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Article

Evidence of Copper and Iron Deposits of the Protohistoric City of Temesa

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Abstract: With the name 'Temesa' (Latin Tempsa), the ancients identified a settlement located along the Tyrrhenian coast of Calabria, cited by sources as an international metal exchange emporium. The town is mentioned by Homer as being famous in the ancient world for the production of bronze, and in the I century A.D. Strabo wrote that there were rich copper mines near the city. Many years of study led to the recognition of Temesa as a complex urban system located between the Oliva and Savuto rivers, near Amantea. To confirm this hypothesis, we searched, in the surrounding rocky outcrops, for the presence of minerals useful for the extraction of iron and copper. Samples of 3 different rock stratifications were taken near the protohistoric settlement of Serra Aiello. The observation under an polarized reflected light microscope and the X-ray diffraction patterns revealed the presence of many minerals useful for the extraction of iron and copper in every sample. The heating of samples under both oxidizing and reducing conditions helped us to better quantify copper and iron minerals content causing, at the same time, the appearance of a marked paramagnetic behavior that could be associated with the presence of goethite. X ray fluorescence analysis showed a high concentration of iron and a low copper content.

Keywords: Temesa; protohistoric metallurgy; ore minerals; copper minerals; iron minerals; Odyssey; supergene enrichment; low grade metamorphic rocks; goethite paramagnetic transition; polarized reflected light microscopy; X-ray powder diffraction

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1. Introduction

The problem of identifying the site of Temesa is one of the main nodes of historical and archaeological research in southern Italy. Temesa was the non-Greek town mentioned by Homer in the Odyssey (Od., I 181–184), in which Mente, king of the Tafii, inhabitants of an island traditionally located between the coasts of Acarnania and the Ionian Islands, landed to exchange iron for bronze. According to the Homeric text, it is clear that Temesa was an important copper extraction center, contiguous to rich cupriferous deposits. The placement of the story in the Homeric text leads one to discard the hypothesis of the identification of Temesa with the Cypriot city of Tamaso made by some authors [1] and to consider it more probable that the ancient town was located in Southern Italy. Between the Greeks and the populations of Tyrrhenian Calabria, there were intense commercial relations that began from at least the eighth century BC. Moreover, the Greek savant Strabo wrote about the presence of copper mines, at that time already exhausted, that the indigenous population indicated as to demonstrate the Calabrian location of Temesa (Geographica, VI, 1–5). Until 1981, the question of Temesa's location could only count on hints in classical sources. In that year, an important conference held in Perugia launched the first archaeological and topographical studies with a program of excavations and territorial reconnaissance promoted by several Italian universities, whose results helped to confirm the location of Temesa on the hill between the terminal courses of the rivers Oliva and Savuto [2–4].

A decisive boost to the archaeological research in this area came from the discovery, by Francesco La Torre, of the archaic sacred building of Campora San Giovanni, whose attendance is fixed between the second fourth of the VI and the first fourth of the fifth century BC [5–7]. In 2003 and 2004, a reconnaissance campaign on the hilly ridge of Serra d'Aiello documented the existence of a high-altitude settlement occupied from the early Middle Bronze Age up to the 8th century BC, while in the locality of Chiane di Serra d'Aiello, Fabrizio Mollo investigated a necropolis nucleus datable to the advanced phase of the early Iron Age, which documents the existence of an indigenous community in contact with Oenotrian, Etruscan-Villanovan and pre-colonial Greco-Euboic [8,9]. In the years 2006 and 2007, the same archaeologist investigated some necropolis nuclei in Campora San Giovanni datable to the late-archaic and proto-classical age, abandoned between the third and fourth decades of the fifth century [10].

In 2008, the University of Messina started a research program with excavations in Cozzo Piano Grande in Serra d'Aiello, where important phases of the 7th century BC have been documented along with the thermal system of a Roman villa [11,12].

In the Naghicelle-Romia locality of Serra d'Aiello (39°5′44.3364″ N 16°7′46.2756″ E), near a summit plateau located a few hundred meters from the protohistoric town of Serra d'Aiello, from which it dominates the upper valley of the river Oliva up to Aiello Calabro and Monte Serra Lucerna (Figure 1), the surveys of the University of Messina (2012–2014) identified an area where ceramic fragments and obsidian lithic tools are associated with numerous rocky outcrops, distributed both on the top and on the slopes of the plateau, where iron-hydroxide lumps are well visible. These rock formations attracted our attention, suggesting the hypothesis that they could be what remained of the ancient mines of Temesa. The toponym 'Romia', which generally indicates caves and cavities in rock, could, moreover, be an indication of ancient mining activities. Establishing the presence of copper and iron minerals could provide a decisive element to definitively settle the question of the location of the protohistoric town. We have, preliminarily, identified three rock typologies: a soft dark fine-grained schist, here identified as Temesa 1, a dark gray phyllite crossed by veins of iron oxides and iron hydroxides, here identified as Temesa 2, and a green-gray phyllite, here identified as Temesa 3, with interlayers of iron oxides and hydroxides.

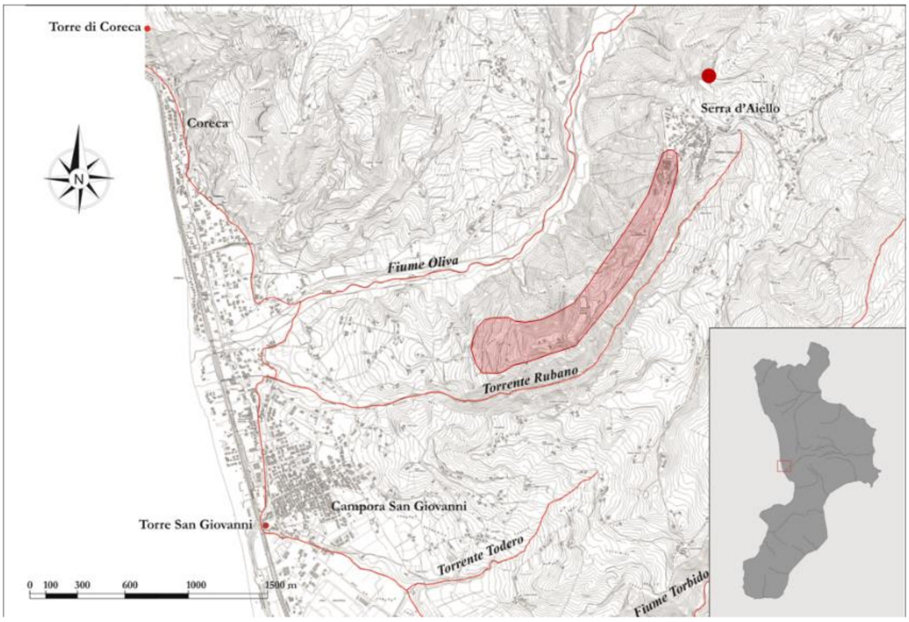


Figure 1. Map of the area between the rivers Oliva and Savuto: the pink shaded area indicates the protohistoric settlement, and the red dot indicates the location of rock sampling.

2. Materials and Methods

Thin sections coming from the three rocks, about 30 µm thick, have been studied with polarized reflected light microscopy (PRLM). Image acquisition has been conducted with a high-resolution digital camera (1920 × 1080 pixels). The camera sensitivity, exposure and white balance have been initially optimized for a representative image and then kept constant. Two aliquots of Temesa 1 have been subjected to heating at around 600 °C for 5 min in an oxidizing and reducing atmosphere, respectively, in order to produce phase transitions that emphasize the copper and iron content.

X-ray powder diffraction (XRD) patterns have been acquired using a Brucker D8 Advance diffractometer equipped with a Cu Kα line at λ = 1.54 Å in the (5–80)° 2θ range with a step of 0.01° and an integration time of 0.1 s.

An X ray fluorescence (XRF) spectrometer was equipped with a Si-PIN detector Amptek XR-100 CR and an Amptek Mini-X2 fluorescence tube with a copper target, operating at 20 kV and 5 mA. Each spectrum was acquired for 500 s. Laboratory references have been used for quantitative analysis.

Magnetic susceptibility has been calculated with the Gouy method [13] using a standard analytical balance and a permanent magnet whose magnetic field was measured with a magnetometer. We estimated the magnetic susceptibility (χ_m) following the Gouy equation [14]:

$$\chi_m = \frac{F \times 2V_{mol}}{A \times H^2} \quad (1)$$

Due to the non-standard instrumental configuration and to the polyphasic matrix, the measured values were conservative.

3. Results

3.1. Temesa 1

The hand sample is finely foliated, earthy, soft and dark-gray-colored (Figure 2a). Thin-section observation under PLRM reveals a mainly cryptocrystalline mass surrounding much bigger quartz grains. The distinction of phases in the matrix is hard, and only those with a strong internal reflection are clearly visible. Goethite (α-FeO(OH)) lumps are widespread (Figure 2b) alongside rare melantherite ($FeSO_4 \cdot 7H_2O$) microcrystals (Figure 2c) and a few microcrystals of chalcanthite ($CuSO_4 \cdot 5H_2O$) [15] (Figure 2e) and cuprite (Cu_2O) [16] (Figure 2c,d). In order to better quantify ore minerals, two aliquots of the sample were heated at around 600 °C for 5 min with wood fire in an oxidizing and reducing atmosphere, respectively [17]. Heating in an oxidizing atmosphere produced, as expected, an increase in chalcanthite content, while heating in a reducing atmosphere caused the appearance of nodules of metal oxides and sulfides. Following heating, both rock aliquots acquired marked paramagnetic behavior which was quantified by measuring the magnetic susceptibility. The measured value of 25.5 × 10^{-8} m^3/kg is compatible with a high content of iron oxides and/or hydroxides. This transition can be attributed, indeed, to the presence of goethite, which has antiferromagnetic properties at room temperature but which becomes paramagnetic at around 400 K (Neél temperature) [18]. The XRF spectrum (Figure 3) of a non-heated sample shows large k iron peaks that account for 27% of the iron content. The very low k copper peaks, which are also present in the spectrum, instead only allow for the quantification of 0.3 % of this metal. However, this quantity can be considered as being largely underestimated because of the matrix effect, primary copper radiation being easily absorbed by iron, which then produces a secondary emission.

The XRD spectrum (Figure 4) of a natural sample shows goethite [19] and cuprite peaks alongside those of magnetite ($Fe^{2+}Fe^{3+}{}_2O_4$) [20,21] and digenite (Cu_9S_5) [22]. The XRD pattern of the heated aliquots in an oxidizing atmosphere indicates the presence of maghemite [23] instead of magnetite, suggesting that the first has been formed starting from the second. The other peaks belong to typical phases of low-grade metamorphic rocks, such as phlogopite ($KMg_3(Al\,Si_3O_5)(OH)_2$) [24], chloritoid ($(Fe,Mg,Mn)_2Al_4Si_2O_{10}(OH)_4$) [25] and quartz ($SiO_2$) [26].

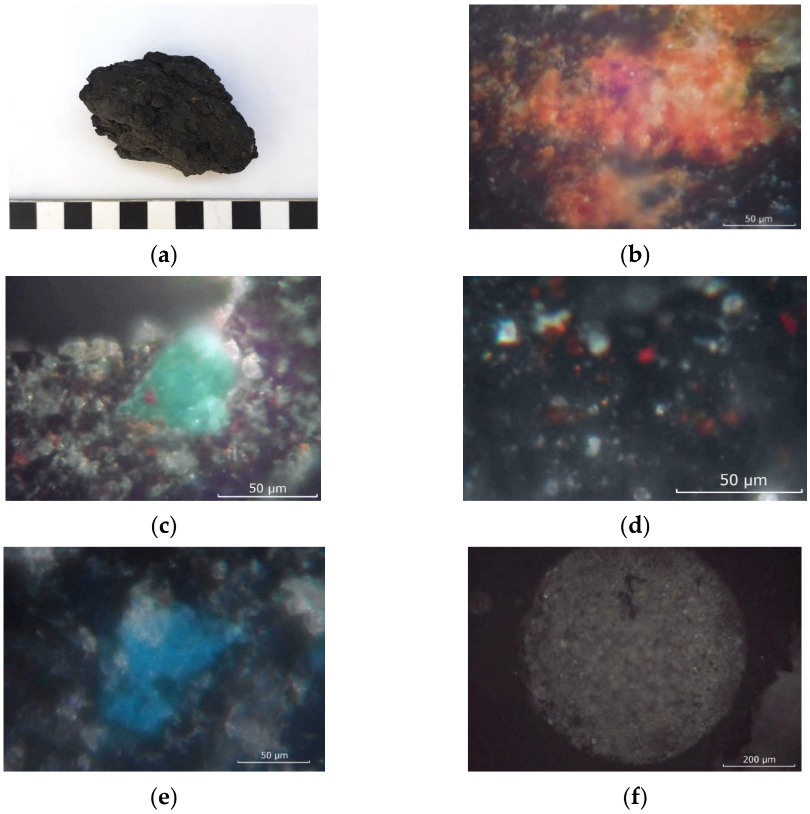


Figure 2. Temesa 1: (**a**) rock sample; (**b**) goethite lump (reflected light micrograph, crossed polarizers); (**c**) melantherite microcrystal surrounded by cuprite microcrystals (reflected light micrograph, crossed polarizers); (**d**) cuprite microcrystals (reflected light micrograph, crossed polarizers); (**e**) sample aliquot heated at around 600 °C in oxidant atmosphere: chalcanthite crystal (reflected light micrograph, crossed polarizers); (**f**) sample's aliquot heated at around 600 °C in reducing atmosphere: nodules of metal oxides and sulfides (reflected light micrograph, crossed polarizers).

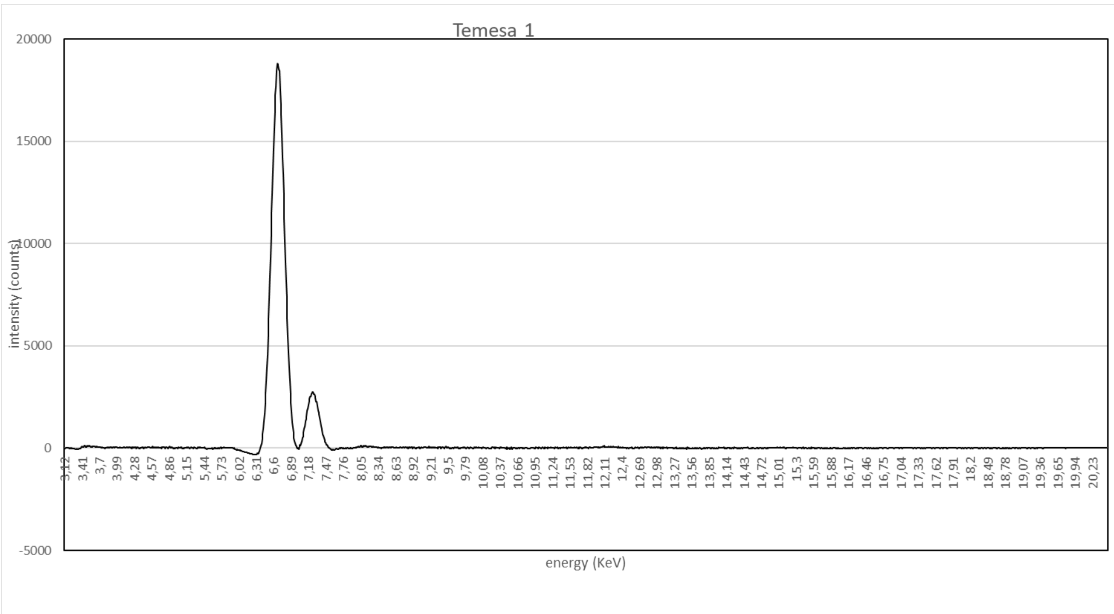


Figure 3. Temesa 1: XRF spectrum.

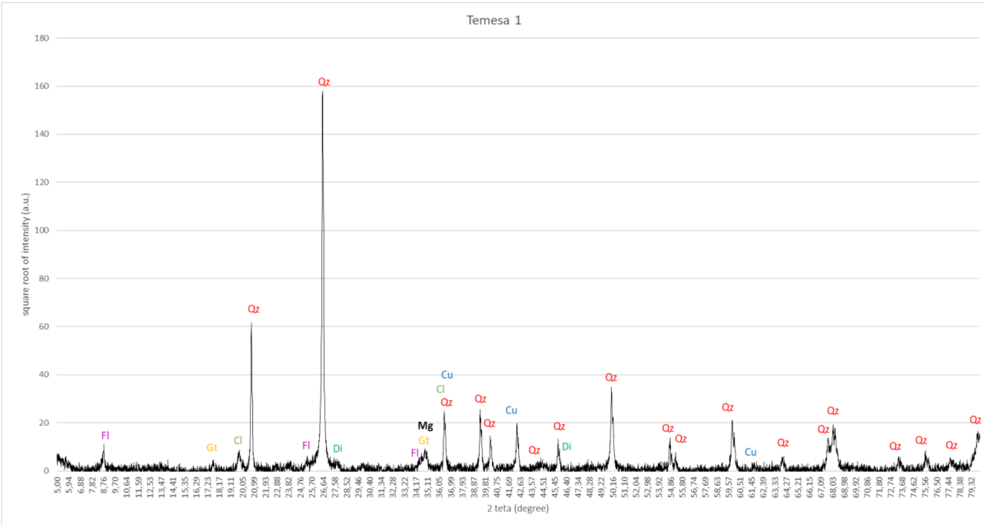
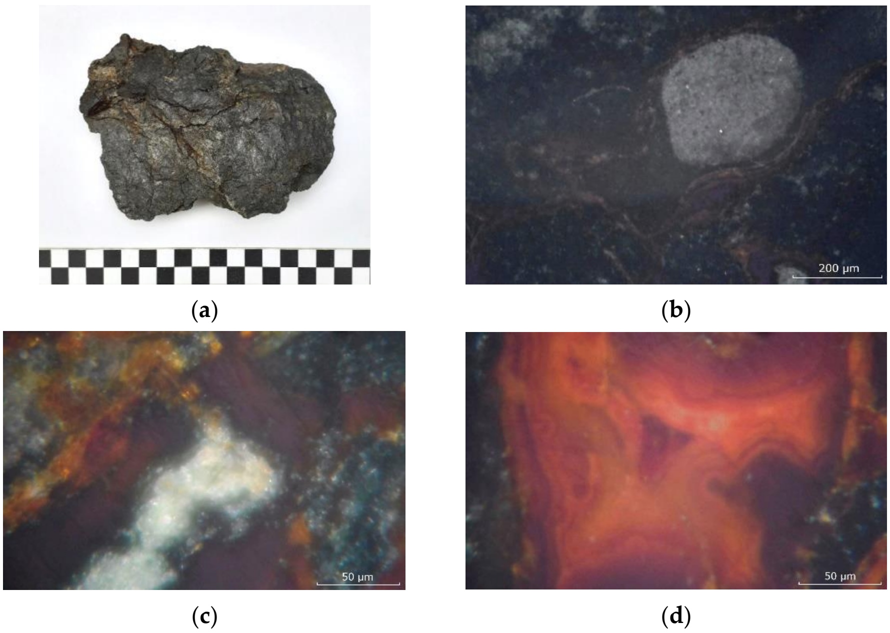


Figure 4. Temesa 1: XRD pattern. Abbreviations: Fl—phlogopite, Gt—goethite, Cl—chloritoid, Qz—quartz, Di—digenite, Mg—magnetite, Cu—cuprite. Square-root intensity scale.

3.2. Temesa 2

The hand sample has a silky glossiness, and it is greasy to the touch and dark-gray-colored (Figure 5a). It is finely foliated and has a good fissility. Thin-sections observation under PLRM reveals a mainly cryptocrystalline mass surrounding much bigger quartz grains nodules of metal oxides and sulfides (Figure 5b), showing great similarities with Temesa 1. In the matrix, the distinction of phases is not possible except for goethite, which is present as little veins (Figure 5c) and colloidal structures (Figure 5d). The XRD pattern (Figure 6) shows many valuable iron and copper mineral peaks, such as cuprite, covellite (CuS) [27,28], digenite, tetraedrite (Cu, Fe)$_{12}$ Sb$_4$ S$_{13}$) [29], jacobsite ($Mn^{2+}Fe^{3+}{}_2O_4$) [30], malachite ($Cu_2CO_3(OH)_2$) [31] and goethite. The other peaks belong to typical phases of low-grade metamorphic rocks, such as phlogopite, chloritoid, quartz, stevensite (($Ca_{0,5}$,Na)$_{0,33}$(Mg,Fe^{2+})$_3Si_4O_{10}(OH)_2\cdot n(H_2O)$) [32] and fosterite ($Mg_2SiO_4$).



(**a**) (**b**) (**c**) (**d**)

Figure 5. Temesa 2: (**a**) rock sample; (**b**) metal sulfides nodule (reflected light micrograph, crossed polarizers); (**c**) goethite veins surrounding quartz microcrystal (reflected light micrograph, crossed polarizers); (**d**) colloidal goethite (reflected light micrograph, crossed polarizers).

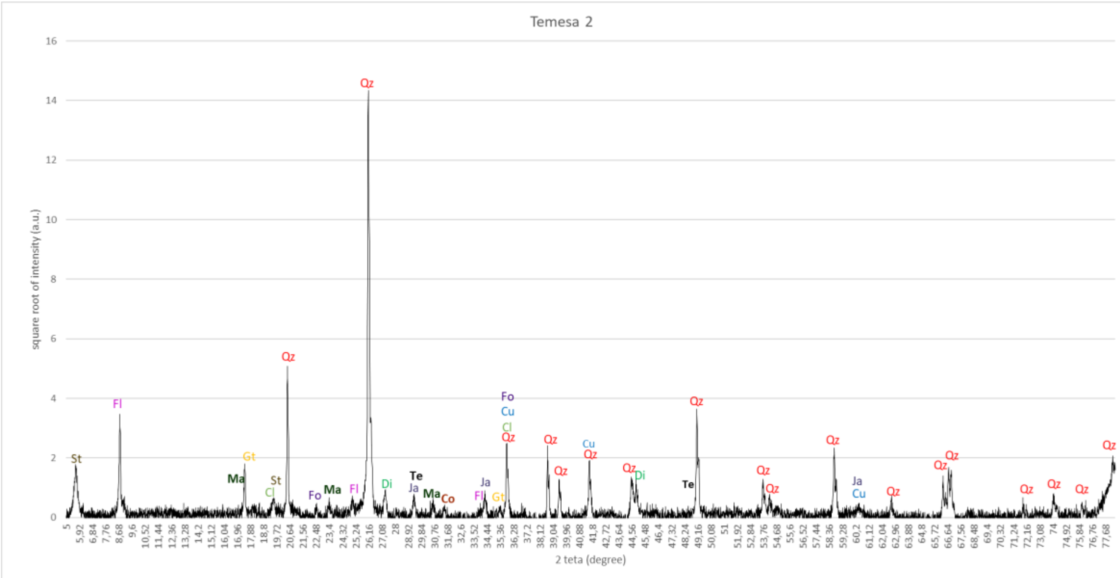


Figure 6. Temesa 2: XRD pattern. Abbreviations: St—stevensite, Fl—phlogopite, Gt—goethite, Cl—chloritoid, Qz—quartz, Di—digenite, Ma—malachite, Cu—cuprite, Fo—fosterite, Te—tetraedrite, Ja—jacobsite, Co—covellite. Square-root intensity scale.

The XRF spectrum of a Temesa 2 sample (Figure 7) is very similar to that of a Temesa 1 sample. The iron content was quantified as 14.5%, while the copper content was quantified as 0.3%.

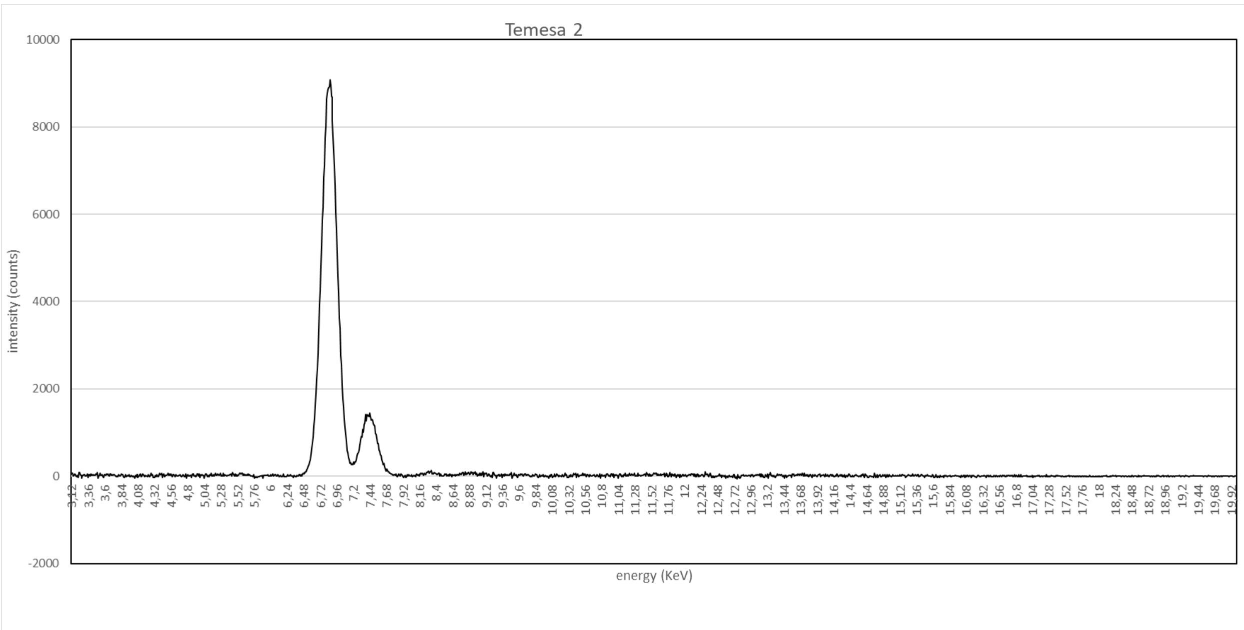
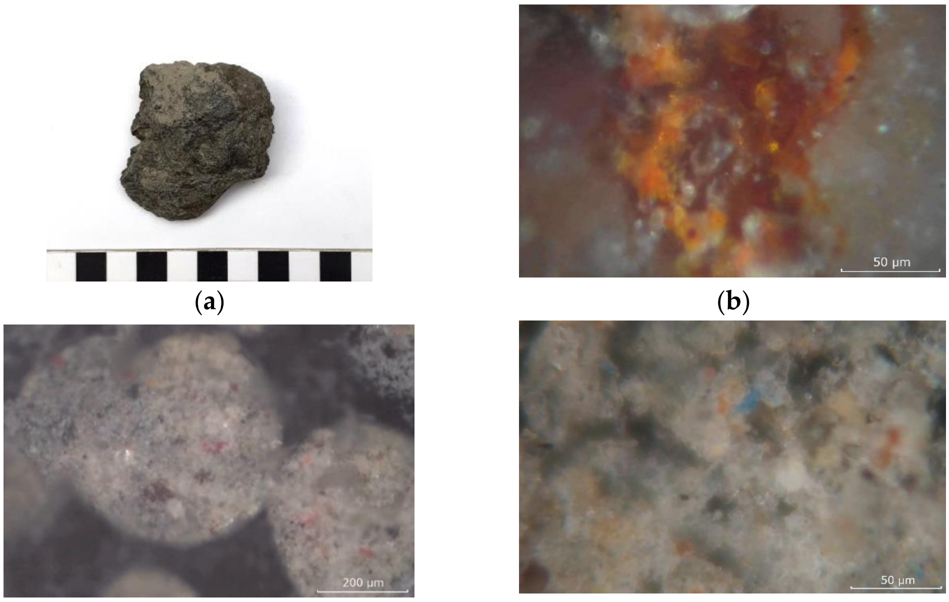


Figure 7. Temesa 2: XRF spectrum.

3.3. Temesa 3

The hand sample has a silky glossiness, and it is greasy to the touch and green-gray-colored (Figure 8a). It is finely foliated and has a poor fissility. Thin-sections observation under PLRM reveals a mainly cryptocrystalline mass surrounding quartz grains and many nodules of metal oxides and sulfides (Figure 8c). In the matrix, the distinguishable phases are only those with a strong internal reflection, such as goethite, chalcanthite and cuprite. Goethite is present as a lump or little veins (Figure 8b), while crystals of chalcanthite

(Figure 8d) and cuprite (Figure 8c) are micrometric. The XRD pattern (Figure 9) shows many valuable iron and copper mineral peaks, such as cuprite, covellite, digenite, chalcanthite, djurleite ($Cu_{31}S_{16}$), tetraedrite, jacobsite and goethite. The other peaks belong to typical phases of low-grade metamorphic rocks, such as phlogopite, chloritoid, quartz, corrensite ($(Mg,Fe)_9((Si,Al)_8O_{20})(OH)_{10}{\cdot}nH_2O$) [33], clinoferrosilite ($Fe^{2+}{}_2Si_2O_6$) [34] and brindleyite ($(Ni,Mg,Fe^{2+})_2Al(SiAl)O_5(OH)_4$) [35].



(**a**) (**b**) (**c**) (**d**)

Figure 8. Temesa 3: (**a**) rock sample; (**b**) goethite lump (reflected light micrograph, crossed polarizers); (**c**) cuprite microcrystals inside nodules of metal oxides and sulfides (reflected light micrograph, crossed polarizers); (**d**) cuprite and chalcanthite microcrystals inside nodules of metal oxides and sulfides (reflected light micrograph, crossed polarizers).

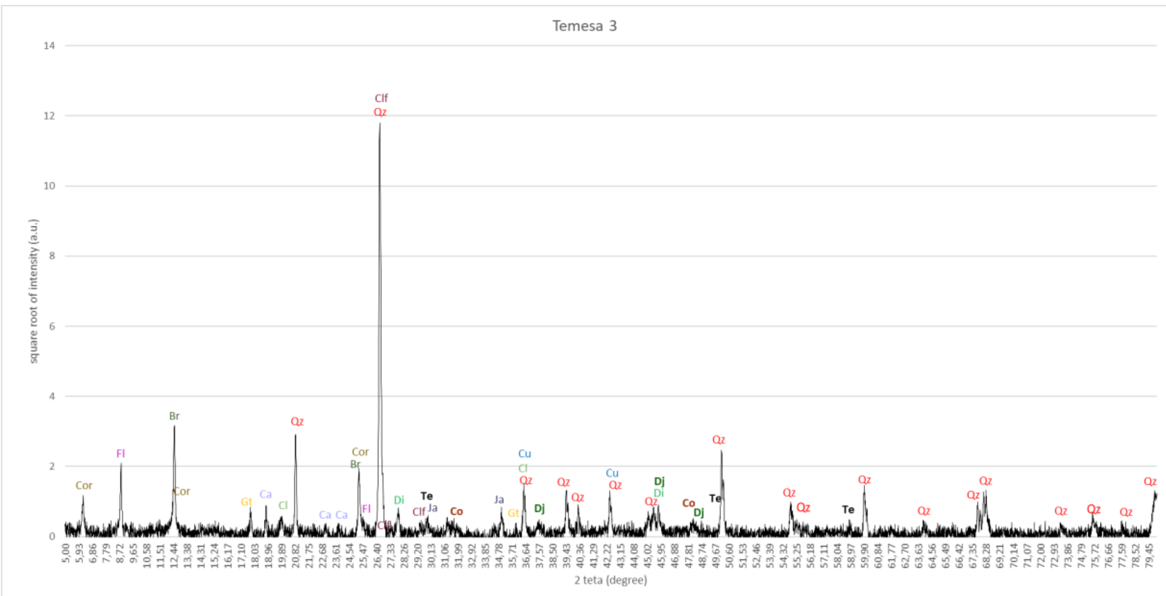


Figure 9. Temesa 3: XRD pattern. Abbreviations: Cor—corrensite, Fl—phlogopite, Gt—goethite, Cl—chloritoid, Qz—quartz, Di—digenite, Dj—djurleite, Cu—cuprite, Clf—clinoferrosillite, Br—brindleyite, Ca—chalcanthite, Te—tetraedrite, Ja—jacobsite, Co—covellite. Square-root intensity scale.

The XRF spectrum of a Temesa 3 sample (Figure 10) is similar to that of the other two samples. The iron content was quantified as 27%, while copper was not detected.

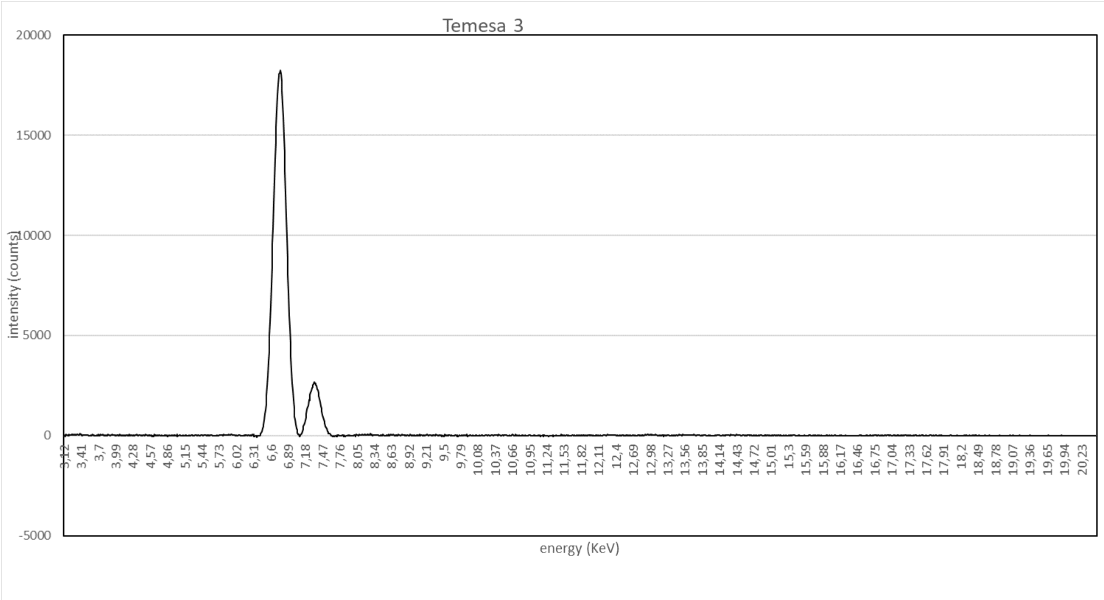


Figure 10. Temesa 3: XRF spectrum.

4. Discussion

Mineralogical and petrographic analyses indicate a common geological genesis of the samples. The formation is a low-grade regional metamorphic rock in which micas and clay minerals are widely represented alongside other typical species of phyllades, such as quartz, forsterite [36] (as serpentine alteration), clinoferrosillite and chloritoid. The rock has undergone copper and iron supergene enrichment [37] resulting in the presence of many copper sulfides, copper and iron sulfides, copper and iron oxides, iron hydroxides, and copper and iron sulphates.

Supergene enrichment is a secondary accumulation of metals via electrochemical oxidation, which transforms primary sulfides, oxides or native metals into soluble metal species that are, then, deeply transported by runoff water and re-precipitated by reduction, supersaturation, or cation-exchange below the water table. Copper is among the most common metals that undergo supergene enrichment, and it is mainly re-precipitated so as to form copper sulfides (covellite, chalcocite, digenite, djurleite) and, under more reducing conditions, copper and iron sulfides (chalcopyrite, bornite) or iron sulfides (pyrite, marcasite, greigite). The process is generally accompanied by capping, the precipitation of goethite and limonite (polyphasic compound roughly indicated with the formula $FeO(OH){\cdot}nH_2O$) in the leached zone, from which copper is removed. Between the leached zone and the enriched zone, there is an oxidized zone that contains copper and iron sulphates (chalcanthite, melanterite, brochantite ($Cu_4SO_4(OH)_6$), antlerite ($Cu_3SO_4(OH)_4$)), copper and iron carbonates (malachite, azurite ($Cu_3(CO_3)_2(OH)_2$), siderite ($FeCO_3$)), and copper and iron oxides (cuprite, tenorite (CuO), hematite (Fe_2O_3)).

Copper extraction from sulfides was widely practiced in the Italian peninsula in the early first millennium BC [38,39]. On the other hand, iron oxides and hydroxides, such as goethite, hematite and, to a lesser extent, magnetite, have always constituted the best minerals for iron smelting. Copper minerals are quantitatively very scarce, while iron minerals seem to be abundant. We must remember, however, that the historical sources report that Temesa mine was already exhausted in the first century BC. The presence of minerals useful for the extraction of copper and iron in a territory that literary and archaeological sources allow one to identify with the ancient Temesa constitutes an important element of confirmation. The area from which the rock samples come is about 600 m, as the crow flies, from the protohistoric necropolis of Chiane, which constitutes the northern limit of a vast highland settlement that occupied, starting from the 9th century BC, an area of over 40 hectares, similar in extension to that of the most advanced centers of the Calabrian Early Iron age [40,41].

An acquisition of particular interest is the large presence of iron minerals. The information provided by Strabo (VI, 1, 5) on the Temesa copper mines always led scholars to interpret the well-known passage of the Odyssey (I, vv. 180–184) as a testimony relating to an emporium in which copper was exchanged for iron of foreign origin. The presence of iron minerals could, instead, explain the Etruscan-Villanovan and Euboic-pre-colonial interests, which are well-documented [42]. Thanks to its iron deposits, Temesa could have attracted, around the middle of the eighth century, the interests of Euboic prospectors in search of metals, mainly iron, along the route that, crossing the Strait of Messina, headed towards Campania, the terminal of the first Euboic commercial interests [43].

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