McNair Scholars Journal

Volume 2 | Issue 1 Article 17

1-1-1998

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ABSTRACT:

The status of research on Middle Woodland clay samples from the Grand River basin as of September, 1997 is summarized providing background on the Middle Woodland era and use of neutron activation analysis in their study of elemental analysis of ceramics found at archaeological sites in the Grand River basin.

The Middle Woodland Period in Western Michigan

Prehistoric settlements in the Grand River basin with Early and/or Middle Woodland ceramics are rare in southwestern Michigan. Ceramics are important for determining the age of sites because distinctive ware patterns and decoration tend to correlate with certain time periods. Pottery had its early beginnings in the Early Woodland period; by the Middle Woodland period, when most of eastern North America was dominated by the Hopewell culture (200BC–400AD), ceramics had evolved in aesthetic quality. The middle Woodland marks a time when people and ideas came to southwestern Michigan from the Illinois region. Classifying and identifying particular ceramic zones is only the beginning of understanding the meaning of Hopewell (Fitting, 1975).

The Grand River basin is the largest drainage system in Michigan, but only thirty-five camps, seven mound and mound groups, and five find spots have been documented within this system. Seventy percent of these Middle Woodland archaeological sites are found within the lower Grand and Thornapple areas (Brashler and Mead, 1996). Some of these sites include the Spoonville site (20OT1), the Norton Mounds site (20KT1), the converse mound group (20KT2), and the Grandville site (Brashler and Mead, 1996). Upstream, ceramics are rare and sites tend to consist of small amounts of lithics (Brashler and Mead, 1996). However, the Prison Farm site, located in the eastern areas of the Thornapple sub-area (five miles west of the confluence of the Maple River with the Grand River), is a substantial campground/village site rich in a variety of lithics, ceramics, bone, and other artifacts.

Middle Woodland pottery characteristics are quite varied. Zone of broad trail lines, thin small inclusions, random incisions, cord-wrapped stick impressions, large cross hatch incisions, nodes, shallow indentation, straight dentate stamps, brushing or combing, hemiconal punctates, conical punctates, single occurrences of dentate rocker stamping, plain rocker stamping, knotted cord impressions, plain stamp, small rectangular stamp, and twisted cod impressions used as zone markers are all characteristic of Hopewell pottery from at least one Middle Woodland site on the Grand. Middle Woodland pottery can be a combination of all these designs (Brashler, 1995). Similar types have also been found in the Norton Mounds. Pot thicknesses vary as well; sherds can be as thin as 5–8 millimeters, or as thick as 10 millimeters or more.

Research Method and Materials

To examine the relationship between sites in the Grand River basin and the clay sources, the research focused on existing collections and collected clay source samples and archaeological samples. Archaeological samples were collected as part of a field school excavation at the Prison Farm site (20IA58). Here, pottery sherds were recovered, in addition to other debris, including stone tools, remnants of hearths, and animal bone and small-scale plant remains, which indicate diet.

In addition to excavation, 25 samples of clay were also collected from three counties (Kent, Ottawa, and Ionia) along streams and in upland areas, to get a representative sample of the kinds of raw materials prehistoric people may have used for their pottery production. Sample sites were located by examining USDA Soil Conservation Service maps. Samples were taken by wither, digging the sample by hand or with a bucket auger. The samples were places in sealed plastic bags and labeled with an identification code keyed to the location on the map.

Most archaeological samples came from the 1996 excavations at the Prison

Farm site, but samples were also selected from previously excavated Norton Mounds, Spoonville, and Paggeot site collections in the Grand Valley State University Anthropology lab. In all, 57 samples were collected. Sample preparation included labeling, weighing, documenting, and mapping samples, which were shipped in June of 1997 to the Missouri University Research Reactor (MURR) for analysis.

Instrumental Neutron Activation Analysis

In 1936, Levi and Hevesy found that certain elements become radioactive after being exposed to neutrons. These experiments led to the development of Neutron Activation Analysis (NAA), a highly sensitive technique employing isotope decay to identify certain elements within a substance. The main reasons for investigating the composition of different pottery collections are (a) to identify clearly distinguishable ceramic types or groups for insightful archaeological interpretation, (b) to identify pottery of unknown origins with previously classified groups, or (c) to determine the validity of a previous identification (Glascock, 1992). Only Instrumental Neutron Activation Analysis was used to complete this task; INAA is purely the instrumental technique of NAA, whereas RNAA (Radiochemical Neutron Activation Analysis) employs chemical separation of elements after the use of INAA.

Sample preparation at the MURR lab includes scraping the inner and outer surfaces of a $2cm^2$ sherd, brushing it clean, washing it in deionized water, and allowing it to dry. Clay samples are dried prior to analysis. Both clay and pottery samples are individually crushed and homogenized into fine powder and stored in glass vials separately. The samples are then dried in a 100°C oven for 24 hours and stored in a dessicator.

Samples are then irradiated to reveal percentages of trace elements present in the clays. The most common type of nuclear reaction used for NAA, the n, gamma reaction, entails that a sample be bombarded with neutrons. Compound nuclei form in an excited state from this non-elastic collision, and these nuclei will almost immediately begin to decay. During this decay, particular gamma rays are emitted that are characteristic of certain elements. Prompt NAA (PGNAA) is used to measure elements that decay too rapidly to be measured by delayed NAA (DGNAA), elements that only reproduce stable isotopes, or elements with low gamma ray intensities. Samples are irradiated for 5 seconds at thermal neutron flux of $8x10^{13}$ $n/cm^2/s$. The samples are then allowed to decay for 25 minutes, and then are counted by a germanium detector to collect the different gamma rays of short-lived elements (which may include Al, Ba, Ca, Dy, K, Mn, Na, Ti, or V) (Glascock, 1992).

Following this initial counting is a two-week cooling period. This next step involves DGNAA, which is the conventional method used to identify most elements that create radioactive nuclides. After two weeks, the sample is reweighed and then transferred to quartz vials. The samples are then wrapped in aluminum foil with four primary standards and two quality control standards and placed in an aluminum canister. This bundle is then irradiated for 24 hours at a neutron flux of $5x10^{13}$ $n/cm^2/s$. After another week, the samples are again washed and each counted for 2000 seconds by a germanium detector combined with an automatic sample changer. This counting detects other elements (which may include Ce, Cr, Cs, Eu, Fe, Hf, Rb, Sb, Ta, Tb, Th, Zn, or Zr). The advantage to DGNAA is selectivity; this method is time-flexible so that if a shorter-lived radionuclide interferes with a long-lived radionuclide, the measurement can be made after the interfering isotope has decayed (Glascock, 1992).

The standard comparator method provides the most sufficient analysis. This method entails standard materials with known element concentrations being irradiated and measured under identical circumstances with the unknowns. Using this method, either accuracy or the occurrence of systematic errors can be detected. Element concentrations in an unknown sample are determined by comparing the measured decay per unit weight of the unknown to those known measurements of the reference standard (Glascock, 1992).

Use of Results

Results of the neutron activation analysis of the initial samples are not available at this time. When available, they will provide the basis for understanding clays used by prehistoric peoples in the Grand River basin by providing a baseline characterization of the elements present in local clays. Furthermore, since this is the initial phase of a larger scale research project involving several sites from different areas, the results of the first sample analyses will provide preliminary information on several questions, including:

(1) Are the clays used in any or all of the potter sherds submitted from local sources, based on current samples of raw materials?

(2) If an archaeological sample is different than raw material samples, is it likely due to trade, or is it due to inadequate sampling of the clay resources?

(3) Is there a common type of clay used more than other types of clay to produce pottery? If there is a common type of pottery, are these sherds from one collection or from different collections?

These questions will be explored in further research.

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