




## Article

# Archaeometric Analyses of *dolia* of the Final Bronze Age/Early Iron Age Found at Gagliato (CZ)–Calabria–Southern Italy

Anna Maria De Francesco <sup>1,\*</sup>, Antonina Maria Tucci <sup>2</sup>, Adriano Guido <sup>1</sup>, Anna Rao <sup>1</sup> and Donatella Barca <sup>1</sup>

<sup>1</sup> Dipartimento di Biologia, Ecologia e Scienze della Terra (DiBEST), Università della Calabria, 87036 Rende, Italy; adriano.guido@unical.it (A.G.); anna.rao@unical.it (A.R.); d.barca@unical.it (D.B.)

<sup>2</sup> Independent Researcher, 88100 Catanzaro, Italy; amtucci@libero.it

\* Correspondence: anna\_maria.defrancesco@unical.it

**Abstract:** In the hinterland of Soverato (in the Catanzaro province CZ) in Calabria, Italy, in the territory of Gagliato, on a series of terraced plains, the remains of an extensive settlement have been identified with archaeological evidence that includes the various phases of the protohistoric period and the Greek age. In the settlement, numerous protohistoric ceramic finds consisting in fragments of vascular shapes of various sizes and large *dolia* were recovered. This paper presents the preliminary results of the mineralogical, petrographic and paleontological analyses performed on *dolia* samples selected on the basis of the typology and the characteristics of the ceramic impasto. The *dolia* analyses were obtained through various analytical techniques. Petrographic and micropaleontological studies were performed using polarized optical microscopy (POM). The mineralogical analyses were carried out using X-ray diffractometry (XRD), both on selected ceramics and on the clayey and sandy sediments sampled for comparison around the settlement area. Furthermore, micromorphological observations have been performed, using scanning electron microscopy (SEM), on selected foraminiferal tests picked up from the clay sediments collected in the study area. Data highlight the remarkable compatibility between the mineralogical composition of the *dolia* and the sampled sediments, and they confirm that the raw materials for ceramic production may have occurred in an area not far from the settlement, as assumed by archaeologists.

**Keywords:** provenance; *dolia*; Bronze Age; Italy



**Citation:** De Francesco, A.M.; Tucci, A.M.; Guido, A.; Rao, A.; Barca, D. Archaeometric Analyses of *dolia* of the Final Bronze Age/Early Iron Age Found at Gagliato (CZ)–Calabria–Southern Italy. *Minerals* **2023**, *13*, 1422. <https://doi.org/10.3390/min13111422>

Academic Editor: Lluís Casas

Received: 24 July 2023

Revised: 3 November 2023

Accepted: 5 November 2023

Published: 8 November 2023



**Copyright:** © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

## 1. Introduction

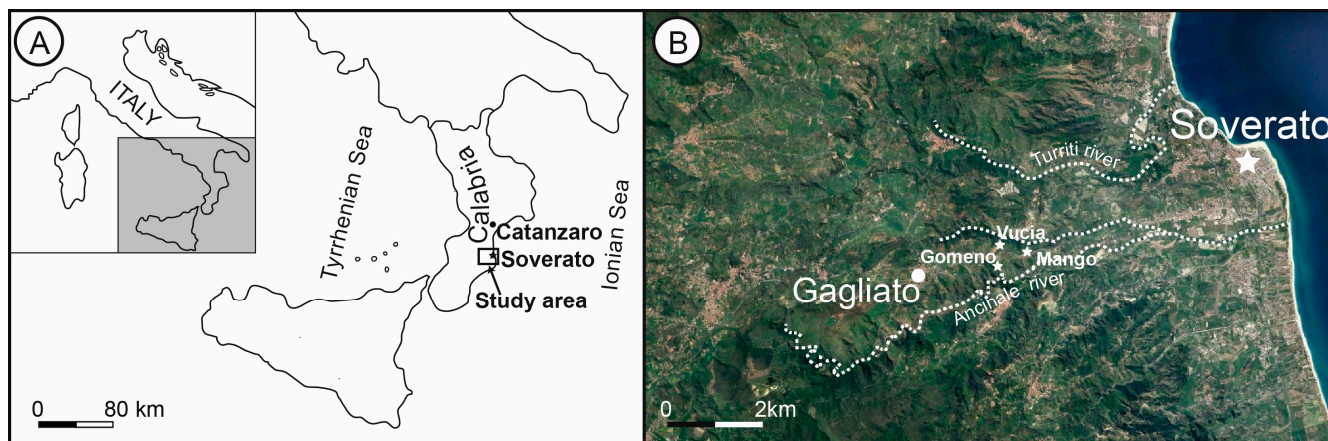
Since the mid-80s of the last century, the vast hilly area near the present town of Gagliato (CZ) has been the subject of surface research.

From a morphological point of view, the area is characterized by a few terraced plains, oriented in an East–West direction, delimited to the South by the Ancinale river and to the North by the Turriti torrent and by steep slopes, which represent a good natural defense (Figures 1 and 2).

The protohistoric material has been recovered in several adjacent sites: Gomeno, Mango and Vucia, located a few hundred meters apart from each other in the Gagliato territory (Figure 1). This complex settlement was widely occupied in the final Bronze Age; therefore, it falls into the category of “major” sites known in other areas of Calabria and Southern Italy during the Bronze Age [1–5].

The material collected during numerous archaeological campaigns in the Gagliato area includes fragments of tableware, mainly cups, truncated conical bowls and ollas, trays, pots, handles of various kinds and large *dolia*, the production of which continued even in the early Iron Age [2]. Previous studies [2,5] were devoted to the typological characterization of *dolia*. In detail, thirty fragments have decorations that allow the reconstruction of the shape, and ten fragments are bottoms and handles. There are also numerous *dolia* fragments whose shape it was impossible to identify. The diameter of the mouth varies between

36 and 60 cm, the overall profile of the body ranges from ovoid to globular and the average thickness of the walls is 3 cm. Some pieces have decorations with bands engraved in a herringbone pattern, horizontal bands of grooves, horizontal bundles of multiple furrows forming wavy motifs and three equidistant domes [2,5].



**Figure 1.** (A) Location of the study area in the Calabria region-Italy. (B) Detail of archaeological sites in the area between the towns of Soverato and Gagliato.

In Southern Italy, the large *dolia* in *impasto* ceramic production, between the final Bronze Age and the early Iron Age, also concerned other sites in the Gulf of Squillace, Crotona area, Tropea, Sicily, south-western Sibaritide and Campania, while the fine ceramics were found in northern Sibaritide, in the Matera area and in the Puglia region [6].

The role of these containers, comparable to the *pithoi* of Aegean tradition, was to store or transport large quantities of particularly valuable foodstuffs, such as oil and wine, even if it cannot be excluded that they contained other provisions. It is very probable that oil was stored or transported in all *dolia*, due to the wide mouth (36–60 cm) not being suitable for storing and transporting wine [5].

Previous studies [6] suppose that in Southern Italy, these large containers were made on site with the use of raw materials collected in the area [6]. This is hypothesized also by their dimensions, which made transport difficult [4,5] and it is confirmed by preliminary mineralogical and petrographic studies [7] which suggest the use of local sediments.

The samples analyzed in the present study consist of fourteen ceramic fragments selected among specimens of different *dolia* manufactured with a mixture of clay and sand with pebbles from medium to large-sizes, also evident on the external surface [5].

The present work aims to verify the local production of the protohistoric *dolia* of Gagliato through mineralogical, petrographic and paleontological techniques.

The area on which the protohistoric settlement developed consists of sedimentary deposits of clays, sands and conglomerates of the Plio-Pleistocene age, derived from the alteration of the intrusive and metamorphic rocks of the Massiccio delle Serre [8–10].

For this purpose, the sediments outcropping in the area were sampled and analyzed using X-ray diffractometry (XRD) to evaluate their possible use as raw materials in the ceramic production.

The local production of ceramics would confirm the importance of the area as a hub for trade between the coast and inland. Indeed, the interest in this archaeological complex is certainly due to its position for the strategic control of the area for the exploitation of the resources and for the distribution of local products over a larger scale since Prehistoric times.

The results obtained will certainly be very useful for stimulating the necessary interest aimed at a systematic excavation of this area, and the developments of such research could open up new interpretations on the role of the Soverato area in the Mediterranean during the Bronze Age.

For now, this paper provides preliminary data on the technology and raw materials used for the production of these large dolia, and it represents an example of the use of micropaleontological methodology for archaeometric purposes.

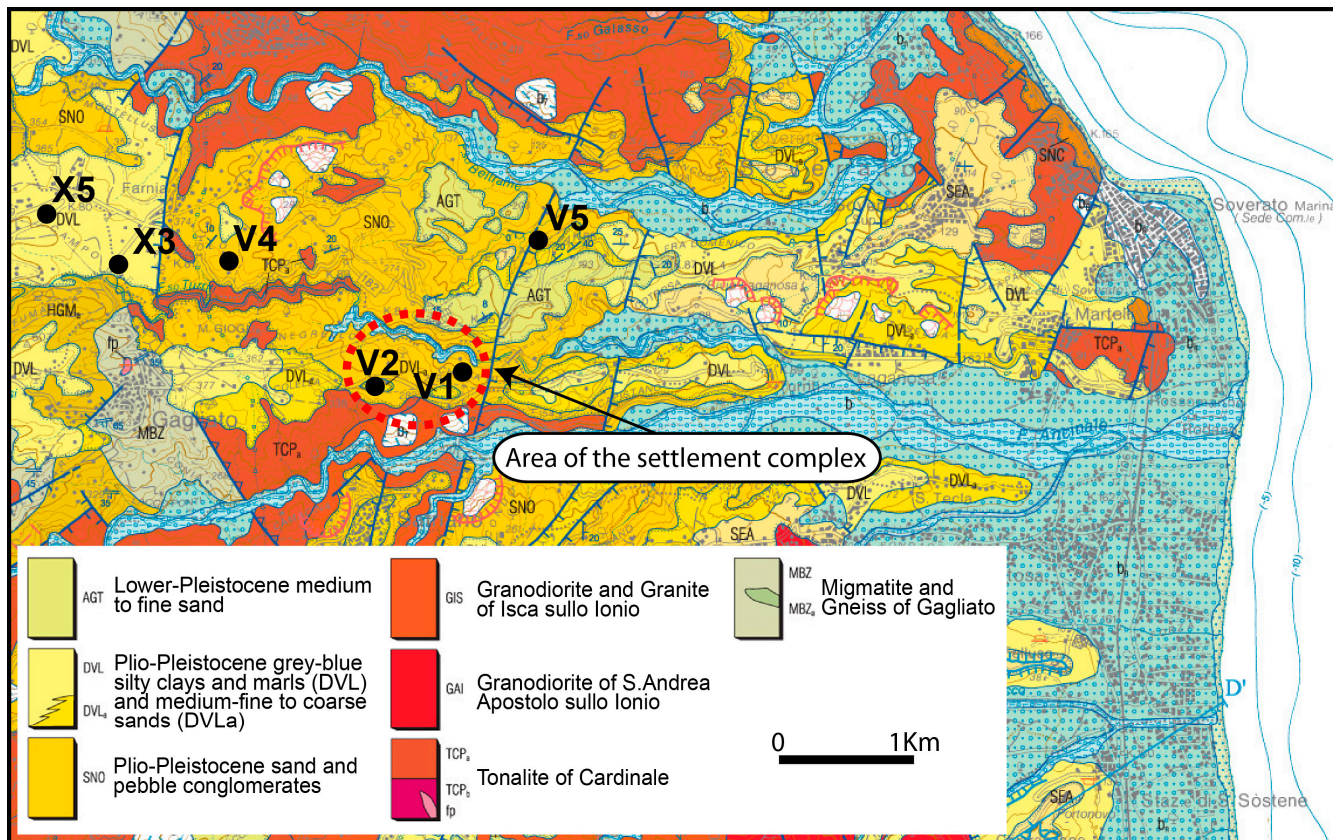


Figure 2. Geological map of sampling area modified from Foglio 580 Soverato [8].

## 2. Materials and Methods

Fourteen *dolia* fragments representative of the different shapes and decorations, coming from the settlement area of Gagliato, were selected: seven come from Mango (GA1, GA5, GA7, MG1, MG5, MG9 and GO20), five from Vucia (GA2, GA3, GA4, GO17, and VG4) and two from Gomeno (GO22 and GO23) (Figures 1 and 3). The list of fragments, their descriptions and bibliographic references are given in Table 1.

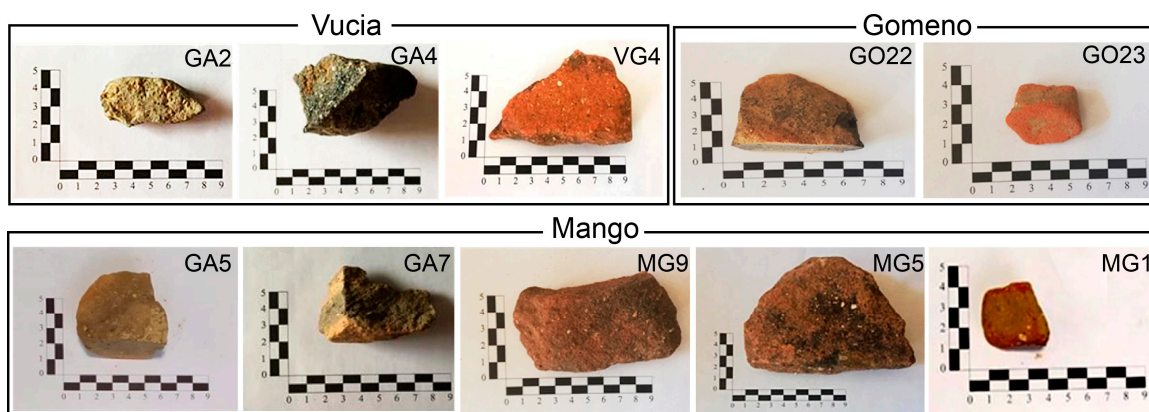
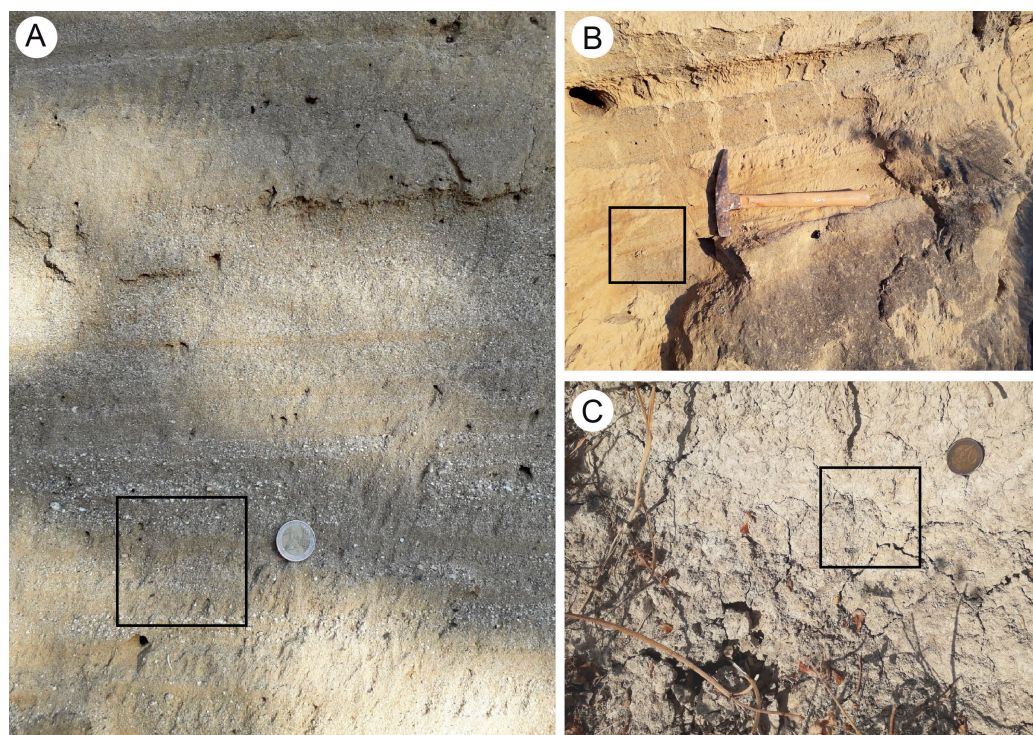


Figure 3. Photo of selected samples of dolia fragments studied here.

**Table 1.** List of the *dolia* and geological samples collected in the Gagliato area, with the description of type and locality.

<i>Dolia</i> Samples	Locality	Type
First group		
GA2	Vucia	Small vertical stick handle [5: Figure 3.6].
GA3	Vucia	Small vertical stick handle [5: Figure 3.5].
GA4	Vucia	Fragment deformed by heat [Tucci 2006: 16].
GO17	Vucia	Shoulder decorated with herringbone pattern [5: Figure 3.10].
VG4	Vucia	Short truncated conical neck; rim with traces on the surface of wheel-fashioning technique [5: Figure 2.7].
GO23	Gomeno	Bottom of a dolium.
MG9	Mango	Flattened rim with enlarged and squared internal and external margins [5: Figure 2.12].
Second group		
GA1	Mango	Concave bottom with triangular decoration [5: Figure 2.1].
GA5	Mango	Banded rim with triangular decoration [5: Figure 2.1].
GA7	Mango	Circular support with triangular decoration [5: Figure 3.3].
MG1	Mango	Brimmed edge [5: Figure 2.5].
MG5	Mango	Brimmed rim and narrow mouth [5: Figure 2.9].
GO20	Mango	Rim of globular to ovoid dolium body [5: Figure 2.4].
GO22	Gomeno	Bottom of a dolium [4: Figure 4].
Geological samples	GPS Lat. Long.	Sediment
X3	38.68573 16.46064	Clay—Plio-Pleistocene grey-blue silty clays and marls (DVL in the geological map Figures 2 and 4)
X5	38.69936 16.45578	Clay—Plio-Pleistocene grey-blue silty clays and marls (DVL in the geological map Figures 2 and 4)
V1	38.67866 16.48318	Sand and conglomerate—Plio-Pleistocene sand and pebble conglomerates (SNO in the geological map Figures 2 and 4)
V2	38.67747 16.48213	Sand—Plio-Pleistocene medium-fine to coarse sands (DVL <sub>a</sub> in the geological map Figures 2 and 4)
V4	38.68656 16.46803	Sand and conglomerate—Plio-Pleistocene sand and pebble conglomerates (SNO in the geological map Figures 2 and 4)
V5	38.68769 16.49667	Sand and conglomerate—Plio-Pleistocene sand and pebble conglomerates (SNO in the geological map Figures 2 and 4)



**Figure 4.** Photos of the outcrops with location (squares) of some of sampled sediments (A) V1: Plio-Pleistocene sand and pebble conglomerates (SNO); (B) V2: Plio-Pleistocene medium-fine to coarse sands (DVL<sub>a</sub>); (C) X3: Plio-Pleistocene grey-blue silty clays and marls (DVL).

In order to verify the hypothesis of the local production of the studied *dolia* [5] and to identify the possible raw materials, clay and sand sediments in the Gagliato area were sampled using geological maps and data [8–10]. Two samples of clay (X3 and X5), one of fine sand (V2) and three of sandy conglomerates (V1, V4 and V5), were collected during the field survey from the Plio-Pleistocene sediments of the area (Figures 2 and 4 and Table 1). The location of sampled sediments is shown in the geological map of Figure 2.

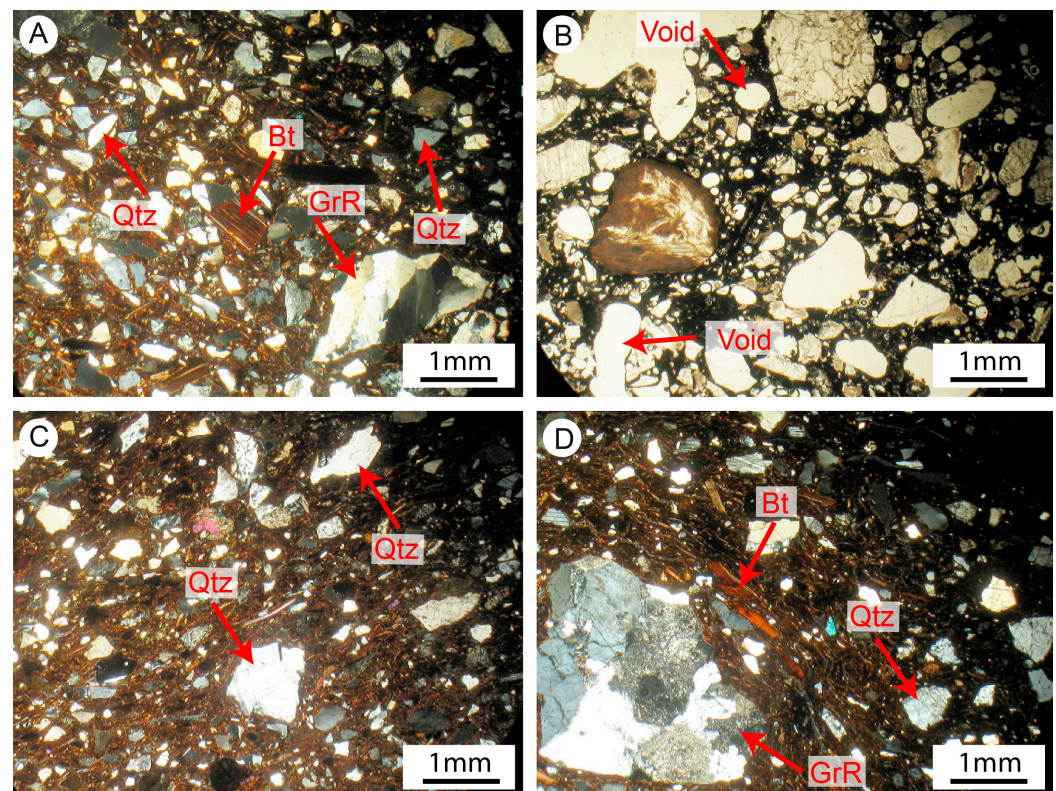
The fourteen selected ceramic fragments and the six sampled raw materials were characterized with mineralogical and petrographic analytical methods. The analyses were conducted in the laboratories of the Department of Biology, Ecology and Earth Sciences of the University of Calabria (Rende, CS). Petrographic and micropaleontological analyses were performed using polarized optical microscopy (POM) on the thin sections of all fourteen ceramic fragments using a Zeiss Axioskop 40 microscope and a Zeiss Axioplan II Imaging coupled with an UV apparatus for epifluorescence, equipped with four objectives that allow observation at  $\times 40$ ,  $\times 100$ ,  $\times 200$  and  $\times 400$  magnification, to obtain information on the matrix, on the textural and petrographic characteristics of the inclusions and, where present, on the fossil shells. The mineralogical analysis was carried out on seven representative *dolia* fragments and on three Plio-Pleistocene clayey and sandy sediments, using a Bruker D8 Advance X-ray diffractometer (XRD). The XRD analyses were executed with Cu K $\alpha$  radiation on pressed powder of the selected samples obtained via an agate mill. X-ray diffraction patterns were taken in the range  $5^{\circ}$ – $60^{\circ}$   $2\theta$ , with steps of  $0.02^{\circ}$   $2\theta$  and step-times of 1 s/step. Furthermore, with the aim of identifying the foraminifera species, micro-morphological analyses were carried out on the shells of selected microfossils in the particle size fraction greater than 63 microns of the sampled clays using Scanning Electron Microscopy (SEM), FEI-Philips ESEM-FEG Quanta 200F.

### 3. Results

#### 3.1. Petrographic Analysis

The textural and compositional features of the ceramic fragments, observed via POM, allowed us to distinguish two main groups. In particular, the presence or absence of microfossils in the matrix was used as the main discriminating criterion for grouping the samples. On the contrary, the mineralogical composition of the inclusions shows a great homogeneity.

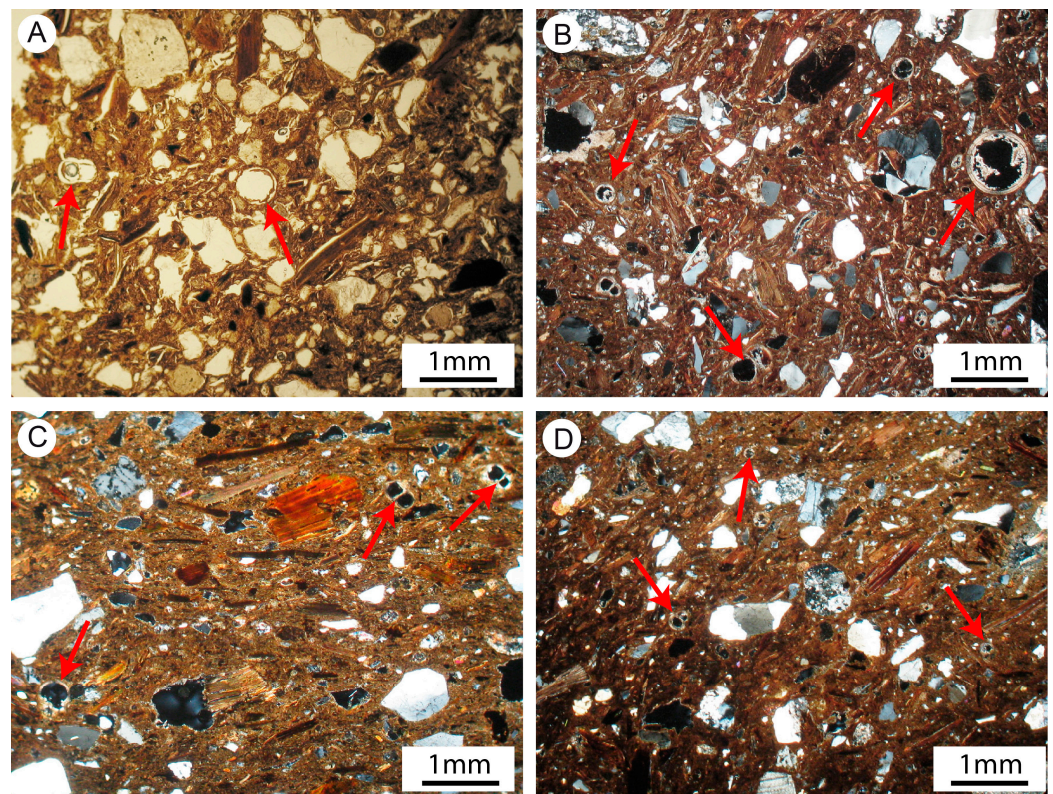
The finds of the first group (GA2, GA3, GA4, GO17, VG4, GO23 and MG9), are non-fossiliferous. The matrix shows a not-homogeneous orange-brown color, often dark red-brown in the central portion (Figure 5A). The optical activity of matrix is extremely variable, ranging from positive up to medium-low or absent as in GA4, showing also a completely fused and blistered structure (Figure 5B). The voids, often irregular, are both elongated and rounded in shape (Figure 5B). The inclusions are present in variable percentages, up to 30%, with grain sizes ranging from 0.2 to 2 mm; the smaller grains show mainly a sharp edge, while, on the contrary, the larger ones are sub-rounded. As regards the composition, all samples show similar inclusions consisting of quartz, plagioclase, K-feldspar (microcline) and biotite as the main minerals (Figure 5); epidote, garnet, amphibole, oxides and muscovite are present in subordinate quantities. There are also some fragments of granitic and metamorphic rocks, sometimes up to approximately 3–4 mm in diameter (Figure 5A,D).



**Figure 5.** Microphotographs of *dolia* fragments of the 1st group observed in thin section via POM ((A,C,D): cross-polarized light view; (B): polarized light). (A) sample GA2; (B) sample GA4 showing a typical fused and blistered structure; (C) sample GO23; (D) sample MG9. **Qtz**: quartz; **Bt**: biotite; **GrR**: granitic rock inclusion.

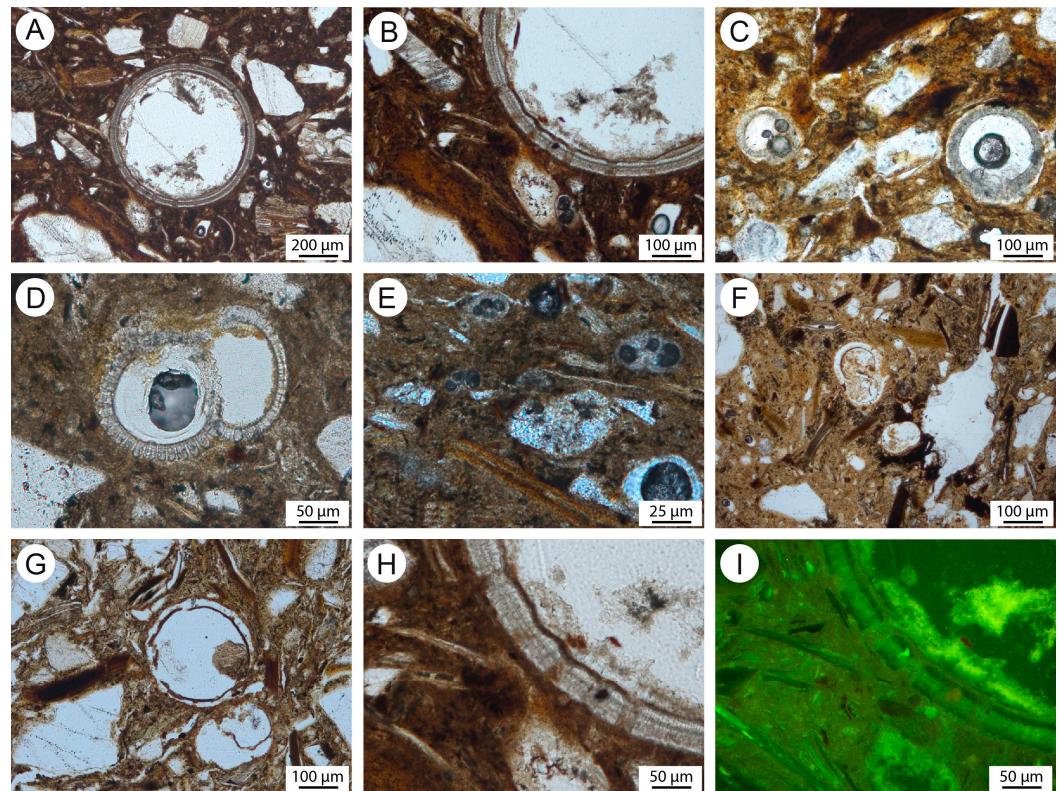
The finds of the second group (GA1, GA5, GA7, MG1, MG5, GO20 and GO22) are characterized by a clayey fossiliferous matrix with foraminifera micro-fauna, showing different degrees of preservation of the shells (Figure 6). The matrix is mica-rich and heterogeneous; its color varies from red-orange to reddish-brown to brown. The optical

activity varies from positive (MG5 and GA5) to medium-low (GA1 and GA7). The voids, often irregular, are both rounded and elongated. Inclusions show variable quantity, from 10% to 20%, and different grain size, from 0.2 to 1.4 mm; as in the first group the larger grains are sub-rounded while the smaller ones are angular (Figure 6). The composition of the inclusions is very similar to that of the first group and consists of quartz, plagioclase, K-feldspar (microcline) and biotite, which represent the most abundant minerals (Figure 6), while epidote, sillimanite, garnet, amphibole, oxides and muscovite are scarcely present or rare. Inclusions of granitic and metamorphic rocks up to 4 mm in diameter are frequent. Calcite and scattered grains of carbonate rocks are found in MG5 and GA5 ceramic fragments, though absent in the samples of the first group.



**Figure 6.** Microphotographs of *dolia* fragments of the 2nd group observed in thin section via POM ((A): polarized light and (B–D): cross-polarized light view). (A) sample GA1; (B) sample GA5; (C) sample GO22; (D) sample MG5. The red arrows point to the calcareous shells of microfossil, showing different degrees of preservation.

The second group is characterized by the presence of calcareous microfossils represented by planktonic and benthonic microforaminifera. The morphological characteristics allow us to tentatively attribute the foraminifera mainly to Globigerine, Orbuline and Globorotalie in variable quantities and with different degrees of preservation (Figure 7). In some samples, it is possible to observe the pristine lamellar/perforate wall structures and carbonate composition of the planktonic foraminifera, as in the sample GA5 (Figure 7A,B), while in many others, the shells are completely transformed (Figure 7C–E) or have disappeared and only ghosts attributable to foraminifera shells are present (Figure 7F,G). The different degrees of preservation of the foraminifera are also detectable through epifluorescence observations (Figure 7I); the well-preserved foraminifera show organic matter molecules strictly related to the crystals forming their walls, as evidenced by the bright fluorescence of the skeletons under UV excitation (Figure 7H,I). The foraminifera showing a lower degree of preservation do not emit fluorescence under UV light and often denote recrystallization of the walls, transforming in dog-tooth-like structures (Figure 7D).



**Figure 7.** Planktonic microforaminifera observed in thin section of the analyzed *dolia*. (A,B) *Orbulina* sp. showing very well preserved lamellar/perforate wall structures (sample GA5). (C–E) Microforaminifera showing recrystallized skeletons; note the dog-tooth-like structure of the wall in (D) ((C): sample GO20; (D): sample GO22; (E): sample MG5). (F,G) Trace of microforaminifera; the microfossils are observable only as ghosts ((F): sample GA7; (G): sample GA1); (H,I) Sample GA5: Detail of a microforaminifera skeleton observed in transmitted light (H) and UV epifluorescence (I); note the epifluorescence of the wall due to the preservation of organic molecules trapped among the crystals of the skeletons, which denote a low degree of alteration during the firing procedure of the *dolia*.

### 3.2. Mineralogical Analysis

Table 2 shows the mineralogical phases identified using XRD (S1 supplementary XRD patterns) in selected archaeological fragments and sediments. The ceramic samples were selected considering the petrographic and paleontological differences both between the two groups and within each group. Therefore, on the basis of the greater mineralogical and paleontological variability, we decided to analyze a major number of samples of the second group. GO17 and VG4 samples were selected for XRD analyses as representative of the first group and GA1, GA5, GA7, GO20 and MG5 were selected from the second group. Quartz, k-feldspar, plagioclase and biotite are the main mineralogical phases of all ceramic samples. In the second group, calcite is present in GA5, MG5 and GO20; on the contrary, in GA1 and GA7, the calcite disappeared and traces of diopside appeared. As concerns the geological samples, three sediments were selected and analyzed: one sandy sediment and two samples of fossiliferous clay (X3 and X5) considered representative of the geological source. The sandy sediment (V1) shows the presence of quartz, k-feldspar, plagioclase, biotite, illite and chlorite, whereas in the clay samples (X3 and X5), calcite is the prevalent mineral, together with subordinate quantities of quartz, chlorite and illite (Table 2).



**Table 2.** Mineralogical phases found in selected *dolia* samples and in the sediments identified via XRD. Qtz: quartz; Pl: plagioclase; Bt: biotite; Kf: K-feldspar; Cal: calcite; Di: diopside; Ill: illite; Chl: chlorite.

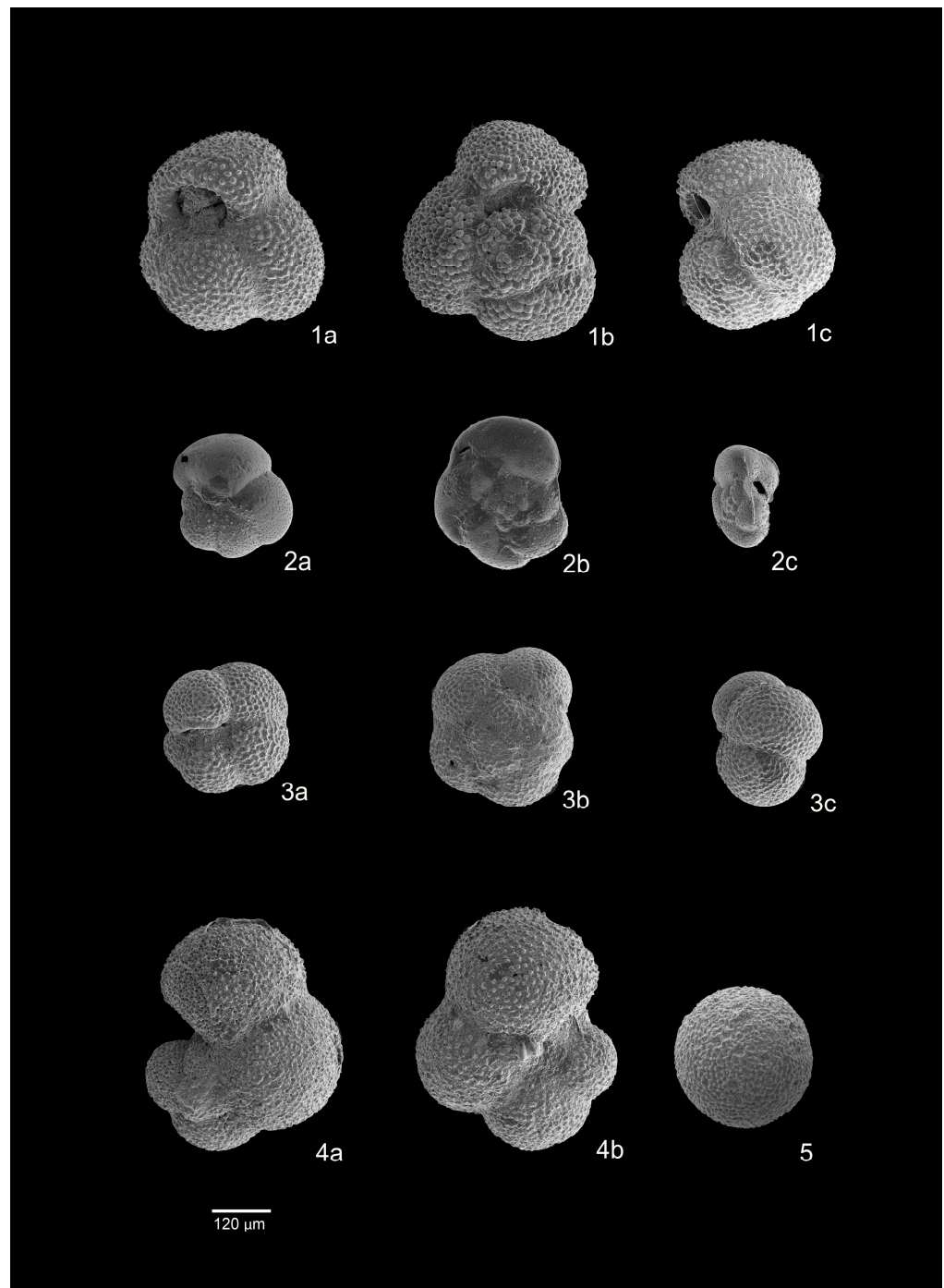
	Ceramics	Group	Qtz	Pl	Bt	Kf	Cal	Di		
GAGLIATO	MANGO	GA1	2	***	**	**	*	-	Tr	
		GA5	2	****	**	**	*	**	-	
		GA7	2	****	**	***	*	-	Tr	
		MG5	2	****	***	***	**	*	-	
		GO20	2	****	**	**	*	*	-	
	VUCIA	GO17	1	****	**	**	*	-	-	
		VG4	1	****	*	**	*	-	-	
		<b>sediments</b>		<b>Qz</b>	<b>Pl</b>	<b>Bt</b>	<b>Kf</b>	<b>Illite</b>	<b>Cal</b>	<b>Chl</b>
		V1		****	***	*	*	*		*
		V4		****	***	*	*	*		*
	V5		****	***	*	*	*		*	
	X3		**	-	-	-	Tr	****	Tr	
	X5		**	-	-	-	**	****	**	

\*\*\*\* = very abundant; \*\*\* = abundant; \*\* = present; \* = rare; Tr = traces.

### 3.3. Micropaleontological Analysis

The clay (X3) and fine sand (V2) collected around the settlement area were tested for micropaleontological analyses. The residues for planktonic foraminifer analyses were obtained from 200 g of dry sediment disaggregated in normal water and washed in 63 µm sieve. After the picking, the foraminifera were observed at high magnification in optical and electron microscopy for taxonomic recognition. The analyses revealed an association mainly constituted of planktonic foraminifera in discrete states of preservation for sample X3, while sample V2 was sterile. The microforaminifera association is constituted by the following: *Globigerinoides obliquus obliquus*, *Globigerinoides obliquus extremus*, *Globigerinoides* cfr. *trilobus*, *Neogloboquadrina acostaensis* sx, *Neogloboquadrina acostaensis* dx, *Orbulina universa*, *Globigerina bulloides*, *Hastigerina siphoniphera*, *Globorotalia scitula* sx, *Globorotalia scitula* dx and *Globorotalia* cfr. *margaritae* (Figure 8).

The presence of the index species in sample X3, ascribed to the early Pliocene, allows us to attribute the sediment to the *Globorotalia* cfr. *margaritae* Biozone MPI2 [11].



**Figure 8.** Main microforaminifera observed in the clay sample X3. (1) *Globigerinoides obliquus obliquus*. (2) *Globorotalia scitula* dx. (3) *Neogloboquadrina acostaensis* dx. (4) *Hastigerina siphoniphera*. (5) *Orbulina universa*. View: (a) umbilical; (b) spiral; and (c) lateral.

#### 4. Discussion

The optical microscopic observations of the *dolia* fragments allowed the distinction of two different ceramic groups, on the basis of the absence/presence of microfossil shells in the matrix.

The two groups share homogeneous composition of the grain inclusions, consisting mainly of rock fragments and crystals of quartz, plagioclase, K-feldspar and biotite, with garnet, sillimanite and amphiboles in very small quantities. This mineralogical association is consistent with the granitic and metamorphic rocks outcropping around the area, as

evidenced also by the presence of these rock fragments in the inclusions. The second group differs because of the presence of microfossils in the matrix; indeed, planktonic foraminifera are recognizable, showing different degrees of conservation of the shells.

The presence/absence of fossils therefore suggests the use of different types of clay sediments for the production of the Gagliato *dolia* between the first and the second group. The dark brown colors of the ceramic samples, sometimes black with reddish hues, indicate that the artefacts were fired in kilns with poor circulation of oxygen [12] and inhomogeneous temperature distribution. The porosity is to be attributed to excess water in the mixture but also to rapid evaporation with the formation of thin fractures. The optical activity of the ceramic matrix, from positive to negative, suggests different firing temperatures [13] and therefore non-standardized working procedures. In fact, in some samples (GA5 and MG5), the matrix is optically positive, and we can suppose that the firing temperatures did not exceed 750/800 °C [14]. In the GA4 sample of the first group, the matrix is optically negative or completely fused and blistered, indicating higher firing temperatures, over 900 °C [15], probably reached accidentally.

The XRD results demonstrated that the mineralogical assemblage of sandy sediment is perfectly coherent with the mineralogical composition of the aplastic inclusions observed via POM, and the clay sediments are calcitic.

The XRD analyses of seven representative archaeological samples showed the presence of calcite only in some samples of the second group (GA5, MG5 and GO20). This mineral is preserved at firing temperatures lower than 750–800 °C [16–18], but it is transformed into other mineralogical phases at higher temperatures. In the other two samples of the second group (GA1 and GA7), calcite is absent and diopside traces are found. Diopside forms at high temperatures; therefore, its presence suggests firing temperatures higher than 900–950 °C [14,16,18].

Numerous studies on the experimental firing of fossiliferous clays [19–21] have allowed us to observe the systematic changes in the shell structure of fossils with the increasing of the temperature. In particular, the authors of [19–21] report no morphological changes in foraminifer skeletons up to the calcite decomposition temperature, which ranges from 750/800 °C to 900 °C. In samples fired at 900 °C, “dog-tooth-like” structures substitute the foraminifer walls. These structures resemble those which develop during carbonate diagenesis [22]. Privitera et al. [20] hypothesize that after the decomposition of the calcite walls at 900 °C, if the microsystem is closed, the calcium oxide (CaO) reacts with the carbon dioxide (CO<sub>2</sub>) during the cooling phase and after firing, re-precipitating calcite in form of dog-tooth cements. Voids (shrinkage rim) between the matrix and the microfossil appear at a temperature of 1000 °C. Due to the complete or partial dissolution of the original walls at these temperatures, the microfossils are recognizable only by the external shape [20]. At a firing temperature of 1100 °C, the void thickness increases due to the decrease in volume of the ceramic paste. The microfossil remains, not yet decomposed, and undergoes a progressive wall thinning, which appears as “micritic clots”. At these conditions, the loss of all original morphologies make it impossible to identify microfossils at the genus level. At 1200 °C, the ceramic paste acquires a glass aspect with bubbles (sinterization) due to the loss of the granular appearance, and there is no possibility of observing any trace of microfossils.

As regards the ceramic samples studied here, based on the different transformations of the shell structures of microfossils of the second group (from those preserved in GA5 to those completely transformed in GO22), the temperatures can be estimated between 750/800 °C and 950 °C [19–21]. Therefore, the well-preserved structure of the foraminifera skeletons of the samples GA5, the recrystallized walls observed in the samples GO22 and the ghost observed in the samples GA7 allow us to hypothesize that the firing procedures of the *dolia* were not homogeneous.

Moreover, the microfossils analyses allowed us to also suggest a local provenance of the raw materials utilized for the ceramic production. Actually, even if an exact systematic attribution is not possible in thin section observations, the presence of foraminifera

tentatively attributed to *Globorotalia*, *Orbulina* and *Globigerina* in the ceramic sample GA5 is coherent with the association observed in clay X3, mainly characterized by *Globorotalia scitula* s.s., *Orbulina universa* and *Globigerina bulloides*.

## 5. Conclusions

These mineralo-petrographic and paleontological analyses conducted on the *dolia* and sediments sampled in the Gagliato area have provided important information on the possible raw materials and on the production technology of these ceramics. The study of the samples using POM made it possible to distinguish two groups of ceramics characterized by similar aplastic inclusions but with a different plastic fraction: non-fossiliferous and fossiliferous. Indeed, the plastic fraction of the second group of *dolia* fragments is rich in shells of microfossils, mainly planktonic foraminifera. The mineralogical composition of the aplastic inclusions of both ceramic groups is consistent with the granitic and metamorphic rocks outcropping in the area, as evidenced by the presence of minerals and fragments of these rocks in the impasto. Firing temperatures between 750/800 °C and over 950 °C were estimated using different analytical methodologies, based on the optical activity of the matrix under the POM and on the mineralogical phases detected using XRD. In addition, the state of conservation of the structure of the microfossil shells observed using POM highlighted different degrees of transformation, with microfossil shells ranging from perfectly preserved to completely decomposed as a function of the firing temperature. The ceramics showing well-preserved foraminifera (lamellar perforated microstructures and bright fluorescence) were fired at temperature below 750/800 °C, whereas ceramics with foraminifera showing recrystallized structures (dog-tooth-like) were fired at temperature ranges between 750/800 °C and 950 °C. The ceramics with ghosts of foraminifera probably reached temperatures major than 950 °C. The brown color of the *dolia* suggests that they were fired in kilns with poor circulation of oxygen and inhomogeneous temperature distribution, while the oriented and thin porosity could indicate the use of a slow lathe and rapid evaporation of the water with the formation of thin fractures. The results of the micropaleontological analysis of the clay sampled in the Gagliato area indicate a strong compatibility with the microfossils of the plastic fraction of the ceramics of the second group. The analyses conducted on the sandy-conglomerate sediments also showed strong similarities with the aplastic inclusions of the finds, so it is possible that these sandy sediments were used together with the clays in variable and non-standardized proportions. All observations indicate inaccurate production technology and variable firing temperatures in rather rudimentary kilns.

These new data improve the knowledge about the Gagliato archaeological area, and although it is quite difficult to determine the quarrying places of the raw material, it is possible to suppose that local sedimentary formations were employed, confirming the hypothesis suggested by the archaeological observations. Moreover, the possible local provenance of raw materials for the *dolia* production indicates that the inhabitants reached a good technological level in the production of ceramics.

**Author Contributions:** Fieldwork, A.M.D.F. and A.M.T.; Conceptualization, A.M.D.F. and D.B.; methodology, A.M.D.F. and D.B.; validation, A.M.D.F. and D.B.; formal analysis, A.M.D.F., A.G. and A.R.; investigation, A.M.D.F., A.G. and A.R.; resources, A.M.D.F. and A.M.T.; data curation, A.M.D.F., A.G., A.R. and D.B.; writing—original draft preparation, A.M.D.F., A.G., A.M.T., A.R. and D.B.; writing—review and editing, A.M.D.F. and D.B.; supervision, A.M.D.F. and D.B. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research received no external funding.

**Data Availability Statement:** The data underlying this article are available in the article.

**Conflicts of Interest:** The authors declare no conflict of interest.

## References

1. Tucci, A.M. *L'età del Bronzo nel Comprensorio di Soverato (CZ), Atti Della XXXVII Riunione Scientifica, Preistoria e Protostoria della Calabria, Scalea, Papisidero, Praia a mare, Tortora, 29 Settembre–4 Ottobre 2002*; Istituto Italiano di Preistoria e Protostoria: Florence, Italy, 2004; pp. 843–847, ISBN 978-88-6045-044-9.
2. Tucci, A.M. *L'età del Bronzo Nelle Province di Catanzaro, Vibo Valentia e Crotone. Studi e Materiali di Geografia Storica della Calabria*; Givigliano, G.P., Ed.; Due Emme Edizioni: Torre del Greco (NA), Italy, 2002; pp. 3–160.
3. Aisa, M.G.; Tucci, A.M. Il sito fortificato di Gomeno (Gagliato, CZ) tra età arcaica ed ellenistica. In *Centri Fortificati Indigeni della Calabria dalla Protostoria all'età Ellenistica*; Atti del Convegno: Napoli, Italy, 2017; pp. 489–491.
4. Aisa, M.G.; Tucci, A.M. *Nuove Prospettive di Ricerca nel Territorio di Gagliato*; Deputazione di Storia Patria per la Calabria: Reggio Calabria, Italy, 2014; pp. 101–114, ISSN 0393-022X.
5. Tucci, A.M. *Dolii Protostorici di Impasto dal Territorio di Gagliato (CZ) in Preistoria del cibo. L'alimentazione nella Preistoria e nella Protostoria*; Studi di Preistoria e Protostoria Italiana; Damiani, I., Cazzella, A., Copat, V., Eds.; Riunione Scientifica dell'Istituto Italiano di Preistoria e Protostoria: Florence, Italy, 2021; pp. 991–996, ISBN 978-88-6045-088-3.
6. Levi, S.T. *Produzione e Circolazione della Ceramica nella Sibaritide Protostorica, I. Impasto e dolii*; Grandi contesti e problemi della protostoria italiana 1, a cura di; Peroni, R., Vanzetti, A., Cazzella, A., Eds.; All'insegna del Giglio: Florence, Italy, 1999; ISBN 9788878141391.
7. De Francesco, A.M.; Barca, D.; Tucci, A.M.; Scarpelli, R. Provenance study of the protohistoric impasto pithoi in the Soverato area (Calabria–Southern Italy) by LA-ICP–MS analysis of individual biotite grains, Il tempo del pianeta Terra e il tempo dell'uomo: Le geoscienze fra passato e futuro, Abstract Book. In Proceedings of the Congresso SIMP-SGI-SOGEI 2019, Parma, Italy, 16–19 September 2019; p. 166. [[CrossRef](#)]
8. Paglionco, A.; Festa, V. *Note Illustrative Carta Geologica d'Italia ISPRA Foglio 580-Soverato*; ISPRA, Servizio Geologico d'Italia: Rome, Italy, 2015.
9. Ortolano, G.; Pagano, M.; Visalli, R.; Angi, G.; D'Agostino, A.; Muto, F.; Critelli, S.; Cirrincione, R. Geology and structure of the Serre Massif upper crust: A look into the late-Variscan strike-slip kinematics of the southern European Variscan chain. *J. Maps* **2022**, *18*, 314–330. [[CrossRef](#)]
10. Russo, D.; Fiannacca, P.; Fazio, E.; Cirrincione, R.; Mamtani, M.A. From floor to roof of a batholith: Geology and petrography of the north-eastern Serre Batholith (Calabria, southern Italy). *J. Maps* **2022**, *19*, 2149358. [[CrossRef](#)]
11. Lirer, F.; Foresi, L.M.; Iaccarino, S.M.; Salvatorini, G.; Turco, E.; Cosentino, C.; Sierro, F.J.; Caruso, A. Mediterranean Neogene planktonic foraminifer biozonation and biochronology. *Earth Sci. Rev.* **2019**, *196*, 102869. [[CrossRef](#)]
12. Rathossi, C.; Pontikes, Y. Effect of Firing Temperature and Atmosphere on Ceramics Made of Nw Peloponnese Clay Sediments. Part I: Reaction Paths, Crystalline Phases, Microstructure and colour. *J. Eur. Ceram. Soc. It.* **2010**, *30*, 1841–1851. [[CrossRef](#)]
13. Whitbread, I.K. *Greek Transport Amphorae: A Petrological and Archaeological Study*; British School at Athens, Fitch Laboratory Occasional Paper 4: Athens, Greece, 1995.
14. Maggetti, M.; Neururer, C.; Ramseier, D. Temperature evolution inside a pot during experimental surface (bonfire) firing. *Appl. Clay Sc.* **2011**, *53*, 500–508. [[CrossRef](#)]
15. Maniatis, Y.; Tite, M.S. Technological examination of Neolithic–Bronze Age pottery from central and southeast Europe and from the Near East. *J. Archaeol. Sci.* **1981**, *8*, 59–76. [[CrossRef](#)]
16. Cultrone, G.; Rodriguez-Navarro, C.; Sebastian, E.; Cazalla, O.; De La Torre, M.J. Carbonate and silicate phase reactions during ceramic firing. *Eur. J. Mineral.* **2001**, *13*, 621–634. [[CrossRef](#)]
17. Maritan, L.; Nodari, L.; Mazzoli, C.; Milano, A.; Russo, U. Influence of firing conditions on ceramic products: Experimental study on clay rich in organic matter. *Appl. Clay Sc.* **2006**, *31*, 1–15. [[CrossRef](#)]
18. Riccardi, M.P.; Messiga, B.; Duminuco, P. An Approach to the Dynamics of Clay Firing. *Appl. Clay Sc.* **1999**, *15*, 393–409. [[CrossRef](#)]
19. Maritan, L.; Mazzoli, C.; Freestone, I. Modelling Changes in Mollusc Shell Internal Microstructure during firing: Implications for Temperature Estimation in Shell-Bearing Pottery. *Archaeometry* **2007**, *49*, 529–541. [[CrossRef](#)]
20. Privitera, A.; Guido, A.; Mastandrea, A.; Rao, A.; Russo, F. Morphological and mineralogical evolution of microfossils during the heating process: A contribution to the archaeometric study of ceramics. *Rend. Fis. Acc. Lincei* **2015**, *26*, 499–512. [[CrossRef](#)]
21. Daghmehchi, M.; Guido, A.; Mastandrea, A.; Salahi, M.A.; Omrani, M.; Nokandeh, J. Thermal analysis of ancient ceramics using the microchemical and microstructural alterations of foraminifera. *Mater. Character.* **2017**, *130*, 81–91. [[CrossRef](#)]
22. Flugel, E. *Microfacies of Carbonate Rocks, Analysis, Interpretation and Application*; Springer: Berlin/Heidelberg, Germany; New York, NY, USA, 2004.

**Disclaimer/Publisher's Note:** The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.