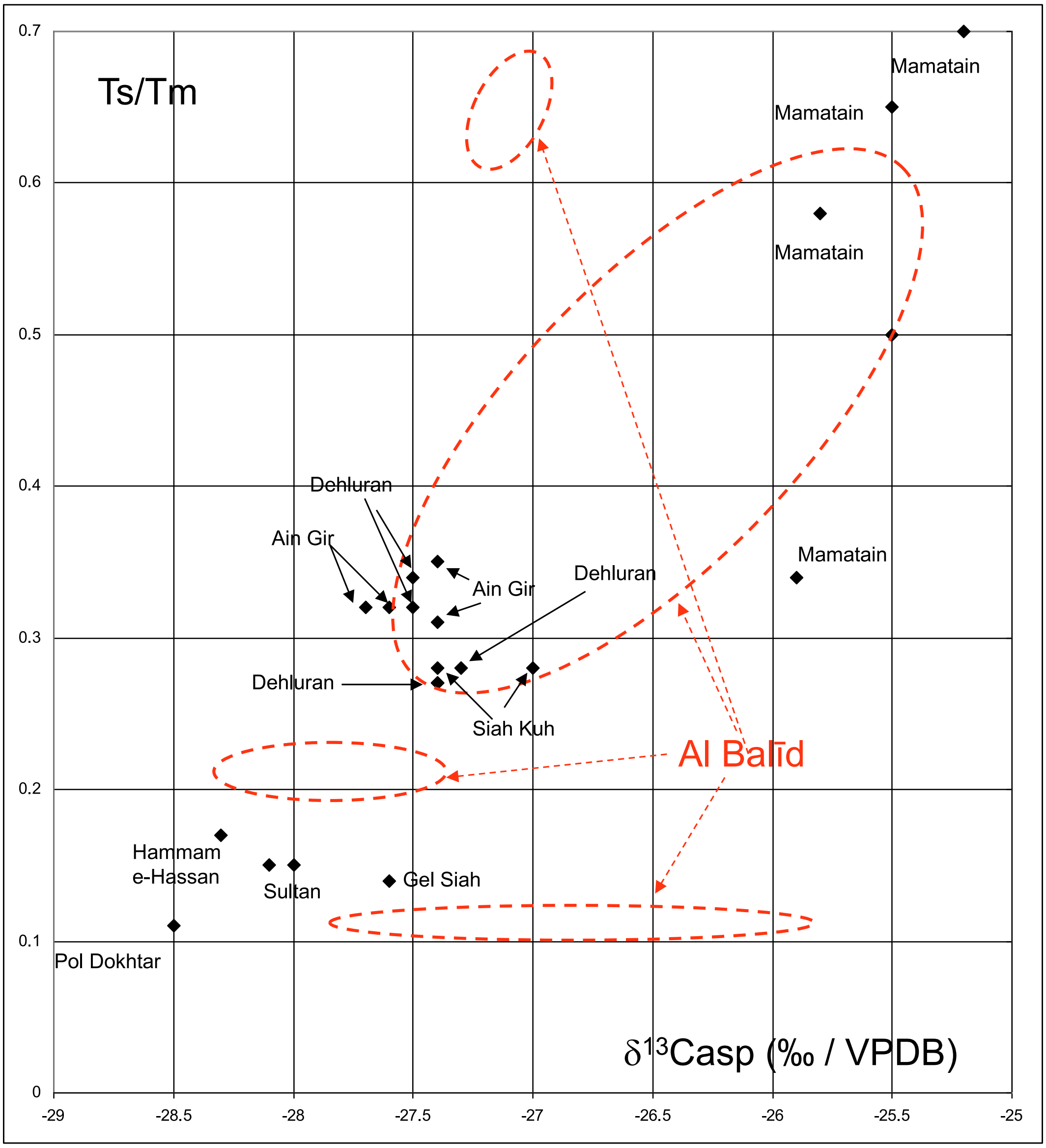

Fig. 22. Plot of Ts/Tm *vs.* δ^{13}Casp (‰ /VPDB) for the oil seeps of Iran: comparison to the al-Balīd samples.

discovered in al-Balīd, might also be a clue for these activities at the site premises. Bitumen could have been imported to al-Balīd and there mixed with other materials to create the amalgam to lute the ships built or repaired at the site.

Lastly, one should also consider that the bitumen amalgam on the al-Balīd timbers might not have necessarily been applied during the construction of the vessel, but at a later stage. The sewing cordage of sewn boats had to be regularly replaced, and it is also possible that the luting on the al-Balīd timbers might have been smeared on when the vessels had been re-sewn.

8. Conclusion

Chemical and mineralogical analyses of the luting of the al-Balīd timbers offer information about the sources, processing and use of bitumen in the Indian Ocean during the medieval period. They also yield insights into boatbuilding materials and practices in the region, deepening our understanding of sewn-plank construction during the middle Islamic period. The identification of the timber luting as a bitumen-based compound suggests that bitumen was the predominant material used by the coastal communities of the western Indian Ocean to seal the planking of their vessels. The data from al-Balīd also indicates that the use of bitumen amalgams to waterproof hulls of vessels in the region, generally associated to much earlier periods, continued in the 10-15th centuries, revealing the key role played by bitumen in the Indian Ocean boatbuilding context for millennia.

Only a limited area of al-Balīd has been excavated, and a large number of ship timbers are visible in the masonry and rubbles of the buildings. Future excavations will offer the opportunity for further analysis to determine whether there are changes in bitumen sources and variation in its mixture through time.

CRediT authorship contribution statement

Jacques Connan: Writing – original draft, Writing – review & editing. **Alessandro Ghidoni:** Writing – review & editing. **Tom Vosmer:** Writing – review & editing. Elodie Mezzatesta, Céline Joliot and Carole Mathe carried out the analysis of fat, resin and bitumen. Renaud Gley and Isabelle Bihannic did the mineralogical analysis. Alexia Pavan provided samples. Michael H. Engel acquired the isotopic data and was involved in the review-editing. Alex Zumberge did the biomarker analyses.

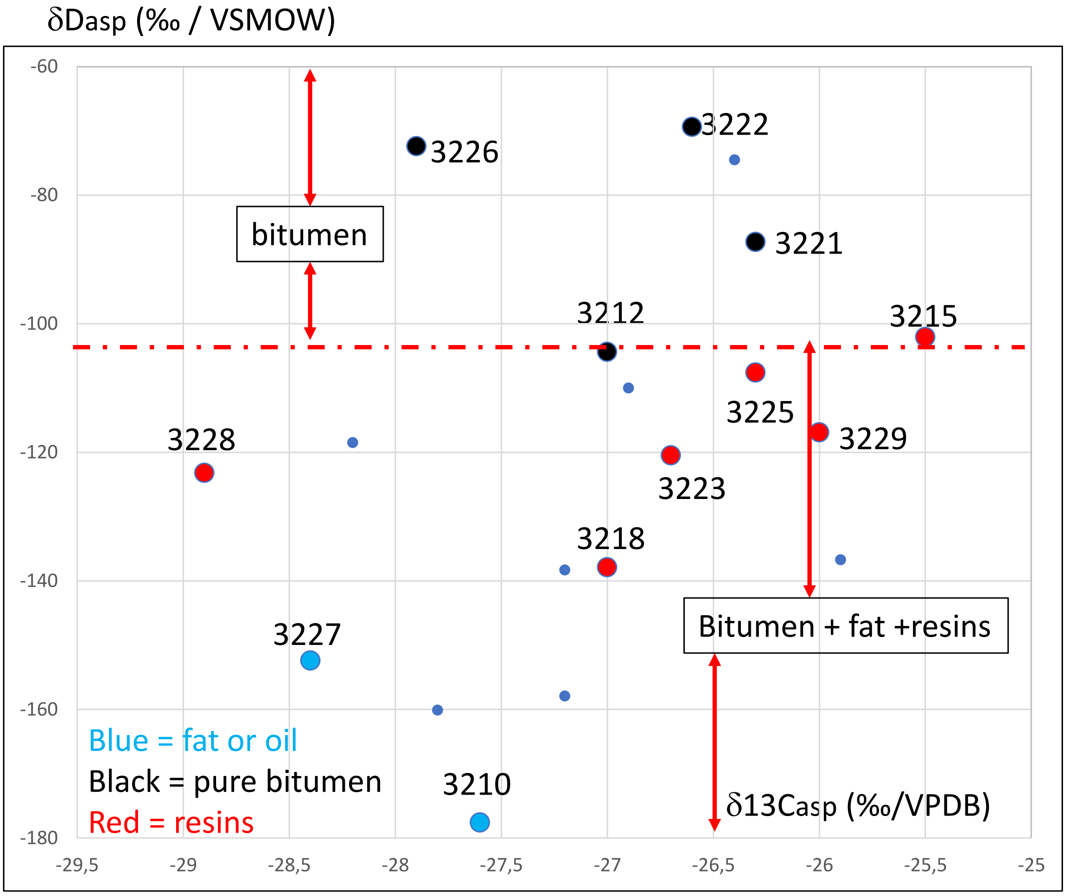

Fig. 23. δDasp (‰ / SMOW) *vs.* δ^{13}Casp (‰ /VPDB): modified Fig. 13 according to the results of complementary chemical investigations.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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