PROVENANCING OF RED FERRUGINOUS ARTEFACTS AND RAW MATERIALS IN PALAEOLITHIC SOCIETIES

JOANNA TRĄBSKA

Key words: iron artefact, provenance, iron raw materials, haematite, Palaeolithic

An interest in provenance of ferruginous raw materials at Palaeolithic sites has been dated for many years, though only the latest thirteen years provided wider research programmes and results. Nature of ferruginous raw materials: their genetic diversity and abundance along with macroscopic similarity of representatives of various geological formations compels to recognise a number of fingerprints – more numerous than for other raw materials - and their sets.

Application of ferruginous raw materials may have been very wide in numerous sacral and everyday contexts, so that not only unprocessed but also processed materials must be considered. The latter would cover powders: intentionally produced or 'side effects' at various processes. They also need to be provenanced, though a procedure may be more difficult to perform due to: a) their processing, b) low amount of material, c) poor representativity of a material accessible only in a low volume. Nevertheless, provenance studies of both large pieces and powders, processed and unprocessed may answer a question on an ability of exploration of variable environments (not only to collect material but also to gather knowledge of other areas that would provide better life conditions\(^1\), on a range of group contacts, on reasons for diversity of it\(^2\), also to explain a use of exotic raw materials.

Last but not least, elements of 'châine opératoire' can be deduced due to recognition of a raw material and recognition of it's features after some transformation (e.g. heating; experimental works must be involved here). Moreover, it can be stated for sure that numerous applications of ferruginous materials are unavailable to our interpretation. For example, hard haematite or haematite rocks pieces not producing a streak, found not rarely at sites: is it an ordinary remnant or intentionally brought material? For what purposes then?

In Central Europe numerous varieties of red ferruginous raw materials are known. They can be divided into two groups: a) bound to determined lithostratigraphic levels, where palaeoclimatic conditions were a dominant factor of their creation, like, for example, sedimentary rocks of Lower Devonian, Lower Permian, Lower and Upper Triassic, terra rossa of Devonian, Permian, Triassic, Tertiary and recent age, b) connected to determined geological formations, where mineral and rock origin processes were involved, like haematite bearing scarns, hydrothermal veins, weathering sheets on volcanic rocks, specific sedimentary basins and their sediments (e.g. Carpathian variegated shales), metamorphic rocks (e.g. BIF and others). For all them internal variability (a degree of homogeneity) of a statistically determined level is obvious.

It can be assumed that all could have been accessible to Palaeolithic people, cut into valleys or

\(^1\) HENSILWOOD 2008, 36.
\(^2\) In the South African Middle Stone Age context an increase in raw material diversity and greater colour variation is observed (HENSILWOOD 2008, 40) Such a diversity was observed by the author for some Central European Upper Palaeolithic 'red' assemblages, among others, Magdalenian Dzierżysław-35 site (Upper Silesia, Poland, TRĄBSKA 2010).
uncovered through landslides, even if their counterparts are not accessible to us now.

In this work a term “ochre” will not appear (even if it were introduced by quoted authors) because it defines only a part of ferruginous raw materials. A term Iron Red Raw Materials will be used (further in the text as IRRM). When using the term ‘red’ all ‘near red’ colour scale is understood (light pink, pink, cherry dark and light, violet, purple, orange, etc).

A review of so far research, presented in this work, comprises several representative, to the author opinion, articles issued after 1990, they are organised according to chronological order. The time caesura was accepted because earlier works were based mostly on macroscopic observations. Significant number of fingerprints to discuss and compare appeared in the nineties with wider accessibility of research methods, especially the ones allowing examination of a 'low quantity' material.

In the 90-ties mostly Australians concentrated on IRRM provenance research. In 1997 an article of Smith and Pell appeared, where concentration of oxygen isotopes in quartz grains in IRRM bearing sediments was considered as an indicator. Geological material of various origin was examined and δ18O relative to V-SMOW standard was calculated. Calculated values were not distant from standard error or fell into it. Very similar isotope ratio were obtained for two outcrops, whereas the other two were distinctively different. Very low amount of samples, however, was investigated, it was admitted by the authors themselves who suggested application of complementary methods. The samples were processed only according to the method procedure. Discrimination between samples representing the same geological context was impossible but differences between samples from various formation gave a satisfactory result.

Jercher et al. published another article on provenancing IRRM; again raw materials from several outcrops of Aboriginal interest were screened, samples were collected from museum inventories. It, to a certain degree, limited research possibilities, especially in regard to a problem of raw material variability, but yet, this is a common situation in archaeometric works. Geological origin varied, three of the six outcrops represented the same formation but one of the outcrops was remote 600 km from the others. Samples in the field were also collected, representing analogous geological formations. Macroscopic features were described, phase composition and size of elemental haematite cells were examined with X-ray diffraction (XRD), chemical composition was measured with X-ray fluorescence (XRF). Trace elements in haematite crystal lattice were excluded and chemical composition revealed differences in composition and concentration within the samples, especially for the ones from various geological contexts. In some cases untypical, according to the authors, minerals appeared, like natrojarosite and bassanite. Further statistical analyses might be fruitful, according to the authors suggestion, yet, even at the completed stage it is possible to point at a source (even an outcrop) for a sample. Samples were not intentionally prepared, only following to a routine method procedure.

Magnetic properties were considered as a set of fingerprints for IRRM provenancing also for Australian samples, three of them were examined previously by Smith and Pell. One sample came from an archaeological trench, the other two were of unknown origin. Samples were not preprocessed and the methods were non-destructive. Numerous parameters were considered, like magnetic susceptibility, para- (dia)magnetic susceptibility, frequency dependent susceptibility, isothermal remanent magnetisation, saturation isothermal remanent magnetisation, anhysteretic remanent magnetisation, saturation magnetisation, coercive force, S-ratio. They allowed to gain information on variety, concentration and size of ferrimagnetic (e.g. magnetite), antiferromagnetic (e.g. haematite), paramagnetic (e.g. iron silicates) and diamagnetic (e.g. quartz) minerals. Differences of the parameters were significant so that ascribing a sample to an ore was possible.

Another research concerns a material from Arizona. 110 samples from 22 outcrops were collected and three geological contexts were considered to investigate a variability and homogeneity of the ores. Samples were preprocessed: the finest fraction was separated and

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3 A specific property of a mineral or of a rock, observable in all examined rock type but differing significantly in the varieties.
5 JERCHER et al. 1998.
6 MOONEY et al. 2003.
8 POPELKA-FILCOFF et al. 2008.
then INAA (Instrumental Neutron Activation Analysis) chemical analysis was conducted. Statistical analyses enabled to reveal the elements connected with iron and to explore their mutual dependencies (e.g. As/Fe and Sb/Fe); Pearson's correlation was applied and 90% confidence interval was accepted. PCA (Principal Component Analysis) and CDA (Canonical Discriminant Analysis) analyses allowed to find out a „geochemical signature” within the set, even for the samples provenancing from the same geological context.

Another approach in IRRM provenancing has been applied for red pigments from cave, stone and other paintings as well as red surfaces from other contexts. Many research provide information only on a pigment variety, without further works on its origin, due to various reasons. Some proceed further, an example comes from the Obłazowa Cave, Poland, famous for an Early Gravettian mammoth tusk boomerang9. Red painted (or covered by a red fluid in another mode) artefacts surfaces were examined along with red sediments from the cave of terra rossa type and terra rossa from the cave vicinity. XRD and SEM/EDS were performed10 and indicated that actually terra rossa was a painting material.

Aurignacian stone paintings from Grotta di Fumane, Monte Lessini (Italy) were examined by Broglio et al.11 Samples were collected from loose material, optical microscopy (PLM), scanning microscopy (SEM/EDS), X-ray diffraction (XRD) and gas chromatography were applied. Casual, natural pigmentation was excluded. Based on results of chemical analyses red residual rocks from the vicinity were pointed at as a pigment source. But it was not a sole one: „probably haematite” was also confirmed. In Aurignacian layers IRRM were also found: fine crystalline haematite and another IRRM supposedly from Provence, as it was supposed on the basis of macroscopic observations.

The author and colleagues12 examined a powdery, thin layer on a small pebble from Aurignacian layers from Klissoura, Greece. The origin of the powder: natural or intentional was a problem to solve. Terra rossa, ubiquitous in the site and with a wide red shade spectrum, might have coloured a pebble but comparison of haematite crystallite size of the powder and adjacent terra rossa allowed to exclude the suspicious rock.

Composition of red pigments in cave and stone paintings used to be ascribed to haematite from a cave outside or terra rossa-like rocks from a cave inside or vicinities. This is only a rough estimation: detailed analyses of red cave rocks and neighbourhood reveal far more complex image. A work of Iriarte et al.13 inspects the problem. Fingerprints of IRRM from two caves and the adjacent areas were searched for to point at potential raw materials for cave paintings from the area of Pyrenees (Northern Spain). Research were conducted with the use of relatively accessible methods (PLM, SEM/EDS, XRD, ICP-MS). 24 samples from each cave (both from walls and floor) were collected and preprocessed: they were crushed and sieved (sieve diameter 0.15 mm) and the finest fraction was analysed. The red rocks were of various origin, microstructure and chemical patterns, distinction of pigments made of them is, according to the authors, would be possible.

From 2006 on, the author with colleagues has been examining samples representing IRRM from various geological context, chronology and locality, mostly from Central Europe. The only selection criterion has been red colour. Field samples has been supplemented by IRRM from archaeological sites and red ferruginous artefacts. Data, still being completed, are a base for a red rocks database, an example of the construction is contained in the Table 1, complementary proposition, based on the Magdalenian Dzierżysław-35 site was issued elsewhere14.

Completed data allow to define some selected fingerprints for each rock (here: microstructure, haematite crystallite size, chemical composition). Samples have been stored and additional measurements for other fingerprints may be introduced, so that the database has an open character. Analogous database has been constructed for IRRM from archaeological sites and 'red' artefacts if they could have been examined.

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9  VALDE-NOWAK 2003.
14  TRĄBSKA 2010.
Table 1.: An example sheet of an IRRM database

<table>
<thead>
<tr>
<th>Record definition</th>
<th>Description/photograph, diagram or reference to a set of them</th>
<th>References, if necessary</th>
</tr>
</thead>
<tbody>
<tr>
<td>Loco (name)</td>
<td>Bečov</td>
<td></td>
</tr>
<tr>
<td>Region</td>
<td>Most Basin</td>
<td></td>
</tr>
<tr>
<td>State</td>
<td>Czech</td>
<td></td>
</tr>
<tr>
<td>Coordinates</td>
<td>Not measured</td>
<td></td>
</tr>
<tr>
<td>Geological origin and chronology</td>
<td>Porcelanite (caustically changed clays), Tertiary</td>
<td>Tyráček 2005(^{15}), (with other references)</td>
</tr>
<tr>
<td>Geological map</td>
<td>Cháb et al. 2007(^{16})</td>
<td></td>
</tr>
<tr>
<td>Sample code</td>
<td>Bec-10</td>
<td></td>
</tr>
<tr>
<td>Macroscopic description of a raw sample</td>
<td>Light cherry, pink, poorly concised, moderately soft rock of pelitic and fine psamitic components and chaotic texture</td>
<td>Reference to 'PLM' data set(^{17})</td>
</tr>
<tr>
<td>Microscopic (PLM) description and photograph of a raw sample.</td>
<td>Fine, rectangular grains of feldspar and quartz, inhomogeneously dispersed in very fine grained, porous mass composed of pyroxene and mullite. Secondary opal concentrations are present. Crossed nicols</td>
<td>Reference to 'SEM' data set</td>
</tr>
<tr>
<td>Microscopic (SEM) description and photograph of a raw sample.</td>
<td>Secondary kaolinite. Magn. 6000 X</td>
<td>Reference to 'SEM' data set</td>
</tr>
<tr>
<td>Chemical analysis of a raw sample (EDS, XRF).</td>
<td>EDS: Si, Al, O; K, Fe in a very low concentration</td>
<td>Reference to 'Chemical analysis' data set</td>
</tr>
<tr>
<td>Phase composition of a raw sample.</td>
<td>Mullite, CT-opal, enstatite, anortite, hercinitite, haematite, Na2Ca(SiO)4 phase</td>
<td>Reference to 'Phase composition' data set</td>
</tr>
<tr>
<td>Haematite crystallite size of a raw sample.</td>
<td>Measurement impossible due to coincidence of haematite and mullite</td>
<td>Reference to 'Crystallite size' data set</td>
</tr>
<tr>
<td>Information of processing a sample in experiments.</td>
<td>Experiments on colour durability, covering and astringent properties</td>
<td>Reference to 'Processing and Experiments' data set. Previous experiments: Sajnerová-Dušková et al. 2009(^{18})</td>
</tr>
</tbody>
</table>

\(^{15}\) Tyráček 2005.  
\(^{16}\) Cháb et al. 2007.  
\(^{17}\) In practice always more than one analysis was performed with a use of a method, so that the record is linked with a separate one with adequate information.  
\(^{18}\) Sajnerová-Dušková et al. (2009)
Table 2.: Fingerprints established for the three genetically various haematite bearing rocks

<table>
<thead>
<tr>
<th>Lithostratigraphic units – rock differences</th>
<th>Sedimentary Lower Triassic siltstone, Poland</th>
<th>Carpathian variegated shales, Poland</th>
<th>Terra rossa, Croatia, Greece, Poland</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical composition</td>
<td>Chemical composition</td>
<td>No analyses</td>
<td></td>
</tr>
<tr>
<td>Size of haematite crystallites</td>
<td>Size of haematite crystallites</td>
<td>Size of haematite crystallites</td>
<td></td>
</tr>
<tr>
<td>Image in thin section</td>
<td>Image in thin section</td>
<td>Image in thin section</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sub-lithostratigraphic units – rock differences</th>
<th>Sedimentary Lower Triassic siltstone, Poland</th>
<th>Carpathian variegated shales, Poland</th>
<th>Terra rossa, Croatia, Greece, Poland</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical composition</td>
<td>Chemical composition</td>
<td>No analyses</td>
<td></td>
</tr>
<tr>
<td>Mineralogical features (I/Sm ratio; zeolite presence)</td>
<td></td>
<td>Size of haematite crystallites</td>
<td></td>
</tr>
<tr>
<td>Image in thin section</td>
<td>Image in thin section</td>
<td>Image in thin section</td>
<td></td>
</tr>
</tbody>
</table>

Table 3.: Fingerprints applied at IRRM provenancing, based on quoted references

<table>
<thead>
<tr>
<th>Publication</th>
<th>Sample outcrop</th>
<th>Processed sample artefact</th>
<th>Method</th>
<th>Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMITH–PELL 1997</td>
<td>+</td>
<td>+</td>
<td>Oxygen isotope in quartz</td>
<td>Discrimination between samples from the same region is impossible. Significant results for only some outcrops and samples.</td>
</tr>
<tr>
<td>JERCHER et al. 1998</td>
<td>+</td>
<td>-</td>
<td>Chemical (XRF) and phase analysis (XRD)</td>
<td>Discrimination between outcrops of various origin is possible.</td>
</tr>
<tr>
<td>MOONEY et al. 2003</td>
<td>+</td>
<td>-</td>
<td>Magnetic properties</td>
<td>Distinctive variations in magnetic parameters.</td>
</tr>
<tr>
<td>ŁAPTA–PASZKOWSKI 2003</td>
<td>+</td>
<td>+</td>
<td>Phase (XRD), microstructural and chemical (SEM/EDS) analyses</td>
<td>Local raw materials of red painted (?) shell was identified.</td>
</tr>
<tr>
<td>BROGLIO et al. 2005</td>
<td>+</td>
<td>-</td>
<td>Chemical (EDS) and phase (XRD) analyses of painted materials, macroscopic evaluation of raw materials</td>
<td>Possible to point at local and remote sources of red pigments.</td>
</tr>
<tr>
<td>POPELKA-FILCOFF et al. 2008</td>
<td>+</td>
<td>+</td>
<td>Chemical composition (INAA), statistical analysis (PCA, correlation, CDA)</td>
<td>Distinctive variations in geochemistry within a one geological formation</td>
</tr>
<tr>
<td>TRĄBSKA et al. 2008</td>
<td>+</td>
<td>-</td>
<td>Chemical analysis (XRF, PIXE), statistical analysis (PCA, k-means)</td>
<td>Distinction between artefacts and outcrop samples from various sources of raw materials.</td>
</tr>
<tr>
<td>IRIARTE et al. 2009</td>
<td>+</td>
<td>+</td>
<td>Chemical composition (EDS, ICP-MS), statistical analysis (Al/Si ratio), phase composition (XRD), microstructure analysis (PLM)</td>
<td>Possible to distinguish the examined sources on the basis of textural, mineralogical and geochemical parameters.</td>
</tr>
<tr>
<td>TRĄBSKA et al. 2009</td>
<td>+</td>
<td>-</td>
<td>Chemical composition (XRF), haematite crystallite size (XRD), statistical analysis (cluster analysis)</td>
<td>Distinction between samples from various geological units and possible discrimination within a one unit.</td>
</tr>
</tbody>
</table>

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19 TRĄBSKA et al. 2010
Selected field material and artefacts were thoroughly examined and the results published. Soft rocks, easily covering surfaces (e.g. a palm surface), originating from the Lower and Upper Triassic cherry siltstones from the southern and northern boundary of the Świętokrzyskie Mts. were compared, in regard to chemical analysis, with artefacts from the Magdalenian site Dzierżysław-35. Both assemblages are macroscopically very similar one to the other. Statistical analysis (PCA, K-means, Kohonen neural net) based on chemical composition (XRF, PIXE) of field samples and artefacts revealed a clear distinction between them. This allowed to eliminate the Świętokrzyskie Mts. cherry siltstones as a potential source for Magdalenian artefacts. A conclusion is important because an immense Final Palaeolithic haematite mine was found there in Rydno.

Macroscopically similar rocks from three different geological context were examined in another article both in mega- (distinction between geological formation) and in mesoscale (distinction within sub-formation), distinctive fingerprints are presented in the Table 2. It is likely to discern, on the basis of chemical composition, Lower Triassic siltstones from a southern and northern peripheries of the Świętokrzyskie Mts.; recent and 'old' terra rossa (from older geological epochs) varies one from the other in haematite crystallite size, Carpathian variegated shales from the sub-units also differ (on the basis of chemical composition and mineral indicators). Works on details are being continued.

Provenancing of red ferruginous powders

Provenancing red powders creates a separate methodical problem, examples from cave and stone paintings as well as powdery remnants on stone surfaces were quoted above. Results of research conducted by the author and colleagues from 2006 suggest sufficient representativity of powders in regard to a parent rock, both in microstructure and chemical composition as well as haematite crystalline size, so that it seems possible to point at a source of even scarce powders. One can first identify powders composition, then an assemblage homogeneity, next original rocks provenance, finally a manner of processing (e.g. heating). The latter stage seems to be most difficult. All these remarks are going to be explored thoroughly in articles in preparation.

Discussion and Conclusion

All researchers dealing with provenancing of any raw materials are perfectly aware of problems hidden behind straightforward conclusions. For IRRM the obscurities are multiplied, due to reasons mentioned at the beginning but yet, considering their huge significance for sacral and everyday life of Palaeolithic people, the problem should be explored.

One of problems in IRRM provenancing is their variability, even in a one profile; a question formulated long ago. In many occasions the parameter is impossible to estimate (e.g. when an outcrop does not exist or too many analyses should be performed). Some works suggest, however, that successful solutions are available and provenancing on meso-scale was proved.

Another problem is a relevance between raw materials and processed artefacts. Red ferruginous artefacts may have been heated, ground, mixed with other IRRM or with organic substances. It seems that Popelka-Filcoff et al. pointed constructively at the problem, statistically exploring the elements bound to iron. But what about the procedure if two or more iron bearing raw materials had been mixed? A quest and testing of various fingerprints and sets of them has been summarised and their adequacy for sourcing procedures has been presented. Probably at imminent stages of research a unification of fingerprints for all databases at least in Europe will be possible.

Last but not least an important "archaeological" fingerprint: IRRM and other significant raw materials (first of all flint) cropping out close one to the other. The problem was analysed for example in Poland for Final Palaeolithic.

Development of research methods in geochemistry introduces continuously new perspectives for new IRRM fingerprints. Even if
now their application may be strongly confined, in several years impediments are likely to be defeated. A might be fingerprint can be iron isotopes, $^{56}$Fe and $^{54}$Fe. For the moment now $^{29}$ $\delta^{56}$Fe for iron bearing magmatic and sedimentary rocks seems not to be distinctive, yet with proceeding growth of data the fingerprint may be worth tracing.

$^{29}$ Schoenberg–Blackenburg 2006, 351.
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VASTARTARMÚ VÖRÖS ÁSVányok És KÖZETEK
EREDETMEGHATÁROZÁSA A PALEOLIT KÖZÖSSÉGEKBEN

JOANNA TRÁBSKA

Kulcsszavak: vastartalmú kőzetek és ásványok, származási hely vizsgálat, hematit, öskökor

A paleolit lelőhelyeken előkerült vastartalmú vörös ásványok és kőzetek eredetmeghatározásával kapcsolatosan régóta nagy érdeklődés mutatkozik a kutatásban. Ezeknek a nyersanyagoknak a genetikus sokfélesége és makroszkópos hasonlósága, valamint gyakori előfordulásuk a különféle geológiai képződményekben nehézzé teszi a feladatot. A vastartalmú vörös nyersanyagokat valószínűleg változatos, szakrális és világi kontextusban egyaránt használták, feldolgozva és eredeti állapotukban egyaránt. Találkozunk porított ércekkel is, amelyek szintén keletkezhetnek egy feldolgozási folyamat részeként és véletlenül is. Mind a tömbanyag, mind a porított minták, feldolgozva és feldolgozatlanul, lényegesen gazdagíthatják ismereteinket arról, hogy a paleolit ember mennyire ismerték környezetüket és milyen különleges anyagokat használtak.

Közép-Európában számos vörös vastartalmú ásványi anyagot ismerünk. Ezeket a különféle feltárásokból a paleolitikum embere is ismerhette, még akkor is, ha ma már nem mindegyik lelőhelyről tudunk mintát gyűjteni. A szerző nem használja az "okker" kifejezést, mert ez csak a vastartalmú vörös anyagok egy részére helyes meghatározás. A szövegben inkább a vastartalmú vörös anyagok (Iron Red Raw Materials, rövidítve IRRM) kifejezést használja, ami tágabb jelentésű.


A vizsgálatok során összehasonlított például a Szentkereszt-hegységből (Świętokrzyskie Mts.) származó, puha, jól fedő festékanyagot adó alsó- és felsőtriász korú vörös üledekes közeteket a Dzierżysław-35 magdaléni lelőhelyen talált festékanyaggal, amelyek egymáshoz nagyon hasonlónak mutatkoztak. A mintákon végzett geokémiai vizsgálatok (XRF, PIXE) és ezek statisztikai értékelése azonban jelentős különbségeket mutatott ki.

A vizsgálatokat kiterjesztette a Szentkereszt hegység további triász korú feltárásaira is (ld. 2. táblázat).

A vörös por formájában megtalálható festékanyagok vizsgálata további módszertani problémákat vet fel. Ez esetben a por előállításának és festékanyagként való alkalmazásának módzatait is vizsgálni kell és lehet.

A szerző felhívja a figyelmet a vastartalmú vörös anyagok kutatásával kapcsolatos nehézségekre. Az egyik probléma a variabilitás, akár egy feltáráson belül is. Egy lehetséges megoldás az eredmények középtávon történő értékelése. A másik probléma a nyersanyag feldolgozása - a vörös festékeket esetenként pörkölnek, örlíknak, és keverik más festékanyaggal vagy szerves kötőanyagokkal. A lehetséges kompozit anyagokról ad áttekintést a 3. táblázat.

Végül, de nem utolsó sorban, foglalkozik a régészeti lelőhelyekről előkerülő vörös festékanyagok vizsgálatával is, késő paleolit lengyelországi példákon. A vas izotópok ($^{56}$Fe és $^{54}$Fe) szintén hozzájárulhat az IRRM típusú anyagok pontosabb azonosításához.