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# Mineralogy of Iza Cave (Rodnei Mountains, N. Romania)

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# Abstract:

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The secondary minerals from Iza Cave result from the interactions of karst water and/or cave atmosphere with a variety of sedimentary and metamorphic rocks. The cave passages expose at various extents Eocene limestones and conglomerates, Oligocene black shales, Upper Precambrian micaschists, marble and dolomitic marble and associated ore deposits. Twelve secondary minerals identified in the cave (carbonates, sulfates, phosphates, oxides and hydroxides, and silicates) are presented in this study. Calcite, aragonite, gypsum, brushite and hydroxylapatite are the components of common speleothems in the limestone, dolomite and conglomerate areas of the cave. Ankerite crusts are related to areas with pyrite mineralization within the metamorphic carbonate rocks. Goethite, jarosite, hematite and gypsum form various speleothems in the sectors within micaschists and conglomerates. Large weathering deposits occurring in passage areas developed within micaschists consist of illite, kaolinite, jarosite, goethite, gypsum and alunite. The extent of the weathering deposits occurring on non-karst rocks in the underground environment makes this cave a particularly interesting site for studies of water-rock interactions.

Keywords: cave minerals, contact cave, lithology, weathering deposits, Rodnei Mountains, Romania

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# INTRODUCTION

Minerals forming speleothems are the results of complex interactions between bedrock, circulating water, and sediments of various sources (e.g. Hill & Forti, 1997; Onac, 2000; Forti et al., 2006; Ford & Williams, 2007; Palmer, 2007). In most caves formed in limestones, the minerals resulted from bedrock water interaction are calcite, less often aragonite, and gypsum (Hill & Forti, 1997). Pestera Iza (Iza Cave) from the Rodnei Mountains (Maramures county, East Carpathians, N Romania) crosses Eocene - Oligocene sedimentary and Precambrian metamorphic rocks: limestones, conglomerates, black shales, micaschists, marble, dolomitic marble and associated ore deposits (Fig. 1), and is so far the only cave known in Romania to develop in such a complex lithological setting (Tămaş, 2009a; Tămaş et al., 2009). Micaschists, conglomerates and the ore deposits exposed by the cave streams are subject to weathering due to the high humidity of the subterranean environment, thus

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<sup>4</sup>Electron Microscopy Center, Department of Experimental Biology, "Babeş-Bolyai" University, Clinicilor 5, 400006 Cluj-Napoca, Romania (lbarbu@biolog.ubbcluj.ro). creating large weathering deposits in places above the present level of the cave stream. Samples from one such weathering deposit in the cave had been previously analyzed by means of X-ray diffraction (XRD) and transmission electron microscopy (TEM) (Viehmann et al., 1979, 1981).

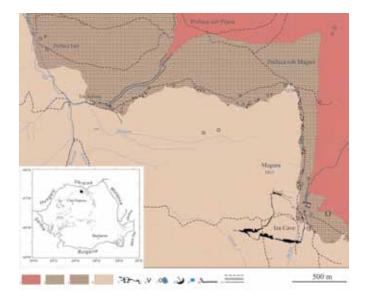


Fig. 1. Map of the Izvorul Izei karst area (modified from Mureşan, 2010); 1. Upper Precambrian crystalline rocks (undifferentiated): micaschist, quartzite, marble, dolomitic marble; 2. Lutetian conglomerate; 3. Priabonian limestone; 4. Oligocene shales and sandstones; 5 caves; 6. potholes; 7. dolines 8; ponors; 9. karst springs; 10 cliffs and gorges; 11. roads. Inset: Location of Izvorul Izei karst area (black square) on the map of Romanian karst.

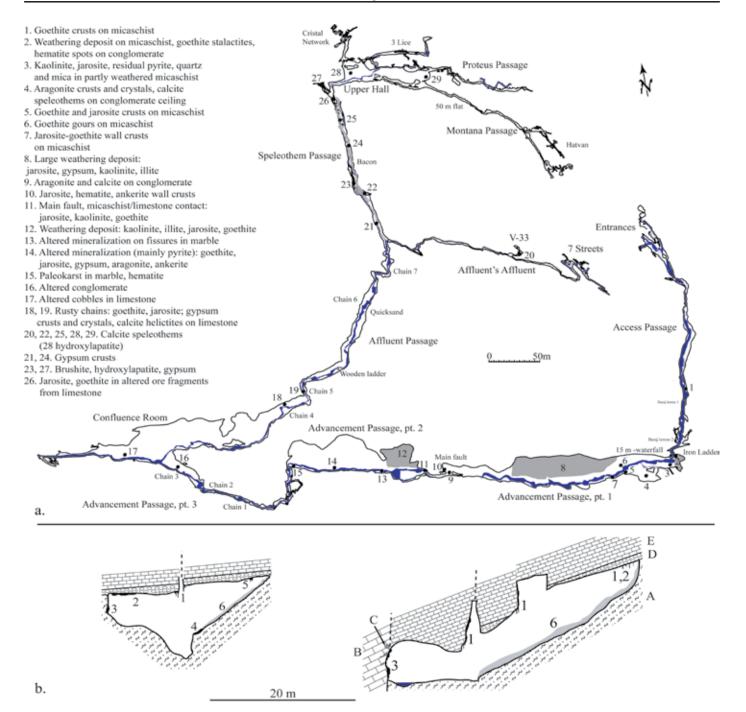


Fig. 2. a) Plan of Iza Cave showing the location of secondary deposits and various mineral occurrences (numbered); b) Typical cross - sections of the Advancement Passage showing the location of various speleothems in relation to the lithology of the host rocks (A – micaschist, B – marble, C – pyrite mineralization, D – conglomerate, E – limestone; 1, – calcite speleothems; 2 – aragonite speleothems; 3 – goethite and jarosite speleothems; 4. goethite speleothems; 5. goethite, calcite, hematite speleothems; 6. weathering deposit).

In 1979, the explored length of the Iza Cave was 2.5 km. In the '80's the entrance passage was blocked by tree trunks and visitors no further reached its deeper parts until 2000, when the cave got reopened. Since then, the members of the *Iza Cave Project* have mapped 4.4 km of passages, providing a detailed picture of the lithologic contacts between the formations that make up the geologic setting (Sahy et al., 2008; Tămaş et al., 2009; Mureşan, 2010). The purpose of this paper is to describe the secondary minerals from the Iza Cave and their relation with the lithology of the host rocks. In this respect, over a hundred samples have been collected from various secondary deposits within

the cave, especially from the Advancement Passage, although cave deposits similar to the ones sampled were identified along the whole cave (Fig. 2a).

# SITE LOCATION AND GEOLOGIC SETTING

The Iza River is an important left-side tributary to the Tisa, which forms a part of the northern border of Romania. Its origins are in the NW part of the Rodnei Mountains, in the territory of the villages of Moisei and Săcel (East Carpathians, Maramureş County). *Izvorul Albastru al Izei* (The Blue spring of Iza, 1025 m a.s.l.), or shortly *Izvorul Izei* (Iza spring) is the wellknown karst source of the river and drains the SE half of an Upper Eocene (Priabonian) limestone band. The main entry point for the surface waters in the system is the Iza Cave (1250 m a.s.l.) (Fig.1).

The crystalline basement of the area belongs to the Rebra Series, Upper Precambrian (Bercia et al., 1976; Kräutner et al., 1982) and consists of quartz micaschists with garnet, which underwent a Variscan retromorphism to the greenschist facies (Balintoni, 1997; Strutinski et al., 2006). The micaschists are associated with crystalline limestones and dolomites containing in places stratiform lead-zinc pyrite mineralization (Kräutner et al., 1978, 1982; Udubaşa et al., 1983). Although in the northern part of Rodnei Mountains they don't form economically important accumulations, on the southern side of the mountains the ore-containing carbonates were mined at Valea Blaznei and Guset (Socolescu et al., 1961; Udubaşa et al., 1983). The components of the mineralizations are pyrite, sphalerite and galena, with subordinate chalcopyrite, pyrrhotite, magnetite, rutile, ilmenite, arsenopyrite, tetrahedrite, barite and cymrite (Udubaşa, 1981).

The Priabonian limestone band is approximately 10 km long, 3 km large and 40-50 m thick and overlies a thin discontinuous bed of Lower Eocene (Lutetian) conglomerates disposed transgressively over the crystalline rocks (Fig. 1). The limestones are covered by Oligocene black shales and sandstones (Kräutner et al., 1982; Mureşan, 2010). Most of the carbonate succession has a high siliciclastic input (Sahy et al., 2008). The sedimentary rocks are disposed in a monocline dipping westward at 10-20° and discordantly overlie the crystalline basement (Kräutner et al., 1982).

# **CAVE DESCRIPTION AND LITHOLOGY**

Iza Cave (Figs. 1, 2a) is an active ponor cave consisting of several cave streams which create a dendritic network. The two main cave streams, each collecting several tributaries, join near a sump situated 181 m below Entrance 3. The three entrances are opened within the Priabonian limestones. Detailed descriptions of the Iza Cave were published first by Viehmann et al. (1979) and more recently by Tămaş (2009a, b) and Tămaş et al. (2009). The contact between the sedimentary and the metamorphic rocks is reached at 50 m from the entrance and can be followed at various levels in the walls along most of the cave, up to the Upper Room, developed only in limestones. The largest cross-sections occur in passages developed in the metamorphic rocks (15 m high, 40 m large).

The Advancement Passage of the cave (Fig. 2a, b) consists of 3 very large sectors, developed E-W along faults and connected by narrow canyons. The cave passages expose micaschists and metamorphic carbonate rocks, as well as conglomerates and limestones (Figs. 2b, 3). Pyrite mineralization occurs at various sites within the cave, as lenses in the metamorphic carbonates and micaschists, and blocks or cement in brecciated areas of the walls (Fig. 4 a). Pebbles, large cobbles and even boulders of the metamorphic rocks and associated ore deposits can be



Fig. 3. Geological setting at the junction of the Access Passage with the Advancement Passage in Iza Cave (The 15 m waterfall - sampling points 2 and 3 in Fig. 2a): the waterfall starts from a 1 m ledge of conglomerates separating the Priabonian limestones above and the crystalline rocks below (photo by A. Posmoşanu).

observed all along the cave walls in the conglomerates and to a lesser extent in the limestones (Fig. 4b). The floor of the Advancement Passage dips at 30-40° SW generally following the micaschist dipping. In the large sectors situated in the upstream part of the passage, the weathering deposits have formed on the exposed bedrock, along the areas not affected by flooding and where the micaschists or conglomerates are not covered by detrital sediments. In a rough estimate of its extent, the largest weathering deposit covers a surface of more than  $5000 \text{ m}^2$  and is in places more than 30 cm thick. The degree of weathering is variable: in parts large blocks still keep the older internal structure of the host rock. On longer, less steep slopes, where trickles of water cross the conglomerate ceiling, mass flows can form (Fig. 5a). The color of the weathering deposit varies from white to yellow, grey to reddish brown, depending on the dominance of a certain secondary iron mineral.

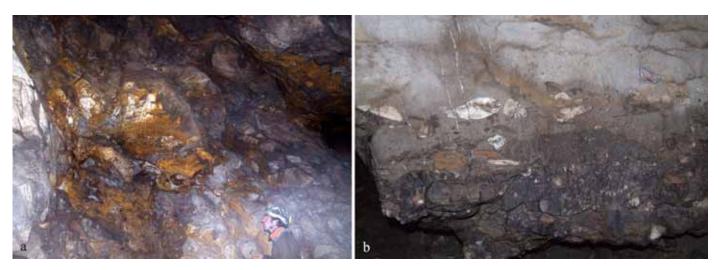


Fig. 4. a) Mineralized marble breccia with extensive goethite deposits on the left wall of the Advancement Passage; b) Lutetian conglomerates and transition to Priabonian limestones in the left wall of the Confluence Room: the "rusty" cobbles near the top of the conglomerates consist of oxidized mineralization (karabiner on top right for scale).

On the steep, almost vertical southern walls of the Advancement Passage, water seeping along schistosity or at the limestone-conglomeratemicaschist contacts has deposited brown-black stalactites, reddish powdery crusts and layered crusts of yellow-brownish crumbly material with either orange or blood-red earthy spots or stains composed of iron oxides and sulfates (Fig. 5a).

Along the Affluent series, until the Upper Hall, all passages follow the contact between micaschists and limestones, the conglomerates being replaced by scarce micaschist and/or marble blocks in a carbonate cement. The Fossil Level - Speleothem Passage complex crosses the whole limestone sequence, exposing also the Oligocene black shales in the passage ceilings. Weathering deposits are less extensive here due to the smaller size of the passages, which also are developed less and less in metamorphic rocks as one climbs upstream and calcite and gypsum speleothems predominate.

#### ANALYTICAL METHODS

Sample separations were made with a Nikon SMZ1500 stereo zoom microscope, equipped with a fiber-optic ring illuminator and a DS-5M standard CCD camera. The samples were analyzed by means of X-ray powder diffraction (XRD), using a Philips PW1710 diffractometer and a Bruker D8 Advance diffractometer (both with  $CuK_{\alpha}$ ,  $\lambda \alpha^{1} = 1.54060$  Å), with a step of 0.02° 20 for particle-oriented clay fraction, and  $0.04^{\circ} 2\theta$  for bulk samples. Mineral species were identified based on the PDF2 (2005) database. The samples were examined by transmission and scanning electron microscopy at the Electron Microscopy Center, University of Cluj on a JEOL - JEM 1010 (TEM) operated at 80kV, with samples sonicated in distilled water and deposited onto carbon coated Cu 400 mesh grids, and a JEOL JSM5510LV (SEM) operated at 15kV, with samples deposited onto carbon sticky tabs and 5 nm gold sputter-coated (Agar Automatic SputterCoater, Agar Scientific, UK).

# **RESULTS AND DISCUSSION**

The secondary mineral associations found in the Iza Cave comprise so far carbonates (calcite, aragonite, ankerite,), phosphates (brushite, hydroxylapatite), oxides and hydroxides (goethite, hematite), sulfates (gypsum, jarosite, alunite) and silicates (illite and kaolinite). The identification of these minerals was based mainly on their diffraction patterns. Electron microscopy was used in some cases to determine crystal shapes and morphologies.

In addition to the minerals above, dolomite, pyrite, muscovite and quartz have also been found in some samples, but they are not of secondary origin. Dolomite together with pyrite occurs in the areas of the cave walls where breakdown has exposed unaltered mineral deposits included in crystalline carbonate rocks. Quartz and partly muscovite and pyrite are the main residual minerals in the weathering deposits formed on the micaschists and conglomerates in the areas where these rocks were exposed through erosion by the cave streams.

#### Carbonates

*Calcite* (CaCO<sub>3</sub>) occurs in common speleothems such as stalactites, stalagmites, flowstones and also as pool crystals and crusts. Stalactites are more common along the faults from the ceiling of the Advancement Passage (where they have no corresponding stalagmites, which have been probably periodically washed away by the cave stream) and in the upper part of the Affluent section, especially along the Speleothem Passage and in the Upper Hall, the best decorated parts of the cave (Fig. 2a).

Aragonite (CaCO<sub>3</sub>) speleothems were found in several places along the Advancement Passage, on conglomerate ceilings and walls, where it occurs as needle-like crystals, crusts and globular formations (Fig. 2a). In secondary crusts associated with pyrite mineralization, aragonite forms levels along with gypsum, calcite and ankerite. In all occurrences aragonite formed from Mg-rich solutions resulted from the dissolution of metamorphic dolomite occurring either as levels in the crystalline rocks or as pebbles in the conglomerates.



Fig. 5. a) "Mass flow" in a weathering deposit (sampling point 8 in Fig. 2a); b) Weathered micaschists and Upper Eocene limestones at the Main Fault point (sampling point 10 in Fig. 2a); .

Ankerite  $[Ca(Fe,Mg,Mn)(CO_3)_2]$  uncommon in caves, was identified in Iza Cave in two occurrences: a) grey carbonate and sulphate crusts associated with the ore deposits, and b) reddish white crusts formed on conglomerate walls (Fig. 2a). It was identified by its XRD pattern (Fig. 6a). In the former occurrence, where it is associated with gypsum, quartz and aragonite its origin is not clear, in the second it is most probably a supergene mineral, occurring in the vicinity of goethite and hematite spots. The source of cations is the Lutetian conglomerates, which include pebbles of both pyrite mineralization and metamorphic carbonate rocks. This is the third occurrence of ankerite in Romanian caves, after the ones in Peştera Mare din Cheile Runcului (Onac, 1992) and Movile Cave (Diaconu & Morar, 1993). Other world occurrences include caves in Ukraine, South Korea and Turkmenistan (Hill & Forti, 2007).

#### Phosphates

Brushite (CaHPO<sub>4</sub>·2H<sub>2</sub>O) occurs either as small nodules in fine sands, associated with gypsum crusts and crystals, or as powdery deposits on the walls of the Speleothem Passage (Fig. 2a), whereas hydroxylapatite [Ca<sub>5</sub>(PO<sub>4</sub>)<sub>3</sub>(OH)] forms black crusts covering pebbles or calcite speleothems in the Speleothem Passage and in the Upper Hall (Fig. 2a). Both these phosphates are common cave minerals and resulted from the interaction of limestones and/or calcite speleothems with small bat guano deposits in slightly different pH conditions - below 6 for brushite and above 6 for hydroxylapatite (Hill & Forti, 1997).

#### Iron oxides and hydroxides

*Goethite* [FeO(OH)] is widely spread in the Iza Cave on a great variety of substrates (Figs. 2a, 7): micaschists, marble, conglomerates, limestones and various consolidated cave sediments, even on the iron chains used for rigging during the first exploration of the cave in the '70's. Cryptocrystalline goethite is the main component of stalactites, gours and microgours and may also form yellow, brown or black layered crusts in the weathering deposits (Figs. 6b, 7).

Hematite ( $Fe_2O_3$ ) occurs at the Iron Ladder and in several other places along the Advancement Passage as dark-red powdery spots or crusts covering the weathered micaschist or conglomerate walls (Figs. 2a, 6c, 7a). Although it may sometimes indicate hydrothermal conditions (Hill & Forti, 1997; Hill, 1999), in the Iza Cave hematite is most likely a lowtemperature mineral, formed through the dehydration of iron hydroxides in strongly ventilated areas of the passages. Most hematite samples analyzed by XRD show a low degree of crystallinity.

## Sulfates

Gypsum (CaSO<sub>4</sub>·2H<sub>2</sub>O) is the most widespread mineral throughout the cave, occurring on all types of substrates as tiny acicular crystals, always under 1 cm long, associated with iron hydroxides in areas within the WD and in weathered cobbles inside the conglomerate; crusts and acicular crystals in micaschists; crusts on limestones and sediments in the Affluent section. In the Fossil Level, millimeter-size transparent gypsum crusts cover completely the

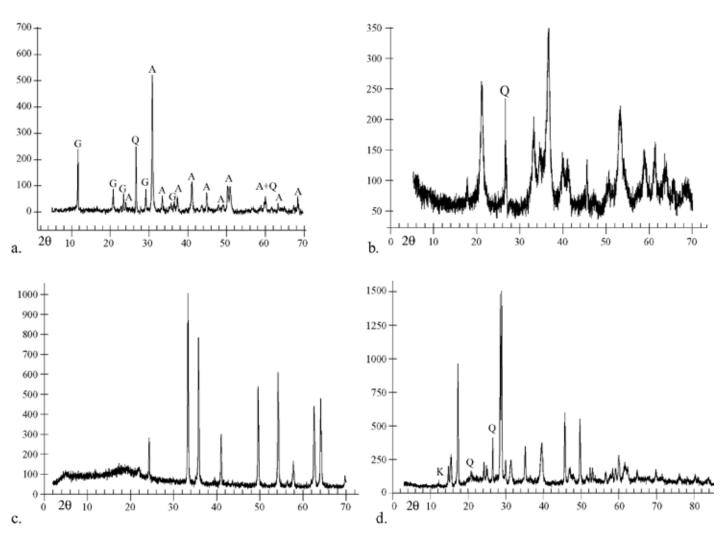


Fig. 6. XRD patterns of: a) ankerite (A); b) goethite; c) hematite; d) jarosite (Q quartz, G – gypsum, K – kaolinite).

limestone walls of a canyon sector. Gypsum resulted from the oxidation of pyrite, present in the metamorphic rocks and associated ore, as well as in the limestones and in the Oligocene black shales covering the limestone all over the cave area.

Alunite  $[(K,Na,H_3O)Al_3(SO_4)_2(OH)_6]$  is a minor constituent in the weathering deposits and it was determined only by its most intense XRD lines in two samples (Tămaş & Ghergari, 2003) and its presence is therefore somewhat ambiguous. It probably resulted from the transformation of muscovite in the presence of sulfuric acid resulted from pyrite oxidation.

Jarosite  $[(K,Na,H_3O)Fe_3(SO_4)_2(OH)_6]$  forms milimeter to centimeter-sized yellow-orange dusty nodules and crusts in various areas of the altered nonlimestone cave walls (Fig. 7). Small (1 - 3 µm), almost isometric rhombohedral crystals occur in the weathering deposits and in distinct levels in well-rounded pebbles inside the conglomerate (Fig. 8). Jarosite as a cave mineral has been reported from few caves in the United States, South Africa, Italy and Nepal, originating from sulfur bearing solutions, with the iron provided either by pyrite mineralization, basalts or schists (Hill & Forti, 1997). The only other cave occurrences of jarosite in Romania are Fântâna lui Pavel (Tămaş & Ungureanu, 2010) and Turia Cave (Onac & Forti, 2011). In the Iza Cave, jarosite formed in a strongly acidic environment from the oxidation of pyrite, provided by the mineralization associated with the crystalline carbonate rocks, or disseminated in micaschists and conglomerates.

#### Silicates

The silicates identified so far by us in the Iza Cave are *illite*  $[K(A1,Mg,Fe)_2(Si,A1)_4O_{10}(OH)_2,(H_2O)]$  and *kaolinite*  $[A1_2Si_2O_5(OH)_4]$ , occurring in the large weathering deposits formed on micaschists along the Advancement Passage (Figs. 5a, 8b). They are definitely authigenic and have formed through weathering on the expense of muscovite and feldspars present in the micaschists. Illite and kaolinite have also been reported by Viehmann et al. (1979, 1981), in the so-called "*Iza silicate complex*", where they are associated with muscovite and quartz, residual minerals from the original host rocks.

#### CONCLUSIONS

Twelve secondary minerals belonging to five classes have been so far described from the Iza cave. Calcite, aragonite, and gypsum, common in normal limestone caves, occur in the passage sectors developed in limestones and conglomerates. Calcium phosphates occur in sections of the cave where bat



Fig. 7. Speleothems in the Advancement Passage of Iza Cave: a) Goethite stalactites (about 20 cm long); note the different coloured zones on the wall: reddish (predominantly hematitic), yellow (jarosite and goethite) and white (clay minerals and muscovite); b) Wall crusts and microgours; the upper yellowish crust is made up mainly by jarosite, while goethite and an amorphous mineral (probably allophane) are deposited below, indicating pH increase along the flow path; c) Goethite flowstone on retromorphosed micaschist; d) "Banded" weathering deposit; part of the original rock may be noticed in the bottom left corner.

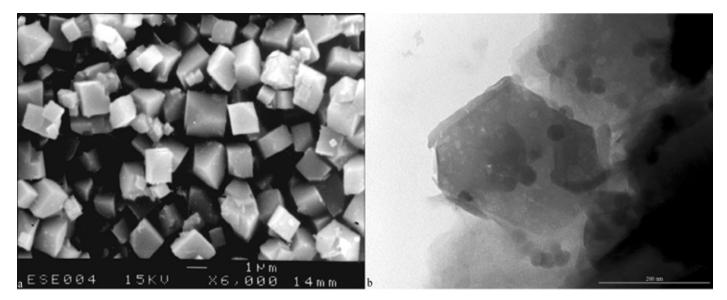


Fig. 8. a) SEM image of jarosite crystals; b) TEM; kaolinite crystal and "spheres" of amorphous material.

guano supplied phosphate ions. Ankerite occurs in areas where the cave walls consist of metamorphic carbonate rocks and is deposited from magnesium and iron-rich carbonate solutions. Gypsum is the most ubiquitous mineral, occurring on all types of substrates, both in limestone areas and in zones within micaschists and conglomerates, where it occurs inside the weathering deposits.

The large amount of secondary iron minerals discovered is due to the presence of pyrite concentrated in lenses or disseminated in all the other rocks forming the walls of the cave. The occurrences of these minerals within various lithologic settings give valuable information about their genetic environments. Jarosite forms in strongly acidic environments, while goethite requires weakly acidic or neutral environments. In many occurrences in the cave, one can observe a gradual transition from one mineral to the other (jarosite – goethite, ± gypsum if calcium available) indicating gradual changes in the acidity of the solutions.

Two main factors are responsible for the cave minerals resulting from the non-karst rocks exposed in the cave. The first is the seepage water crossing micaschists and conglomerates and depositing mainly goethite crusts, flowstones and stalactites, with accessory jarosite, hematite and gypsum. The second is the humidity of the cave atmosphere, which creates extended weathering deposits on non-karst rocks no longer exposed to flooding by the cave streams. This resulted in a mineral association consisting of illite and kaolinite as main minerals, with variable amounts of jarosite, goethite and gypsum. The occurrence of these minerals, together with the extent of the weathering deposits developed on non-karst rocks make the Iza Cave an important site for the study of the karst water interaction with different bedrock lithologies.

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