Determine the mineralogical source of currently unidentified diffraction peaks.

Comparison of samples from known Savanna terrace clay sources both locally and along the Mississippi River Valley in general.

Samples ground into uniform powder to measure all possible atomic spacing (Fig. 4a below).

Data processing

Copper x-ray source (\(\lambda = 1.5418 \text{ Å}\)).

Mounted onto clear plastic slide.

XRD data collected via Siemens D500 Powder X-ray Diffractometer (Fig. 4b).

Data obtained from 2θ to 120θ.

Comparison of peaks used to calibrate the data.

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 incident onto a crystalline material at an incident angle (\(\theta\)). 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 wavy nature of light, an x-ray reflecting off adjacent crystalline planes may either constructively or destructively interfere upon reflection due to the difference in path length traveled by the respective waves. This path length difference is twice the product of the distance between crystal planes (\(d\)) and the sine of the angle of incidence. The condition for constructive interference is given by Bragg’s Law:

All features observed in the XRD pattern of locally sourced clay are observed in data obtained from the Pammel Creek site (not presented) suggest similar results.

Table 1. Oneota phase descriptions and associated archaeological sites.

<table>
<thead>
<tr>
<th>Site</th>
<th>Phase</th>
<th>Time Period</th>
<th>Pottery Style Description</th>
<th># of Samples</th>
<th>Clay Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Olson/North Shore</td>
<td>Early</td>
<td>AD 1300-1400</td>
<td>Shell-tempered pottery</td>
<td>9</td>
<td>Local clay source</td>
</tr>
<tr>
<td>Hillpost</td>
<td>Early</td>
<td>AD 1300-1400</td>
<td>Shell-tempered pottery</td>
<td>9</td>
<td>Local clay source</td>
</tr>
<tr>
<td>Valley View</td>
<td>Early</td>
<td>AD 1300-1400</td>
<td>Shell-tempered pottery</td>
<td>9</td>
<td>Local clay source</td>
</tr>
<tr>
<td>Valley View</td>
<td>Middle</td>
<td>AD 1530-1625</td>
<td>Finer shell-tempered pottery</td>
<td>9</td>
<td>Local clay source</td>
</tr>
<tr>
<td>Valley View</td>
<td>Late</td>
<td>AD 1625-1650</td>
<td>Finer shell-tempered pottery</td>
<td>9</td>
<td>Local clay source</td>
</tr>
</tbody>
</table>

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 a 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 calcite 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 (\(\lambda = 1.5418 \text{ Å}\)).
- Data obtained from 2θ to 120θ.
- Data processing:
  - Step-step refinement and background using March-Poulter Powder X-ray Diffraction software
  - Overlay loss profiles for qualitative comparison of peak locations.

Results

Discussion

The known local clay source, sharing a similar mineral composition with numerous samples, is suggested to derive from the Savanna Terrace formation from the Wisconsin glacial-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.

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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