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Characterizing 'Chocolate' Flint Using Reflectance Spectroscopy

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The study details a pilot experiment in which samples of 'chocolate' flint from four procurement sites in Poland and chert from the United States were characterized spectrally and distinguished using reflectance spectroscopy and multivariate statistics. The characterization of 'chocolate' flint and the successful differentiation of sources has been, and continues to be, a major research focus for understanding prehistoric consumption, use, and distribution of this favored lithic resource. Reflectance spectroscopy potentially provides an analytical methodology for identifying artefact source by successfully distinguishing spatially and compositionally unique deposits. Initial results from the study show that 'chocolate' flint can be distinguished from other silicite tool stone resources, regional lookalike materials, and by individual deposit. Future studies will test a more robust sample size of 'chocolate' flints and conduct experiments on surface weathering

KEY-WORDS: reflectance spectroscopy, 'chocolate' flint, source in Poland, Visible Near-infrared (VNIR), Fourier Transform Infrared (FTIR)

INTRODUCTION

The following study is a preliminary experiment on the application of reflectance spectroscopy to characterizing variability in 'chocolate' flint at two scales of analysis, between type formations and within a type formation. Recent studies highlight the possibility of characterizing 'chocolate' flint to a degree benefiting provenance research (Přichystal 2013; Brandl *et al.,* 2016). The current study largely builds upon the previous research and positive results presented in Parish (2016a) that demonstrated the ability of reflectance spectroscopy to differentiate between a large sample of cherts outcropping across the Southeastern United States of America. In order to assess the application of reflectance spectroscopy as a provenance technique in other regions, a preliminary sample of 'chocolate' flint was analysed. However, the results of the study should first be contextualised within a theoretical and methodological framework.

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Provenance Theory

The 'Provenance Postulate' is lauded as the fundamental principle guiding current archaeological source studies. The provenance postulate stipulates that in order for a deposit to be distinguished from all other potential sources, the variability found within the deposit must not exceed the variability found outside it (Banks and Wigand 1978). Though originally articulated in the context of ceramic provenance studies, the postulate is equally applicable to lithic provenance studies. Three main related components of this fundamental principle can be identified, spatial scale, variation, and characterization.

The spatial scale of the provenance study is important to define and is determined by the human behavioural question. The spatial scale of the study could range from an examination of inherent variation with a single artefact, deposit/outcrop, or geological formation. At different scales of analysis, the nature of variation may range considerably from homogeneous to heterogeneous. It can be expected that the variation within a single artefact would be less than that found within a deposit, and again that found within an entire geological formation, though analyses may show that such assumptions may in some cases be unfounded. Finally, the postulate stipulates the need to characterize the range of variation at the spatial scale in question in order to successfully determine if the source can be differentiated between others in the sampling universe. Therefore, it is these 'characterizations of variation' or brackets of variation ranges which the provenance researcher is attempting to distinguish and subsequently match the artefact's characterization to.

The characterization of the range of variation at various spatial scales (i.e. artefact, deposit, formation) and differentiation of the source(s) are the goals regardless of the provenance method utilized. The ranges of variation are important to know, irrespective of whether the researcher is using macroscopic attribute analysis, petrographic, or geochemical techniques. The three main components of the provenance postulate dictate the sampling strategy, the technique utilized, and the statistical methods of the provenance study. However, it is necessary to broaden our understanding of not only the presence of variation within the lithic source but also the nature of the variation.

Mechanism of Variation

The paleodepositional environment at the time of deposit formation and subsequent post-depositional alterations provide the researcher with a mechanism to study variation in lithic sources. The unique climate, geological conditions, and environment at the time of deposition impart the lithic source with a potentially diagnostic signature. This environmental signature in the form of chemistry, mineralogy, crystalline structure, … etc. within the parent formation may be inherited by the secondary forming lithic material or primary deposit of silica. In this manner, terrestrial and marine inputs may be spatially unique. The depositional signature in lithic sources is further altered regionally by weathering. The diagenetic process of flint may also be regionally specific. Though beyond the scope of the current study, further research in the areas of paleodepositional environments and postdepostional processes is warranted in order to understand the possible means by which one lithic source is differentiated from another.

The term silicite is used here to denote sedimentary siliceous rocks formed as a result of biochemical, chemical, or diagenetic precipitation of SiO_2 (Přichystal 2010). Silicite occurs as nodules forming in void spaces or as bedded planes and lenses in carbonate formations such as limestone and dolomite. The Kimmeridgian/Oxfordian 'chocolate' flint, Jurassic-Cracow flint, and Fort Payne chert discussed in the study are categorized broadly as silicite.

'Chocolate' Flint

Much literature is available on the geology of the 'chocolate' flint in preceding volumes (Krukowski 1920; Budziszewski and Michniak 1995; Hughes *et al.*, 2016). However, a brief summary is given as context in order to examine the potential reason why spectral variation within 'chocolate' flint is spatially unique.

'Chocolate' flint occurs and in prehistoric times was exploited along the northeastern slopes of the Świętokrzyskie (Holy Cross) Mountains of central Poland. The irregular, flat, and tabular nodules occur in late Jurassic carbonate deposits (Schild 1971; Hughes *et al.*, 2016). The carbonate deposits were laid down in shallow marine environments near the shore. The various cycles of silica formation occurred sporadically as shorelines stabilized in between transgression and regression phases (Budziszewski and Michniak 1995: 11). The precise parent formation for 'chocolate' flint is currently elusive, either being at the top margins of the Oxfordian, the base of the Kimmeridgian, a transitional boundary in between, or occurring in both limestone formations (Hughes *et al.*, 2016). Regardless of the position of the 'chocolate' flints in the regional geological sequence, the shallowing marine reservoir environment created a sedimentary boundary between the 'chocolate' flint horizon and the overlying banded Turonian flints (Budziszewski and Michniak 1995). The shifting terrestrial contribution, sea chemistry, and temperature regimes were only a few of the variables affecting the diagenetic process of flint formation. It is possible that more localized deposits, later exploited in prehistoric times, are internally more compositionally homogeneous due to their proximity to similar formation conditions and later alterations.

The 'chocolate' flint deposits were heavily utilized as a lithic source both regionally and much further afield. The first reported use of the lithic resources is thought to have been by communities in the Middle Palaeolithic (Schild ed., 2005) but continue to the end of the Bronze Age (Schild *et al.*, 1985). The utilization of 'chocolate' flint reflects a much broader spatial and temporal trend in the region as other lithic sources shared a similarly long period of use and distribution. The widespread distribution, consumption, and utilization of 'chocolate' flint outcropping in a relatively small geographic area makes the development of an accurate sourcing technique desirable.

Fort Payne chert

Fort Payne chert is found in the Fort Payne formation of the Southeastern United States. This is a carbonate formation laid down during the Mississippian geological subsystem (354–324 mya) at a time when much of the interior United States was covered by an inland sea. The internal structure of Fort Payne chert is very uniform, composed of cryptocrystalline silica with small amounts of chalcedonic silica and irregularly shaped pherulites (Marcher 1962). A large amount of iron oxides and brown organic material are present distinguishing it from the parent limestone. The material occurs in two forms. One type of Fort Payne chert occurs as large rounded masses of dense, dark chert. The second type occurs closer to the top of the formation and is described as highly porous, fossiliferous chert identical to the overlying Warsaw formation (Marcher 1962).

Fort Payne chert was an important lithic resource for the entire prehistoric record of the Southeastern United States. The chert was first exploited by late Pleistocene hunter-gatherer inhabitants and utilized by subsequent culture groups up until the 19th Century. The medium to fine white, tan, brown, and black-grained chert contains significant variability across deposits spanning 1500 linear kilometres. Despite efforts to distinguish Fort Payne chert qualitatively and quantitatively, the large amount of variability has frustrated provenance researchers until the application of reflectance spectroscopy.

Reflectance spectroscopy

Reflectance spectroscopy is a broad term describing any method that studies and records the interaction of electromagnetic radiation with matter. The description of reflectance spectroscopy in general is covered more thoroughly in Clark 1999; Hollas 2002; Smith 2011; and specifically applied to archaeological sourcing studies in Hawkins *et al.*, 2008; Morin 2012; Parish and Butler 2017. However, it is important to note that reflectance spectroscopy data is a measurement of the amount of radiation reflected from the surface and near-surface of a specimen by wavelength across a portion of the electromagnetic spectrum.

Reflectance data is expressed as a percentage reflectance value between 0 and 1 per wavelength unit. For example, a specimen of 'chocolate' flint might have a reflectance value of 0.058234 at wavelength 350 nm. Each reflectance value per wavelength is recorded by the spectrometer generating thousands of values. The current study uses two spectrometers whose range extends from 350 to 15,419 nm, encompassing the visible, near and into the middle infrared regions of the electromagnetic spectrum.

Expressed graphically, each sample's spectrum is a series of curves with peaks and valleys (see Parish 2016a: Fig. 1). A spectrum's peaks and valleys (features) at specific wavelength positions are indicative of atomic electron shield structure and molecular bonding. The presence of identifiable spectral features is indicative of mineral composition and slight changes in the silica matrix. Reflectance spectroscopy is very sensitive to minute compositional features and, when coupled with multivariate statistical techniques, is proving to be an effective, cost efficient, and fast method for accurately sourcing silicite tool stone resources. One of reflectance spectroscopy's more valuable potential characteristics is that analysis is non-destructive to the artefact.

METHODS

'Chocolate' flint has to date been analysed by a few different methods (Přichystal 2013; Grafka *et al.* 2015; Brandl *et al.*, 2016; Hughes *et al.* 2016; Werra *et al.* 2018). The most useful results were obtained thanks to the use of Multi-Layered Chert Sourcing Approach (MLA) by Michael Brandl (Brandl *et al.*, 2016) and stereomicroscopic appearance made by Antonín Přichystal (2013: 108).

Experiment design

In order to examine the possible application of reflectance spectroscopy to characterize and differentiate 'chocolate' flint from similar varieties of Jurassic-Cracow flint and 'chocolate' flint from the Udorka valley, two spectrometers were used to analyse a comparative sample of these and a Fort Payne chert control group. A total of 34 samples were analyzed of 'chocolate' flint from Wierzbica 'Zele', Radom distr. (n=11), Orońsko, Szydłowiec distr. (n=13), and Polany II, Radom distr. (n=10). In addition 11 samples of Jurassic-Cracow flint from Sąspów, Cracow distr., and 30 samples of Fort Payne chert from the Southeastern United States of America were analysed as lookalike controls. Therefore, the reflectance spectra of 75 flint/chert samples were recorded in order to test whether enough spectral variability existed to distinguish one material type from another and one deposit from another.

All samples were fractured via hard hammerstone percussion producing an interior less weathered surface for analysis. Just prior to analysis, a lens tissue was used to lightly wipe any dust or contaminants from the sample's surface. The two spectrometers used to gather spectral data included a PSR+ (made by Spectral Evolution), recorded radiation in the visible and near-infrared, and a FTIR 4300 (made by Aligent) in the middle-infrared. The use of both spectrometers provided high-resolution spectral data from 350 to 15,419 nm, or 3,051 raw reflectance values per sample analysed. A total of 228,825 reflectance values were collected from the 75 geological samples, each reflectance value potentially diagnostic to parent formation and/or deposit location.

Processing of the spectra was necessary due to sample surface to probe angular differences, instrument and atmospheric noise, and spectral feature intensity differences. A more thorough discussion on spectral processing can be found in Parish *et al.,* 2013; Morin 2012, and Smith 2011. The current study utilized spectroscopic, normative, and derivative transforms. The spectroscopic transform is used to convert the reflectance values into absorption in order to highlight subtle features. The normative transform minimized sample surface to probe angle differences by providing a baseline correction, in other words, standardizing the 75 flint/chert spectra. Finally, the derivative transform function was utilized in order to smooth atmospheric and instrumental noise and to highlight subtle slope change features in the spectra indicative of slight compositional characteristics.

Visual comparison of the graphic depiction of the results from each flint type and each deposit type is not often informative; this is because the spectrum of flint, compositionally dominated by silica, is similar (Fig. 1). Therefore, a stepwise discriminant function model was run to create discriminant functions between flint/chert

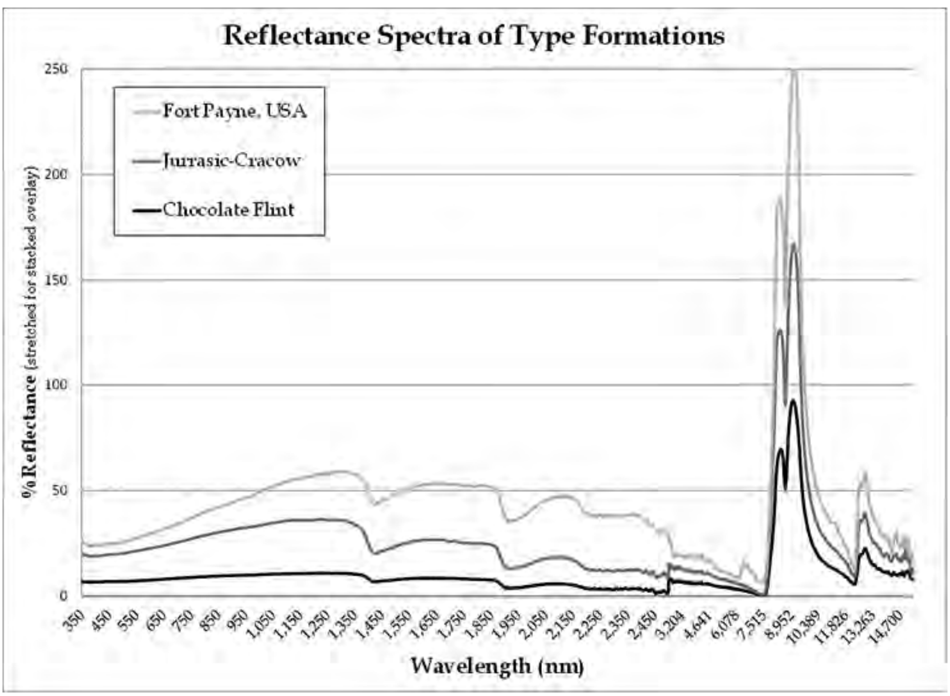

Fig. 1. Overlayed spectra in the visible, near, and middle-infrared of the three material types analysed in the study. Note the similarity of the spectral features. Spectra stacked for visibility. Computer graphics: R.M. Parish.

types and individual deposits. The stepwise discriminant function assesses the power of each reflectance value (variable) in the spectrum of a sample for group membership. An f-value of the reflectance variable is calculated and if the f-value exceeds a set threshold than the variable is not retained in the resulting function. As a result, only the most discriminatory reflectance values are used to calculate group membership. The model generates a predicted group membership nominal variable, discriminant function scores, and a scatter plot. This multivariate statistical method is demonstrated to be effective in analysis of flint/chert spectra though best applied to larger sample sets (Speer 2013, 2014; Parish 2016b).

The initial test examined whether Kimmeridgian/Oxfordian formation 'chocolate' flint could be statistically distinguished from Jurassic-Cracow flint and Fort Payne chert. The significance of the initial test was to gauge inter-formation variation between three flint/chert sources forming under similar geological conditions in marine carbonate formations. Though separated by space and time, the base mineralogy of the tool stone materials was largely the same, being composed of micro to cryptocrystalline quartz grains. The experiment was designed to characterize the spectral variation found within separate geological units. The second test examined the intra-formation variation within the Kimmeridgian/Oxfordian parent formation(s). The Jurassic-Cracow samples from the Sąspów deposit were retained for a regional control. The goal of the intra-formation experiment was to characterize variation within individual deposits (Wierzbica 'Zele', Orońsko, Polany II) of 'chocolate' flint and to distinguish one from the other using spectral data.

The two preliminary experiments were a necessary first step approach in assessing the nature of variation between and within formation types. The two tests also examined the ability of reflectance spectroscopy to characterize this variability at two scales of analysis. Finally, the application of discriminant function analysis in differentiating the tool stone materials statistically was evaluated as a mechanism to quantify the atomic and molecular bonding differences. The results of these experiments highlighted the significant results that could be attained by the application of reflectance spectroscopy in distinguishing between look-alike flint types in the Świętokrzyskie (Holy Cross) Mountains of central Poland.

RESULTS AND DISCUSSION

Inter-formation

All 75 flint/chert samples were organized into three groups; Kimmeridgian/Oxfordian formation 'chocolate' flint, Jurassic-Cracow flint and Fort Payne chert. The discriminant function model successfully assigned each sample to its respective group based upon the diagnostic reflectance values retained in the step-wise model. No samples

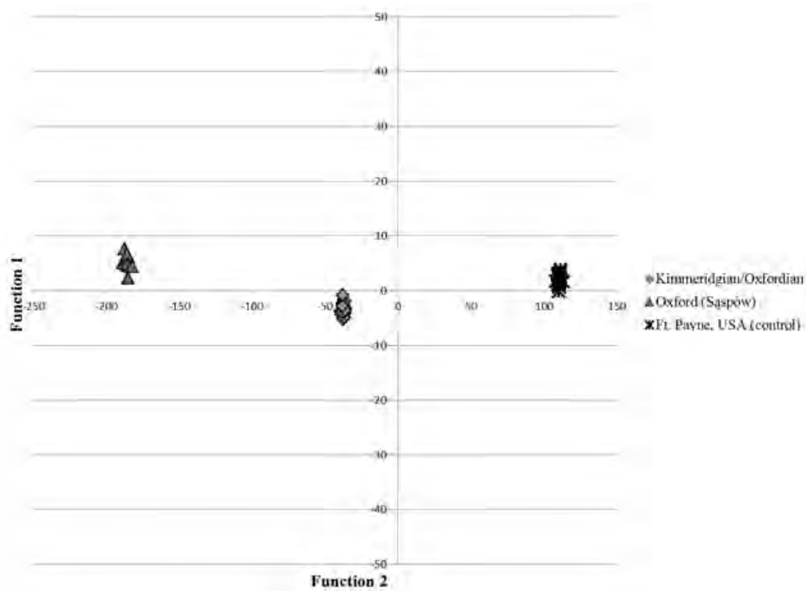

Fig. 2. Discriminant Function scatter plot depicted clear separation between all three formation type materials analysed in the study. Computer graphics: R.M. Parish.

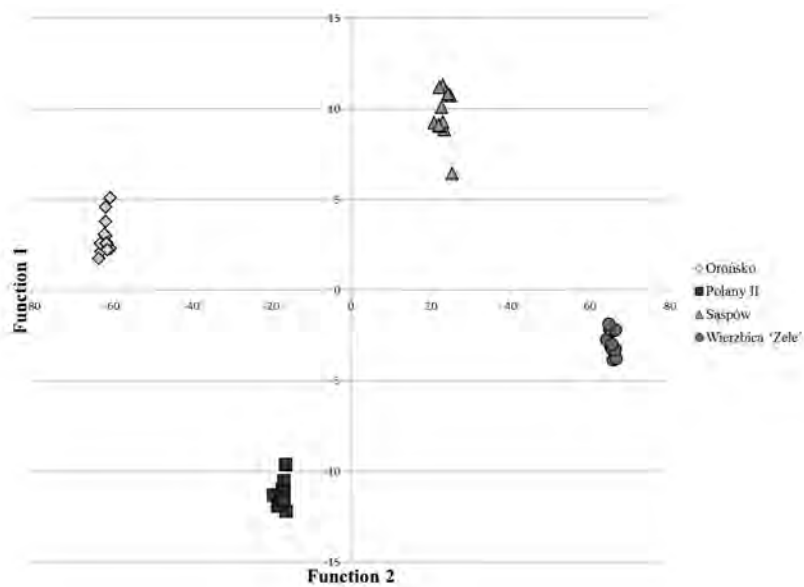

Fig. 3. Discriminant function scatter plot depicting clear separation between all three deposits of 'chocolate' flint (Orońsko, Szydłowiec distr., Wierzbica 'Zele' and Polany II, Radom distr.) and Jurassic-Cracow (Sąspów, Cracow distr.) flint. Computer graphics: R.M. Parish.

Table 1. Diagnostic reflectance wavelength (nm) variables retained in both discriminant function models.

Electromagnetic Range	Inter-formation test	Intra-outcrop test	Diagnostic Attributes Detected
Visible, Near-Infrared	556, 590, 610, 920, 1159, 1258, 1304, 1309, 1315, 1374, 1446, 1484, 1697, 1805, 1825, 1861, 1913, 1997, 2284, 2396, 2480	441, 801, 878, 892, 977, 1053, 1238, 1247, 1301, 1314, 1612, 1808, 2249, 2304, 2388	Unbound electrons, atomic structure, mineral impurities Silica phase, formation conditions Kaolinite, Illite, Montmorillonite, Limonite and other clay minerals
Middle-Infrared	2730.27197, 3635.57397, 3721.79321, 3865.49194, 4282.21533, 5417.41602, 5431.78564, 5575.48193, 5661.69971, 5776.65674, 6150.26709, 6265.22412, 6581.35596, 6753.7915, 6796.90039, 7156.14111, 7256.72852, 11222.84863, 12257.49707, 12286.2373, 12602.37988, 12760.45117, 13996.28125, 14269.31348	3132.62842, 4770.78271, 5259.3501, 6710.68262, 7601.59961, 8708.07813 8938, 9196.66211, 10073.23926, 10389.38184, 12702.9707, 13004.74316 14844.11816	Kaolinite, Illite, Montmorillonite, Organic compounds, Dolomite and Calcite, Dolomite, Smectite, Rutile, Goethite, Limonite, Glauconite, Pyrite, Manganite, Brucite, Hematite, Magnetite

were misclassified in the predicted group membership output. The model generated a scatter plot based upon function 1 and function 2 (Fig. 2). The plot shows a clear separation of the three flint/chert groups in two-dimensional vector space. Had the samples not contained enough diagnostic reflectance values, the accuracy of the model would have been severely compromised and the resulting scatter plot graph would have shown no clear separation of the sample groups. All 46 diagnostic reflectance values retained in the inter-formation model are listed in Table 1.

Intra-formation

The intra-formation model consisted of four flint groups of 45 samples from the 'chocolate' flint deposits of Wierzbica 'Zele', Orońsko, Polany II and the single group of Jurassic-Cracow samples from Sąspów. The Fort Payne chert samples were not included in this stage of the analysis. The discriminant function analysis generated a model consisting of 28 diagnostic reflectance values (Table 1) and no misclassified samples. All of the 'chocolate' flint samples were identified to their respective site deposits. The Sąspów samples were similarly classified correctly. The scatter plot of function 1 and function 2 illustrate clear group separation in two-dimensions. Therefore, the results of the model indicate that 'chocolate' flint can be distinguished from other look-alike materials and can also be distinguished from separate deposits along the strike of the prehistorically exploited geological formation.

Though the scope of the current study was small, the implication of the results is significant. The 75 samples incorporated in the study can be characterized based upon a series or range of diagnostic reflectance values through the use of multivariate statistics. Slight absorption features due to dipole molecular bonding indicate the presence of micro-mineral groups that when measured have a spatial component. Additionally, the position of the mineral impurities within the silica matrix may be related to the position of the materials within the geological formation and the conditions within which the deposit was originally deposited. Later diagenetic processes acting on the flint deposits may also have a spatial component. This means that the regional conditions and variables affecting the shallow marine deposits and later weathering of these potentially imparted a characteristic signature not previously noted by petrologic or geochemical analyses. The extreme sensitivity of reflectance spectroscopy to slight compositional and structural changes allows the differentiation of both deposits and formation types.

The reflectance spectral data do not differentiate between micro-regional weathering conditions. Though the analysis of the samples is primarily upon the surface with slight penetration to a few micromillimetres, the samples were prepared by hard-hammer percussion exposing an interior surface. It is also readily apparent that weathering conditions within a single deposit of 'chocolate' flint are variable, not uniform. Also, the samples were collected from surface debitage immediately surrounding the deposit, not from an in situ horizon exposed within the geological profile. It is however, hypothesized that the long-term diagenesis of flint, coupled with the regional deposition of the parent formation, explains the spatially distinctive spectral variation.

The relatively small sample size of the study prohibits exploring the full range of characterization by formation and by deposit. The use of discriminant function analysis is more appropriately performed on larger sample databases. Therefore, it is a bit premature to make wide ranging theoretical assumptions regarding the nature of the variation in and between the 'chocolate' flint deposits. A larger sample is needed to assess the nature and spatial patterning of the diagnostic reflectance features identified in this study and the robustness of the multivariate statistical method.

In addition to a larger sample database, future directions of the research will entail a surface weathering test that examines the affects that patina has on the accuracy of the technique for source determination. As reflectance spectroscopy has the potential to be a non-invasive provenancing method, a focused examination on analysis of artefacts compared to a geological sample database is needed. Though the characterization of flint source and differentiation of other potential sources is a necessary first step, the accurate sourcing of artefact assemblages back to the resource is the ultimate objective if an understanding of human behaviour is to be realized.

CONCLUSIONS

The study successfully demonstrated the application of reflectance spectroscopy in characterizing variation between 'chocolate' flint and Jurassic-Cracow flint sources as well as differentiating individual deposits of 'chocolate' flint along the slopes of the Świętokrzyskie (Holy Cross) Mountains of central Poland. The results demonstrate the potential for additional provenance studies the aim of which is to analytically identify the source of flint artefact assemblages. The deposits of flint investigated in the study contain a range of diagnostic reflectance values in the visible, near- and middle-infrared portions of the electromagnetic spectrum created by atomic electron transforms and molecular bonding. The spectral features are indicative of micro-mineral groups and possibly also the structure of the silica matrix. The clear separation of 'chocolate' flint by formation type and deposit may be explained by the palaeo-environmental conditions at the time of formation of the deposit and the resulting post-depositional alteration of the flint through micro-regional diagenesis. This is the fundamental principle of provenance studies; that variability in a deposit is geologically and spatially distinct from other exploited resource locations.

The positive results of the pilot study raise a great deal of optimism for the success of future sourcing programs the goal of which would be the characterization and differentiation of tool stone resources. The application of reflectance spectroscopy is shown to be a promising methodology in future provenance studies. The cost-efficient, fast, accurate, and non-destructive characteristics of reflectance spectroscopy are desirable traits in collecting source data. The PSR+ (VNIR) and 4300 FTIR devices used in the study are also portable, encouraging onsite analysis at the curation facility. Additionally, the spectral data are easily assembled into sharable databases and libraries encouraging the continuous sampling and adding of deposits across the landscape. Our broadened view of the lithic landscape will similarly expand our understanding of prehistoric resource, selection, distribution, and consumption across space and through time.

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