Archaeologia Polona, vol. 54: 2016, 67–82
PL ISSN 0066-5924

Erratic Flint from Poland: Preliminary results of petrographic and geochemical analyses

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The paper presents the preliminary results of petrographic and geochemical analyses of erratic flint found throughout present-day Poland. Three different methods have been applied: electron probe micro analysis (EPMA), scanning electron method (SEM) and energy-dispersive x-ray fluorescence (EDXRF) spectrometry. The results of the EPMA and SEM analyses of erratic flint have revealed a largely homogeneous mineral composition, which suggests that mineral composition will be of limited utility in in distinguishing erratic flint. However, EDXRF analysis of a small sample of erratic flint has identified differences in calcium (Ca) and iron (Fe) content between and among samples of erratic and 'chocolate' flint but a much larger sample of erratic flint specimens needs to be analysed to determine the range of chemical composition they contain.

KEY-WORDS: erratic flint, geochemical analysis, EDXRF, SEM, EPMA

INTRODUCTION

Archaeological sites in Poland, and elsewhere in western Europe, document that flint and chert have a long history of use. The most commonly knapped raw materials throughout the Stone Age and Early Bronze Age, flints from a number of different sources were used for making tools and weapons, fire-lighting tools, etc. Given their utilitarian properties, raw flint and chert continued to be exploited in the Modern Period, serving as gunflints for flintlock guns (de Latour 2009) and even today, flints

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are utilised in the production of pottery, glass, and in pharmaceutical and cosmetic industries (Muszyński 2008: 348).

But from an archaeological perspective, detailed knowledge of the outcrops of siliceous rocks and the identification of the mineral and the chemical composition of raw materials is prequisite to archaeological studies devoted to documenting long distance distribution, exchange networks, and mobility patterns in the Stone Age (Sulgostowska 2005: 8). Fast developing methods of instrumental analysis, e.g. Prompt Gamma Activation analysis (PGAA; Révay and Belgya 2004), Energy-Dispersive X-ray Fluorescence (EDXRF) analysis (Hughes *et al.*, 2012), and Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry (LA-ICP-MS; Pettit *et al.*, 2012) have all been employed to help determine the origin of raw materials used to produce a given artefact and to chemically distinguish between and among individual varieties of siliceous rocks. With respect to identification of Polish raw materials, until recently most has been limited to macroscopic determination. Since the late 1980s, however, the situation has been gradually changing in Poland (Schild 1976; Lech 1980; Kozłowski and Pawlikowski 1989; Pawlikowski 1994; Högberg *et al.*, 2014; Hughes and Werra 2014; Sobkowiak-Tabaka *et al.*, 2015) and in Lithuania and Belarus (Baltrūnas *et al.*, 2006; Hughes *et al.*, 2011).

This paper is an initial attempt to investigate the petrographic-geochemical characteristics of Erratic flint, based on Scanning Electron Method (SEM), Electron Probe Micro Analysis (EPMA) and Energy-Dispersive X-ray Fluorescence (EDXRF). The results provide a context for a wider discussion on the possibilities, usefulness, and restrictions of the applied methods for recognition of links between outcrops, raw material and flint artefacts.

ERRATIC FLINT – CHARACTERISATION AND OCCURRENCE

Flint is a siliceous sedimentary rock composed of opal, chalcedony, quartz with few accessory minerals; it may also contain sponge spicules (Muszyński 2008: 343). The colour and other properties of flint result from its mineral composition, e.g. red, brown or yellow colours manifest the presence of goethite, lepidocrocite or hematite; grey and bluish show the presence of iron sulphides (mostly pyrite and marcasite); dark grey to black colour attest to the presence of hydrocarbons. Several colouring minerals typically co-exist in the rock, occurring in the trace elements (Pawlikowski 1989: 8). Formation of flint is believed to occur as a result of the deposition of siliceous skeletons elements, the precipitation of the silica from aqueous solution, the concentration of silica minerals as a consequence of transformation of siliceous rocks or sedimentary rocks, and long-term weathering processes (Muszyński 2008: 330–336).

Cretaceous flint is the most widespread raw material in the area of present-day Poland, which has seen several glaciations throughout the Quaternary Period (Mojski 2005: 42).

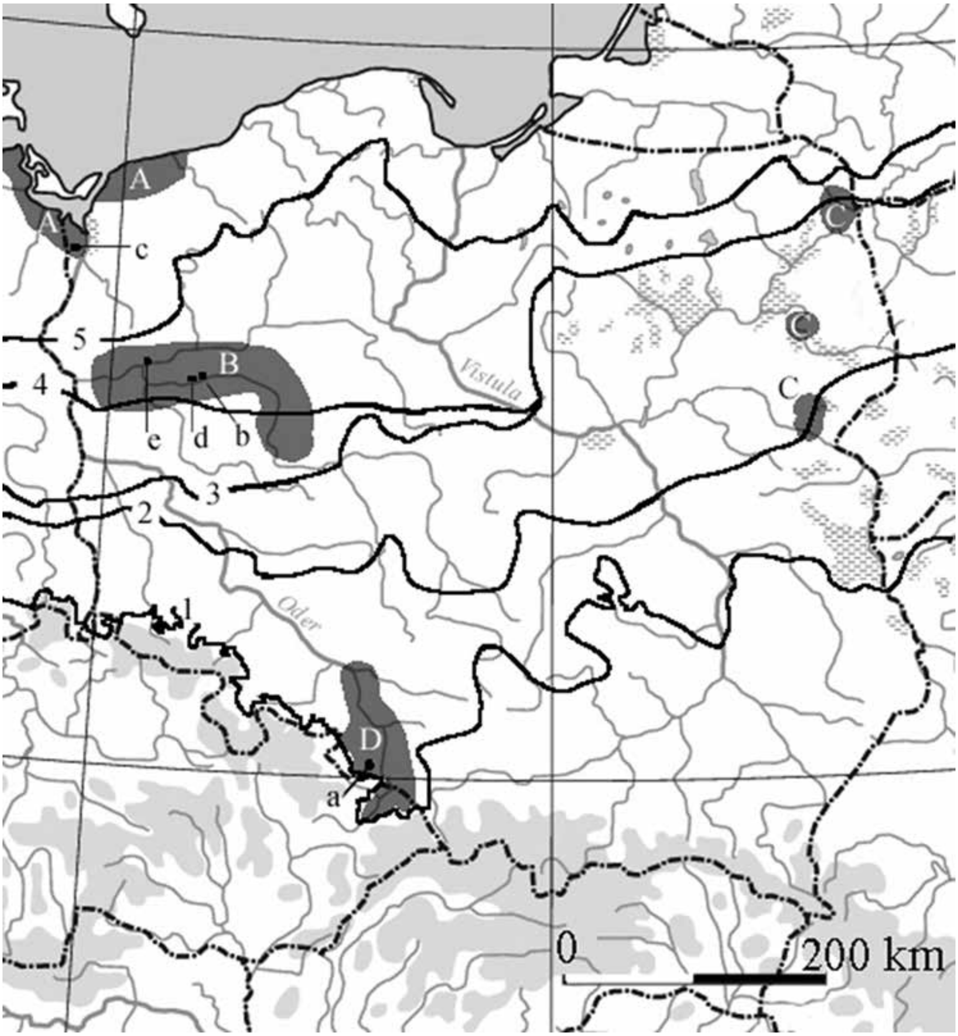

Fig. 1. Location of concentration of glacial rafts with Cretaceous flint (A, C) and concentration of glacial rafts with Cretaceous flint (B, D). 1 – Odra Glaciation (maximum of the Riss), 2 – Warta Glaciation, 3 – Leszno Stadial of the Vistulian, 4 – Poznaństadaial of the Vistulian, 5 – Pomeranian of the Vistulian; location of investigated flint samples: a – Racibórz, district of Racibórz, b – Radgoszcz, Międzychód district, c – Warnik, Police district, d – Łężce, Międzychód district, e – Gorzów Wielkopolski, Gorzów Wielkopolski district.

Owing to glacial movements, moraine deposits covering the bedrock contain rocks from different sources. Unlike comparatively well-defined sources of crystalline and sedimentary erratic from Scandinavia deposited by a glacier in the territory of present-day Poland

(Górska-Zabielska 2008), the detailed localisation of primary sources of Erratic flints in Poland is not yet clear.

Two kinds of sources of Cretaceous flint have been recognised on the Polish Lowland. The first has been found in glacial rafts (Wilczyński 1962: 9) and the second, owing to its rich occurrence in fluvioglacial and till deposits or just on the surface (Bobrowski and Sobkowiak-Tabaka 2012), is referred to as erratic flint (Krukowski 1920). As to the former, Cretaceous flint is known from a few sites in north-western Poland, e.g. from a quarry in Trzciągowo, Kamień Pomorski district, where grey or greyish nodules of flint with lenses or thin layers of chalcedony and opal containing sponge spicule, foraminifera and *Pithonela ovalis,* dated to the Upper Turonian (Alexandrowicz 1966: 30–31) occur. In Western Pomerania *in situ* deposits of Jurassic flint also have been identified. Fragments and nodules of black flints have been identified in limestone layers dated to Upper Jurassic times in quarries in Czarnogłowy, Świętoszewo, Goleniów district, Kłęby and Kamień Pomorski, Kamień Pomorski district (Czekalska and Krygowski 1957: 354; Wilczyński 1962: 30).

The dispersion of secondary sources of Cretaceous flints in Quaternary formations is closely related to Pleistocene glacial activity (Fig. 1). Rich occurrences of lithic raw materials are known from the Toruń-Eberswald marginal-ice valley, the central Warta Valley, and the region of the Silesian-Moravian border. Widely distributed, these raw flints vary in size, colour, and the presence of cortex, and, having been possibly transported by an ice sheet from several geographical locations, they come from various geological periods. This is particularly true for western Polish materials (Bobrowski 2009: 143–145; Högberg and Olausson 2007: 17).

GEOCHEMICAL ANALYSIS OF FLINT IN POLAND

The Vistula basin area is rich in siliceous rocks and it contains deposits of several varieties of flint. A 'chocolate', grey white-spotted, a striped (banded) variety, Volhynian flint on its eastern borders, and erratic flint, the latter occurring mostly in secondary deposits, were all widely used in prehistoric times.

Previous attempts at linking lithic artefacts recovered from archaeological sites to particular flint deposits have met with limited success. Thus far, the description of siliceous rocks occurring in Poland has been mostly confined to macroscopic observations (Krukowski 1920, 1922; Samsonowicz 1923; Schild 1971, 1976; Balcer 1976; Kaczanowska and Kozłowski 1976; Kaczanowska and Lech 1976; Kozłowski and Pawlikowski 1979; Lech 1980; Pelisiak 1987; Budziszewski and Michniak 1989; Michniak 1989; Schild and Sulgostowska 1997; Kamińska-Szymczak and Szymczak 2002; Pieńkowski and Gutkowski 2004; Krajcarz and Krajcarz 2009; Přichystal 2009, 2013; Krajcarz *et al.*, 2012) and classification was based typically on the colour of raw flint, the size of organic inclusions, transparency, the character of fracture, the shape of a nodule and the type of cortex (Schild 1971, 1976).

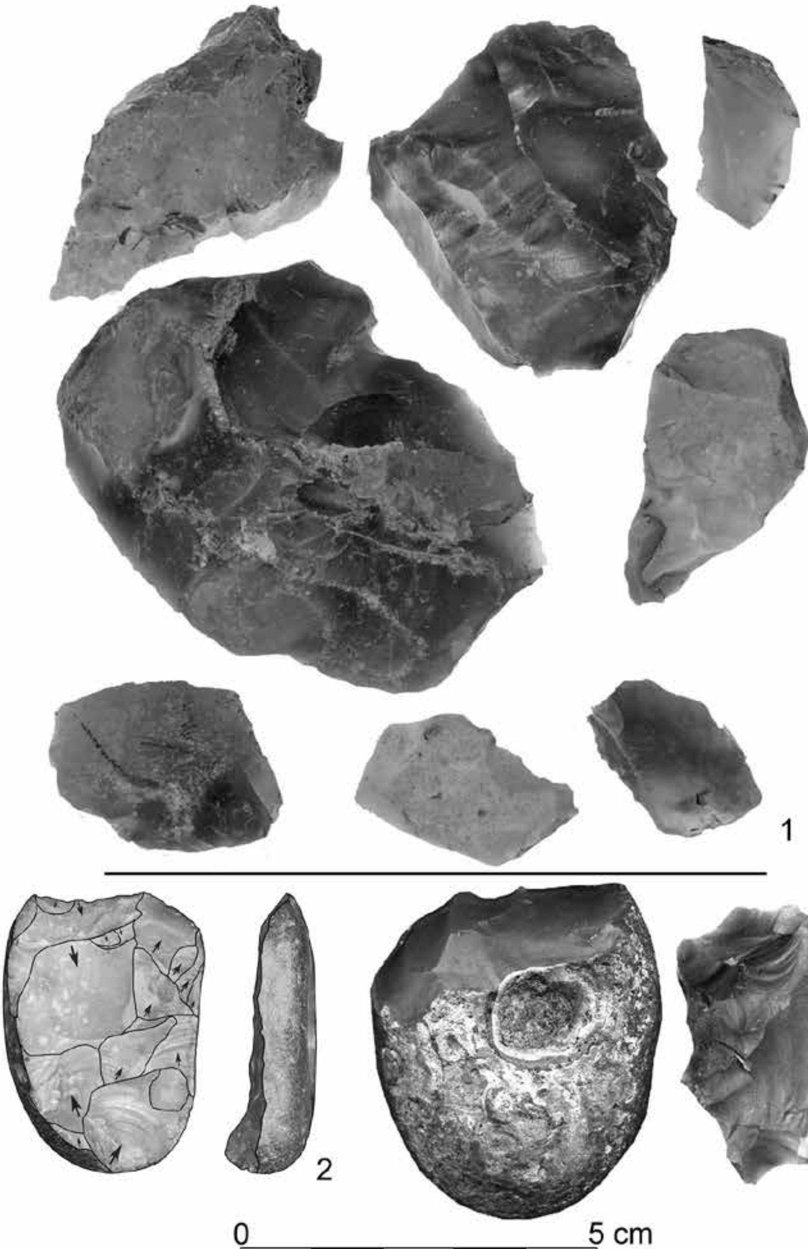

Fig. 2. Erratic flint samples: 1 – nodules of erratic flint from Chrzypsko Wielkie, Międzychód district (photo by P. Szejnoga); pebbles of Pomeranian flint (the so called *swallow eggs*); 2 – Kopydłowo, site 6, Konin district (photo by M. Gembicki); 3 – Dąbki, site 9, Sławno district. Photo: A. Tabaka.

In some cases, a general microscopic analysis of thin sections of flint samples by polarized light (Schild 1971; Přichystal 2009; 2013) and scanning electron microscopic studies (Kamińska-Szymczak and Szymczak 2002; Kamińska *et al.*, 1993) also were carried out.

To overcome some of the shortcomings of macroscopic approaches, a range of petrographic and geochemical methods were employed to define the diagnostic features of siliceous rocks (e.g. de Sieveking *et al.*, 1972; de Sieveking and Hart 1986; de Sieveking and Newcomer 1987). In recent years, a project has been developed using various instrumental methods to distinguish different varieties of siliceous rocks, with special attention devoted to a more precise description of diagnostic features of 'chocolate' flint. Supported by funds from the National Science Centre in Poland (PRELUDIUM 2; UMO-2011/03/N/HS3/03973), the project has employed several different methods, i.e., the Scanning Electron Microscope (SEM), analyses of molecular composition of organic compounds, Cathodoluminescence, energy dispersive X-ray fluorescence (EDXRF), Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS), Electron Probe Micro Analysis (EPMA) and a micropaleontological analysis (Grafka *et al.*, 2014; Werra and Siuda 2015; Grafka *et al.*, 2015; Brandl *et al.*, in print).

Until the present pilot study, erratic flint has never been a specific subject of research, although some limited analyses have been published (Kozłowski and Pawlikowski 1989; Kamińska-Szymczak and Szymczak 2002: 302–303). Even in a recent publication, Antonín Přichystal presents only materials from Maków (Racibórz district; Přichystal 2013: 101). This perhaps stems from the fact that among several varieties of flint used by prehistoric people inhabiting the region of present-day Poland, erratic flint is the most challenging raw material because of its high variability in colour, the presence of fossil microorganisms and heterogeneous composition. Last but not least, its sources are unknown (see Instrumental Analysis section below).

Several visual classifications of Erratic flint have been presented so far, based mostly on size, colour, the presence or lack of microfossils (e.g. Bryzoe) and other properties (e.g. Balcer 1983: 49–50; Dmochowski 2006; Jurys 2006). In general, two main types of Cretaceous flint have been distinguished: the so-called variant A – bluish-grey nodules (Fig. 2.1), and variant B – Pomeranian flint in the form of pebbles (the so-called *swallow eggs*), yellowish-brown or pink in colour (Balcer 1983: 49–51; Fig. 2.2–3).

INSTRUMENTAL ANALYSIS[1]

Five erratic flint samples were selected for intrumental analysis based on the homogeneity or heterogeneity of the material, a uniform flat surface, and colour (Table 1, Fig. 1).

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[1] The analysis were funded by the National Science Centre in Poland (PRELUDIUM 2; UMO-2011/03/N/HS3/03973).

The samples did not have inclusions or patina (see Hughes *et al.*, 2012: 781, 786). For the EPMA analysis, thin sections were prepared from flint from Radgoszcz, Międzychód district. To undertake the SEM analysis, small flakes of flint not larger than 20 mm in diameter were required. We used flint samples from Racibórz, Racibórz district, Radgoszcz, Międzychód district, and Łężce, Miedzychód district. Specially prepared samples from all five types of flint (Racibórz, Racibórz district, Radgoszcz, Międzychód district, and Łężce, Międzychód district, Warnik, Police district, and Gorzów Wielkopolski, Gorzów Wielkopolski district) were subject to EDXRF analysis. The samples analyzed by EDXRF were cut in cubes, 30 mm in diameter and at least 5 mm thick.

Table 1. List of Erratic flint geological samples from Poland presented in text.

Lab no.	Location	Origin of the sample
NB 1	Racibórz, Racibórz district, Silesia Province	Archaeological site no. 423
NB 2	Radgoszcz, Międzychód district, Wielkopolska Province	From the surface
NB 3	Warnik, Police district, West Pomerania Province	From the surface
NB 4	Łężce, Miedzychód district, Wielkopolska Province	Gravel-pit
NB 5	Gorzów Wielkopolski, Gorzów Wielkopolski district, Lubuskie Province	From the surface

Three methods were employed to examine the chemical distinctions between erratic flint samples from different parts of present-day Poland, deposited in secondary resources.

The Scanning Electron Method (SEM) is useful for the analysis of materials that are too small to be resolved by light microscopy. It provides spot microchemical data on uniform grains, heterogeneous rock fragments and soils, coatings and other pedofeatures (Goldberg and Macphail 2011: 362).

The Electron Probe Micro Analysis (EPMA) provides a high accuracy qualitative and quantitative chemical microanalysis and helps identify accessory elements. Polished blocks or thin sections are analysed in the electron microprobe (Goldberg and Macphail 2011: 363). The chemical composition in micro area in thin sections was analysed in the Laboratory of Electro Microprobe in the Institute on Mineralogy, Geochemistry and Petrology, Geology Department, Warsaw University, using EMPA – Elecron Probe Microanalyser produced by Cameca. All analyses were conducted by

Fig. 3. Core from Ośno Lubuskie, site 7, Słubice district, with visible inclusions. Photo: A. Tabaka.

Lidia Jeżak, and their results interpreted by Rafał Siuda from the Institute of Mineralogy, Geochemistry and Petrology (Faculty of Geology, University of Warsaw).

The Energy-Dispersive X-ray Fluorescence analysis (ED XRF) seeks to determine the quantitative composition of nine major, minor and some trace elements. This non-destructive method offers high precision for certain constituent elements, but care needs to be taken to avoid inclusions, e.g. fossils or calcium elements from sea creatures' shells (Fig. 3, 4). which might influence the measurements determined. It is noteworthy in this context that sedimentary rocks, such as flints, exhibit a fairly heterogeneous composition (Hughes *et al.*, 2010, 2012).

THE RESULTS OF INSTRUMENTAL ANALYSIS

SEM and EMPA

Results of the SEM and EMPA analysis are complementary and as such will be presented together.

Quartz

The main mineral component of Erratic flint is quartz (as a chalcedony), developed as cryptocrystalline clusters. Transmitted light sometimes shows parallel arrangements of its thin needle crystals. The presence of massive quartz aggregates (up to 100 μm in size)

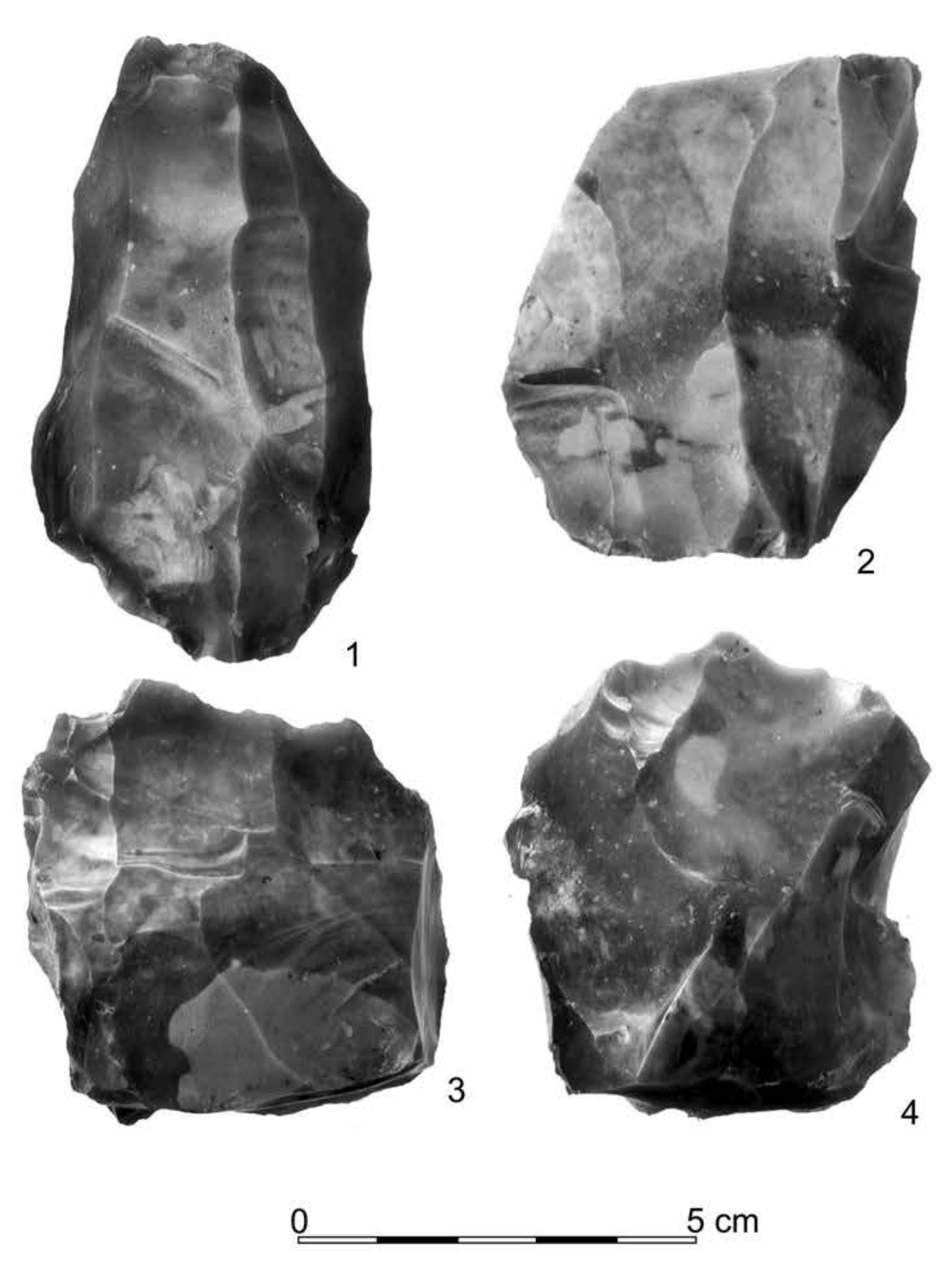

Fig. 4. Cores and perforator from Myszęcin, site 19, Świebodzin district, with visible variety of colour. Photo: P. Szejnoga.

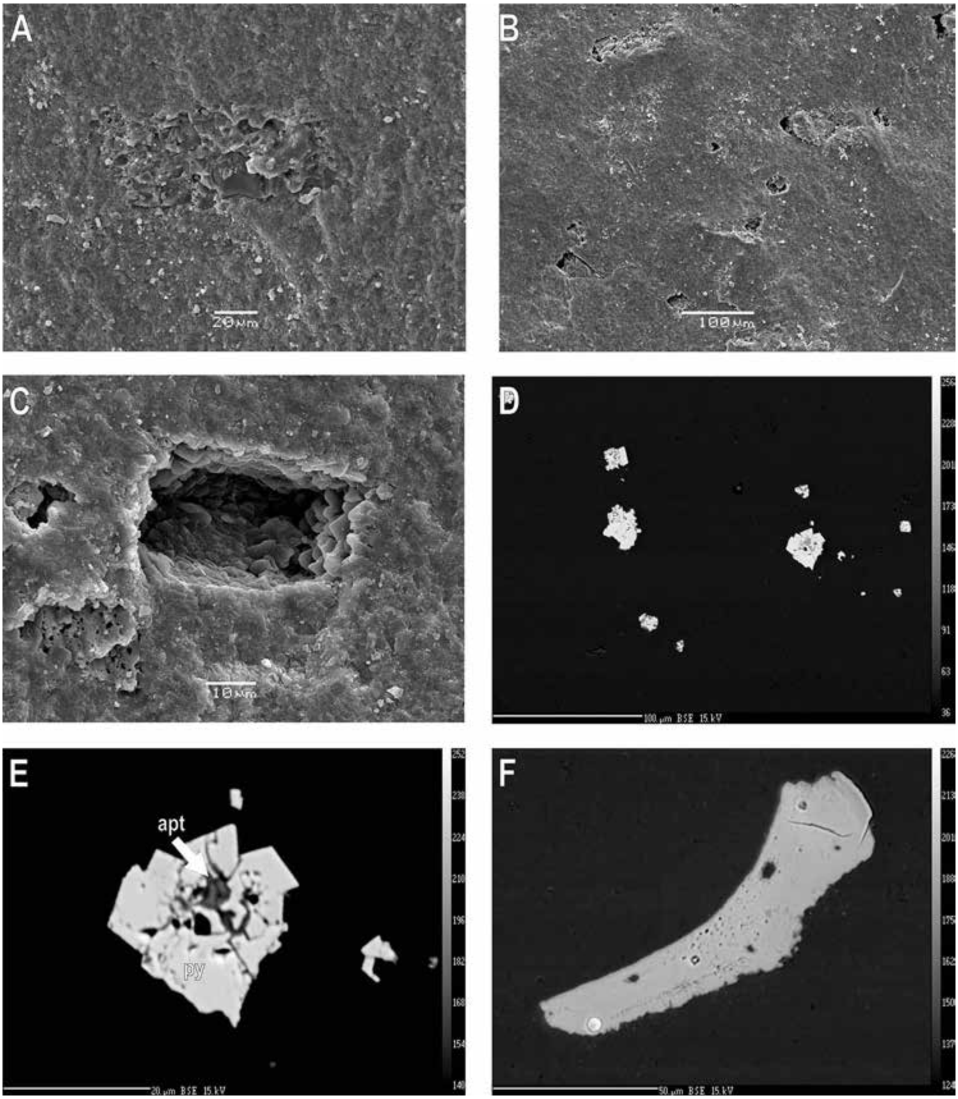

Fig. 5. SEM and BSE images of Erratic flint geological samples. A: SEM image of quartz aggregates; B: SEM image of small cavities in chalcedony matrix; C: SEM image of void with quartz automorphic crystals; D: BSE image of pyrite aggregates; E: BES image of pyrite aggregate with apatite (apt); F: BSE image of fragments of fish skeletal elements. Photo: R. Siuda.

has been observed in some cases. The formation of these aggregates is associated with recrystallization of chalcedony pseudomorphs after the organic remains (Fig. 5A). The last type of quartz contains small hipidiomorphic quartz crystals, reaching up to 5 µm, growing on the walls of small voids scattered in the chalcedony matrix (Fig. 5B, 5C).

Pyrite

The most common accessory mineral in flint, pyrite has been found in all analysed samples. The mineral forms fine automorphic crystals, up to 10 mm in size, such as cubes or octahedrons. Pyrite crystals form specific spherical aggregates, up to 20 mm in diameter (Fig. 5D). Residually preserved apatite aggregates are sometimes present in the central parts of pyrite aggregates (Fig. 5E).

Apatite

Apatite was identified only in one sample of Erratic flint. In flint from Radgoszcz (NB–2), this phase comes in two varieties. Apatite I builds fragments of fish skeletal elements (Fig. 5F), while Apatite II forms irregular aggregates up to 25 μm in size (Fig. 6A). Formation of this latter type of apatite is connected with dissolution of apatite I during the diagenetic processes and formation of flint nodules. In this case, apatite II co-occurs with small amounts of pyrite. Tiny apatite aggregates also are present in central parts of aggregates of pyrite (Fig. 5E). Due to the high porosity of the aggregates of apatite and the instability of the mineral under the electron beam it was not possible to perform micro probe analyses of this phase.

Calcite

Calcite is fairly frequent in Erratic flint. It forms very small aggregates, up 5 μm in diameter, irregularly dispersed in the chalcedony matrix of flint. Calcite identified in the analysed material represents the remains of calcium carbonate, which originally built a calcite sediment within which the flint concretions was formed.

Pseudomorphoses

Very common in the analysed samples are organic debris completely replaced by quartz. These usually include fragments of dinocysts (Fig. 6B), or silicified needles of sponges (Fig. 6C). Sometimes the sponge needles are perfectly preserved, with a visible ornamentation of the spicules. In many cases, the process of replacing microfossils by quartz is so extensive that it is not possible to accurately assign organic remains to a specific group of organisms. Perfectly preserved voids left after leached calcite crystals provide another type of pseudomorphoses (Fig. 6D). Up to 10 μm in size, they show a rhombohedral structure, typical for calcium carbonate. Their walls are overgrown by automorphic quartz crystals.

ENERGY-DISPERSIVE X-RAY FLUORESCENCE ANALYSIS (EDXRF)

As noted in previous studies using non-destructive EDXRF analysis (Hughes *et al.*, 2010, 2011, 2012), contrasts in calcium and iron composition help differentiate among and between different chemical varieties of flint. In this preliminary study, the CaO vs.

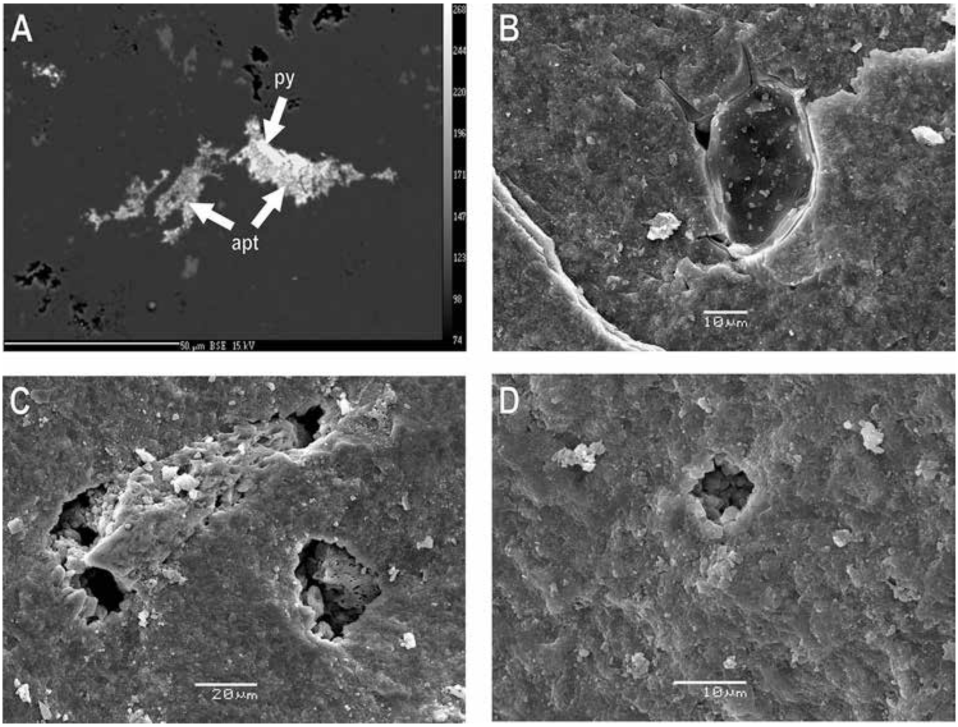

Fig. 6. SEM and BSE images of Erratic flint geological samples. A: BSE image of apatite II and pyrite intergrowths; B: quartz pseudomorph after dinocyste; C: quartz pseudomorph after urchin spike; D: void after calcite crystal dissolution. Photo: R. Siuda.

Fe composition determined for a small sample of erratic flint was compared with that of 'chocolate' flint from four different localities (Orońsko, Tomaszów, Szydłowiec distrit, Wierzbica and Prędocin, Radom district) in the Vistula River basin of south eastern Poland (Fig. 7). As this figure shows, the erratic flints analysed contain significantly lower iron composition than 'chocolate' flint, and the CaO composition for two of the four specimens analysed is greater than measured in 'chocolate' flint.

CONCLUSIONS

Because of its heterogeneous chemical composition, flint is notoriously difficult to source. This is particularly evident in the case of Erratic flint. The results of SEM and EMPA analyses have shown that the mineral composition of investigated samples is largely homogeneous and undiversified. Accessory minerals present in the composition (pyrite, apatite, calcite) are typical of other variants of flint used in the territory of

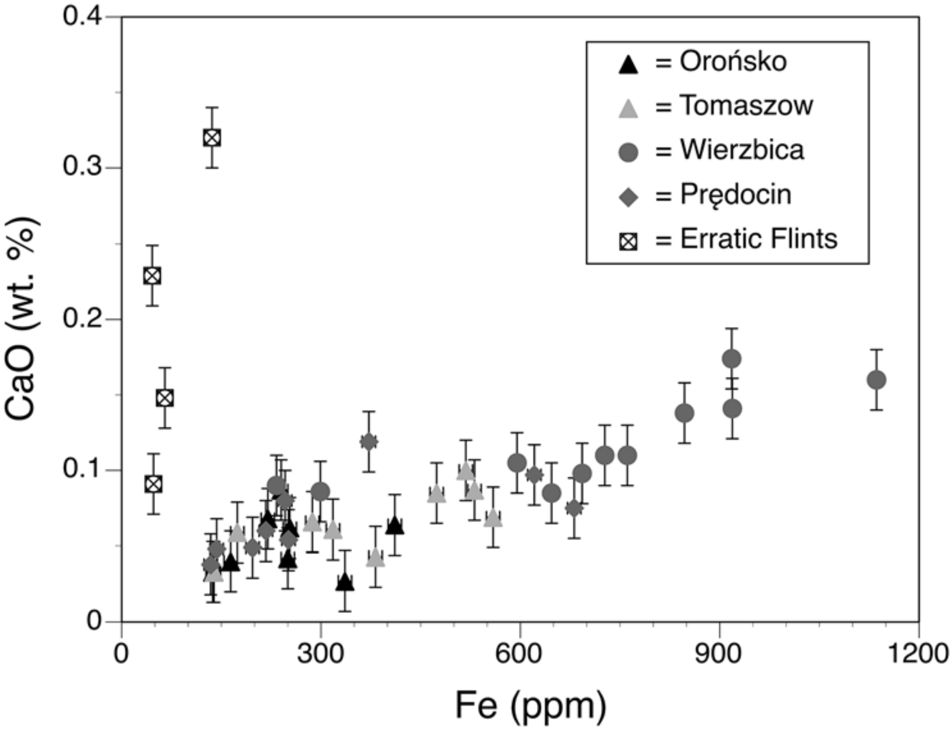

Fig. 7. The CaO versus Fe composition of 'chocolate' and Erratic flint geological samples from Poland.

present-day Poland. The differentiation between single variants of erratic flint by testing their mineral composition using the electron microscope and chemical composition of minerals in microprobe is therefore impossible. The similarity between single variants is caused by primary conditions of sedimentation and formation of flint nodules.

Of the laboratory-based instrumental analyses we have undertaken to date, only EDXRF analysis has offered some hope for differentiating between and among varieties of erratic flint. These pilot results are encouraging insofar as intersource discrimination is concerned, but additional erratic flint specimens need to be analysed before we can identify the range of chemical composition they contain.

ACKNOWLEDGEMENTS

We would like to thank Dr Przemysław Bobrowski (Institute of Archaeology and Ethnology PAS) and Marcin Dziewanowski M. A. (Adam Mickiewicz University) for supplying the samples for analysis.

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