

# Reflectance spectroscopy as a chert sourcing method

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The non-destructive application of reflectance spectroscopy within chert provenance studies is evaluated and the implications of archaeological source determination of chert artifacts are discussed. The combined use of Visible Near-infrared (VNIR) and Fourier Transform Infrared (FTIR) reflectance spectroscopy demonstrate the accurate, fast and relatively low cost for the characterization of geological deposits of chert and the potential identification of source for chert archaeological materials. Reflectance spectroscopy gathers data on the trace and minor mineral components within a sample as identified by subtle absorption peaks and slope changes. The variable range of spectral features per sample, per deposit, per geological formation is potentially diagnostic for a geographically isolated deposit of chert. A chert sample database consisting of 2430 samples from the Midwestern and Southeastern United States is utilized to illustrate the accuracy of reflectance spectroscopy at characterizing chert deposits for archaeological use.

KEY-WORDS: reflectance spectroscopy, chert, provenance research, Southeastern United States, Visible Near-infrared (VNIR), Fourier Transform Infrared (FTIR)

## INTRODUCTION

### *Chert Sourcing*

The objective of this study is to assess the application of reflectance spectroscopy as a chert provenance technique. The accurate determination of source for raw materials utilized by past peoples provides a proxy for modeling a wide range of human behaviors. The source or provenance of the raw material utilized in the manufacture of the particular artifact allows researchers to study human behavior relating to both group and environmental interactions.

Chert provenance research has produced varied results. However, the significant contribution chert provenance research has in archaeology cannot be overlooked and remains an important mechanism in studying past human behavior.

Current chert provenance research involves many methodologies and techniques (Church 1994). The multitude of chert sourcing techniques can be organized into three main groups, macroscopic attribute analysis, petrographic and geochemical.

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Geochemical techniques such as Neutron Activation Analysis (NAA), Inductively Coupled Plasma Mass Spectroscopy (ICPMS), X-ray Fluorescence (XRF) are used in recent chert provenance studies with varying degrees of success. In the study region, research conducted by Jack D. Nance (2000) successfully differentiated Lower St. Louis chert samples from Fort Payne (misidentified as Lower St. Louis in the study) using NAA. However, the majority of chert provenance studies in the Southeastern United States are conducted via qualitative visual identification.

Current chert provenance methods are limited by high cost, level of destructiveness, lengthy analysis, data management, reliability and precision (Frahm 2012; Crandell 2006). Arguably, the most critical issue limiting the use of chert provenance data is accuracy; the ability of the technique to identify the 'true' source of the material used to manufacture the artifact in question. Furthermore, a narrow geological and geographic perspective of tool stone availability limits our anthropological applications of chert provenance data.

A successful chert provenance study must characterize all possible sources at a geographic scale which matches the anthropological question and geological resource base. A chert provenance study must also be able to characterize a potential source through large geological sample sets and be able to differentiate the sampled source from other deposits. In addition, a provenance study must characterize the range of variation within an unknown artifact and match it to a particular range of variation at the source location (Harbottle 1982). Therefore, the 'fingerprint' analogy within provenance research is often a misnomer (Luedtke 1979). The current study examines the use of reflectance spectroscopy to gather spectral data within a chert samples database to specifically address the ability of the method in differentiating chert by parent formation, one deposit from another within the same formation, and to differentiate sub-sections within the same deposit.

### *Reflectance Spectroscopy*

Spectroscopy is the study of the interaction of light (electromagnetic radiation) with matter. A spectrometer is an instrument that measures this interaction (Smith 2011). Reflectance spectroscopy encompasses a wide range of techniques that gather electromagnetic data which is reflected or emitted from matter. The reflected electromagnetic radiation contains information related to atomic and chemical functional groups within a compound. The incident radiation in the visible portion of the spectrum (350–750 nm) stimulates vibration of particular atoms whereas dipole bonded molecules are stimulated in the near and middle infrared (751–25,000 nm) regions. Absorption of the incident radiation is wavelength dependent meaning absorption occurs at the wavelength frequencies corresponding to particular vibration energy states of the atom or molecule present. Therefore, the absorption of the incident radiation at certain frequencies gives the researcher information regarding atomic and molecular

structure and composition of the material. When graphically portrayed, the reflectance values per wave unit produce a line graph composed of Gaussian and Lorentzian curves (Fig. 1). Slight features and imperceptible slope changes in the chert's spectrum related to micro-mineral impurities are proving to be diagnostic for particular chert bearing formations and deposit locations (Table 1). Both qualitative and quantitative relationships can be studied using various mathematical functions, a subset of analytical chemistry termed chemometrics (Morin 2012).

The diagnostic micro-mineral groups causing particular spectral features may be directly related to the paleodepositional environment of the parent geological formation and the diagenetic processes influencing chert formation. Other researchers, using geochemical data, have speculated that chert diagnosis imparts a variable range of diagnostic characteristics (Foradas 2003; Malyk-Selivanova 1998) but the lengthy and costly analysis of large sample sizes have previously restricted efforts to tease out the geological and geographic relationships on a large scale. A variety of provenance studies are demonstrating the potential application of spectroscopy in archaeology (Beck *et al.*, 1965; Emerson *et al.*, 2013; Hubbard 2006; Morin 2012). The studies specifically related to the one presented here are chert provenance applications of both reflectance and transmission spectroscopy (Long *et al.*, 2001; Hubbard *et al.*, 2005; Hawkin *et al.*, 2008; Parish 2011; Hassler *et al.*, 2013; Parish *et al.*, 2013).

Reflectance spectroscopy as a chert source technique differs from current methodologies in four significant ways; 1) data acquired, 2) speed, 3) level of destruc-

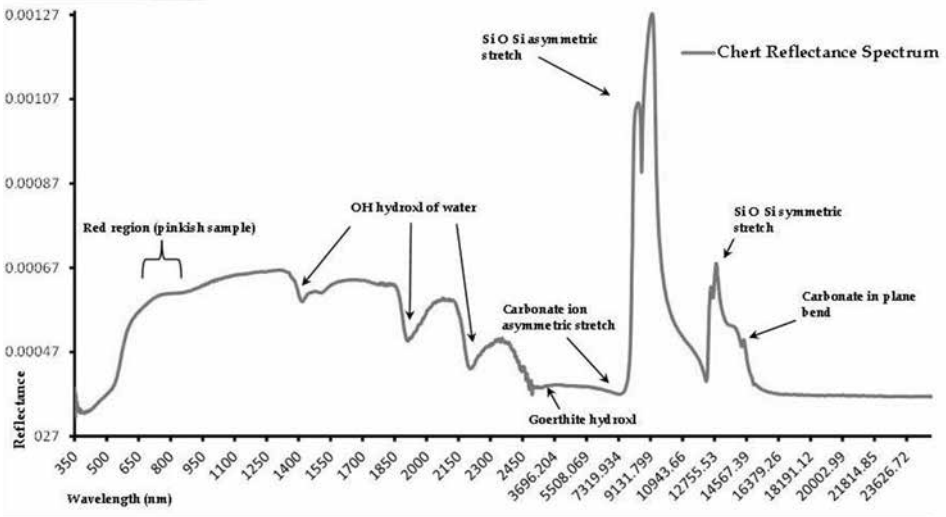


Fig. 1. A typical chert spectrum in the visible near-infrared and middle infrared with some spectral features labeled. The entire spectrum (350 – 15000 nm) is included in the analysis.

Graphic design: R.M. Parish.

Table 1. Some diagnostic spectral features noted in both the current study and in preliminary studies (Hassler *et al.*, 2013; Hawkins *et al.*, 2008; Long *et al.*, 2001; Parish 2013).

Spectrometer	Spectral Feature	Reflectance Peak Locations (nm)	Diagnostic Attributes Detected
VNIR	Color regions	350 – 750	Unbound electrons, atomic structure, mineral impurities
	OH, H-O-H, SiOH bonds	1400, 1900, 2200	Silica phase, formation conditions (Rice <i>et al.</i> 2013)
	Al-OH, M-OH bonds	2200 – 2300	Kaolinite, Illite, Montmorillonite, Limonite and other clay minerals
FTIR	Al-OH bonds	2700 – 2770	Kaolinite
	K-Al-Si-OH bonds	2800, 10100	Illite
	Na-Ca-Al-Si-O-OH bonds	2880 – 2980	Montmorillonite
	CH <sub>3</sub> bonds	3500 – 4080	Organic compounds
	Carbonate asymmetric stretch	6050 – 7200	Dolomite and Calcite
	Carbonate asymmetric stretch	5460 – 5500, 6570, 6860 – 6940, 13750	Dolomite
	Carbonate asymmetric stretch	6680, 11300 – 11340, 14070	Calcite
	Na-Ca-Al-Si-O-OH bonds	10730	Smectite
	Ti-O bonds	10030 – 10100, 14130 – 14190	Rutile
	Fe-O-OH bonds	11010 – 11080, 12600	Goethite
	Fe-O-OH · nH <sub>2</sub> O bonds	11430	Limonite
	Fe-K hydroxide bonds	12320	Glauconite
	Fe-S <sub>2</sub> bonds	14370 – 14400	Pyrite
	Mn-O hydroxide bonds	14460	Manganite
	Mg oxide bonds	14500 – 14550	Brucite
Fe oxide bonds	14900 – 14940, 17540	Hematite	
Fe oxide bonds	18380 – 21650	Magnetite	

tiveness, and 4) cost. Spectral data exists as arbitrary percent reflectance decimal numbers between 0 and 1 per wave frequency. Each reflectance value is a potentially 'diagnostic' variable relating to an atomic or molecular composition. Therefore, reflectance spectroscopy is not attribute information derived from empirical observations, nor is it purely geochemical data in the form of trace or rare earth element quantities.

## METHODS

### *Experiment design*

The application of reflectance spectroscopy to chert provenance research is assessed through three accuracy tests. The accuracy tests are designed to quantify the ability of reflectance spectroscopy in characterizing variation and differentiating ranges of variation both between and within chert bearing formations. The first test examines the accuracy of reflectance spectroscopy in distinguishing chert type, or chert found within different geological formations. The term formation is used in the study to describe geological material containing enough characteristics to distinguish it from adjacent rock. The second test refines the spatial scale of the provenance study through characterization and differentiation of multiple chert deposits within a single geological formation. The third and final test explores intra-deposit variability by differentiating sub-set samples of chert within a single deposit. By conducting internal tests within the chert database of known provenance, the accuracy of source assignment at different levels can be quantified.

### *Sampling*

Arguably the first step in any provenance study is to identify potential sources and assemble a representative database of those sources. An ambitious sampling strategy is adopted for the current study in which 30 samples were selected across the lateral and vertical breadth of each spatially discrete chert deposit. Multiple deposits were sampled per chert bearing geological formation. In two instances 60 total samples were collected from a deposit in separate sub-sections to assess intra-deposit heterogeneity. All deposits sampled were marked using a handheld GPS unit. Additional, field notes and photographs were taken to document the chert deposits. In total 2430 samples from 81 deposits collectively representing seven chert types in the Midwestern and Southeastern United States were analyzed (Fig. 2; Table 2). All samples were derived from Ordovician and Mississippian epoch aged carbonate formations, with chert type sample names reflecting their parent geological formation. The seven chert types investigated were widely used intermittently by prehistoric peoples in the Southeastern United States.

Table 2. Chert deposits analyzed in the study and the results of the three accuracy assessment tests.

Inter-formation accuracy test				
Geologic Formation	<i>Deposits</i>	<i># of samples</i>	Base Model misidentified	<i>10% test set misidentified</i>
<i>Lower St. Louis (Dover)</i>	6	180	8	2
<i>Fort Payne</i>	47	1410	3	4
<i>Tuscaloosa</i>	4	120	17	2
<i>Upper St. Louis</i>	11	330	2	2
<i>Ste. Genevieve</i>	11	330	4	5
<i>Leipers and Catheys</i>	1	30	1	0
<i>Burlington</i>	1	30	0	0
Total	81	2430	35	15
Intra-formation accuracy test				
Geologic Formation	<i>Deposit</i>	<i># of samples</i>	Base Model misidentified	<i>10% test set misidentified</i>
<i>Lower St. Louis (Dover)</i>	control	120	0	0
<i>Upper St. Louis</i>	Adams, KY	30	0	0
	Cook 1, TN	30	0	0
	Dot, KY	30	0	0
	DVD, KY	30	0	0
	Port Royal, TN	30	0	0
	St. Louis 1, KY	30	0	0
	St. Louis 2, KY	30	0	0
	St. Louis 3, KY	30	0	0
	St. Louis 4, KY	30	0	0
	St. Louis 5, KY	30	0	0
	WFDC 1, KY	30	0	0
Total	11 deposits	330	0	0
Intra-deposit accuracy test				
Geologic Formation	<i>Deposit</i>	<i># of samples</i>	Base Model misidentified	<i>10% test set misidentified</i>
<i>Ste. Genevieve</i>	40Pm103a	30	0	1
	40Pm103b	30	0	0
	40Wr48	30	0	0
Total	2 deposits	90	0	1

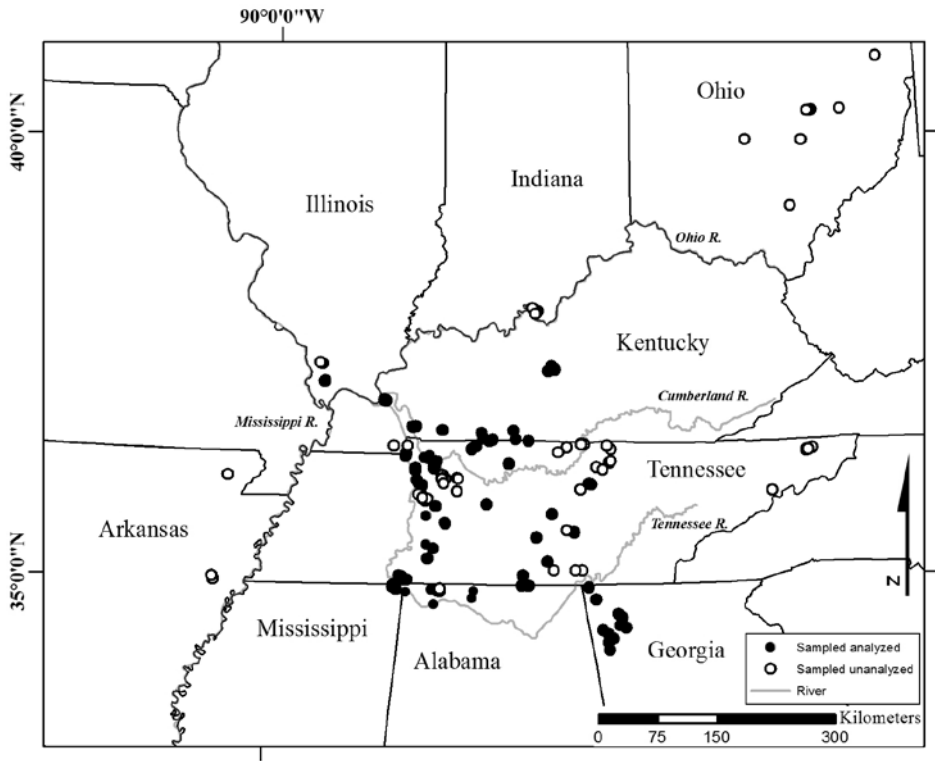


Fig. 2. All chert deposits sampled within the Midwestern and Southeastern United States. Deposits analyzed in the study are solid dots whereas sampled deposits needing analysis are circles. Graphic design: R.M. Parish.

All samples obtained were fractured conchoidally by direct hard hammer percussion using quartzite hammerstones. Six flakes were retained per sample so that the chert type collection can be duplicated six times. Thirty sample bags each containing six flakes collectively represent a single chert deposit. Just prior to analysis, lens tissue was used to lightly wipe the analyzed surface. No other treatments were used in preparing samples for spectral analysis.

*Spectral Analysis*

**VNIR**

Two reflectance spectrometers were used in the study. A FieldSpecPro<sup>®</sup> manufactured by ASD Inc. was used to collect 2150 reflectance values in the visible and near-infrared regions (350 – 2500 nm; Fig. 3). The probe-to-sample surface distance provided an approximate two centimeter diameter field of view upon the sample. The radiation (light) source, a quartz-halogen bulb, was mounted nearby, illuminating the sample tray below



Fig. 3. An ASD Inc. FieldSpec® spectrometer used to obtain spectra in the visible and near-infrared regions. Photo: R.M. Parish.

the detector. The sample was placed under the detector and a spectrum was recorded in less than one second producing a composite spectrum of the area of the specimen. A white reference reading was taken every ten samples to minimize instrument drift and atmospheric interference. All 2430 samples were analyzed in this manner with approximately 100 samples analyzed each hour.

### FTIR

A Bio-Rad FTS 40 spectrometer collected spectra in the middle infrared region (2500 – 25,000 nm; Fig. 4). The device was given time to initialize and a background measurement was taken on a gold standard to calibrate and to minimize atmospheric interference. Spectral resolution of the device is 12 nm, and a total of 64 scans taking just over one minute per analysis provided good signal to noise ratio. Samples were placed under the spectrometer's optical scope and a spot 20 microns in diameter was drawn in to focus prior to rotating the IR detector into place. Three measurements in different locations were taken on each sample and later averaged to provide a more representative spectrum per sample. Ten samples were analyzed in just under an hour. A total of 1867 reflectance values collectively represent a single spectrum in the middle infrared region.



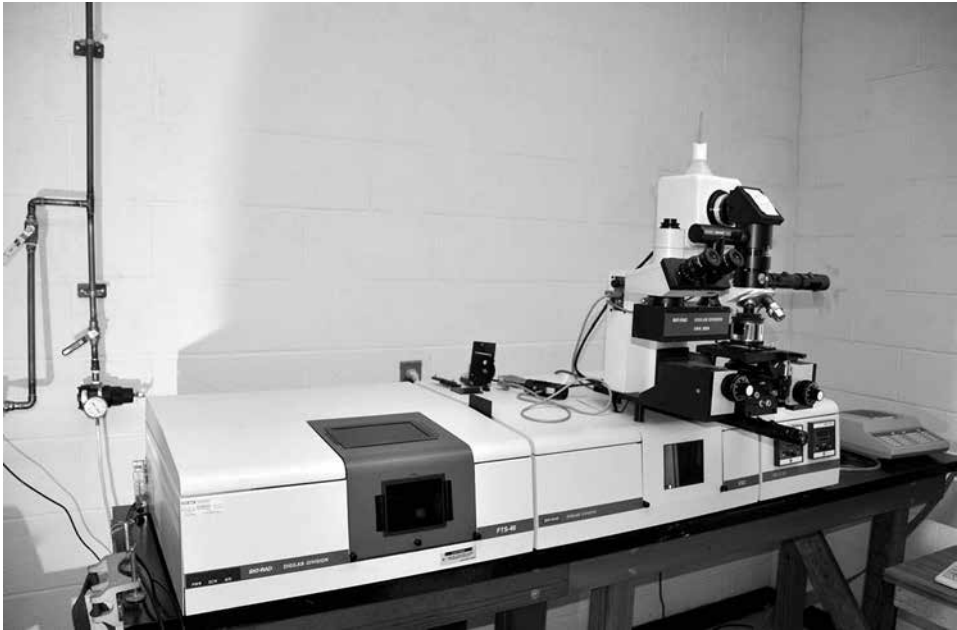


Fig. 4. A BioRad FTS-40 FTIR spectrometer used to obtain spectra in the middle infrared region.  
Photo: R.M. Parish.

## SPECTRAL PROCESSING

All of the raw spectral data were processed using conventional techniques to eliminate or reduce atmospheric interference, instrument noise, sample surface roughness, probe angular effects, and to standardize measurements for comparison. In addition to spectral processing, the reflectance spectra were converted to absorbance and first derivative transformed. Both absorbance and first derivative transforms provide a more robust means for quantitative analysis as well as highlight subtle spectral slope changes. A more detailed discussion regarding spectral processing may be found in Parish (2013: 176–178). The final stage in spectral processing was the combining of the VNIR dataset to the FTIR dataset. Each chert sample's composite spectrum consists of 4017 reflectance values.

### *Statistical Analysis*

A stepwise canonical discriminant function analysis was conducted on the VNIR and FTIR spectral datasets. The potentially diagnostic regions in the VNIR data include most of the visible and a portion of the near-infrared sections. Most of the middle-infrared diagnostic variables used in the study were selected from the 2600 to 7500 nm region; however, additional portions of the middle-infrared signal were also identified as diagnostic regions (Table 1). The discriminant function analysis evaluates

each wavelength variable and enters or removes it from the model. Additionally, the groups were weighted to account for differences in group size. Visual identification of 'diagnostic' spectral features by stacking spectra proved an inadequate methodology as spectral variability prohibited direct presence vs. absence comparisons. Multivariate statistical analysis was necessary to characterize patterns and bracket variation.

## RESULTS AND DISCUSSION

In the first experiment, all 2430 chert samples were grouped according to parent geological formation assessing inter-formation provenance. The second experiment focused on differentiating 11 chert deposits within the Upper St. Louis Formation sampled over a 400 km lateral extent. The third and final experiment examined the spectral differences within a single deposit of Ste. Genevieve chert as samples of prehistoric debitage were obtained in two sectors within a prehistoric procurement site. In each experiment a base discriminant function model was generated with all samples having known provenience followed by a second model where a random ten percent of the samples were treated as having unknown provenience by removal of their grouping variable.

### *Characterizing chert by formation (inter-formation)*

The accuracy assessment by parent geological formation (type) returned 2395 correct source assignments out of 2430 (99% correct classification). A total of 35 samples were misclassified to other geological formations. Upon removal of a randomly selected ten percent sample ( $n = 243$ ), the discriminant function model was run a second time with a reduced training set of samples. A total of 228 (94% correct classification) unknown samples were correctly assigned to their subsequent geological formations. Fifteen samples were misclassified to geological formations other than their correct provenience (Fig. 5a; Table 2).

### *Characterizing chert by deposit (intra-formation)*

The accuracy of intra-formation chert provenance was assessed in the Upper St. Louis Formation among 330 Upper St. Louis chert samples from eleven individual deposits. Lower St. Louis 'Dover' chert samples ( $n=180$  from 6 deposits) were included as a control. The base model successfully classified all 330 samples (100% correct classification) into their source deposits. A 10 percent ( $n = 33$ ) random sample of specimens had their grouping variable removed, and the discriminant function model was rerun. All 33 unknown samples (100% correct classification) were correctly assigned to their spatially discrete source deposits within the Upper St. Louis formation (Fig. 5b; Table 2).

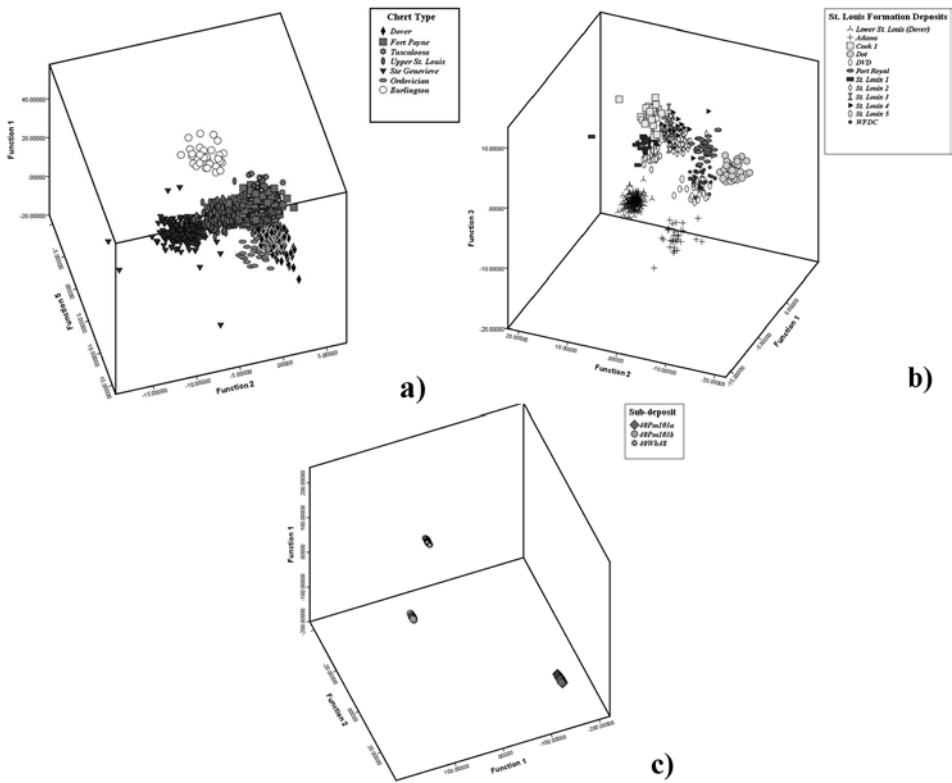


Fig. 5. Discriminant function analysis scatter plot showing; (a) delineation of chert by type/parent geological formation, (b) by deposit within the Upper St. Louis Formation, and (c) by sub-section within prehistoric procurement site 40Pm103. Graphic design: R.M. Parish.

*Characterizing chert within a deposit (intra-deposit)*

Prehistoric site 40Pm103 is a recorded quarry site of unknown cultural affiliation located in Putnam County, Tennessee<sup>1</sup>. Initially, 30 samples of primary chert debitage were collected from the northern section of the site followed by a collection of 30 samples of chert primary debitage from the southern section at a later date. The two groups of samples were treated as sub-samples within the single deposit of Ste. Genevieve chert and run in the discriminant function model. A third sample group of Ste. Genevieve chert from a prehistoric quarry further to the south (40Wr48) was included in the analysis as a control. The resulting intra-deposit assessment of the

<sup>1</sup> The Smithsonian Institution trinomial site system is used to here representing the one hundred and third (103) site recorded in Putnam County (Pm) in the state of Tennessee (40). The reporting protocol adopted by the Smithsonian lists the U.S. state first (in numerical alphabetical order), followed by county (usually a two or three letter abbreviation), followed by the sequential site number.

base discriminant function model correctly classified all 90 samples (100% correct classification). A randomly selected 10% sample ( $n = \text{six}$ ) from the 60 obtained from the  $40\text{Pm}_{103}$  deposit were treated as having unknown provenience. The discriminant function model correctly identified the sub-deposit source of five of the six samples (83% correct classification). The single misclassification identified the unknown sample as being from the control group location (Fig. 5c; Table 2).

## CONCLUSIONS

Chert source is often assigned through the identification of macroscopic attributes often failing to quantify the range of variability between chert deposits with large associated error rates (Calogero 1991, 1992). The aid of type collections as a methodology is potentially limiting because the few samples representing a specific type of chert may constrain the analyst's ability to adequately characterize variation. Additionally, type collections typically limit identification of the chert artifact to relatively few locations on the landscape and it is quite probable that the inability of previous studies to distinguish among and between source relates to small unrepresentative sample sizes (Parish 2013: 214). A sampling strategy which assembles a large range of materials is necessary but unmanageable as a type collection.

The use of analytical techniques such as geochemical methods is restricted by the cost of analysis and the level of destructiveness. The results of the current study demonstrate that reflectance spectroscopy's application within chert provenance research is a viable methodology. The spectral variance within a deposit of chert due to the atomic structural arrangement and composition of molecular compounds is patterned. The results of the accuracy assessment tests demonstrate the ability of reflectance spectroscopy to correctly assign source greater than 90% of the time by formation and deposit and above 80% of the time by sub-deposit (Table 2).

The analysis of 2430 samples from seven geological formations and 81 deposits illustrates the reflectance spectroscopy's ability to identify a range of diagnostic spectral variables, speed of analysis, and low cost. Currently, another 2040 samples obtained from 68 deposits await analysis (Figure 2). The analysis of the entire chert database using conventional geochemical techniques (NAA, ICP-MS) would exceed most research budgets, take multiple years of analysis, and may still not adequately differentiate chert even by geological parent formation due to the heterogeneity of trace elements.

The positive attributes of reflectance spectroscopy promotes the construction of large representative databases of chert resources necessary to bracket variability at various scales of resolution. Additionally, the non-destructive application of reflectance spectroscopy is vital from a preservationist perspective. Future research will

focus on controlled experiments of patinated archaeological materials and expand preliminary studies regarding heat treatment and outer surface weathering effects (Parish 2013). Much of the spectral variance due to surface roughness and outer surface weathering is alleviated through spectral processing which standardizes the spectra for comparison. Preliminary studies on heat treatment demonstrate the oxidation of iron minerals mainly occurs beyond the detectors limits (>15000 nm). However, more controlled experiments are designed to test outer surface weathering and heat treatment potential effects upon spectral source accuracy.

The prehistoric use of chert as tool-stone material across broad regional and temporal spans makes the application of chert source programs of paramount interest. The range of human behavioral questions addressed through chert source research illustrates the need for reliable methods. The capability of reflectance spectroscopy to accurately assign chert source is illustrated by the study and has the potential to track the movement and interaction of prehistoric people via their consumption of tool-stone resources.

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