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# **SPY CAVE**

125 years of multidisciplinary research at the Betche aux Rotches (Jemeppe-sur-Sambre, Province of Namur, Belgium)

Edited by Hélène ROUGIER & Patrick SEMAL

Volume 1

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#### CHAPTER VIII

#### **OOLITHIC IRONSTONES FROM SPY CAVE**

#### Éric GOEMAERE, Cécile JUNGELS & Anne HAUZEUR

#### Abstract

Numerous pieces of oolithic ironstones from Spy cave are described in this chapter. They are supposed to come from the famous "red layer" which occurs in the "second fauna-bearing level". They are deposited in many different collections and the detailed stratigraphic context is mostly unclear. Numerous isolated Fe-ooliths are associated with hematitic powder, regarded as responsible for the red pigmentation of the "red layer". Only one artefact shows polished surfaces. Other pieces have been probably crushed to obtain a red powder. What the red pigment was used for remains unknown. The Belgian geological context of oolithic ironstones is described. The macroscopic comparison between geological and archaeological samples as well as the determination of the magnetic susceptibility allow to propose the Lower Famennian from the Parautochton of Namur, east of Spy, as the main stratigraphical and geographical source for the oolithic ironstones.

#### **INTRODUCTION**

Ochre and hematite are names that archaeologists use to refer to several forms of iron oxide, a type of clay or sandy clay mineral that is found in natural deposits in many regions of the world.

Goethite and hematite are among the first coloured ores from which a use is attested in archaeological records. Early examples include a Mousterian tool made in ochre discovered at Oafzeh (Israel) by Vandermeersch (1969), lumps of red ochre and ochre-stained shells from the same site (Bar-Yosef Mayer et al., 2009), redstained marine shells associated with Iberian Neandertals (Zilhão et al., 2010), and ochre blocs discovered in Blombos cave (South Africa) and wearing scratches interpreted as non-utilitarian (d'Errico et al., 2001). The latter are dated about 77,000 BP. Older dated discoveries in the same area induce questions about their status as functional or symbolic (Barham, 2002; Watts, 2002). But for sure, together with manganese oxides and charcoal, chosen for their black colour, they constituted the early prehistoric "painter's palette" (e.g. Pomiès & Menu, 1999). Whatever their functions during Prehistory, red ores were attractive to such an extent that extraction was organised since the Upper Palaeolithic (Schild et al., 1997; Trąbska et al., 2008).

Ochre comes in a variety of colours, from brown to red to yellow. Red ochre is mainly composed of pulverised hematite (Fe<sub>2</sub>O<sub>3</sub>), while the yellow ochre is composed of limonite (field term for a group of hydrous ferric oxides) or goethite (hydrated iron oxide –  $\alpha$ -Fe<sup>3+</sup>O(OH)).

Sedimentary rocks with more than 15 % of iron, which have usually been worked as ores, are known as ironstones. Phanerozoic ironstones are usually local accumulations of fossiliferous oolithic deposits and are called oolithic ironstones (Adams et al., 1984) and also called in French: "oligiste oolithique", "hématite oolithique" or "fer oolithique". The main iron mineral is hematite and the colour of this rock is deep red in fresh material to brownish in weathered rock (limonite). The mineralogical composition of the oolithic ironstones (acronym: OI) is complex and their relation with stratigraphy can often be specified. Besides hematite, the mineral compounds are quartz, clay minerals (micas, chlorite, chamosite) (Laenen et al., 2002), different types of carbonates (calcite, dolomite, iron carbonates), and marine fossils (crinoids, brachiopods,...). Fossils can be present as rounded fragments,



Figure 1. Oolithic ironstone from Vezin, Andenne.

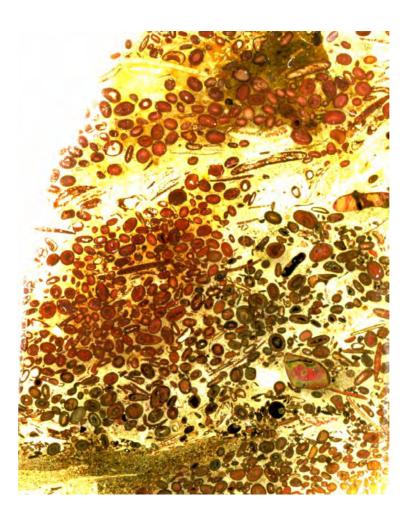


Figure 2. Microscopic view (natural light) of an oolithic ironstone from an old mine in Sclaigneau (Andenne), Lower Famennian, north border of the Namur Parautochton. Sorting of the mm-sized ellipsoidal ferruginous ooliths is good. See the concentric structure and the presence of a nucleus inside some ooids. Fossiliferous fragments are rare. The cement is composed of chamosite.

either broken or whole allochems, or as nuclei for ooids. Bioclasts are often impregnated by iron oxide. Petrographical study allows identifying mineral, texture, structure, and comparison with known geological levels (Figures 1-5; see also the Lexicon at the end of the chapter).

OI artefacts were found and briefly described on Belgian Early Neolithic sites (Linear Pottery Culture or LPC). At the beginning of the 20th century, M. De Puydt (1903) wrote down that the oolithic oligist came from the region of Namur. J. Thisse (Thisse-Derouette & Tomballe, 1957) described and analysed (chemical analyses, microscopy) OI artefacts from ten LPC (or Omalian) sites in the Liège-Hesbaye region. The comparison with geological samples coming from ten old iron mines allowed him to conclude that the artefacts and the iron ores of Couthuin, Waret and Les Isnes villages, located between Spy and Huy, are similar.

#### ARCHAEOLOGICAL CONTEXT

Numerous pieces of OI were discovered during several archaeological excavations of the Spy cave (OISC). OISC pieces are deposited in different scientific institutions and private collections.

In the literature devoted to the excavations at Spy, the "second fauna-bearing level" is generally considered as a layer of variable thickness, totally or partially red coloured. It is a sedimentary breccia coating ivory fragments, knapped flints, charcoal, and crushed limestones (De Puydt & Lohest, 1887: 209 and annexe statement; Fraipont & Lohest, 1887: 664, 668; de Loë & Rahir, 1911: 46). The numerous pieces of hematite – and the powder they could generate – discovered in this layer were at that time already considered as the cause of this coloration (De Puydt & Lohest, 1887: 213; de Loë



Figure 3. Microscopic view (natural light) of an oolithic ironstone from Tailfer (Lustin), Lower Famennian, north border of the Dinant Synclinorium (Dinant Basin). Sorting of the ellipsoidal mm-sized ferruginous ooliths is weak. Fossiliferous fragments (crinoids and rounded fragments of brachiopods shells) are present. The cement is composed of chamosite and calcite.

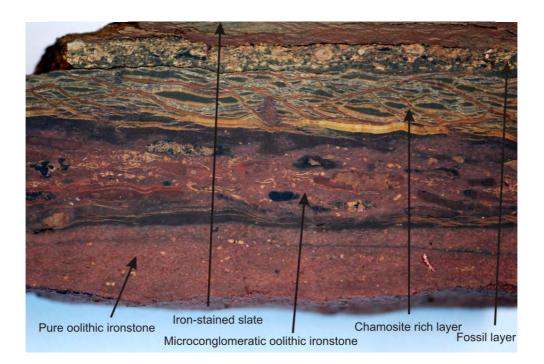


Figure 4. Polished slab of an oolithic ironstone showing different successive centimetre-wide layers; old iron mine place known as "Bure Bodson", Namêche (Andenne, Namur Parautochton north border).



Figure 5. Decimetre-thick layer of oolithic ironstone pitched between greenish Lower Famennian slates, section of Huy, along the Meuse river and the railway section, east of the railway station. South reversed flank of the Namur Parautochton. & Rahir, 1911: 46). Nevertheless, most of the hematites come from Twiesselmann's excavations in the shaken up or waste levels of the slope.

As no archaeological context is assumed for the hematite pieces coming from the slope, an attribution to a later period than Early Upper Palaeolithic cannot be excluded. Actually, the presence of a scrubbed/grinded oolithic hematite (Figure 6), and that of Neolithic human remains coming from destroyed burials (Semal *et al.*, 1996), suggests that part or most of the material could correspond to funeral goods or deposits.

Several lithic and bone artefacts wear traces of this reddish/pink sediment (SF1). Those pieces are mostly associated with the "second fauna-bearing level". The typological diagnostic for the "coloured" lithic artefacts belonging to the De Puydt collection at the *Grand Curtius* Museum (Liège) points out their attribution to the Upper Palaeolithic, in particular to the Aurignacian (Table 1).

The question is asked whether some pieces are coloured because of a long stay in this

"red" sediment or because of an anthropogenic coating. Several pieces of finery seem tinctured with ochre, especially the ear-shaped pendants (De Puydt & Lohest, 1887: 223). Moreover, some bird bone tubes seem to have been used to keep colouring agents. Their inside walls are still strongly impregnated with ochre (Otte, 1979: 312).

## GEOLOGICAL AND GEOGRAPHICAL SETTINGS

The Belgian geological context is described in this volume by Pirson, Delcambre & Goemaere (chapter V).

Since the end of the 19th century, several distinct OI levels have been reported from the Middle Devonian (Eifelian), Upper Devonian (Frasnian and Famennian), and Middle Jurassic (Aalenian, Bajocian) strata of Belgium and bordering countries but their mining as an iron ore is very old. Their geographic distribution, in Belgium and bordering areas, is reported on Figure 7.



Figure 6. Grinded hematite fragment with two opposite flattened surfaces (*Université de Liège*, De Puydt collection, "second fauna-bearing level").

<i>Type of pieces with red sediment</i>	Chrono-cultural attribution	Number	Location	Layer	Number
	Linner Delegelikkin en leter	7	Tamaaa	UL	6
	Upper Palaeolithic or later	/	Terrace	SFBL	1
			Теггасе	UL	8
		130		SFBL	110
	Upper Palaeolithic (sensu largo)			TFBL	1
			IPC	Undet.	4
			Undet.	Undet.	7
	Upper Palaeolithic, Gravettian	1	Undet.	Undet.	1
Lithic artefacts	Upper Palaeolithic, Aurignacian	101	Terrace	SFBL	91
			IPC	Undet.	1
			Undet.	Undet.	9
	Middle Palaeolithic	10	Terrace	SFBL	15
		18	Undet.	Undet.	3
		69	Terrace	UL	4
	TT. I. de musica d			SFBL	60
	Undetermined		IPC	Undet.	2
			Undet.	Undet.	3
Tiddiana aliana	Undetermined	5	Terrace	SFBL	1
Lithic raw pieces		5	Undet.	Undet.	4
Breccia (with undetermined	I Indetermined	4	Terrace	TFBL	2
lithic pieces)	Undetermined	4	Undet.	Undet.	2
Total		335			335

Table 1. Chronocultural attribution for pieces wearing reddish/pink sediment residues and coming from the excavations of M. De Puydt and M. Lohest in 1885-1886 (De Puydt collection, *Grand Curtius* Museum, Liège). The chronocultural attribution is based on typological determination. The layer and the location of the pieces come from the De Puydt collection's inventory, 1920, *Grand Curtius* Museum, Liège. IPC = inner part of the cave; Undet. = undetermined; UL = upper level; SFBL = "second fauna-bearing level";

TFBL = "third fauna-bearing level".

- The *Middle Jurassic OI*, called "*minette*", are considered to be economically more important and were intensively mined in the three border areas (Belgian Lorraine, French Lorraine, and Luxembourg Gutland).
- The *Famennian OI*, considered economically as the second most important ore, have been previously mined almost in the north flank of the Namur Basin (Namur Parautochton<sup>1</sup>; Dreesen, 1982). In this geological unit, the thickness of the ore veins ranges between 0.25 and 1.50 m, but their geological and geographical occurrences are larger. They also occurred at the south border of the Namur Basin, at the northern and eastern border of the Dinant Basin, and in the Vesdre Basin. In the Famennian, ferruginous

ooid-bearing, bioclastic wacke- to grainstones occur interbedded in shales, nodular shales (Verviers) or micaceous silt- and sandstones (Arbre, Profondeville). In the Vesdre Basin (Verviers Synclinorium), four conspicuous closely spaced beds are described, three in the Hodimont Formation (I, II, IIIa levels) and one at the base of the Esneux Formation (IIIb level - 0.15 to 0.70 m thick) (Dreesen, 1982). Delmer (1913) noticed the purple staining of the Lower Famennian shales (Mariembourg and Senzeilles Formations) and mentioned their different macrofacies (siliciclastic and calcareous matrix). The litho- and bio-stratigraphical position, the geographical distribution, the macro- and microfacies analyses of the Famennian OI through the Dinant Basin and the Vesdre Basin were studied by Dreesen (1984). OI consist of different discontinuous levels, interstratified within the green or purplish

<sup>&</sup>lt;sup>1</sup> The Namur Parautochton was formerly named Namur Syncline.

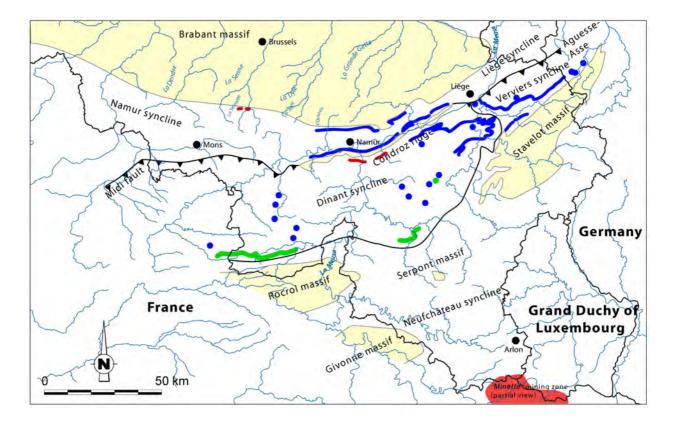


Figure 7. Geographical distribution of the Belgian oolithic ironstones related to their stratigraphical position. The main structural units are shown on this map; the lower Palaeozoic inliers are represented in yellow. Legend: green line: Eifelian (old Lower Couvinian - Coa); red line: Upper Frasnian; blue line:
Lower Famennian (old Fa1b); red area: Aalenian and Upper Bajocian (Belgian Lorraine area, French Lorraine & south of the Grand Duchy of Luxembourg). The Brabant Massif is drawn by removing the thick Tertiary cover. The outcrops are limited to the Dendre, Dyle, Thyle, Senne, Sennette, Gette and Orneau Valleys. The blue circles indicate the localities where oolithic ironstone was found according to the literature.

shales. Each level, ranging in thickness from a few centimetres to more than two decimetres, consists of ferruginised allochems imbedded in fossiliferous shales or lenticular crinoidal-brachiopodal limestones. Impure fossiliferous limestones with iron ooids are also present at the south flank of the Dinant Synclinorium. Note that OI from Héron and Lavoir were considered as Upper Famennian on the base of brachiopods (Ancion et al., 1956). Other levels are more anecdotic, even if they have been mined as an iron ore along the Couvin - Pétigny axis (Marion & Barchy, 2001). Wulff & Schmidt (in Dreesen, 1984) noticed a thin calcareous OI layer within the Upper Famennian sandstones north of the Aachen anticline and a 0.90 m thick red-stained limestone with iron ooids ("oolithisches Roteisenerz") from the Famennian of Walheim (eastern part of the Vesdre Valley).

The sorting, the granulometry (mean grain-size and clasticity), the nature (detrical, carbonates, clayed or mixed) of the intergranular matrix, the abundance of the matrix, the shells (unbroken, broken or rounded), the size and the shape (rounded, flat) of the iron ooids are additional descriptive elements to discriminate the different stratigraphic levels and the lateral variation within the sedimentation basins. As a consequence, oolithic Famennian levels represent true marker beds to correlate distant sections, especially throughout the Dinant and Vesdre basins. Some of them can be traced from the Aachen (Germany) region to the Avesnois (France).

Variations of the macro- and microfacies are related to palaeogeography and as a consequence to geography, bringing keys to ascribe archaeological samples to geological and geographical sources.

If we consider both the weak thickness and the folding of the OI layers, how is it possible to collect this raw material on the field? Its red colour and its availability to stain the surrounding weathered shales are the field criteria. The south flank of the Namur Parautochton is characterised by reversed layers with a high dip. On the other hand, the north flank of the Namur Parautochton is gently inclined to the south. It is important given the decimetre thickness of the ironstones layers because outcropping zones in flat areas decrease with their dip. For example, a 0.30 m-thick layer with a dip of 80° has an outcrop width of 0.303 m while the same layer inclined at 10° has an outcrop width of 1.68 m. So the probability to discover red-coloured OI between green slates and collect them on the field is higher in the north flank of the Namur Parautochton (the northernmost blue line on the geological map of Figure 7) than in other places!

- *Frasnian and Eifelian OI* are poorly understood and no study was performed until today. The iron content is low and the matrix is rich in carbonates.

In addition to their specific fossiliferous contents, Palaeozoic and Mesozoic OI can be differentiated by their mineralogy. Mesozoic ironstones mainly contain goethite and berthierine (Fe-rich silicate mineral of the serpentine group), while Palaeozoic OI contain hematite and chamosite (Fe-rich trioctahedral chlorite). These iron-bearing sheet silicate minerals can be identified via X-ray diffraction.

#### **MATERIAL AND METHODS**

The aim of this study is to test the supposed relationships between stratigraphic levels, geographic location and magnetic susceptibility and to apply this method to archaeological samples. The studied material consists of archaeological artefacts and geological raw materials. Furthermore, we have deliberately included red (hematite), red-brown, and yellow (goethite) OI, which differ from the dominant iron mineral present. Each sample was macroscopically described in order to determine diagnostic descriptive parameters. Shape, dimension, sorting and orientation of ooids, colour, nature of matrix and cement, occurrence of fossils, intraclasts, pyrite, etc. were determined. No destructive analysis methods were used on the archaeological samples.

#### Archaeological samples

The 243 OI samples examined in the framework of this study come from the Royal Belgian Institute of Natural Sciences in Brussels (RBINS), the Royal Museums of Art and History - *Cinquantenaire* Museum in Brussels (RMAH), the *Université de Liège*, the *Grand Curtius* Museum in Liège, the *Musée Archéologique de Namur* and the Beaufays collection<sup>2</sup>. Both description and location of the 243 OI are reported in ST1, while the archaeological context is described in other chapters of this volume of the present monograph. Most of the samples come from Twiesselmann's excavations in the slope deposits (N = 199).

In addition to the samples from Spy cave, some other archaeological samples have been selected from Palaeolithic cave sites, like Goyet, Chaleux and Trou Magrite, and from an Early Neolithic (LPC) open-air settlement at Darion, to have a contrasted reference (ST2). This choice was operated in such a way for several reasons: 1) those caves have also Upper Palaeolithic levels; 2) like Spy, they are close to potential outcrops; 3) all of this material is kept at the RBINS and was easily accessible.

#### **Geological samples**

The geological material used in the study consists of 60 samples coming from three different collections of the RBINS (stratigraphical, mineralogical and Dumont collections); they cover the Famennian (main samples), Frasnian (4 samples) and Aalenian-Bajocian (called "*minette*", 4 samples) from Belgium and 2 samples (Famen-

<sup>&</sup>lt;sup>2</sup> The Beaufays collection was still a private collection when we performed this study. It joined the RBINS collections in 2009 (Semal *et al.*, 2009).

nian) are from Germany (ST3). These samples have been present in the collections for a long time and come from iron mines, outcrops and pits. An additional 17 Frasnian and Lower Famennian OI have been newly sampled in outcrops, mainly on riverbanks and old humps. All these samples constitute a reference collection to compare with ironstones from some archaeological Belgian sites (Spy, Goyet, Chaleux, Trou Magrite, and Darion).

#### Magnetic susceptibility

Mooney and collaborators (2003) describe the magnetic susceptibility (MS) as the magnetic response of a sample when exposed to a (generally weak) magnetic field. The induced magnetisation is reversible, so no remanence is acquired. MS is mainly a function of the concentration and mineralogy of the ferromagnetic (magnetite, maghemite, Fe-sulfides) minerals present, but can also depend on the strength of the applied magnetic field and the particle-size distribution of the magnetic grains.

In the absence of ferromagnetic minerals, MS can be due to antiferromagnetic (hematite, goethite), paramagnetic (e.g. Fe-bearing silicates) or diamagnetic (e.g. quartz, calcite) minerals. MS is also dependent on sample size<sup>3</sup>. MS was measured in 2008 on a Geofyzika Kappa Bridge KLY-3S of the Université de Liège (Prof. F. Boulvain). Samples were cleaned and air-dried. A known mass was put into clean tubes. Samples were weighted with a precision of 0.01 g and three measurements were made on each sawn sample (max. 2.5 x 2.5 cm). Therefore it is customary to present susceptibility values as mass normalised susceptibility. Average MS was calculated and is expressed in m<sup>3</sup>/kg. Only small-sized archaeological samples were selected to stay unchanged during the measurements, while large geological samples were broken to reach suitable size for measurements.

<sup>3</sup> See the website of the *Université du Québec à Chicoutimi* for more theoretical information (www.ens.uqac.ca/chimie/chi-mie-theorique/chapitres/chap\_14.htm).

	RBINS (Twi	esselmann co	ollection): 18	87 samples						
	Weight	Length	Width	Height	L/W(*)					
Average	3.9	12.5	9.1	4.4	1.5					
Median	1.9	16.0	12.0	6.0	1.4					
STD	14.3	9.3	6.3	3.3	0.3					
Minimum	0.1	8	5	2						
Maximum	164	78	45	28	2.6					
Shape	rectangle	triangle	square	trapezoid	hexagon					
Numbers	120	40	24	2	1					
Université de Liège (a. o. De Puydt collection): 18 samples										
	Weight	Length	Width	Height	L/W(*)					
Average	13.9	31.6	24.6	9.9	1.5					
Median	10.4	29.8	24.5	9.0	1.3					
STD	12.7	9.8	9.0	4.1	0.6					
Minimum	1.3	16	8	4	1.2					
Maximum	48	46	38	16	3.0					
Shape	rectangle	triangle	square	trapezoid	hexagon					
Numbers	8	6	2	1	1					
RMAH (de Loë collection): 18 samples										
	Weight	Length	Width	Height	L/W(*)					
Average	17.1	34.4	22.9	7.5	1.7					
Median	8.1	28.5	22.5	6.5	1.5					
STD	30.5	20.3	14.1	3.4	0.6					
Minimum	1.5	12	8	4	1.3					
Maximum	132	100	65	16	3.3					
Shape	rectangle	triangle	square	subrounded						
Numbers	11	3	2	2						

(\*) calculated only for rectangle-shaped samples.

Table 2. Morphology, weight (in g), and dimensions (in mm) of the OISC.

#### MACROSCOPIC CHARACTERISATION OF OOLITHIC IRONSTONES FROM THE SPY CAVE (OISC; SF2-8)

OISC ooids have a hamburger-like shape and a size ranging between 0.5 and 1.2 mm. The sorting is good and the content of carbonate is very low. Fossil remains are rarely observed while they often occurred in Lower Devonian OI outcrops. The general shape of the samples corresponds to a right-angled parallelepiped or presents a triangular shape, lengthened according to the bedding plane (Table 2). OISC samples show a great homogeneity of aspect, colour, granulometry and texture indicating a probable single geological source, but we can also suspect that Prehistoric humans were very selective when choosing their material. ished, sub-perpendicular to the plane of stratification. Angles between these surfaces are smoothed. The two other surfaces are small sides with a broken aspect.

One piece from the *Université de Liège* collection shows two grinded surfaces (the ooliths are flattened; Figure 6). It was found by M. De Puydt in the "second fauna-bearing level". M. Otte also mentioned a piece covered by scrubbing striations (Otte, 1979: 306). This piece was no more found in the collections but an original sketch by M. Otte still exists (Figure 9).

If we cannot completely exclude an alteration on the archaeological site, the rounded or smoothed morphology of some Betche aux



Figure 8. Pentagonal-shaped discoid "nodule" of siderite with goethite and hematite impregnated crust (onion-skin weathering). The sample shows polished surfaces and striation (*Musée Archéologique de Namur*, A.5627).

If around 20 % of the material collected during Twiesselmann's excavations show rounded or smoothed edges, only one specimen showing obvious traces of anthropogenic use was found. It is a pentagonal-shaped lenticular nodule (length of the sides: 26-20-10-12 mm, and 8 mm; width: 9.5 mm; weight: 9.36 g) with six polished surfaces (Figure 8). The two main polished parallel surfaces match with the bedding plane while the four others are small surfaces – two are well polished while the two others are only slightly polRotches samples could be regarded as coming from a surface hand-picking. Siderite nodules frequently occur in the Namurian shales close to the cave. At the outcrop, they present an atmospheric alteration inducing the precipitation of iron oxy-hydroxides at their upper surface.

A histogram of frequency was built based on the weight of the samples (Figure 10). The weights spread out between 0.2 and 164 grams. The average and the median are

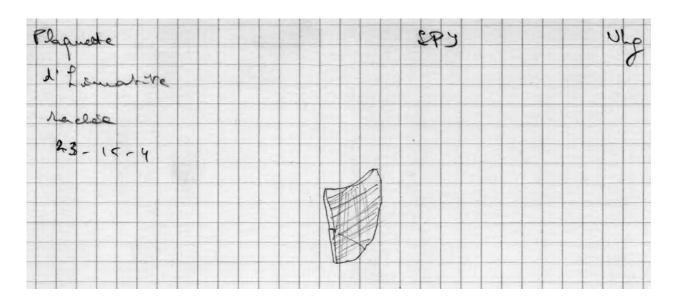


Figure 9. Worked hematite fragment. Original sketch drawn by M. Otte (Université de Liège collection).

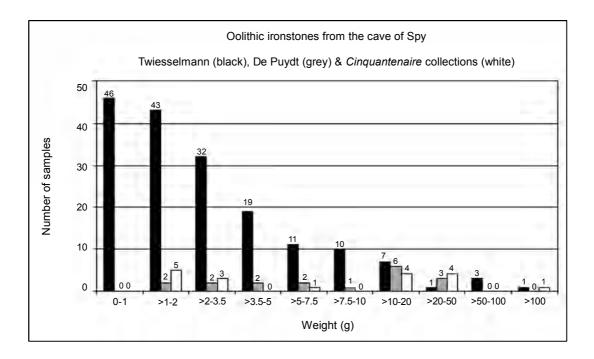


Figure 10. Weight data plotted into a frequency diagram (with variable intervals) to show the probable influence of the methods of the successive excavations on the piece sizes.

respectively 5.1 and 2 grams indicating that the majority of the samples from the Twiesselmann collection are small-sized. The average weight is strongly influenced by the heaviest samples, drawing the standard deviation towards high values (14.6). The same exercise, carried out on the samples preserved at the RMAH, shows an increase of the sizes to higher values (SF7).

The Twiesselmann excavation was exhaustive and was carried out mainly from wastes of the former excavations. Seaving provided little pieces which were not collected by the former excavators partly due to the employed method. This implies an artificial transfer of the results to small values for the samples from the Twiesselmann collection. The largest samples are also both the poorer ones in Fe-ooliths and richer ones in carbonates (cement and shell remains). This observation could be explained in terms of an anthropic behaviour of the time: a secondary selection of the "red rocks" on the Spy cave site with plans to throw away inappropriate material.

The good sorting, the small size of ooids and the lack of hematitised intraclasts exclude the Verviers area as geological and geographical sources of the raw material. Samples are very rich in ooids and the silty matrix is not very abundant; a calcitic cement occurs in some samples. On the other hand, geological samples collected from Landenne, Lavoir or Fraipont show big rounded ooids in strong contrast with OISC. In the Huy outcrops, three different layers of OI can be distinguished. The concentration of ooids, the abundance of the matrix and the fossil content vary vertically (and horizontally to a lesser extent) from one layer to another.

Taking all the descriptive criteria described above into account, a very good correspondence can be found between OISC and Lower Famennian OI from Sclaigneau (Vezin), Vedrin and Flône, and a good analogy with OI from Huy, Layable (Statte), Spy, Rhisnes, Suarlée (Les Isnes) and Gelbressée. All this material comes from the Namur Parautochton Geological Unit.

#### Magnetic susceptibility results

The MS signature of all samples ranges from  $1 \times 10^{-7}$  to  $7 \times 10^{-7}$  kg/m<sup>3</sup> (Figure 11).

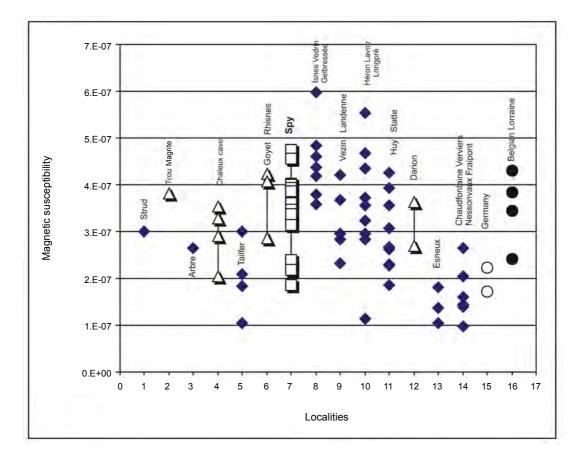


Figure 11. Magnetic susceptibility determined on oolithic ironstones from different localities. Triangles: archaeological stations of Trou Magrite (Walzin, Namur Prov., Middle/Upper Palaeolithic layer), Chaleux cave (Hulsonniaux, Namur Prov., Palaeolithic, Magdalenian layer), Goyet cave (Mozet, Namur Prov., Palaeolithic), and Darion (Geer, Liège Prov., Early Neolithic); squares: Spy cave (Jemeppe-sur-Sambre, Namur Prov., Palaeolithic); diamonds: Devonian Belgian geological outcrops; black circles: Jurassic geological sections; white circles: Devonian German geological sections. Higher values come from samples richer in hematite, and sites with a great number of samples show a large dispersion of values. Frasnian ironstones have lower values than both Lower Famennian and Bajocian-Aalenian ironstones. On the sole basis of MS, it is impossible to distinguish Lower Famennian and Bajocian-Aalenian ironstones while it is very easy by mesoscopic observation. They differ by colours, sizes, morphology of ooids, clay mineralogy and fossil content.

The lowest values (between 1 and 2.8 x  $10^{-7}$  kg/m<sup>3</sup>) match with Lower Famennian ironstones from Eastern Belgium, Germany and Frasnian ironstones. Samples from archaeological sites show values between 2 and 5 x  $10^{-7}$  kg/m<sup>3</sup>.

Archaeological samples from Spy cave can be compared with geological samples. MS values are between 1.85 and 4.56 x  $10^{-7}$  kg/m<sup>3</sup>. Macroscopically, the lowest values (< 2.5 x  $10^{-7}$  kg/m<sup>3</sup>) can be linked to impure OI. We can show that they are more compatible with the Lower Famennian ironstones from the two flanks of the Namur Parautochton structural unit. Geographically, this matches with a geographic area located east of Spy (Western Hesbaye area) from Les Isnes to Huy in the Meuse Valley through the Vedrin, Gelbressée, Vezin, Landenne, and Héron villages. However, we cannot exclude a south-western origin in the Tailfer area (Profondeville, Dinant Basin) for archaeological samples with lower MS values.

Three samples (two from Spy cave and one from Darion) show abnormally high MS values between 3 and 4 x  $10^{-6}$  kg/m<sup>3</sup>. One sample is clearly identified as coming from the level "le foyer" (the hearth) but the two others have no particular additional information. These three abnormal values are interpreted as heated material (voluntary or not). Heating has the property to modify both the crystallinity and the mineralogy, with a direct influence on MS measurements. These samples have not been plotted on Figure 11. Heating experimentation (around 450°C) performed on Lower Fammenian OI samples indicates the same increasing rate of the MS values.

We think however that a geochemical analysis should be performed in order to identify the signature of each geological layer and to confront the archaeological samples with it. For the time being, there are benchmark data neither for the geological samples, nor for the archaeological parts.

#### CONCLUSIONS

At first it is important to notice that hematite is not naturally present in the close vicinity of the cave as the Visean limestone substratum never contains it. As such, hematite must have been imported.

The hematite fragments which were discovered at Spy are often characterised by a lack of archaeological context. We can only record that the oolithic hematite seems to be the main component of the ochre observed on certain – often Aurignacian – archaeological pieces, and at the origin of the pinkish red-brown colour of the sediment and the breccia of the "second fauna-bearing level". Further sediment analyses should prove it.

With rare exceptions, the hematite fragments wear no anthropic traces like scrubbing or scraping striations. Then a crushing action can be imagined from which the smallest fragments may be the waste.

Oolithic ironstones from the Spy cave show great compositional and textural homogeneities. They represent the best level quality of available material and have a high content of hematite. This quality requires a prerequisite sorting of the raw material extracted from the geological outcrops.

OISC artefacts present a particular affinity with OI of the Lower Famennian from the Namur Synclinal, which places the possible sources north of the Meuse, east of Spy. On a first approach, one can exclude oolithic Famennian ironstones coming both from the east of Belgium and Germany (geological continuity of Eastern Belgium ores – Dinant and Vesdre Synclinoria). Close geological sources are thus to support. Among the best candidates, the site of Huy (south border of the Namur Syncline), located on a stiff slope along the Meuse, seems a good one because of its freshness reactivated by the erosive processes. The sites of the Hesbaye plate (north border of the Namur Parautochton), covered with loess deposits, were to be accessible only in the valleys of the small Meuse tributaries.

The oolithic hematite levels having been strongly exploited by mines (north border of the Namur Syncline) during the last centuries to feed metallurgy, it seems difficult to still hope to find traces of an old iron ore mining relating to the Prehistoric times. Today, only small impure, wild wooded ore heaps, easily identifiable in a flat, extensively cultivated landscape, persist.

On the sole basis of the determination of the MS, the method is not sufficiently discrimin-

atory to obtain a precise localisation because of the too strong dispersion of measurements in geological site as well as in archaeological context. Other magnetic parameters should be measured.

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#### Lexicon

- Hematite (or Haematite): common iron mineral of formula Fe<sub>2</sub>O<sub>3</sub>. It occurs in metallic-look, steel-grey or iron-black crystals, in kidney-shaped masses or fibrous aggregates, or in deep red or red-brown earthy forms. Hematite has a distinctive cherry red to reddish brown streak and a characteristic brick-red colour when powdered. Red ochre and oligist are synonymous.
- Oolith (also spelled oölith): a spherical to ellipsoidal body, 0.25 to 2.0 mm in diameter, which may or may not have a nucleus, and has a concentric or radial structure, or both. It is usually calcareous, but may be siliceous, hematitic, or of other composition (American Geological Institute, 1976). Oolite is a sedimentary rock, usually a limestone, made up mainly of ooliths cemented together (Jackson, 1997). An ooid is an individual spherulite of an oolithic rock. The term has been used in preference to "oolith" to avoid confusion with oolite (Jackson, 1997).
- "Ooid" is also used as a general, non-generic term for a particle that resembles an oolith in outer appearance and size.
- Allochem: collective term for one of several varieties of discrete and organised carbonate aggregate that serve as the coarser framework grains in most mechanically deposited limestones, as distinguished from sparry calcite (usually cement) and carbonate-mud matrix (micrite). Important allochems include: silt-, sand-, and gravel-size intraclasts; ooids, pellets and fossils or fossil fragments (Jackson, 1997).
- Intraclast: fragment of a penecontemporaneous reworked limestone and re-deposited to form new sediment.

#### BIBLIOGRAPHY

- ADAMS A. E., MCKENZIE W. S. & GUILFORD C., 1984. Atlas of sedimentary rocks under the microscope, 1<sup>st</sup> edition. Harlow, British Library Cataloguing in Publication Data, John Wiley & Sons, Inc.: 108 p.
- AMERICAN GEOLOGICAL INSTITUTE, 1976. *Dictionary of geological terms*, Revised edition. Anchor Books: 472 p.
- ANCION C., DEMANET F. & VAN LECKWIJCK W., 1956. Découverte d'une faune strunienne au toit de la couche d'oligiste oolithique dite famennienne de Couthuin (bord nord du Synclinal de Namur). Académie royale de Belgique, Bulletin de la Classe des Sciences, 5 (XLII): 506-514.
- BARHAM L. S., 2002. Systematic pigment use in the Middle Pleistocene of south central Africa. *Current Anthropology*, **31** (1): 181-190.
- BAR-YOSEF MAYER D. E., VANDERMEERSCH B. & BAR-YOSEF O., 2009. Shells and ochre in Middle Paleolithic Qafzeh Cave, Israel: indications for modern behavior. *Journal of Human Evolution*, 56 (3): 307-314.
- DELMER A., 1913. La question du minerai de fer en Belgique. Annales des Mines de Belgique, XVII (4/1912), XVII (2/1913): 180 p. + 100 p.
- DE LOË A. & RAHIR E., 1911. Nouvelles fouilles à Spy. Grotte de la Betche-aux-Rotches. *Bulletin de la Société d'Anthropologie de Bruxelles*, **30**: 40-58.
- DE PUYDT M., 1903. Fonds de cabanes néolithiques de la Hesbaye. Le village des Tombes. Fouilles exécutées à Omal par E. Davin-Rigot et M. De Puydt, en 1900-1901. Bulletin de la Société d'Anthropologie de Bruxelles, XXI (Mémoires I): 23 p.
- DE PUYDT M. & LOHEST M., 1887. L'homme contemporain du Mammouth à Spy (Namur). Annales de la Fédération archéologique et historique de Belgique, Compte rendu des travaux du Congrès tenu à Namur les 17-19 août 1886, **2**: 207-240, 10 pl. h.t.
- D'ERRICO F., HENSHILWOOD C. & NILSSEN P., 2001. An engraved bone fragment from c. 70,000year-old Middle Stone Age levels at Blombos Cave, South Africa: implications for the origin of symbolism and language. *Antiquity*, **75** (288): 309-318.

DREESEN R., 1982. Storm-generated oolitic iron-

stones of the Famennian (Fa1b-Fa2a) in the Vesdre and Dinant synclinoria (Upper Devonian, Belgium). *Annales de la Société géologique de Belgique*, **105**: 105-129.

- DREESEN R., 1984. Stratigraphic correlations of Famennian oolitic ironstones in the Havelange (Dinant Basin) and Verviers boreholes (Vesdre Massif) (Upper Devonian, Belgium). Bulletin de la Société belge de Géologie, 93: 197-211.
- FRAIPONT J. & LOHEST M., 1887. La race humaine de Néanderthal ou de Canstadt en Belgique. Recherches ethnologiques sur des ossements humains, découverts dans des dépôts quaternaires d'une grotte à Spy et détermination de leur âge géologique. Archives de Biologie, 7/1886: 587-757.
- JACKSON J. A., 1997. Glossary of Geology, 4th edition. Alexandria VA, American Geological Institute: 768 p.
- LAENEN B., DREESEN R. & ROELANDTS I., 2002. Sequence-stratigraphic significance and comparative REE-fractionation patterns of Rupelian glaucony concentrates and Famennian oolitic ironstones. *In*: P. DEGRYSE & M. SINTUBIN (ed.), *Contributions to the Geology of Belgium and Northwest Europe. Proceeds. Ist Geologica Belgica International meeting*. Aardkundige Mededelingen, **12**: 51-54.
- MARION J.-M. & BARCHY L., 2001. Momignies-Séloignes. Carte géologique de Wallonie, échelle 1/25.000. Carte et notice explicative. Namur, Ministère de la région wallonne, DGRNE Edit: 75 p.
- MOONEY S. D., GEISS C. & SMITH M. A., 2003. The use of mineral magnetic parameters to characterize archaeological ochres. *Journal of Archaeological Science*, **30** (5): 511-523.
- OTTE M., 1979. Le Paléolithique supérieur ancien en Belgique. Bruxelles, Musées royaux d'Art et d'Histoire, Monographies d'archéologie nationale, 5: 684 p.
- POMIÈS M. P. & MENU M., 1999. Red Palaeolithic pigments: natural hematite or heated goethite? *Archaeometry*, 41 (2): 275-285.
- SCHILD R., HALINA KROLIK H. & TOMAS-ZEWSKI J., 1997. A raw material economy of the Palaeolithic and Mesolithic occupants of the Rydno complex. *In*: R. SCHILD & Z. SULGOSTOWSKA, *Man and Flint*. Papers from

the VIIth International Flint Symposium, Warszawa - Ostrowieć Swiętokrzyski, September 1995. Warsaw, Institute of Archaeology and Ethnology, Polish Academy of Sciences: 285-293.

- SEMAL P., JUNGELS C., CREVECOEUR I., ROUGIER H. & PIRSON P., 2009. Acquisition de la collection de Spy de François Beaufays (dit "l'Horloger") par l'Institut Royal des Sciences Naturelles de Belgique. *Notae Praehistoricae*, **29**: 157-164.
- SEMAL P., TWIESSELMANN F., HAUZEUR A. & DEWEZ M., 1996. Étude d'une face humaine découverte dans les niveaux "gravettiens" des dépôts de pente de la grotte de Spy. Fouilles de F. Twiesselmann (1950-1954). Notae Praehistoricae, 16: 171-181.
- THISSE-DEROUETTE J. & TOMBALLE L., 1957. Fouilles partielles d'une agglomération omalienne à Noville-en-Hesbaye. Bulletin de l'Institut Archéologique Liégeois, LXXI: 149-208.
- TRĄBSKA J., WALANUS A., CIESIELCZUK J., SAMEK L. & DUTKIEWICZ E., 2008. Ferruginous raw material sources for Palaeolithic in Poland (Central Europe) - Provenance studies: occurrence, lithostratigraphy and application. In: 9th International Conference on NDT of Art, Jerusalem Israel, 25-30 May 2008: 8 p.
- VANDERMEERSCH B., 1969. Découverte d'un objet en ocre avec traces d'utilisation dans le Moustérien de Qafzeh (Israël). Bulletin de la Société préhistorique française, **66** (5): 157-158.
- WATTS I., 2002. Ochre in the Middle Stone Age of Southern Africa: ritualised display or hide preservative? South African Archaeological Bulletin, 57: 1-14.
- ZILHÃO J., ANGELUCCI D. E., BADAL-GARCÍA E., D'ERRICO F., DANIEL F., DAYET L., DOUKA K., HIGHAM T. F. G., MARTÍNEZ-SÁNCHEZ M. J., MONTES-BERNÁRDEZ R., MURCIA-MASCARÓS S., PÉREZ-SIRVENT C., ROLDÁN-GARCÍA C., VANHAEREN M., VILLAVERDE V., WOOD R. & ZAPATA J., 2010. Symbolic use of marine shells and mineral pigments by Iberian Neandertals. *Proceedings of the National Academy of Sciences USA*, **107** (3): 1023-1028.

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