A preliminary study on coal cubes found at a Roman wreck off the Island of Kasos

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Abstract

In September 2020, during an underwater survey conducted by the Greek Ephorate of Underwater Antiquities and the National Research Foundation, 50 waterlogged coal cubes, measuring 20×20×20 cm, were discovered at a Roman shipwreck off the island of Kasos.

This preliminary study aimed to investigate basic physico-chemical and morphological properties of the constituent material of the cubes. Knowledge of the manufacturing technology and preservation state of the cubes is expected to aid in the design of an appropriate conservation treatment and provide important pieces of evidence in identifying the origin and use of the cubes.

Light and Scanning Electron microscopy was used to document the morphological features of the material, while some basic physical properties were calculated gravimetrically. In addition, solubility and chemical composition of the cubes using Infrared Spectroscopy and Energy Dispersive X-ray analysis were determined.

Based on the results obtained, it appears that the material constituting the cubes is neither a charcoal nor some type of coal. It is indicated that the Kasos wreck cubes are more likely an industrial product which was made by compressing and shaping crushed coal with a binder. Similar cubes, called 'patent fuel' were commonly manufactured in the 19th Century in Cardiff, Wales, for heating purposes or energy production. Hence, the Kasos wreck cubes are unlikely to have originated from the Roman shipwreck. They most probably belong to a contemporary ship that also wrecked at the site.

Regarding the preservation state of the cubes, initial results showed that the material does not shrink during air drying, does not change texture and color and it does not become brittle; hence no immediate conservation actions appear to be needed.

Nonetheless, further analysis and testing are considered necessary for a more precise identification of the material and for a better understanding of its behaviour during and after air-drying.

Keywords: coal cubes, Kasos Roman shipwreck, patent fuel

Introduction

In September 2020, during an underwater archaeological survey conducted by the National Research Foundation and the Greek Ephorate of Underwater Antiquities, a Roman shipwreck was discovered off the island of Kasos (Argiris 2020). Mixed with the ship's cargo and scattered over a large area around the wreck, 50 carbonaceous black cubes were found. The cubes, measuring 20×20×20 cm and weighing 9~10 kg each, appeared to be a type of solid fuel transported via the sea (Argiris 2021).

Solid fuels of this shape and size could be either charcoal or coals. Charcoal is the solid residue derived from the heating of wood over 270 °C under anoxic conditions, a process termed carbonisation (Emrich 1985, Straka 2017). Carbonisation of wood takes place either in pits that are covered with soil, or in kilns made by clay, bricks, concrete, stone or metal (Emrich 1985, Straka 2017, Kammen and Lew 2005). In Europe around the first millennium BC, charcoal-making had already become an important industry

for the recovery of iron and other metals from ores (Richardson 1934, Emrich 1985).

On the other hand, coals are fossil fuels, developed through the process of coalification, where buried fallen plant matter, with the aid of increasing heat and pressure through millions of years, are turned from peat into lignite, sub-bituminous coal, bituminous coal and finally into anthracite (Flores 2014, Speight 2015, Grammelis et al. 2016). Coal was used relatively early in human history. There are reports that the oldest coal mine was in operation in northeastern China around 1000 BC (WCI 2005, Speight 2015). As for Europe, coal cinders found among ruins in Roman England, indicate that the Romans used coal for a variety of works, while records from the Middle Ages provide the first evidence of coal mining on the European peninsula (Smith 1997, WCI 2005, Speight 2015). It was, however, during the industrial revolution in the 18th and 19th Century that demand for coal surged. The great creation of the steam engine by James Watt, patented in 1769, was largely responsible for the growth in coal use at that time (WCI 2005, Fernihough and O'Rourke 2020). Eventually oil overtook coal and became the largest source of primary energy in the 1960s. Coal, however, still plays a vital role in the world's primary energy (WCI 2005, Grammelis et al. 2016).

The use of coals and charcoal as fuels, therefore, appears to have played an important role quite early in human history. However, due to the lack of historical documentation from ancient to medieval times, it is not well known to what extent, with which form and for what purpose these fuels were used.

For this reason, the discovery of the Kasos wreck cubes raised many questions. Were they a man-made product of the Roman times? Were they made of a type of coal or of charcoal? Did they actually belong to the Roman wreck or to another ship that sank in the same area, and finally, how well were they preserved? Did they require conservation?

Therefore, this work was set up to study, on a preliminary basis, the material constituting the cubes and some of its physico-chemical properties in order to help identify their origin and use, and to assess their need for remedial conservation.

Materials and methods

The material investigated

The material examined was derived from one of the cubes retrieved from the Kasos wreck in 2020 (Figure 1). The sample examined was waterlogged, bore deposits on its surface due to the activity of marine organisms and measured ~5.5×5.5×5.5 cm. For comparative reasons, the investigation of the cube sample was conducted in comparison with other coal types including lignite (Greece), bituminous (Poland) and an anthracite sample of an unknown origin.

Figure 1. The coal cube retrieved from the Kasos wreck (with permission by X. Argiris).

Macroscopic examination

The material was first cleaned with a scalpel to remove deposits and then observed macroscopically under a LED Magnifying Desk Lamp 20X. After this, the sample was examined using a Leica Wild M3B stereoscope with lenses 6.4, 16 and 40.

Scanning electron microscopy (SEM)

Samples of cube material from the wreck (0.5×0.5×0.2 cm) were mounted on aluminium stubs with a conductive carbon glue and then coated with gold-palladium in a Polaron Range sputter coater. The examination was performed with an JEOL SEM JSM-6510 LV on a high vacuum, at 20 V.

Moisture content

To determine the % equilibrium moisture content (EMC) and the % moisture content (MC), six samples measuring ~ 1.5×1.5×1.5 cm, were taken from the material. The samples were left to air dry at 65 ± 5% RH and 21 ± 5 ºC, until three successive constant weight measurements were recorded. They were then oven dried at 100 ± 3 ºC until constant weight. Prior to each weighing, samples were kept in a desiccator with silica gel for 10 minutes, until samples reached room temperature. For the calculation of % EMC and % MC, equation 1 was used. The procedure for the % MC determination was an adaptation of the ASTM D 3173–03 and was also followed for lignite, bituminous coal and anthracite samples.

$$\% \text{ EMC or } \% \text{ MC} = [(m_1 - m_2) / m_2] \times 100 \quad \text{Equation 1}$$

where:
m_1 = the initial mass of the sample
m_2 = the final mass (at equilibrium moisture, for % EMC, or after oven-drying for % MC)

True density

To determine the true density of the material, three air-dried samples (~1.5×1.5×1.5 cm) were weighed and their volume was estimated with the water displacement method. Density was then calculated according to equation 2. Along with the cube samples, the density of three samples of each coal type (lignite, bituminous and anthracite) was calculated comparatively.

$$d = m / v \quad \text{Equation 2}$$

where:
d = the true density
m = the air-dried mass of the sample in g
v = the volume of the air-dried sample in cm^3

Relative density

The relative density was also calculated by weighing three samples (1.5×1.5×1.5 cm) of the material in air and in water and then using equation 3. Similarly, relative density was calculated for lignite, bituminous coal and anthracite with three replicates.

$$RD = W_{air}/(W_{air}-W_{water}) \quad \text{Equation 3 (Nebel 1916)}$$

where:
RD = the relative density of the material
W_{air} = the weight of the material in the air
W_{water} = the weight of the material in water

Shrinkage

For the calculation of % shrinkage (β) three waterlogged samples of the cube, measuring ~1.5×1.5×1.5 cm, were used. Two stainless steel insect pins were placed on each sample and the distance between the pins was recorded with a calliper (0,02 mm). Samples were then air-dried at 65 ± 5% RH and 21 ± 5 °C and when their moisture content reached equilibrium (three successive constant measurements) they were measured again with the calliper. Following this, the samples were placed in an oven at 100 ± 3 °C for 48 hours and placed in a desiccator with silica gel. When the samples reached room temperature the distance between the pins was measured again. The % air- or oven-dried shrinkage was calculated using equation 4.

$$\beta\% = (\beta_1-\beta_2)/\beta_1 \quad \text{Equation 4}$$

Where:
β1 = the initial waterlogged dimensions
β2 = the dimensions of the air- or oven-dried material

Hardness

For the calculation of hardness, one sample from the cube (~2×1.5×1.5 cm) was left to air-dry at 65 ± 5% RH and 21 ± 5 °C. Following this, the surface of the sample was made flat on a metallographic grinding polishing machine Planopol-V at 120 rpm with a 120-grit paper. Then Vickers' hardness under a load of 5 kgf (HV/5) was measured on the flat surfaces with a Zwick/ Roell 8187 5LKV, universal hardness testing machine. Five replicate measurements were performed on each sample, with a distance between measurements of 3 times the diagonal length of the indentation based on the ASTM ISO 6507-1. For comparative reasons, the hardness was also measured on lignite, bituminous coal and anthracite samples following the same procedure.

Solubility

To test the solubility of the material, six organic solvents were used: acetone, white spirit, ethanol, benzyl alcohol, benzene and xylene. Six samples (~ 1.5×1.5×1.5 cm) of

the cube material which had been dried at 100 ± 3 °C for 48 hours in an oven to remove their moisture, were immersed in the different solvents and their dissolution was recorded.

Fourier transform infrared spectroscopy (FTIR)

The organic composition of the samples was examined using Fourier transform infrared spectroscopy (FTIR). A sample from the cube material (~1×1×1cm) was air dried and pulverised with a glass mortar and pestle to a grain size of 250 μm (sieve N. 60). It was then mixed with KBr powder to form pellets. Two of the pellets were tested in a BRUKER ALPHA II spectrometer and the spectra were processed with 'SpectraGryph' software. The same process was adopted for samples of lignite, bituminous coal and anthracite.

Energy dispersive X-ray analysis (EDS)

For the investigation of the inorganic composition of the cube, the same samples used for the SEM examination (0.5×0.5×0.2 cm) were analysed. Before gold coating, samples were examined with a SEM JSM-6510 LV equipped with a Pentafet X-act detector (Oxford Instruments). The spectra acquired were processed using the INCA software.

Results and discussion

Macroscopic examination

The macroscopic observation of both air-dried and water-logged material showed that it ranged in colour from black to gray, and was a compact, granular and inhomogeneous mass, which was mostly rough in texture but also presented rarely some vitreous areas (Figure 2). Furthermore, the material did not show any type of organisation on a structural level or in a specific anatomical direction and thus the hypothesis that the material was a type of charcoal could be discounted.

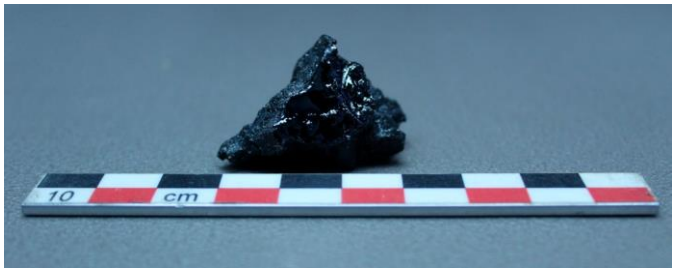

Figure 2. Vitreous areas of the material.

The stereoscopic observation also confirmed the inhomogeneity of the structure of the material and showed that the rough areas were constituted by numerous smaller granular units, whereas the glossy ones were a homogenous vitreous mass (Figure 3). This stereoscopic image gave the impression that the cube was actually made of two materials and was probably created by mixing a crushed solid granular rough material together with a liquid binder which was solidified with time and became smooth and glossy (Figure 3).

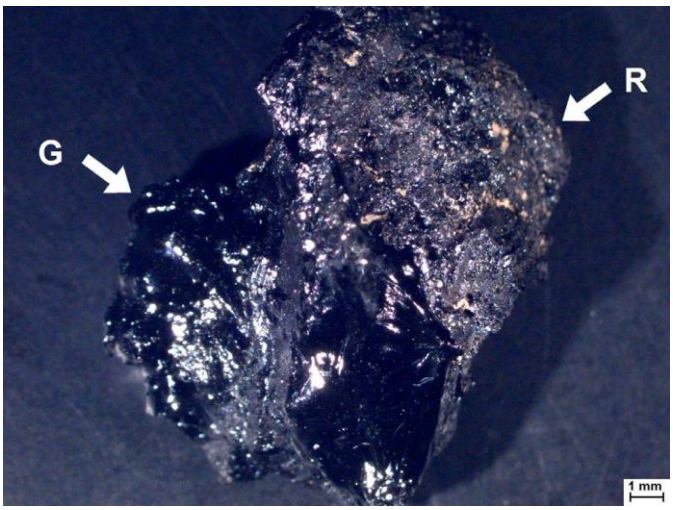

Figure 3. Granular rough (R) and glossy (G) areas observed.

Scanning electron microscopy (SEM)

SEM confirmed the previous visual observation and demonstrated that the rough surface of the material consists of grains of various sizes and shapes (Figure 4a). In contrast, the smooth and glossy surfaces seemed to be scattered in large areas on the material surface and were found embedded in granular parts or inside cavities (Figure 4b). Moreover, the morphology of these glossy surfaces indicated that probably they were once in a liquid phase under heat, and solidified when cooled, often entrapping the granular material (Figure 4c).

Moisture content, density, and shrinkage

The results of the material physical properties investigated are shown in Table 1. The MC of the cube (11%) corresponded to a dry, rather than a wet or even a waterlogged material. When comparing the EMC values (3,9 %) to other coal types it is shown that the cube EMC is similar to bituminous coal. Regarding density, the values recorded are

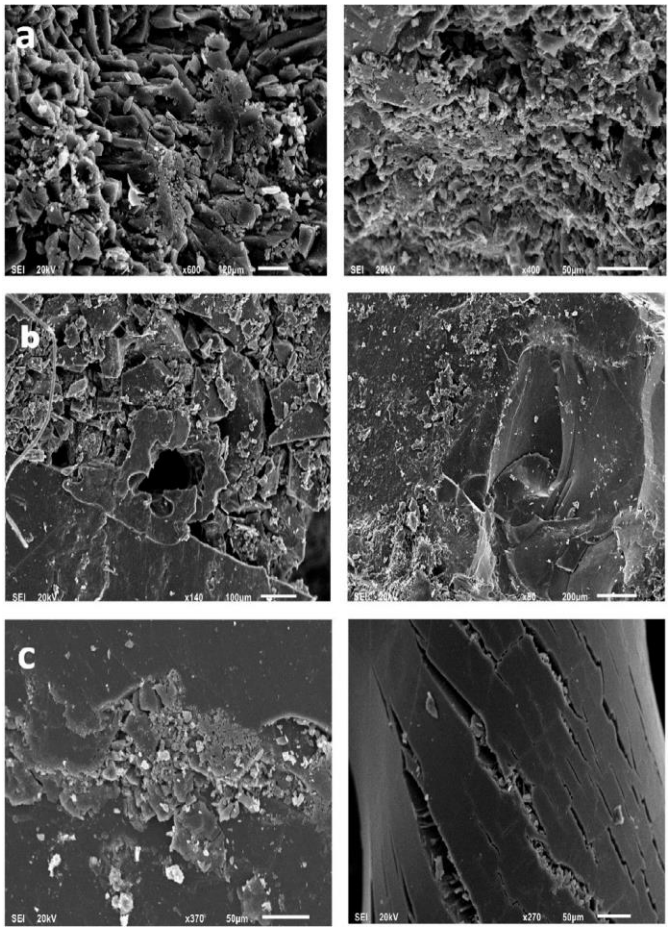

Figure 4. SEM micrographs of a) sample's rough surfaces, b) extent and location of smooth and glossy surfaces and c) granular material entrapped in smooth surfaces.

very similar to the other coal types examined. Nonetheless it should be taken under consideration that the cube is probably a composite material and thus direct comparison to pure coals may have no validity or importance.

The calculation of % shrinkage demonstrated that the material did not show either air-dry shrinkage, from the waterlogged to the air-dry state, nor an oven-dry shrinkage after drying at ~100 °C suggesting that the cubes from the wreck are not in danger of shrinkage or collapse if air-dried.

Hardness

The results of the Vickers' hardness test, are presented in Table 1. The values recorded show that the cube material is softer than the natural coals tested. This could indicate that the cube is made of a lower coal rank, or that the direct comparison to natural coals is not meaningful, as it is probably a composite material.

Table 1. Physical properties investigated for the cube material and the three coal types: lignite, bituminous coal and anthracite. Values are averages of three replicates. Moisture content and shrinkage for the three coal types were not calculated (NC) as the samples were air-dried when received.

	Wreck's cube	Lignite	Bituminous	Anthracite
Equilibrium moisture content (%)	3,9	11,18	1,94	14,72
Moisture content (%)	11	NC	NC	NC
True density (g/cm^3)	0,938	1,132	1,257	1,395
Relative density	1,236	1,282	1,284	1,527
Air-dry shrinkage (%)	0	NC	NC	NC
Oven-dry shrinkage (%)	0	NC	NC	NC
Vickers hardness (HV / 5 kgf)	12,2	13,24	20,26	27,06

Solubility

The results of the solubility of the material of the wreck cube in selected solvents are presented in Table 2 in comparison to bibliographic data for other materials. More specifically, benzene and white spirit did not dissolve the material. Benzyl alcohol and ethanol affected the material slightly, while acetone and xylene dissolved it satisfactorily. No complete dissolution, however, was achieved with any of the solvents tested.

Solubility tests also showed that the solid part of the material was almost unaffected by the solvents used, and that the small part that was dissolved was probably the 'binder' material. The dissolution of the binder in acetone, ethanol, benzyl alcohol and xylene indicate that possibly it is a form of tar.

Table 2. The solubility of the cube in several solvent tested. References used [1] NLM n.d., [2] Remler 1923, [3] Rossi 1988, [4] Pullen 1983, [5] Larsen et. al. 2014, [6] CAMEOa 2020, [7] Masschelein-Kleiner 1995, [8] Cislak 1943, [9] CAMEOb 2020

Materials	Solvents					
	Acetone	Ethanol	Benzene	White spirit	Benzyl alcohol	Xylene
Wreck's cube	X	X			X	X
Bitumen	X[2]			X[5]		
Wood tar	X[2,7]				X[7]	
Coal tar	X[2,1,4,6]	X[4,6]	X[1,4,6]		X[1]	
Creosote	X[3]	X[9]	X[3,9]	X[5]	X[3]	
Plant resins	X[2]		X[8]	X[5]		X[8]

Fourier Transform Infrared Spectroscopy (FTIR)

The results of the chemical analysis, obtained by infrared spectroscopy, are presented in Figure 5. The most significant peaks of the spectrum of the cubes are listed and assigned in Table 3, based on Yao et al. (2011), Coates (2006) and Boyatzis (2022). The majority of the peaks correspond to unsaturated hydrocarbons, including aromatics (peaks 4, 7, 8, and 9), except for peaks 2 and 5 which can be assigned to aliphatic chains. By comparing the infrared spectrum of the material (curve i in Figure 5) with the spectra of anthracite (ii), lignite (iii), and the bituminous sample (iv), it can be seen that all coal types share similar peaks at 2918-21 cm^{-1} and 2851-57 cm^{-1}, assigned to aliphatic hydrocarbon chains. However, from the range 1600 to 744, with the exception of the bituminous coal, there are several differences between the cube spectrum and those of the other coals. The bituminous coal spectrum matches to a great extent the wreck cube spectrum. In particular, the two spectra show similarities at 2918 cm^{-1}, 2856-57 cm^{-1}, 1593-97 cm^{-1}, 1435-37 cm^{-1}, 867-70 cm^{-1}, 809-11 cm^{-1} and 744-45 cm^{-1}, peaks 2, 3, 4, 5, 7, 8, and 9 respectively. It should be noted, however, that the spectrum of the cube shows three clear peaks (3042 cm^{-1}, 1375 cm^{-1} and 1031 cm^{-1}) appearing as shoulders in bitumen. Also, a weak broad band in the range 3800-3000 cm^{-1} is present in the cube but is stronger in the bituminous sample. These small differences between the two spectra are probably due to the processing of the material during manufacture or due to the mixing with a type of binder.

Energy dispersive spectroscopy (EDS)

The EDS showed that the dark areas of the samples are organic, mainly consisting of carbon and oxygen with small inorganic traces of iron, silica, sulfur aluminum and calcium (0.3-0.9%) (Figure 6a). The white/grey areas mostly consist of the same inorganic elements, however aluminium, silica and iron, were found sporadically in much higher concentrations up to 7% (Figure 6b).

Conclusions

Based on the physico-chemical properties of the Kasos wreck cube, it is indicated that no remedial conservation action is required. The sample examined did not show any decrease in dimensions or increase in fragility after

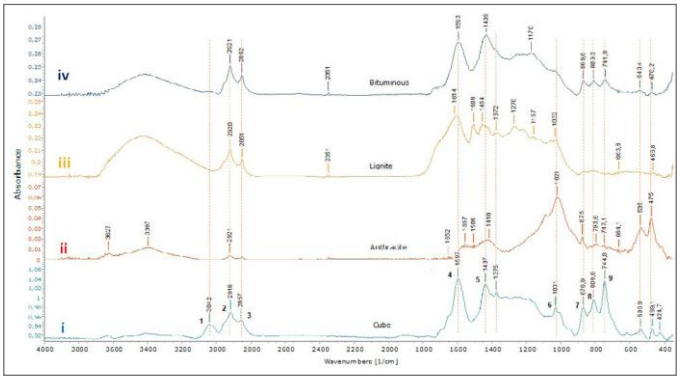

Figure 5. FTIR spectrum of the wreck cube material (i) in comparison to anthracite (ii), lignite (iii) and bituminous coal (iv) spectra after baseline correction.

Table 3. Assignment of the most significant peaks recorded for the cube material.

Peak N.	Maximum (cm^{-1})	Assignment	Comments
1	3042	υ C–H	Aromatic Hydrocarbons
2	2918	υ C–H	Aliphatic Compounds
3	2857	υ C–H	Aliphatic Compounds
4	1597	υ C=C	Aromatic Hydrocarbons
5	1437	δ C–H	Aliphatic Compounds
		υ C=C (Aromatic ring)	Aromatic Hydrocarbons
6	1031	υ C=C (Aromatic ring)	Aromatic Compounds
7	870	δ C=C–H	Aromatic Compounds
8	809	δ C=C–H	Aromatic Compounds
9	744	δ C=C–H	Aromatic Compounds

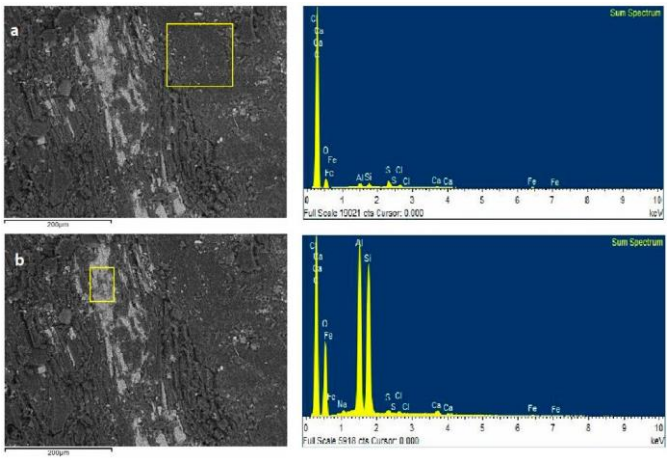

Figure 6. EDS results of a) dark areas of the material and b) grey/white areas of the material.

air drying and generally did not show any macroscopic changes that would prevent its handling, exhibition or storage. Nevertheless, for the safe drying of the wreck cubes, further research is considered necessary in order to predict the behaviour of a whole cube during and after air-drying.

Regarding the manufacturing technology of the cubes, it can be stated with relative certainty, that they are a man-made product. Their main constituent material appears to be a type of coal, mixed with a very small amount of a binder that is possibly a kind of coal pitch tar.

The morphological and physico-chemical properties of the cubes, along with their dimensions (20×20×20 cm), point out that they are probably a type of 'patent fuel'. Patent fuel was a 19th century artificial fuel made in Wales, England by mixing in a hydraulic press coal and binding substances, like pitch (Lathrop 1917, Hallett 1920). There were many factories producing this product with the same recipe, thus the only way of identifying them is their shape and imprinted logos. The most well-known patent fuel factories were the Star Patent fuel Co., the Crown Patent fuel, the Cardiff Patent fuel etc. (Davies 1908).

The dimensions and weight of the wreck cubes also indicate that they are meant for industrial or military use and not domestic. Several factories might have manufactured this kind of product around the world like the UNION in Germany, the Crown Patent fuel in England etc. Nonetheless, the Crown Patent fuel stands out from the rest due to the cubic shape of some of its products (Greene and Perkin 1922).

Therefore, the question arose why these cubes were found on and around a Roman shipwreck. To answer this question, only theories can be formulated at present. These include the possibility that the cubes may belong to two Greek steamers, 'Eleni' and 'Athinai' which were both hit by enemy ships during World War I and sunk at the Kasos Island strait (Helgason n.d.), very near where the Roman wreck was discovered. Another theory is that an Italian or Turkish steamer was hit during the seizure of the Dodecanese islands by the Italians in 1911 (Speronis 1955). Both parties were important buyers of patent fuel during that time (Davies 1908, Lathrop 1917, Hallett 1920). Last but not least there is a small possibility that a collier, a bulk cargo ship designed or used to carry coal, was sunk or just damaged and lost cargo in the specific area (Piozet 2018). The nationality of the collier could not be known, nonetheless it could be Greek as Crown Patent factory's archive show that Greece was a buyer of this product at least from the 1900s (Davies 1908, Lathrop 1917).

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