RESEARCH ARTICLE – VÝZKUMNÝ ČLÁNEK

Social networks around the Orońsko flint mining area (Central-Southern Poland) during the Late Palaeolithic: The first results of the SEM-EDS analysis of flints and pigments

Sociální sítě v okolí těžebního areálu Orońsko (středojižní Polsko) v pozdním paleolitu: První výsledky SEM-EDS analýzy silicitů a pigmentů

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*Orońsko site 2 is a Palaeolithic shaft mine located in the area of chocolate flint deposits in Central-Southern Poland. This raw material was highly valued by Stone Age communities and widely distributed during the Late Palaeolithic. The mine is associated with the Arch-Backed Pieces (*Federmesser*) and Tanged Point Technocomplexes. This article aims to explore the connections between artefacts made of chocolate flint found at various Late Palaeolithic sites located outside the chocolate flint deposits (*Całowanie and ochre quarry at Rydno*) with the Orońsko mine and to investigate a possible connection between the ochre found in the Orońsko mining shaft with the Rydno quarry. Macroscopic and SEM-EDS analyses were conducted on selected samples of these raw materials and artefacts from the Orońsko mine, the Orońsko workshop, Rydno, and Całowanie, all of which can be chronologically linked to one another. The results suggest that some of the flint may have been transported from the Orońsko mining area to Rydno or Całowanie. However, the reverse transport of ochre from Rydno to Orońsko remains unclear and requires further research, including a broader range of analytical methods.*

chocolate flint – pigment – ochre – mine – Palaeolithic – Orońsko – SEM-EDS

*Orońsko 2 je paleolitický šachtový důl, který se nachází v oblasti nalezišť čokoládového silicitu ve středojižním Polsku. Tato surovina byla komunitami doby kamenné vysoce ceněna a v pozdním paleolitu široce rozšířena. Dolování je spojeno s technokomplexy s obloukovitě retušovanými hroty (*Federmesser*) a řapovými hroty. Cílem tohoto článku je prozkoumat souvislosti mezi artefakty vyrobenými z čokoládového silicitu nalezenými na různých pozdně paleolitických lokalitách mimo naleziště čokoládového křemene (*Całowanie a okrový lom u Rydna*) s dolem v Orońsku a prozkoumat možnou souvislost mezi okrem nalezeným v Orońské těžební šachtě s lomem v Rydně. Byly provedeny makroskopické a SEM-EDS analýzy vybraných vzorků těchto surovin a artefaktů z dolu Orońsko, Orońské dílny, Rydna a Całowanie, které spolu mohou chronologicky souviset. Výsledky naznačují, že část křemene mohla být transportována z oblasti dolu Orońsko do Rydna nebo Całowanie. Reverzní transport okru z Rydna do Orońska však zůstává nejasný a vyžaduje další výzkum, včetně širšího spektra analytických metod.*

čokoládový silicit – pigment – okr – důl – paleolit – Orońsko – SEM-DES

Introduction

The detailed characterisation and diversification of siliceous rocks, as well as other materials such as pigments, are key issues for understanding the economy, mobility, and interactions of Stone Age communities. To effectively link the origins of the artefacts or pigments to

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their extraction points, particular macroscopic and microscopic observations along with chemical analyses are essential. By utilising these advanced techniques, we can create a strong framework connecting distinct settlement areas, enhancing our understanding of Palaeolithic social networks and cultural exchanges. This, in turn, provides insight into the complexities of prehistoric human interactions. In recent years, efforts have been made to characterise some types of flint, including chocolate flint and pigments like ochre. These studies have yielded interesting and valuable results (see *Weinstein-Evron – Ilani 1994*; *Hughes et al. 2010*; *Hughes et al. 2011*; *Högberg et al. 2012*; *Přichystal 2013*; *Roldan et al. 2015*; *Werra et al. 2015*; *Brandl et al. 2016*; *Sobkowiak-Tabaka et al. 2016*; *Fiers et al. 2019*; *Velliky et al. 2021*; *Werra – Siuda 2022*; *Mandera et al. 2024*).

The term 'flint' is a commonly used name in Polish archaeology for describing siliceous rocks, such as cherts/silicites, including chocolate flint, Świeciechów flint, striped flint, and others (see, for instance, *Přichystal 2013*; *Matyszkiewicz-Kochman 2020*). As previously noted in the literature, chocolate flint was a highly significant raw material during the Palaeolithic, particularly for Late Palaeolithic communities. It was distributed over distances of several hundred kilometres during this period (*Sulgostowska 2005*; *2008*). Its deposits were first described in the 1920s by S. Krukowski and J. Samsonowicz (*Krukowski 1922*; *Samsonowicz 1923*). The term 'chocolate flint' was originally referred to as 'waxy-chocolate' by *S. Krukowski* (*1922*; *1923*), due to its brownish colour and waxy lustre. The first attempt to characterise and classify varieties of chocolate flint was made by R. Schild, based on samples collected during surface surveys focused on mining sites (*Schild 1971*; *1976*). Schild categorised the raw material into 11 groups, each with further subgroups, based on macroscopic characteristics supplemented by features observable under an optical microscope. In recent years, D. H. Werra and her team have continued this research. They analysed samples of chocolate flint obtained from mining fields and natural exposures, employing various specialised methods in addition to macroscopic characteristics. Studies have included descriptions of micropaleontology through organic compound analysis (*Grafka et al. 2015*) and geochemical techniques such as laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) (*Brandl et al. 2016*), X-ray fluorescence spectrometry (EDXRF) (*Hughes et al. 2016*), reflectance spectroscopy (VNIR – Visible Near Infrared), and Fourier Transform Infrared spectroscopy (FTIR) (*Parish – Werra 2018*). New research is also being conducted in the Krakow-Częstochowa Upland, where exploitation of chocolate flint has been confirmed, along with initial analyses of flint samples (*Mandera et al. 2024*). These findings have significantly advanced the methods for characterising flints and have enhanced our understanding of the distribution of chocolate flint.

The ochre mine located in Rydno was discovered by S. Krukowski and later excavated by various research teams, primarily under the direction of R. Schild. The name 'Rydno' was also given by S. Krukowski for the complex of Late Palaeolithic and Mesolithic sites located in the Kamienna River valley, clustered around the ochre outcrops. Rydno is located near the belt of chocolate flint deposits, about 25 km south of Orońsko (*Fig. 1*).

While the ochre quarry has not yet been the focus of extensive research, some analyses have already been conducted on this material (e.g. *Schild et al. 2011*). The previous research showed that the material in question is composed mostly of iron (30.73–70.12 wt%), which is also responsible for its shade. Moreover, further elements were noted, such as silica (8.00–27.00 wt%) and aluminium (1.70–4.40 wt%), followed by magnesium, titanium and calcium with levels below 2.00 wt% (*Schild et al. 1981*; *Hensel 2011*, 406). It was assessed

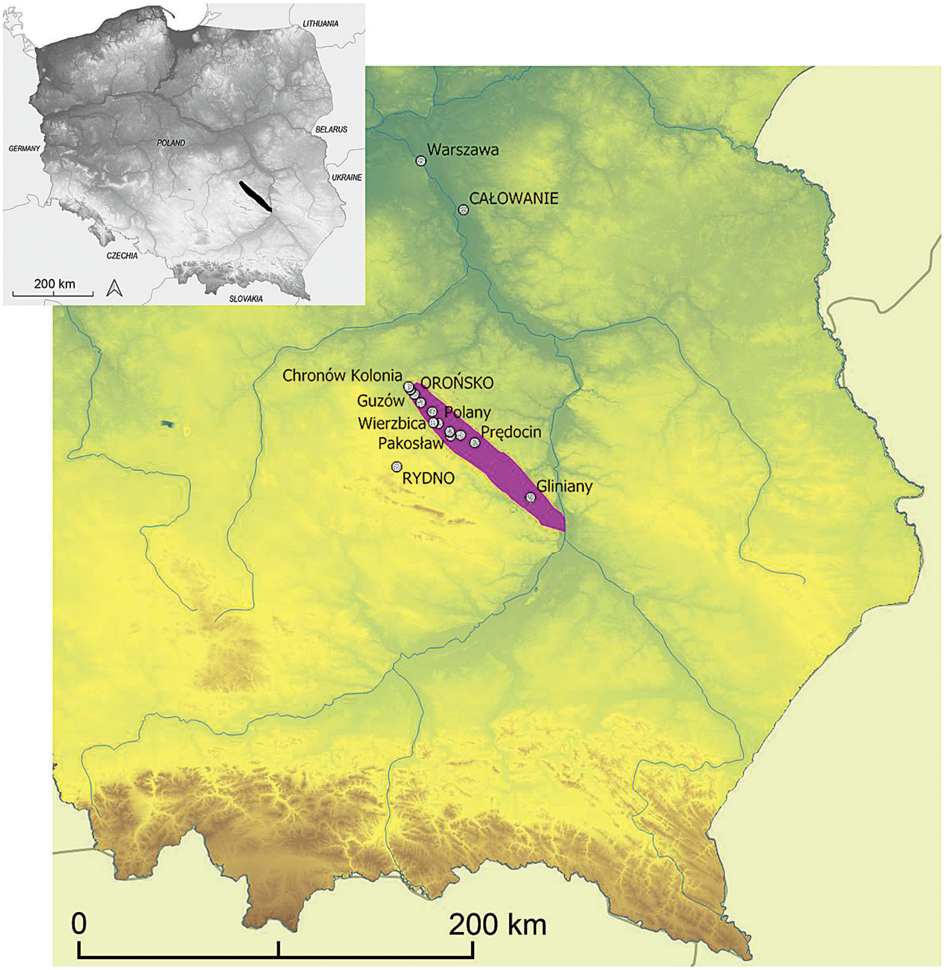


Fig. 1. Location of Orońsko site complex and the chocolate flint outcrops with the main exploitation points and the sites mentioned in the text (by K. Kerneder-Gubała, S. Buławka, and N. Buławka).

that the iron-bearing materials were mostly hematite, occurring in the form of gravels, and hematite-bearing conglomerates, such as sands and clays (*Schild et al. 2011*, 54–58).

Additional research has been performed on ochre samples from Orońsko mining shaft no. 2 (see *Osipowicz et al. 2019*). Given the proximity of both sites and the prominence of the Rydno complex, a hypothesis has been formed regarding the possible transportation of ochre to Orońsko. This article aims to present the preliminary results of comparative analyses of chocolate flint from the mine alongside artefacts from distant workshops, all dated to the same period. The second objective is to analyse and compare pigments from Orońsko with local clay variants and samples derived from Rydno. The research includes both macroscopic and laboratory analyses, specifically using SEM-EDS. Through meticulous work, this study aims to address the reconstruction of social networks during the Palaeolithic in the microregion around Orońsko, explore the possibilities and limitations

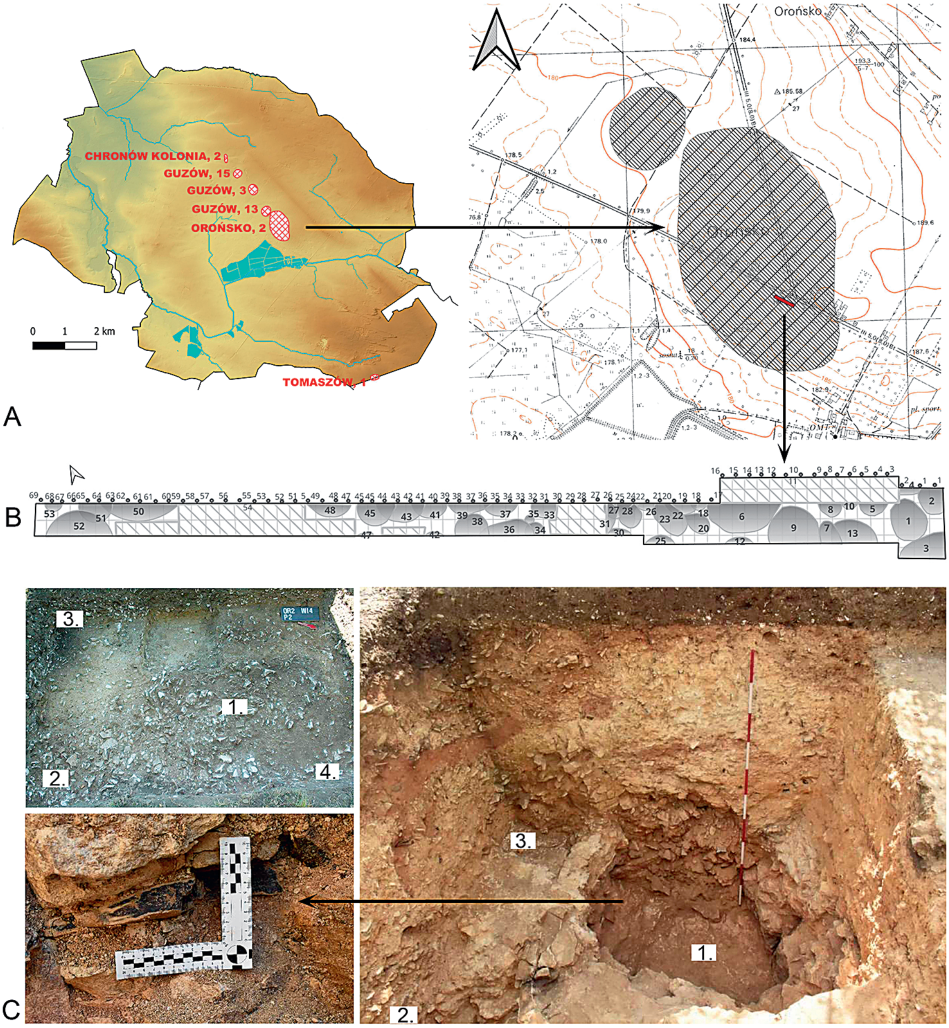


Fig. 2. Location of the trench I4/2016–2024 at site 2 in Orońsko (A, B) and the mining shafts with flint-bearing layers (C; by K. Kerneder-Gubała, S. Buławka, and N. Buławka).

of using SEM-EDS for analysing flint and pigment samples, and propose future directions for research.

Materials and methods

Orońsko is located in the northwestern part of the chocolate flint outcrops, in the Oronka River valley. Chocolate flint deposits are situated along the Upper Jurassic belt, which separates the Kielce-Sandomierz Upland from the Masovian Lowland at the northern edge of the Holy Cross Mountains (*Fig. 1*; *Schild 1971*, 19–38; *Schild et al. 1977*; *1985*, 13–16;

Budziszewski 2008). S. Krukowski and J. Samsonowicz identified most of the known chocolate flint outcrops. Research in this area began in the 1920s with a surface survey around the Orońsko 2 site and the adjacent area (*Krukowski 1922*; *1923*; *1939–1948*). In 1935, as a result of excavations, S. Krukowski discovered the remains of mining shafts sunk into flint-bearing layers of residual clays at the Orońsko 2 site (*Krukowski 1939–48*; *Kozłowski 2008*). Further research in the region was conducted by Romuald Schild and his team (*Schild 1971*), who performed extensive surface surveys and excavations at neighbouring Neolithic and Bronze Age mining sites such as Tomaszów and Polany Kolonie (*Schild 1995a*; *1995b*; *Schild et al. 1977*; *1985*). During this time, multiple samples of flint from sites near Orońsko (*Schild 1971*) were also collected and subjected to an initial study. Extensive excavations were conducted at other sites located in the chocolate flint outcrops, such as Wierzbica Zele and Polany II, both of which were located in the mining fields (*Lech – Lech 1984*; *Chmielewska 1988*; *Herbich – Lech 1995*; *Lech – Werra 2019*). The entire area was later investigated by Janusz Budziszewski and other researchers, though mainly through surface surveys (*Budziszewski 2008*).

Pigments have been identified in archaeological layers at Orońsko particularly in the form of loose powders found in the sediments, on bone tools, and on flint artefacts excavated in the fills of mining shafts. Notably, these pigments were primarily reddish to pinkish in hue. In the immediate vicinity of the site, accumulations of ferruginous clays with yellowish to dark orange hues were observed. Additionally, ochre mining pits and artefacts covered with ochre were noted in the Rydno complex (see *Schild et al. 2011*). For the research presented here, samples of loose-pigmented materials were collected and analysed for comparison.

The Orońsko chocolate flint mine, trench I4/2016–2024

The latest excavation campaign at the Orońsko 2 site conducted since 2016 has led to the discovery of new mining shafts (*Kerneder-Gubała 2019*; *Osipowicz et al. 2019*; *Werra – Kerneder-Gubała 2021*). Dozens of new mining shafts were uncovered in trench I4. These shafts were found in a layer of weathered rock as well as at the level of the limestone bedrock, where flint-bearing layers were identified (*Fig. 2*). Both the archaeological artefacts and radiocarbon dating confirmed that the shafts were used in the Late Palaeolithic period, specifically the late Allerød and Younger Dryas periods (GI-1a-GS1, *Kerneder-Gubała 2019*; *Osipowicz et al. 2019*; *Werra – Kerneder-Gubała 2021*). Flint artefacts from the mining shafts discovered by S. Krukowski were also assigned to the same period. These artefacts exhibit features characteristic of the Arch-Backed Pieces and Tanged Point Technocomplexes (comp. *Krukowski 1939–48*; *Schild 1971*; *Kerneder-Gubała 2019*).

Late Palaeolithic workshops outside the chocolate flint outcrop

In our study of exchange networks, we also focused on sites that are distant from Orońsko. We analysed flint samples from Calowanie, located approximately 80 km to the north, and flint and ochre samples from the Rydno site complex, situated about 25 km to the south of Orońsko (see *Fig. 1*). Rydno is a complex of Late Palaeolithic and Mesolithic sites that served as flint workshops, camps, and an ochre mine (*Schild et al. 2011*). In contrast, Całowanie is a multicultural site with layers dated from the Late Pleistocene to the Early

CHOCOLATE FLINT SAMPLES												
No	Site	Site function	Trench	Chronology	Colour (Munsell scale)	Lustre	Transparency (1-3)	Smudges/ streaks	Stains	Cortex – colour	Cortex – form	Romuald Shild's classification number
FLINT 1	Orońsko 2	flint mine	I4/shaft no. 1	Late Palaeolithic / OTP	10 YR 3/2 very dark greyish brown	waxy, matt	2	+	-	10YR 8/2-8/1 white	irregular, rough, very thick	VIb
FLINT 2	Orońsko 2	flint mine	I4/shaft no. 1	Late Palaeolithic / OTP	5YR very dark grey	waxy	1	-	+	10YR 8/2-8/1 white	irregular, rough, thick	IX / VI
FLINT 3	Orońsko 2	flint mine	surface	Late Palaeolithic	10 YR 3/2 very dark greyish brown	waxy	1	+	+	10YR 6/4 light yellowish brown	little irregular	VIIa
FLINT 4	Orońsko 2	flint mine	I4/shaft no. 1	Late Palaeolithic / OTP	5YR very dark grey, 2/1 dark reddish brown	waxy	1	-	+	10YR 8/2-8/1 white	irregular, rough, thick	IX / VI
FLINT 5	Orońsko 2	flint mine	I4/shaft no. 1	Late Palaeolithic / OTP	5YR very dark grey, 2/1 dark reddish brown	waxy	1	-	+	10YR 8/2-8/1 white	irregular, rough, thick	IX / VI
FLINT 6	Orońsko 1	workshop	Surface	Late Palaeolithic	dark grey to light grey – 4/1 to 6/1	waxy	1	+	-	5YR 7/6 reddish yellow	irregular, rough, thick	VIId
FLINT 7	Całowanie	workshop, camp	trench IX, level IVb	Late Palaeolithic, ABP	5YR 4/4 REDDISH BROWN	waxy	1	+	-	10 YR 8/2 – 8/1 white	smooth, thick, irregular	VI
FLINT 8	Rydno	workshop, camp	trench X/1959	Late Palaeolithic, ABP	dark grey to light grey – 4/1 to 6/1	waxy	1	+	+		little irregular	VIId
FLINT 9	Rydno	workshop, camp	trench IV/1960	Late Palaeolithic, OTP	dark grey to light grey – 4/1 to 6/1	waxy	1	+	+	5YR 7/6 reddish yellow	smooth, thick, irregular	VIId
FLINT 10	Rydno	workshop, camp	trench IX/1959	Late Palaeolithic, OTP	dark grey to light grey – 4/1 to 6/1	waxy	1	+	+		little irregular	VIId

A

OCHRE SAMPLES				
No	Site	Trench	Object	Sample description
OCHRE 1	Orońsko 2	I4	6	sediment
OCHRE 2	Rydno	RK-77	-	sediment – sand
OCHRE 3	Rydno	RK-77	-	sediment – gravel
OCHRE 4	Orońsko 2	I4	1	flint flake
OCHRE 5	Orońsko 2	I4	1	bone tool
OCHRE 6	Orońsko 2	I4	6	limestone
OCHRE 7	Orońsko 2	I4	1	sediment
OCHRE 8	Orońsko 2	I4	6	sediment
OCHRE 9	Rydno	RK-77	-	sediment – gravel

B

Tab. 1. List of samples: A – flint sample macroscopic characteristic, B – list of ochre samples.

Holocene, situated a few dozen kilometres from the chocolate flint outcrop (*Schild 2014*). Some Late Palaeolithic assemblages from this site were crafted from mined chocolate flint. Based on macroscopic features, it was suggested that some of these artefacts could have originated from the chocolate flint found in Orońsko or, more broadly, from the western group of chocolate flint outcrops (*Tomaszewski et al. 2008*; *Schild 2014*). Also, the initial results of the comparative chemical analysis of ochre from the Całowanie site and Rydno showed some similarities (*Hensel 2011*, 407).

All analysed flakes were excavated at Late Palaeolithic sites associated with the Older Tanged Point Technocomplex (OTP/Bromme culture) at Rydno X/1959 (sample FLINT 8), Rydno IV/1960 (sample FLINT 9) and the Arch-Backed Pieces Technocomplex (ABP) at Całowanie (trench IX, level IVb, sample FLINT 7) and Rydno X/1959 (sample FLINT 10). These sites correspond chronologically to the period in which the mine in Orońsko was in operation.

Methods and samples

Ten samples of chocolate flint and nine pigment samples were selected for microstructural and compositional analysis using a scanning electron microscope (SEM) with an energy-dispersive X-ray spectrometer (EDS) (see *Fig. 3*; *Tab. 1*). The chocolate flint group consists of both artefacts and natural nodules collected from flint-bearing levels of the mine and from the surface of the Orońsko area (samples FLINT 1–FLINT 6). Additionally, four samples (FLINT 7–FLINT 10) were obtained from flint workshops located outside the known regions of chocolate flint occurrence, specifically from Całowanie and the Rydno complex. Qualitative EDS analysis of the chocolate flint samples (FLINT 1–FLINT 10) was conducted using a Hitachi electron microscope (PGI-NRI, Warsaw). The analysis was performed on the rough surfaces of each sample under variable vacuum conditions. However, the FLINT 4 and FLINT 5 samples were cut, polished, and carbon-coated to obtain high-resolution SEM images and semiquantitative EDS analysis, which were performed using a ZEISS microscope combined with an Oxford EDS ISIS 300 detector (PGI-NRI, Warsaw). For comparative purposes, the samples were also described in detail through petrographic analysis. The SEM images and semiquantitative EDS analysis of the pigment samples (OCHRE 1–OCHRE 6) were collected using a TESCAN VEGA GMS scanning electron microscope, coupled with an Aztec Live Advanced UltimMAx 40 series EDS detector (PAS, Warsaw). The OCHRE 1 sample was collected from the sediment of shaft no. 6 in Orońsko, while OCHRE 2 and OCHRE 3 were sampled from sand and gravel in the Rydno area, respectively. These three samples were ground, homogenised, and compressed into tablet form. The OCHRE 4–OCHRE 6 group includes samples of distinctive red traces on the surfaces of various objects: a flint flake (OCHRE 4), a bone tool (OCHRE 5), and a piece of natural limestone slab from shaft no. 6 in Orońsko (OCHRE 6). The chemical composition of three additional samples (OCHRE 7–OCHRE 9) was analysed using a Tescan VEGA XM scanning electron microscope with an energy-dispersive X-ray spectrometer (JU, Cracow). The selected samples varied as follows: OCHRE 7 was obtained from feature 1 at the Orońsko 2 site and was orange in hue; OCHRE 8 was collected from feature 6 and was dark orange; OCHRE 9 was sampled from the Rydno complex, with storage number R-K/77, and was red in colour. These samples underwent pulverisation with carbon dust in a turbomolecular duster and were stabilised with carbon glue.

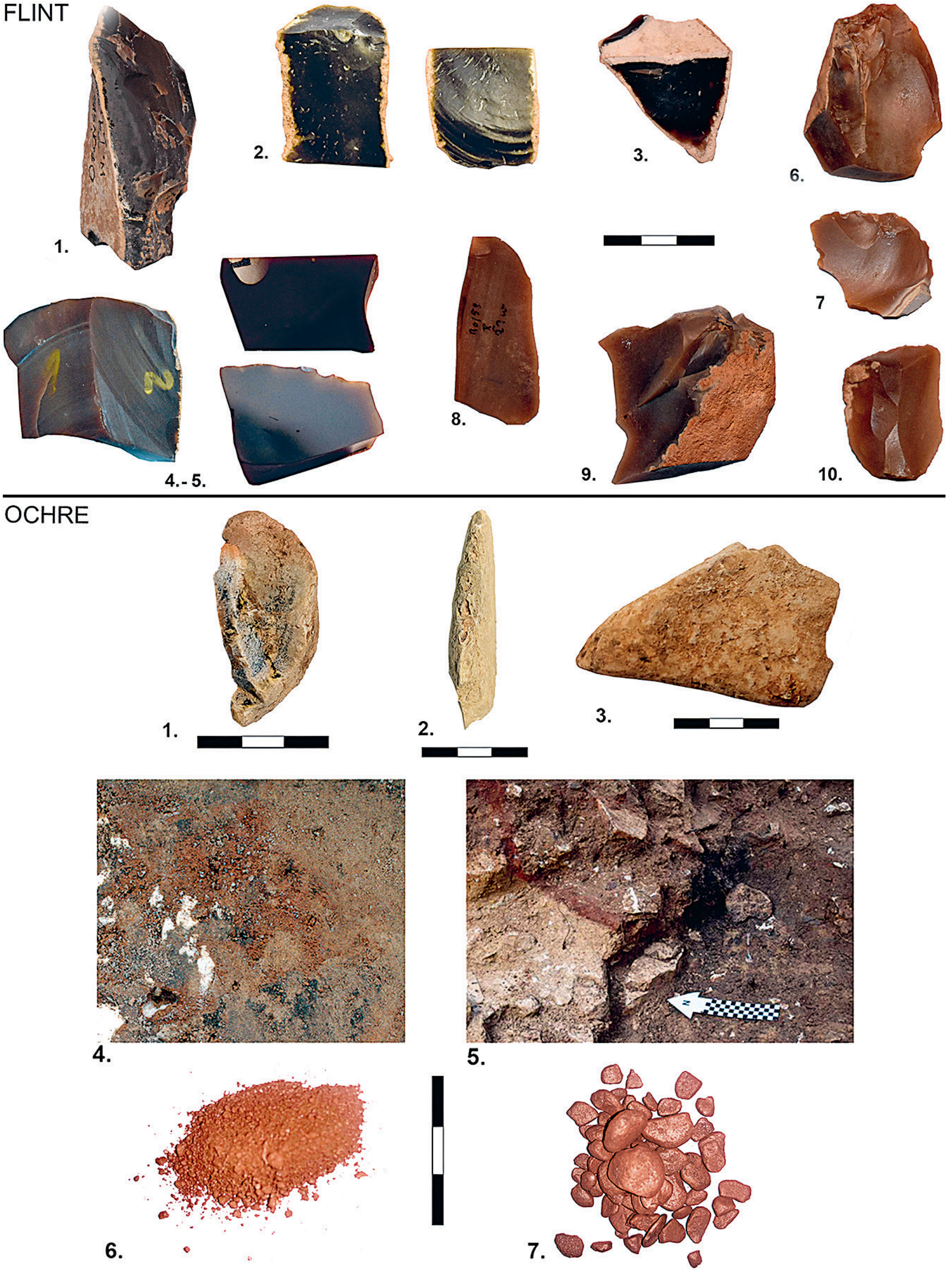


Fig. 3. Flint and ochre samples. FLINT: 1, 2, 4, 5 – Orońsko, site 2, shaft no. 1, flint sample; 3 – Orońsko, site 2, surface, artefact; 6 – Orońsko, site 1, workshop, artefact; 7 – Całowanie, tr. IX, level IVb, artefact; 8 – Rydno, tr. X/59, artefact; 9 – Rydno, tr. IV/1960, artefact; Rydno, tr. IX/1959. OCHRE: 1 – Orońsko, site 2, shaft no. 6, sediment; 2 – Rydno ochre mine, R-K/77, sand; Rydno ochre mine, R-K/77, gravel; 4 – Orońsko, site 2, shaft 1, flint artefact; 5 – Orońsko, site 2, shaft 1, bone tool, 6 – Orońsko, site 2, shaft 6, piece of limestone; 7 – Orońsko, site 2, shaft 1, sediment; 8 – Orońsko, shaft 6, sediment; 9 – Rydno, ochre mine, R-K/77, sediment (photo by authors).

Results

Characteristics of the flint deposits from Orońsko

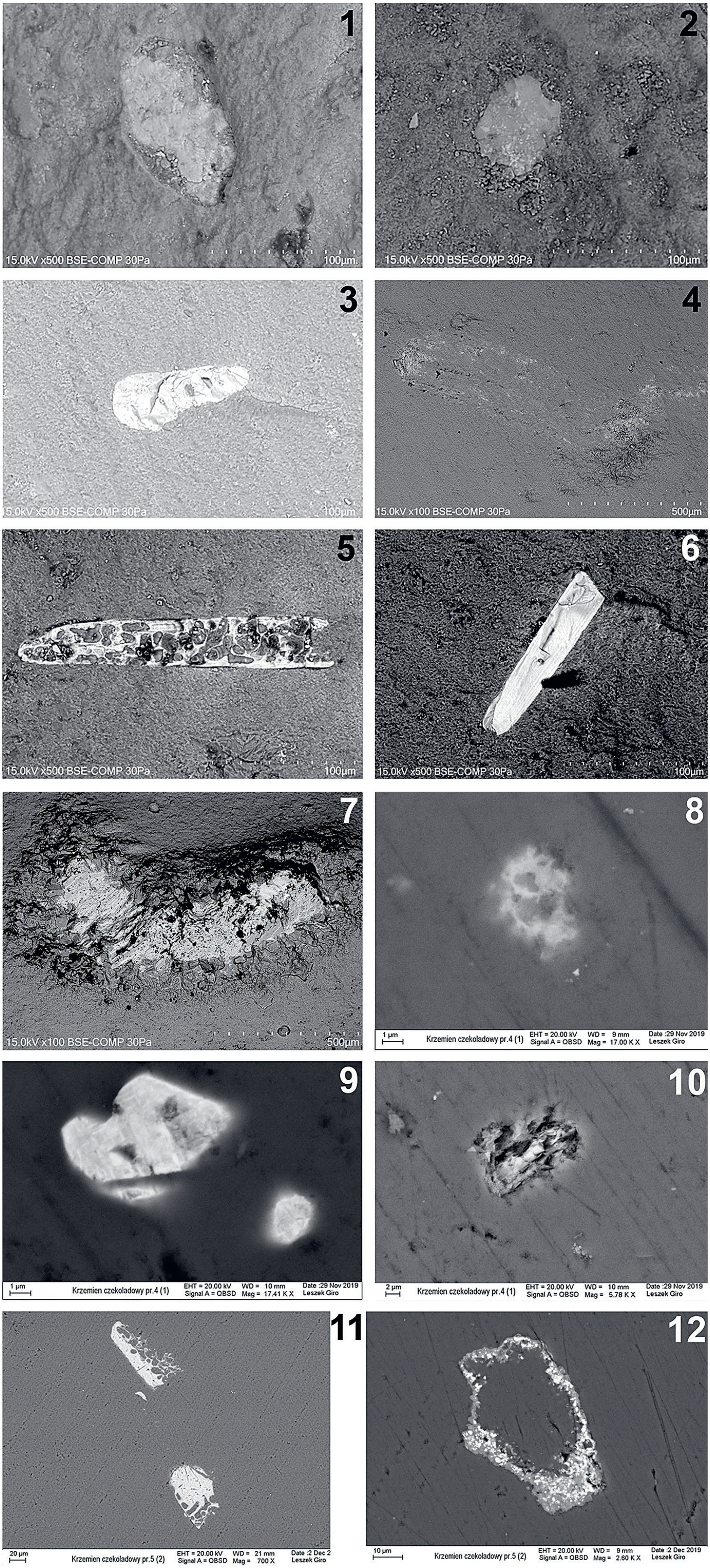
Chocolate flint primarily occurs in layers or nodules found in limestone or marly rocks from the Upper Oxfordian and Lower Kimmeridgian stages of the Upper Jurassic Belt, specifically in the Northern Margin of the Holy Cross Mountains (*Schild 1971*; *Gutowski 1998*; *Budziszewski 2008*) and in the Kraków-Częstochowa Upland (*Krajcarz et al. 2012*; *Sudoł-Procyk et al. 2018*; *Sudoł-Procyk 2022*; *Mandera et al. 2024*). These formations consist of flat, plate-like layers containing flint nodules. In the Orońsko area, flint can be found in layers of residual clays, and the use of mining shafts and pits has been confirmed thanks to the excavations conducted by S. Krukowski. These shafts were up to 3.4 metres deep but did not reach the limestone bedrock and thus the primary layers (*Krukowski 1939–1948*; *Schild 1971*). Recent studies indicate that chocolate flint was also obtained from primary deposits and a flint-bearing layer found in limestone in the Orońsko area. The differences between raw materials derived from primary and secondary deposits can be attributed to various weathering geological processes. These processes result in the degeneration of the cortex and a smoothing effect observed on the natural surfaces.

Petrography of chocolate flint

The variety of chocolate flint is extensive. The types of this sedimentary rock primarily composed of polycrystalline quartz have been classified into 11 categories, with subcategories based on macroscopic features. The light brown to grey-brown flint from Orońsko 2 corresponds to groups VIb, VIIa, VIIc, and VIId (*Schild 1971*, 7–17). A light beige layer beneath the cortex, along with delicate spots and streaks, has been observed (see macroscopic features of the flint samples presented in *Tab. 1*; *Fig. 3*). The samples from Orońsko 1 (located about 1.5 kilometres from the flint outcrop in Orońsko 2) and Rydno (samples FLINT 8 and 9) are noteworthy. According to *Schild* (*1971*), these refer to group VIId. The artefact from Rydno has a slightly more brownish hue (group VII). The samples from Orońsko and Rydno exhibit a range of greyish hues with varying degrees of saturation. Artefact FLINT 7 (group VIb) from the Całowanie site is associated with the raw material obtained from Orońsko and also shows a slightly reddish hue (5YR 4/4), likely interpreted as a result of soil acid interaction. Similar flint has been noted on the surface of the northern part of the mining field in Orońsko (R. Schild's and K. Kerneder-Gubała's observations). Based on macroscopic features, several preliminary conclusions can be drawn, partially confirming previous assumptions regarding the import of chocolate flint from the Orońsko region.

Observation of chocolate flint using a scanning electron microscope (SEM) and analysis of EDS spectra

The flint samples were imaged using scanning electron microscopy (SEM) and analysed for mineral composition (see *Fig. 4*). Several mineral phases were identified in the Orońsko flint. Barite was found in samples FLINT 1 and FLINT 6, while clusters of clay minerals were also present in these samples. Sample FLINT 2 contained calcite. Rutile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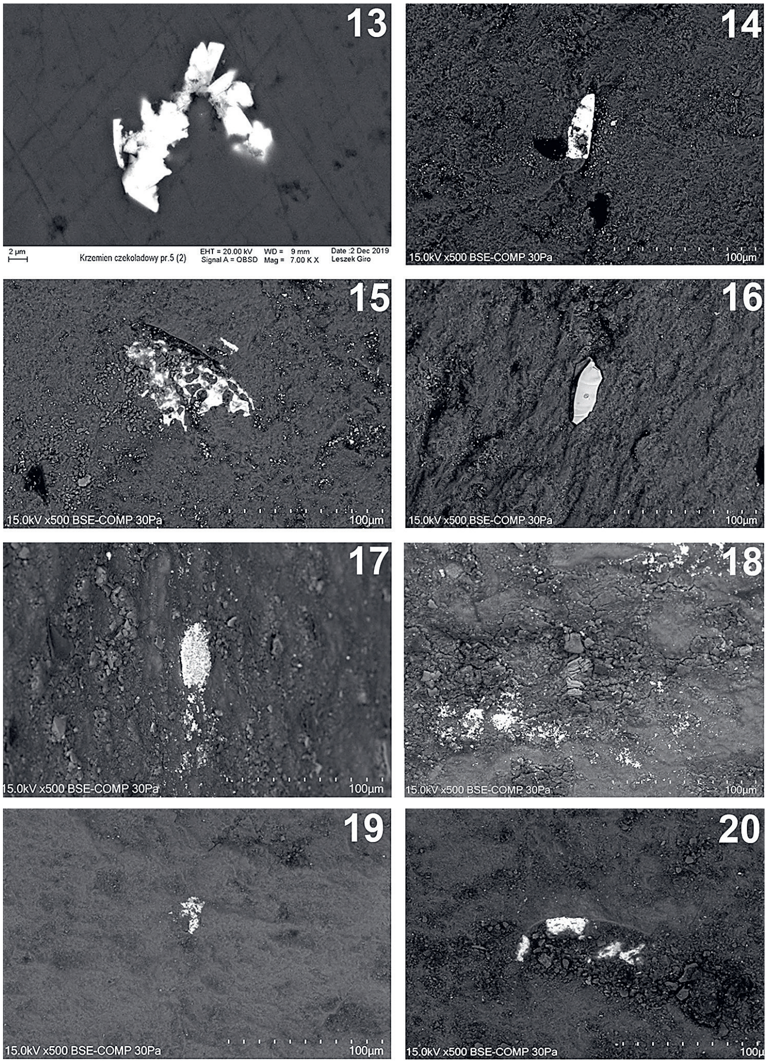


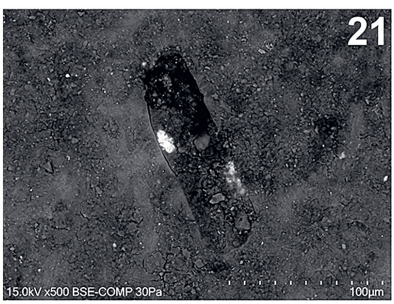


Fig. 4. Microphotography of selected samples. FLINT 1: 1 (apatite), 2 (bioapatite); FLINT 2: 3, 5 (apatite), 4 (calcite); FLINT 3: 6 (apatite), 7 (calcite with Mg); FLINT 4: 8 (apatite), 9 (Fe oxides), 10 (clay minerals); FLINT 5: 11 (apatite), 12 (Fe oxides), 13 (rutile); FLINT 7: 14, 15 (apatite with REE), 16 (potassium feldspar); FLINT 8: 17, 18 (barite); FLINT 9: 19, 20, 21 (apatite with REE) (photo by D. Gurba).

was identified in samples FLINT 3, FLINT 4, and FLINT 5; iron oxides were found in samples FLINT 2, FLINT 4, FLINT 5, and FLINT 6. Additionally, bioapatite was detected as tiny inclusions in all samples from the mine (primary layer).

The polished samples, FLINT 4 and FLINT 5, were examined in different orientations: FLINT 4 was cut parallel to the microlayer and is partially foliated, showing inclusions of clay minerals, iron oxides (in aggregates), rutile, and apatite. The Back Scattered Electron (BSE) image of sample FLINT 5 revealed silica microveins, indicative of quartz recrystallisation. This sample also contained bioapatite, very fine iron oxides or iron hydroxides, rutile, and aggregates of iron and rutile oxides. The samples from the OTP workshop in the Rydno complex (FLINT 8 and FLINT 9) exhibited inclusions of aluminosilicates, iron oxides, and clay minerals. In the sample from the ABP workshop (Rydno X / 1959, No. FLINT 10), rutile and barite were documented. Feldspar was identified in sample FLINT 7 from the Całowanie site (ABP).

Characteristics of pigments from Orońsko and Rydno

The deposits of Rydno quarry

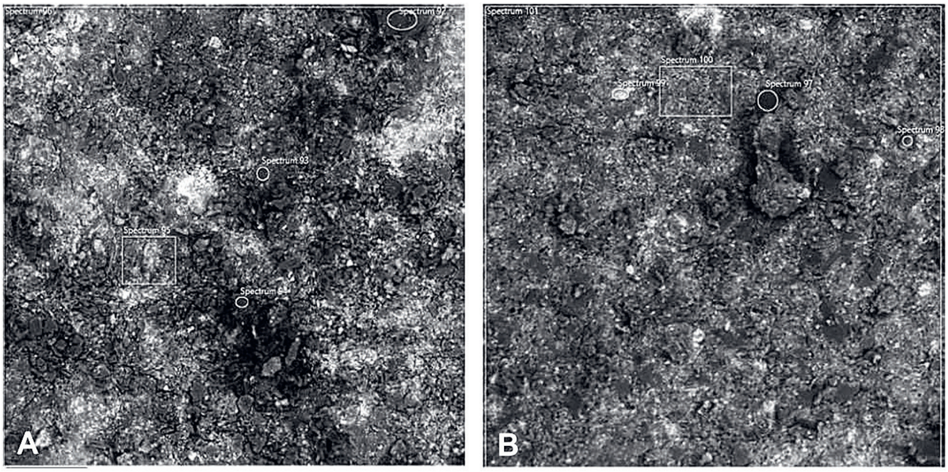
At Rydno the pigment was obtained from natural layers in the form of gravels, sand, or larger plastic elements such as clays. They are composed of hematite and lepidocrocite, as primary works have shown (comp. *Hensel 2011*, 407). The samples chosen for analysis were taken from the well-dated mining pits in trench I/77. The colour of the selected samples is red, with most of them being dark red in hue (*Fig. 3*). The samples were taken from a conglomerate bed consisting of gravel, including quartz, pebbles, hematite, chert, and sandstone in clay. This conglomerate lies on the slope between variegated sandstone of the Upper Raethian of the Triassic period. The conglomerate is formed from eroded deposits of older sediments (*Schild et al. 2011*, 55).

Orońsko mining shafts and clays from the immediate vicinity

Pigments from Orońsko consist of two distinct varieties. One was found in the shafts near the charcoals, suggesting the presence of fire. This material was likely composed of natural clay sediments with a significant iron content. Through fire treatment and dehydration, it changed to darker tones, resulting in a hue that can be described as light red to orange (*Fig. 3*). The second variety present in Orońsko is a natural clay component that exhibits a darker orange colour. This variant is also found in natural clay deposits.

Macroscopic description of ochre

The samples chosen for the SEM-EDS research displayed a variety of characteristics. The sample from Orońsko shaft 1 (OCHRE 7) was a pale orange colour and had a lump form that could easily be crumbled in the hand. In contrast, the samples from Orońsko shaft 6 (OCHRE 1, OCHRE 8) were of a dark orange hue, also in lump form, but these contained visible grains that could be observed macroscopically. The sample from the Rydno complex (OCHRE 2, OCHRE 3, and OCHRE 9) was dark red and came in stable lumps, also with discernible grains.



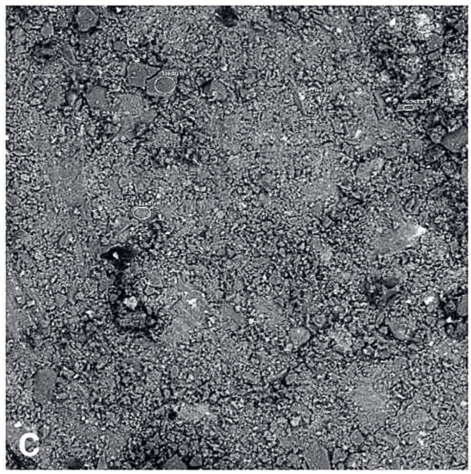


Fig. 5. SEM photographs of the samples with marked areas under analysis: A – OCHRE 1, B – OCHRE 2, C – OCHRE 3; 500x magnification (photo by M. Słoniewski).

SEM-EDS analyses of red material samples

The results obtained for samples OCHRE 1–OCHRE 3 were notably distinct (*Tab. 2*; *Fig. 5*). Each sample exhibited a relatively high mass percentage of silica oxide, with the highest recorded for the OCHRE 1 sample at 81.2 wt%. A significant distinguishing factor was the iron oxide content: OCHRE 1 ranged from 3.65 wt% to 4.08 wt%, OCHRE 2 from 27.42 wt% to 28.42 wt%, and OCHRE 3 from 38.16 wt% to 39.58 wt%. Additionally, the calcium oxide content was higher in OCHRE 1 (4.33–4.36 wt%) compared to the other two samples. The flint artefact displaying visible red staining (OCHRE 4, *Tab. 3*) showed typical elements found in cherts. A slight discrepancy was noted in the Fe_2O_3 levels between sections with macroscopically observable staining (OCHRE 4-1 and OCHRE 4-2) and those devoid of a red coating (OCHRE 4-3 and OCHRE 4-4). It appears that the red coating was not thick enough to be analysed using the selected method, especially without an additional conductive coating. Upon closer examination of areas with a greater accumulation of red material at higher magnifications, elevated levels of iron oxides were present

No		Na_2O	MgO	Al_2O_3	SiO_2	SO_3	K_2O	CaO	TiO_2	Fe_2O_3	Cr_2O_3	NiO	MnO
OCHRE 1	Oxide %	0.45	0.49	7.11	80.44	0.34	1.61	4.33	0.52	4.08	x	0.47	0.16
	Oxide % Sigma	0.03	0.03	0.06	0.16	0.04	0.03	0.05	0.04	0.07	x	0.05	0.04
OCHRE 1	Oxide %	0.45	0.51	7.25	81.29	0.3	1.62	4.36	0.46	3.65	x	x	0.11
	Oxide % Sigma	0.03	0.03	0.05	0.14	0.04	0.03	0.04	0.04	0.06	x	x	0.03
OCHRE 2	Oxide %	0.17	0.32	13.7	56.24	0.3	0.95	0.42	0.47	27.42	x	x	x
	Oxide % Sigma	0.05	0.05	0.11	0.2	0.06	0.04	0.04	0.05	0.17	x	x	x
OCHRE 2	Oxide %	0.17	0.3	12.53	56.17	0.27	0.89	0.43	0.47	28.42	0.14	0.21	x
	Oxide % Sigma	0.03	0.03	0.07	0.14	0.04	0.02	0.03	0.04	0.12	0.03	0.05	x
OCHRE 3	Oxide %	x	0.21	4.84	54.6	x	0.46	x	0.32	39.58	x	x	x
	Oxide % Sigma	x	0.04	0.07	0.18	x	0.03	x	0.05	0.16	x	x	x
OCHRE 3	Oxide %	x	x	4.95	55.92	0.15	0.45	0.11	0.26	38.16	x	x	x
	Oxide % Sigma	x	x	0.06	0.16	0.04	0.02	0.03	0.04	0.14	x	x	x

Tab. 2. The outcomes of the EDS analyses of samples OCHRE1 to 3. These samples were grounded and pressed and analysed without coating of conductive layers.

OCHRE4	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	MnO	Fe_2O_3	NiO
Ochre4-1	-	-	4.80	93.39	-	-	-	0.56	-	-	1.25	-
Ochre4-2	-	0.26	4.94	92.95	-	-	-	0.57	-	-	1.28	-
Ochre4-3	-	-	3.26	95.68	-	-	-	0.32	-	-	0.74	-
Ochre4-4	-	-	3.28	95.79	-	-	-	0.27	-	-	0.66	-
Ochre4-5	-	0.39	6.71	67.67	0.19	0.59	0.72	16.23	0.56	0.20	6.56	0.19
Ochre4-6	0.21	1.07	13.8	63.31	0.34	0.39	2.07	3.63	1.19	0.11	13.87	-
Ochre4-7	0.21	1.09	13.77	58.90	0.39	0.39	2.13	4.09	1.56	0.12	17.36	-

Tab. 3. Results of EDS analyses of the OCHRE 4 sample. Ochre 4-1 and Ochre 4-2 were taken on parts of the artefact with visible red traces, Ochre 4-3 and Ochre 4-4 were taken on the surface without visible red staining, Ochre 4-5 to Ochre 4-7 were taken on restricted areas with observed macroscopically accumulations of red material.

OCHRE 5	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	Fe_2O_3
Ochre5-1	0.31	0.78	7.87	42.67	6.44	1.02	35.31	1.00	4.61
Ochre5-2	-	1.59	6.25	48.42	4.35	0.81	34.59	0.50	3.48
Ochre5-3	-	1.19	5.26	14.60	20.41	0.52	54.91	0.55	2.55
Ochre5-4	0.39	1.17	6.21	15.54	21.72	0.62	51.81	-	2.54
Ochre5-5	0.34	1.15	7.15	17.90	18.85	0.74	51.35	-	2.51

Tab. 4. The EDS results taken on the bone tool OCHRE 5. Ochre 5-1 and Ochre 5-2 were taken on the surface visibly stained with red pigments, Ochre 5-3 was taken on flat surface without distinctive staining, Ochre 5-4 and Ochre 5-5 were taken on the distal end of the tool without visible red staining.

OCHRE 6	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	Fe_2O_3
Ochre6-1	1.02	10.57	37.64	0.46	1.30	42.90	0.60	5.50
Ochre6-2	0.99	9.43	36.29	0.45	1.22	45.88	0.66	5.09
Ochre6-3	1.06	9.02	32.77	-	1.21	50.22	0.67	5.06

Tab. 5. The EDS results taken on the OCHRE 6 sample, which was a piece of limestone with red stains. The analyses were done on the spots with clearly observable red traces.

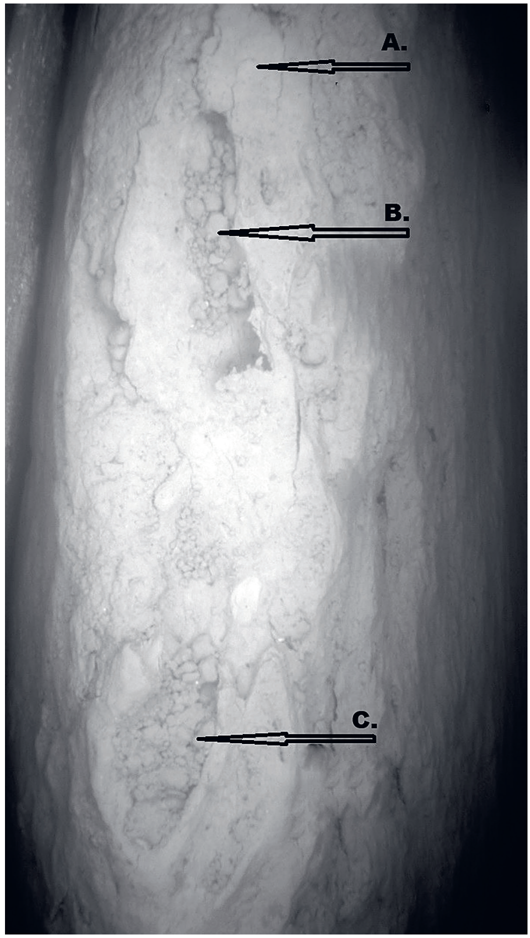


Fig. 6. Occurrence of iron on the surface of analysed the OCHRE 4 sample with macroscopically observable red coating: A – point without red pigment, B – pit 1, C – pit 2 (photo by M. Słoniewski).

(OCHRE 4-5 to OCHRE 4-7, *Tab. 3*). Sample OCHRE 4 was additionally analysed a second time after pulverisation with a carbon layer, allowing for the creation of elemental distribution maps on the surface of the artefact (*Fig. 6*). The red staining appeared to be of a discontinuous nature, with larger accumulations observed in the material's bends. Further analyses were conducted on a bone tool identified as sample OCHRE 5 (*Fig. 7*; *Tab. 4*). Notably, the results for OCHRE 5-3 to OCHRE 5-5 showed significantly elevated levels of calcium oxide and phosphorus oxide, alongside reduced silica oxide levels. These findings should be interpreted as representative of the bone material. Conversely, the results for OCHRE 5-1 and OCHRE 5-2 exhibited notably higher levels of SiO_2 and Fe_2O_3, attributed to the red staining on the surface. However, due to the persistence of notable P_2O_5 and CaO levels, these results should be understood as representative of both the staining and the background material. The OCHRE 6 limestone fragment was also visibly coated with a rusty-red layer. The outcomes indicated iron oxides at levels exceeding 5.00 wt%, likely linked to the observed surface hue (*Tab. 5*). Samples OCHRE 7 to OCHRE 9 were analysed meticulously, with an examination of the various portions of the sample surfaces.

Analysis no	Si	Ti	Al	Fe	Mg	Ca	K
Oro1-1 (24)	44.05	0.00	8.71	5.91	1.04	37.95	2.34
Oro1-2 (25)	17.86	0.00	3.78	2.63	1.01	74.71	0.00
Oro1-3 (26)	9.88	0.00	4.22	2.66	0.67	82.57	0.00
Oro1-4 (27)	86.37	0.00	2.48	1.33	0.00	9.82	0.00
Oro1-5 (28)	34.35	0.00	7.45	4.26	1.06	51.30	1.58
Oro1-6 (29)	55.83	0.56	14.16	7.04	1.76	17.20	3.45
Oro1-7 (30)	85.15	0.00	7.03	2.99	0.95	2.62	1.28
Oro1-8 (31)	58.53	0.00	7.40	3.75	1.23	27.63	1.45
Oro1-9 (32)	27.84	0.00	9.20	14.15	1.47	45.47	1.86
Oro1-10 (35)	8.73	0.00	4.08	2.55	1.41	83.22	0.00
Oro1-11 (36)	9.56	0.00	4.14	2.98	0.85	82.47	0.00
Oro1-12 (37)	10.23	0.00	3.81	4.32	0.68	79.98	0.98

Tab. 6. EDS results of the OCHRE 7 sample with excluded carbon (due to the pulverisation with carbon dust) and oxygen (due to insufficient sensitivity of the equipment). Presented in wt%.

Analysis no	Si	Ti	Al	Fe	Mg	Ca	K	Na	S
Oro2-1 (38)	29.21	0.67	11.61	9.05	1.23	45.49	2.28	0.00	0.45
Oro2-2 (39)	54.21	0.97	22.57	13.05	2.24	2.83	4.14	0.00	0.00
Oro2-3 (40)	55.98	0.97	19.32	14.27	1.89	3.84	3.73	0.00	0.00
Oro2-4 (41)	63.28	0.76	16.69	10.62	1.73	2.78	3.28	0.56	0.31
Oro2-5 (42)	36.62	1.33	15.99	37.76	1.93	2.90	3.47	0.00	0.00
Oro2-6 (43)	69.27	0.59	15.43	8.94	1.62	1.60	2.53	0.00	0.00
Oro2-7 (44)	58.83	0.75	20.10	10.97	2.04	3.79	3.23	0.00	0.29
Oro2-8 (45)	71.40	0.46	15.07	7.91	1.59	1.49	2.07	0.00	0.00
Oro2-9 (46)	9.49	0.00	4.48	2.84	0.55	82.63	0.00	0.00	0.00
Oro2-10 (47)	99.27	0.00	0.73	0.00	0.00	0.00	0.00	0.00	0.00
Oro2-11 (48)	93.18	0.00	3.62	1.96	0.51	0.25	0.48	0.00	0.00
Oro2-12 (49)	15.49	0.00	5.81	3.92	0.71	72.76	0.97	0.00	0.34
Oro2-13 (50)	63.94	1.12	19.02	8.99	2.33	1.68	2.92	0.00	0.00
Oro2-14 (51)	42.49	1.85	16.09	29.04	1.57	3.40	5.32	0.00	0.25
Oro2-15 (52)	49.49	0.94	25.08	14.41	2.12	4.59	3.37	0.00	0.00

Tab. 7. EDS results of the OCHRE 8 sample with excluded carbon (due to the pulverisation with carbon dust) and oxygen (due to insufficient sensitivity of the equipment). Presented in wt%.

Analysis no	Si	Ti	Al	Fe	Mg	Ca	K	Na
Ryd1-1 (53)	36.17	0.52	13.38	48.18	0.00	0.30	1.44	0.00
Ryd1-2 (54)	34.81	0.72	15.24	47.16	0.00	0.42	1.64	0.00
Ryd1-3 (55)	35.71	0.62	15.76	45.95	0.00	0.47	1.48	0.00
Ryd1-4 (56)	32.48	0.62	16.28	47.82	0.70	0.88	1.22	0.00
Ryd1-5 (57)	29.18	0.94	14.43	52.77	0.50	0.00	1.72	0.46
Ryd1-6 (58)	74.45	0.00	4.38	20.50	0.00	0.00	0.66	0.00
Ryd1-7 (59)	69.00	0.00	5.30	25.09	0.00	0.00	0.61	0.00
Ryd1-8 (60)	38.40	0.50	17.25	41.58	0.40	0.83	1.05	0.00
Ryd1-9 (61)	27.28	0.55	17.41	52.85	0.00	0.57	1.33	0.00
Ryd1-10 (62)	31.77	0.58	17.52	48.83	0.00	0.00	1.30	0.00
Ryd1-11 (63)	90.89	0.00	1.43	7.45	0.00	0.00	0.23	0.00
Ryd1-12 (64)	34.88	0.48	19.59	44.29	0.00	0.00	0.77	0.00

Tab. 8. EDS results of the OCHRE 9 sample with excluded carbon (due to the pulverisation with carbon dust) and oxygen (due to insufficient sensitivity of the equipment). That sample presents a notably lower amount of calcium, followed by relatively high outcomes for iron. Presented in wt%.

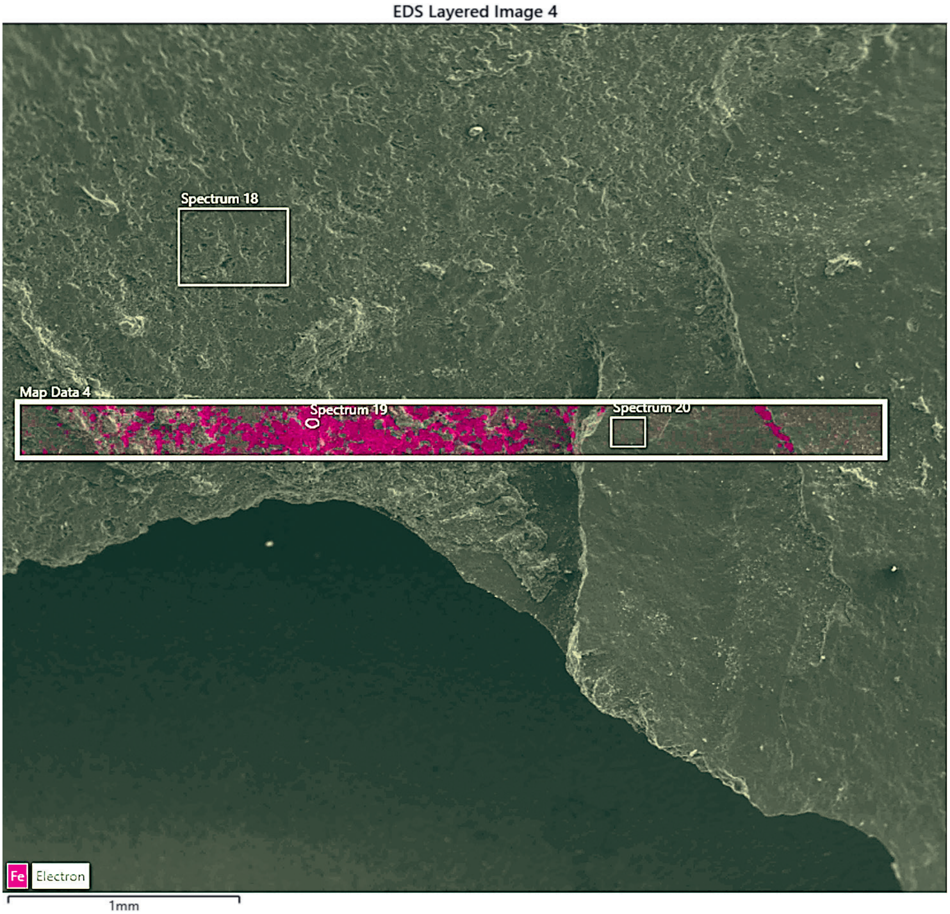


Fig. 7. Photography of the bone tool, named OCHRE 5, with traces of red pigments. Arrows point to spots of conducting EDS analyses (photo by M. Słoniewski).

The OCHRE 7 and OCHRE 9 specimens each received 12 individual EDS analyses, while OCHRE 8 received 15 analyses. The results were presented as weight percentages for each element. For the OCHRE 7 sample, the iron content was relatively low, with an average value of 4.55 wt%. The most abundant elements were calcium, averaging 49.58 wt%, followed by silica at 37.36 wt% and aluminium at 6.37wt%. The remaining components – magnesium, potassium, and titanium – averaged around 1.00 wt% or below that level (*Tab. 6*; *Fig. 8*). The OCHRE 8 sample showed a higher iron content, averaging 11.58 wt%. In addition, silica (54.14 wt% on average), calcium (15.34 wt% on average), and aluminium (14.11 wt% on average) were the major components. Other elements, including titanium, potassium, sulphur, magnesium, and sodium, were noted at average levels below 3.00 wt% (*Tab. 7*; *Fig. 8*). The last sample, OCHRE 9, was significantly enriched with iron, showing an average outcome of 40.20 wt%. Silica, at an average level of 44.59 wt%, and aluminium, at 13.16 wt%, were also abundant, while the remaining recognised elements presented mean values below 1.20 wt% (*Tab. 8*; *Fig. 10*).

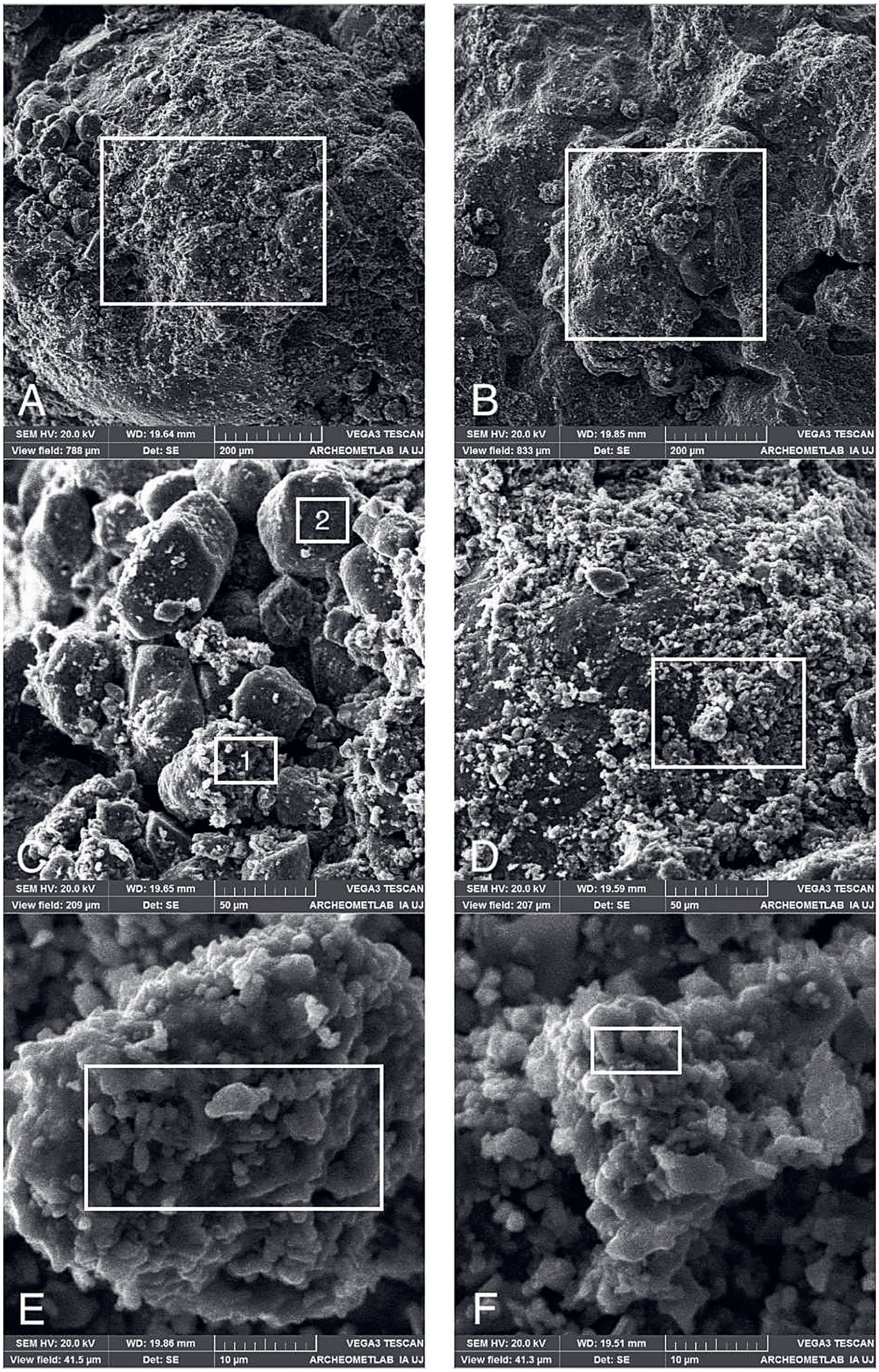


Fig. 8. The SEM microphotographs of the OCHRE 7 sample with marked spots of analyses. A – analysis Oro1-1; B – analysis Oro1-2; C – analysis Oro1-6 (point 1) and Oro1-7 (point 2); D – analysis Oro1-5; E – analysis Oro1-12; F – analysis Oro1-10 (photo by J. Kościuk-Załupka).

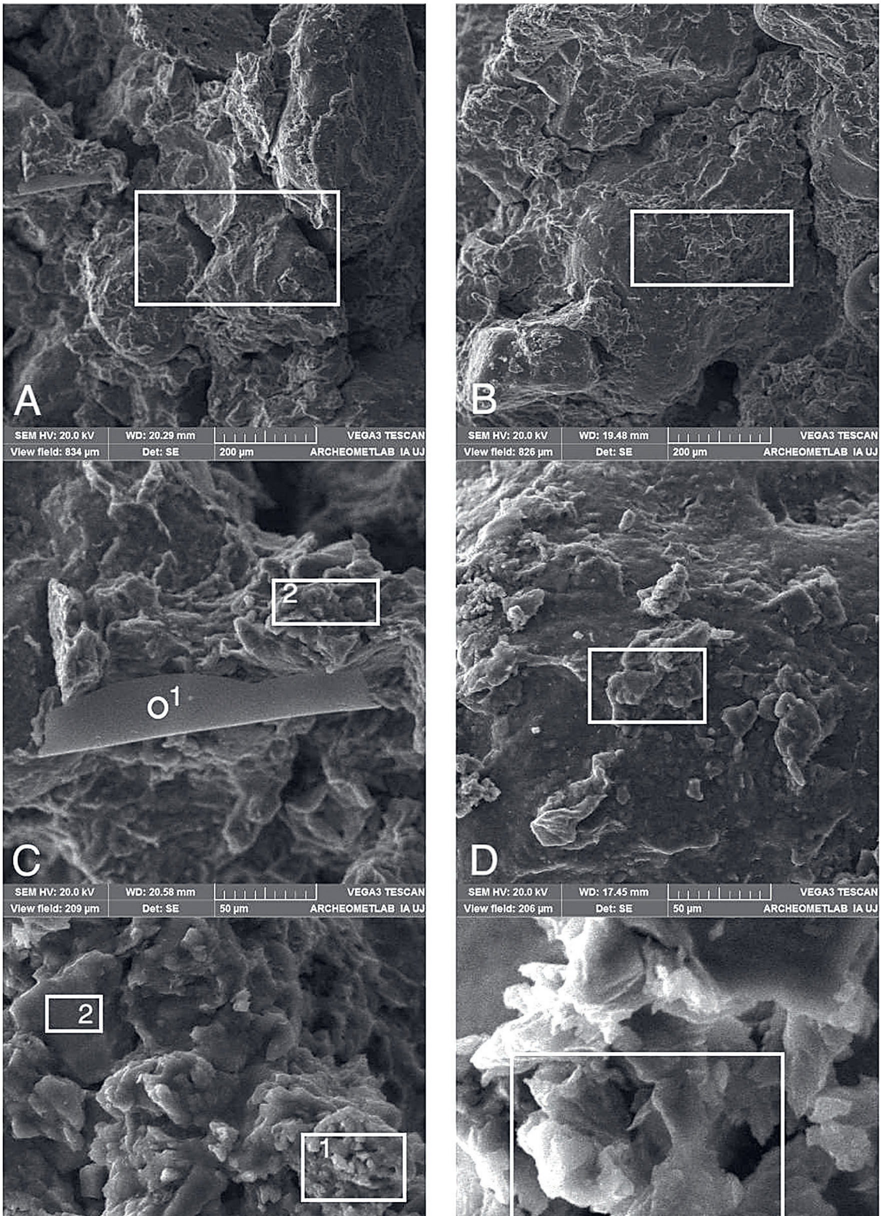


Fig. 9. The SEM microphotographs of the OCHRE 8 sample. with marked spots of analyses. A – analysis Oro2-3; B – analysis Oro2-2; C – analysis Oro2-5 (point 1) and Oro2-6 (point 2); D – analysis Oro2-7; E – analysis Oro2-9 (point 1) and Oro2-10 (point 2); F – analysis Oro2-14 (photo by J. Kościuk-Załupka).

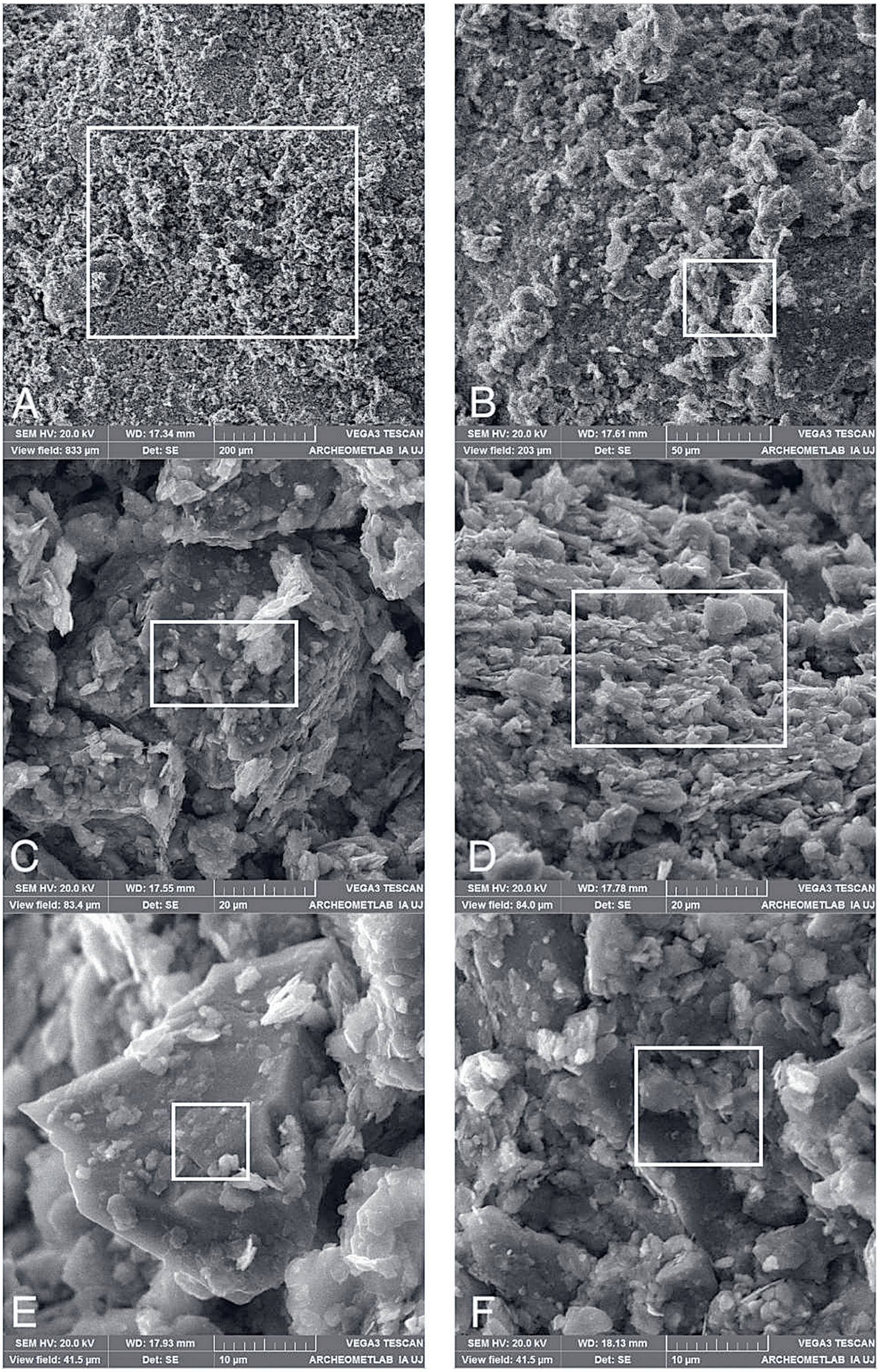


Fig. 10. The SEM microphotographs of the OCHRE 9 sample, with marked spots of analyses. A – analysis Ryd1-2; B – analysis Ryd1-4; C – analysis Ryd1-7; D – analysis Ryd1-9; E – analysis Ryd1-11; F – analysis Ryd1-12 (photo by J. Kościuk-Załupka).

Discussion

Chocolate flint

We made an initial attempt to characterise the flint from the northernmost part of the chocolate flint deposits and to compare it with the earlier published results on this subject (see *Hensel 2011*; *Werra – Siuda 2015*; *Brandl et al. 2016*; *Hughes et al. 2016*; *Sobkowiak-Tabaka et al. 2016*). The material was initially identified by its light hue, which included a range of grey and light brown shades featuring characteristic streaking, delicate lighter streaks, or smaller, more expressive spots of varying sizes and intensities. Most specimens were concentrated in the Orońsko area, and they were primarily classified into groups V, VI, VII, and IX. Additionally, darker specimens were identified, lacking additional characteristic grey stripes and displaying hues of brown, dark brown, and dark brown-grey. However, macroscopic characteristics alone cannot definitively identify the source of specific raw materials. Therefore, an effort was made to compare the specimens – especially those from Orońsko – with samples from more distant sites based on their elemental and mineral constituents using SEM-EDS equipment. The results showed the presence of apatite in each analysed sample, which was noteworthy. Furthermore, the samples contained rutile, barite, iron oxides, and clusters of clay minerals. The analysis of the chemical composition of chocolate flint in the micro-area has revealed the presence of hematite, pyrite, gypsum, barite, calcite, rutile, ilmenite, zircon, monazite, churchite, and apatite. According to previously published data, hematite, barite, and pyrite were the most common minerals, with apatite now considered a probable determinant of chocolate flint (see *Werra – Siuda 2015*), as observed in the samples described in this article. It is important to note that the elements responsible for the colour (Cr, Mn, Fe, Ni) may not be useful for distinguishing the sources of the raw material (*Brandl et al. 2016*, 130). Conversely, the results of the EDXRF analysis of chocolate flint from the modern quarry in Wierzbica (published elsewhere, e.g. *Hughes et al. 2016*) revealed discrepancies in these component proportions when compared to specimens from mining shafts at varying stratigraphic positions. The presence of pyrite also indicated this differentiation (*Hughes et al. 2016*, 109). In other previous studies on chocolate flint (*Brandl et al. 2016*), the combination of various components revealed that the calcium content among samples in Orońsko and Tomaszów was low, while the aluminium content was high (*Brandl et al. 2016*, 144). Analyses conducted on samples of other varieties of flints, including erratic flint, indicated differentiation among the samples based on the calcium versus iron elements (*Sobkowiak-Tabaka et al. 2016*). This approach appears to be a promising method for future research, including the analysis of the distribution of chocolate flint from the Orońsko mine during the Palaeolithic.

Red materials

In the analysis of ochre, several key points should be emphasised. The first issue concerns the comparison of the samples. Initial assessments indicate that the samples from the Rydno site (OCHRE 2, OCHRE 3, and OCHRE 9) show increased levels of iron and iron oxides, particularly when compared to the specimens collected at Orońsko. In contrast, the samples from the shafts in Orońsko (OCHRE 1, OCHRE 7, and OCHRE 8) exhibit higher calcium and calcium oxide levels. These differences may serve as distinctive features that allow for preliminary differentiation between the sets of samples from these two locations. Additionally, three items with visible ochre stains were analysed: a flint flake (OCHRE 4), a bone tool (OCHRE 5), and a limestone fragment (OCHRE 6). In these cases, the results for the underlying background material influenced the outcomes (e.g., elevated P_2O_5 in OCHRE 5), complicating the analysis of the red material. Nonetheless, the red stains showed elevated levels of iron and iron oxides compared to the background materials, validating their identification as ochre traces. Another noteworthy observation is that despite using various analytical modes and presenting outcomes as elemental or oxide compositions, the initially observed trends remained consistent. This consistency allows for the comparison of results derived from different laboratories and presented in various formats, which is particularly useful for pigment analysis and provenance studies. These observations suggest a preliminary hypothesis that the pigments were not transported from Rydno to Orońsko, at least not during the Late Palaeolithic period of occupation in the Orońsko mine area. More samples must be analysed with various techniques and better resolution to compare minor and trace elements. This would facilitate a more detailed characterisation, potentially revealing additional distinguishing features.

Conclusion

The ongoing research into the provenance of raw materials is yielding results by comparing various siliceous raw materials using a combination of macro- and microscopic techniques. Preliminary conclusions can be drawn based on the macroscopic features of the chocolate flint samples from the analysed sites, partially confirming earlier assumptions about the export of chocolate flint from the Orońsko region. Further studies will continue using a broader range of analytical methods and a larger number of samples from the chocolate flint mine in Orońsko and other sites dated to the same period but located away from the chocolate flint deposits in the northern slope of the Świętokrzyskie Mountains, as well as the Cracow-Częstochowa Upland (*Krajcarz et al. 2012*; *Sudoł-Procyk 2022*; *Mandera et al. 2024*). Regarding flint, it has been suggested that some of it could have been transported to Rydno or Całowanie from mining areas in or near Orońsko. Also, some of the results of red pigment analysis from Całowanie has shown some similarities with the Rydno quarry (*Hensel 2011*, 407), indicating a close relationship between these communities. In contrast, the reverse transport of ochre from Rydno to Orońsko remains unclear. The SEM-EDS analysis of red pigments showed no similarity to the samples of ochre taken from the Rydno mine, which was proposed initially as the original source. It is possible that communities in the Orońsko mining area used local red pigments during the Paleolithic, despite direct contact with societies inhabiting the Rydno and Całowanie sites, which may be inferred from the similarity of the flints. Further research with the use of particular chemical and geological methods will certainly help to confirm or refute this hypothesis.

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