X-Ray Diffraction of Oneota Pottery

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Introduction

In 2010, research was initiated to investigate the mineralogical relationship between Oneota pottery recovered from three major archaeological sites in La Crosse, Wisconsin. The Oneota represent the last prehistoric culture that inhabited the upper Midwest between A.D. 1300 – 1625; prior to European contact.

One of the most distinguishing artifacts that characterize the Oneota is shell-tempered pottery (Fig. 1). There are three phases of Oneota occupation within the La Crosse area. Each phase is defined by a shift in decorative styles of pottery (Fig. 2). The Valley View, Olson/North Shore, and Pammel Creek sites selected for sample collection each represent a different phase of Oneota culture within the La Crosse region (Table 1).

Figure 1: Oneota shell-tempered pot (http://www.wisconsinhistory.org/museum/artifacts/archives/pot.jpg).

The underlying question of the present research is whether or not the pottery uncovered at each of the three archaeological sites within La Crosse came from a single source of local clay?

Clays occur throughout this region as floodplain deposits, glacial outwash deposits and on older bluff tops (Boszhardt, 2008), thus, multiple clay sources possibly exist. A local clay sample was collected from Heaven's Gate Cemetery, which is located on a river terrace, to be used as a local control sample.

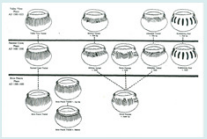


Figure 2: Schematic diagram of Oneota pottery type evolution at La Crosse, c.a. 1300 - 1625 (Boszhardt, 1994).

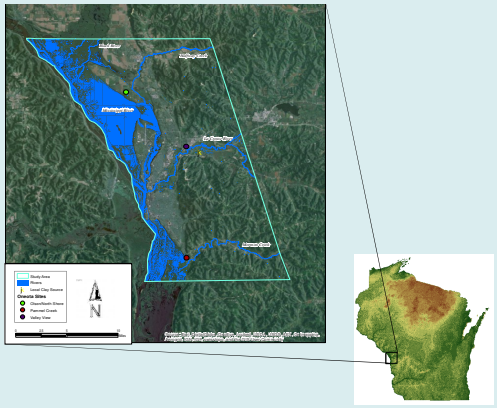
The research presented here applied the technique of powder x-ray diffraction (XRD) to preform qualitative analyses on the pottery samples and local clay sample. The goal was to provide a mineralogical "fingerprint" of each sample that would be compared across samples, and to the local clay source., to determine if there is a common clay source used in the production of Oneota pottery, and if the local clay tested was a possible source.

Table 1. Oneota phase descriptions and associated archaeological sites.

Site	Phase	Time Period	Pottery Style Description	# of Samples Collected
Olson/North Shore	Brice Prairie	AD 1300-1400	Inner lip/rim decoration, shoulder motifs include punctate-bordered elements and handles that attach at lip	3
Pammel Creek	Pammel Creek	AD 1380-1520	Transition between the Brice Prairie and Valley View phases, bold impressed lip patterns, defined by finger or tool notching, inner lip/rim decoration disappears, wide decorated strap handles (some attached to lip)	9
Valley View	Valley View	AD 1530-1625	Finer lip decoration, shoulder patterns, tool trail width decreases, patterns less bold	2

Location

The study area is located within the Upper Mississippi River Valley of the western part of La Crosse County, Wisconsin. The area contains the Olson/North Shore, Valley View, and Pammel Creek sites, as well as the local clay source used in this study.



X-Ray Diffraction

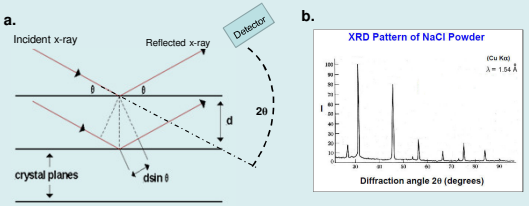


Figure 3.: (a) A schematic diagram of the XRD process , and (b) an example XRD pattern obtained from powdered NaCl (adapted from http://univers-review.ca).

The x-ray diffraction process is shown schematically in Fig. 3a, with data characteristic of pure sodium chloride powder presented in Fig. 3b. In an XRD experiment, an x-ray of known wavelength is send incident onto a crystalline material at and incident angle (θ). This x-ray is then reflected off of the crystalline planes of the sample in a specular manner such that the angle of incidence equals the angle of reflection. Due to the wave nature of light, x-rays reflecting off adjacent crystalline planes may either constructively or destructively interfere upon reflection due to the difference in path length traveled by the respective x-rays. This path length difference is twice the product of the distance between crystal plane (d), and the sine of the angle of incidence. The condition for constructive interference is given by Bragg's Law :

Bragg Law:

$$2d\,(sin\theta) = \lambda_0$$

Where,

d = spacing between layers of atoms (crystal planes)
θ = angle between refracted rays and the surface of sample (Bragg angle)
λ = wavelength of the x-rays

When taking an XRD scan, the angle of incidence is varied along with the position of the x-ray detector to determine at what angles Bragg's Law is met which results in high intensity peaks in the XRD pattern. In the actual experiment the detector is moved twice as fast as the sample to maintain the necessary geometry. Therefore XRD data is plotted as an function of relative intensity versus the detector angle of 2θ as shown above.

Once an XRD pattern is obtained, two basic methods of analysis may be performed:

1. The distance between all possible crystal planes may be determined via Bragg's Law, and the mineral phase identified.
2. The pattern may be used to "fingerprint" a material of unknown crystalline composition since the locations of the set of Bragg peaks are unique.

In the current study, a combination of these methods was used. Quartz and calcium carbonate peaks were located within the samples of interest, after which a qualitative comparison was made between the of the angular locations of diffraction peaks observed in pottery samples and those observed from samples of locally sourced clay.

Methods

- All specimens photographed before preparing for XRD analysis
- Sample Preparation:
  - Samples ground into uniform powder to measure all possible atomic spacing (Fig. 4a below)
  - Mounted onto clear plastic slide
- XRD data collected via Siemens D500 Powder X-ray Diffractometer (Fig. 4b)
  - Copper x-ray source ($\lambda = 1.54$ Å)
  - Data obtained from 20 to 120° 2θ
- Data processing
  - Stripped $K\alpha_2$ and removed background using Match! Powder X-ray Diffraction software
- Overlay line profiles for qualitative comparison of peak locations

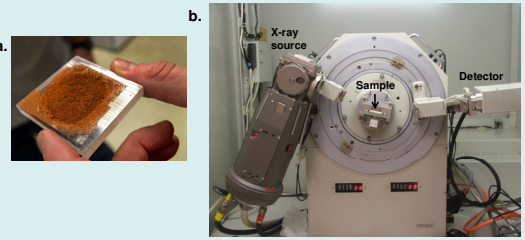


Figure 4: Photos of (a) mounted powder sample, and (b) the D500 Powder X-Ray Diffractometer.

Results

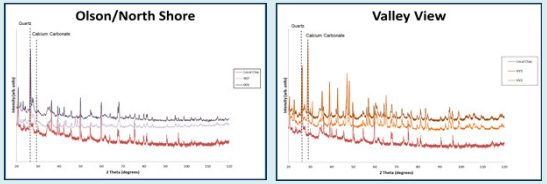


Figure 5: XRD Patterns obtained for samples from the Olson/North Shore (left) and Valley View (right) sites compared to a sample of locally sourced clay. Dashed lines represent the quartz and calcium carbonate peaks used to calibrate the data.

Fig. 5 shows characteristic data obtained from the Olson/North Shore site, and the Valley View site.

Since quartz is present in both the pottery samples of study, and the locally sourced clay, a known high intensity diffraction peak corresponding to quartz was used as reference. A calcium carbonate peak, originating from the shell temper, was used as an additional reference between pottery samples. Both lines are labeled above. Using these peaks as reference, a qualitative comparison of the lineshapes may be performed by looking at the location and intensity of peaks which appear above the background.

The results of such a comparison suggest:

1. All features observed in the XRD pattern of locally sourced clay are observed in data obtained from pottery samples suggesting they are of similar origin.
2. Obvious changes occur in the relative intensity of many of these peaks. This is attributed to the degradation of the clay crystal structure through firing of the pottery.

Data obtained from the Pammel Creek site (not presented) suggest similar results.

Discussion

The known local clay source, sharing a similar mineral composition with numerous samples, is suggested to derive from the Savanna Terrace Formation, a late Wisconsinan glacio-fluvial terrace. The Savanna Terrace has been mapped along the Mississippi River and its tributaries, extending from Pepin, Wisconsin to Jackson, Illinois (Fig. 6).

The Savanna Terrace is also present within the American Bottom (the central home of the Mississippian culture) with mineral composition of clays in this region being slightly different. This provides an opportunity to compare the mineralogy of pottery between Oneota and Mississippian cultures and consider cultural interactions, such as trade, at a larger scale

Figure 6: Locations of the Savanna Terrace Formation (yellow) in relation to the American Bottom (red). Modified from Flock (1983).

Future Work

- Determine the mineralogical source of currently unidentified diffraction peaks
- Quantitative analysis determining detailed mineralogical composition of pottery samples
- Comparison of samples from known Savanna terrace clay sources both locally and along the Mississippi River Valley in general

References

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