

Prehistoric Ceramic Production: Raw Materials and Firing Methods of the La Crosse Locality Oneota

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ABSTRACT

The Oneota who lived in large farming communities in the La Crosse locality from approximately A.D. 1300 to 1625 are recognized by the characteristic ceramics they produced. They manufactured shell-tempered ceramic vessels that were much larger and more finely made than the grit-tempered pottery of the Woodland cultures that inhabited Wisconsin prior to A.D. 1300. The use of shell to decrease the porosity, improve the thermal efficiency and increase the durability of the resulting pottery presents a special problem. Under certain conditions when the calcium carbonate in the shell is heated its chemical composition changes to a more unstable form known as calcium oxide. Calcium oxide is hygroscopic, meaning it absorbs moisture from the atmosphere. The absorption of moisture by the calcium oxide causes fired pottery to crack and spall, weakening the strength of the pot and often causing it to crumble. Some attempts have been made to replicate the prehistoric pottery found in the La Crosse area yet more experimentation is needed to understand the techniques and materials used to produce ceramics with properties similar to those of Oneota pottery. The replicas produced thus far were fired in the controlled environment of a kiln and do not exhibit the same properties as Oneota pottery and attempts to temper the clay with shell have met with spotty success. This research focuses on the problems associated with shell tempered ceramics through the production and testing of clay tiles made from local raw materials and fired using open-air firing techniques and the comparison of these tiles with actual Oneota pottery sherds.

INTRODUCTION

Prior to European settlement of the La Crosse area, the region was occupied by a group of people known to archaeologists as the Oneota. The Oneota occupied the La Crosse locality from approximately A.D. 1300 to 1625, living in large farming communities. The Oneota are recognized by the characteristic ceramics they produced. They manufactured shell-tempered ceramic vessels that were much larger and more finely made than the grit-tempered pottery of the Woodland cultures that inhabited Wisconsin prior to A.D. 1300 (Boszhardt 1994: 3 and 14).

Temper is material potters add to a clay mixture to improve the workability of the wet clay, improve the drying process and/or improve characteristics of the resulting vessels. Tempers can correct stickiness, increase porosity, reduce shrinkage, decrease drying time, reduce deformation on drying, and improve firing characteristics (Rice 1987:74).

Using burned and crushed shell as temper, as the Oneota did, presents some problems. Clay tempered with shell is stiffer, less plastic, difficult to work, and difficult to fire. However because shell temper was so widely used in Eastern North America it likely provided benefits that outweighed these difficulties. To date researchers have discovered that shell tempered ceramics have improved thermal efficiency, are stronger and more durable, and are more impact and thermal shock resistant than grit tempered ceramics (Budak 1991, Bronitsky and Hamer 1986, Feathers 1989, and Bronitsky 1989).

The stability of shell temper presents a special problem. Under certain conditions when the calcium carbonate in the shell is heated its chemical composition changes to a more unstable form known as calcium oxide. Calcium oxide is hygroscopic, meaning it absorbs moisture from the atmosphere. The absorption of moisture by the calcium oxide causes the fired pottery to crack and spall, weakening the strength of the pot and often causing it to crumble (Feathers 1989:580). The Oneota were able to overcome the problem of shell stability and master the manufacture of durable shell tempered pottery.

The attempts made to replicate the prehistoric pottery found in the La Crosse area to date have met with spotty success. Most replicas produced thus far were fired in the controlled environment of a kiln and do not exhibit the same properties as Oneota pottery and attempts to temper the clay with shell have produced pots that crack and spall. The Oneota did not have access to the type of firing environment provided by a kiln and only limited and informal attempts have yet been made to replicate pottery using the open-air firing techniques that were available to them. More experimentation is needed to understand the techniques and materials used to produce ceramics with properties similar to those of Oneota pottery. There are still uncertainties regarding the type of clay used, how the temper was preconditioned prior to mixing with the clay, the proportions of temper mixed with the clay, the superiority of one temper over another, and the firing conditions.

The research presented in this paper explores the questions of the type of clay used, the amount of shell temper added, the benefits of shell temper in the drying process, and firing temperature and conditions. The research was done through the production and testing of clay tiles made from local raw materials and fired using open-air firing techniques. The experiments performed include:

- An analysis of the amount of shell temper used in Oneota ceramics;
- A comparison of the air drying properties of tiles tempered with shell, grit, and no temper;
- An evaluation of the firing temperature and firing conditions that result in tiles resistant to spalling;
- A comparison of raw clays from a source on the south side of La Crosse and one on the north side of La Crosse; and
- A comparison of the properties of tiles made from local raw materials with actual Oneota sherds.

METHODOLOGY

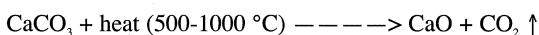
Background

Clay tempered with shell is stiffer, less plastic, difficult to work, and difficult to fire. However because shell temper was so widely used in Eastern North America it likely provided benefits that outweighed these difficulties. Budak found that water penetrated the walls of a grit tempered vessel over three and a half times more quickly than a similar shell tempered

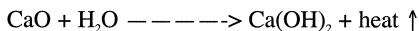
vessel (13 minutes for the grit tempered vessel versus 46 minutes for the shell tempered vessel). This is significant when using a vessel for cooking in that energy must be used to evaporate the water seeping to the exterior of the vessel walls, making this energy unavailable for heating the contents of the pot. Therefore more heat is required to cook the contents of a grit tempered pot than to cook the contents of a shell tempered pot. Budak demonstrated this difference in heat conductivity and improved thermal efficiency by measuring the temperature of water heated in grit and shell tempered vessels under similar conditions. Water heated in the shell tempered vessel reached the boiling point (100 degrees C) in 11 minutes whereas water heated in the grit temper vessel never boiled and plateaued at 98 degrees C after 13 minutes (Budak 1991). Additionally a number of experiments have been performed suggesting that shell tempered ceramics are stronger and more durable than grit or sand tempered ceramics. Though there is some dispute over the validity of the methods used, the general conclusions indicate that shell tempered ceramics are significantly more impact and thermal shock resistant than sand tempered ceramics (Bronitsky and Hamer 1986, Feathers 1989, and Bronitsky 1989).

The stability of the shell temper presents another problem. Under certain conditions when the calcium carbonate in the shell is heated its chemical composition changes to a more unstable form known as calcium oxide. Calcium oxide is hygroscopic, meaning it absorbs moisture from the atmosphere. The absorption of moisture by the calcium oxide results in the formation of calcium hydroxide which occupies a larger volume than calcium oxide. This expansion in volume causes the fired pottery to crack and spall, weakening the strength of the pot and often causing it to crumble (Feathers 1989:580). The chemical reactions involved in this process are as follows:

1. Calcium carbonate (CaCO₃) is heated and under certain conditions is converted to calcium oxide (CaO) and carbon dioxide (CO₂) which is evolved in this process.



2. After firing the calcium oxide (CaO) absorbs water from the atmosphere forming the hydration product calcium hydroxide (Ca(OH)₂) which occupies a greater volume than calcium oxide. This is an exothermic reaction and heat is evolved during the process.



A wide range of temperatures from 500 °C to 1000 °C are given for the temperature at which calcium carbonate changes to calcium oxide (Betts 2000:138, Bronitsky and Hamer 1986:97, Budak 1991:54, Rice 1987: 410, and Tankersley and Meinhart 1982:228).

The Oneota were able to overcome the problem of shell stability and master the manufacture of durable shell tempered pottery. Bronitsky and Hamer and Feathers suggest that burning the shell prior to crushing and using as temper may help to reduce the cracking and spalling problem (Bronitsky and Hamer 1986:97 and Feathers 1989:580). They agree that this benefit was likely a secondary and unknown benefit as the primary purpose of burning the shell appears to have been to make it more friable and easier to crush (Feathers 1989:581 and Bronitsky 1989:590). Archaeological evidence strongly suggests that the La Crosse Locality Oneota did indeed burn their shell prior to crushing and using as temper. Unfired

clay deposits containing burned shell and deposits of burned shell have been found at sites in the La Crosse area (Arzizian et. al. 1989:69).

Experiments

I received an Undergraduate Research Grant to study the problems associated with shell tempered ceramics and produced and tested a number of clay tiles using open-air firing techniques during the summer and fall of 2001. Clay tiles tempered with shell and grit in varying proportions were prepared and analyzed to examine the different properties of each to gain insight into why one may have been used over the other. Variables such as plasticity, workability, shrinkage, porosity, color, and hardness were considered. The shell temper used was pre-burned under open-air conditions and mixed with clay in various proportions. The test tiles were fired at various temperatures and under a variety of open-air conditions. Some tiles were fired in an oxygen rich atmosphere and others were fired in a low oxygen atmosphere created by covering the tiles with organic material. The effect of these different firing conditions on the final properties of the tiles was evaluated. In addition to the above variables, the following were recorded during the course of preparing and firing the test tiles: drying time, firing time, firing temperature, cooling time, type and amount of fuel used, and ambient conditions. The properties of each tile were evaluated and compared with the properties of Oneota pottery sherds obtained from the Mississippi Valley Archaeology Center. The properties evaluated include shrinkage, color (both before and after firing), hardness and porosity. Detailed data sheets were kept documenting the raw material preparation, tile production, and firing techniques used, as well as the variables listed above.

- The specific experiments performed include:
- An analysis of the amount of shell temper used in Oneota ceramics (tile batches 2 and 4);
- A comparison of the air drying properties of tiles tempered with shell, grit, and no temper (tile batch 1);
- An evaluation of the firing temperature and firing conditions that result in tiles resistant to spalling (tile batch 3);
- A comparison of raw clays from a source on the south side of La Crosse and one on the north side of La Crosse (tile batches 2 and 4); and
- A comparison of the properties of tiles made from local raw materials with actual Oneota sherds (tile batches 2 and 4, and Oneota sherds from the Mississippi Valley Archaeology Center).

Raw Materials

Clay. Clay is defined as “fine-grained, earthy material that becomes plastic or malleable when moistened...Clays are sedimentary deposits that ... are comparatively recent accumulations of the products of weathering and disintegration of much older rocks” (Rice 1987: 36). Clay from two local sources was used to prepare test tiles. The first clay used was obtained from the researcher’s property located just south of the City of La Crosse north of county highway K in a small intermittent, tertiary drainage of the Mississippi River which drains into Chipmunk Creek. The second clay used was obtained by the Mississippi Valley Archaeology Center from the north side of La Crosse. Clay from this source has been used in other pottery replication experiments.

The clay found on the researcher's property is secondary (transported or sedimentary), fluvial clay. The clay results from the chemical and mechanical weathering of parent rock that is transported and redeposited by the action of flowing water. The action of weathering, transportation and redeposition results in a relatively homogeneous, fine textured material with argillaceous properties (Rice 1987: 37).

The clay from the Mississippi Valley Archaeology Center is from a source in north La Crosse which is Pleistocene outwash fines from a Lake Superior glacial flood event that spilled back into the La Crosse valley from the main trench. It occupies and caps a terminal Pleistocene outwash terrace that was deposited approximately 12,000 to 9,500 years ago. These deposits alternate with sands and gravels and grey clay bands which correlate with Glacial Lake Aggaziz outbursts (Boszhardt 2002). The clay was dried and ground to the consistency of a fine powder prior to being mixed and formed into clay tiles.

A sample of each clay used was submitted to the Milwaukee Soils Laboratory for particle size and chemical analysis. The clay from the north side of La Crosse had a higher percentage of clay-size particles (less than 2 microns) than did the clay from the Carter property, 50.7 percent by weight versus 19.0 percent by weight respectively. Figures 1 and 2 present a breakdown of the particle size of each clay into clay, silt, and sand particle size ranges. Typically "clay" is defined as a soil having at least 35 percent of its particles smaller than 2 microns. However, soils containing as little as 15 percent particles smaller than 2 microns exhibit plasticity (Rice 1987:39).

Figure 1. Breakdown of North Clay by Particle Size

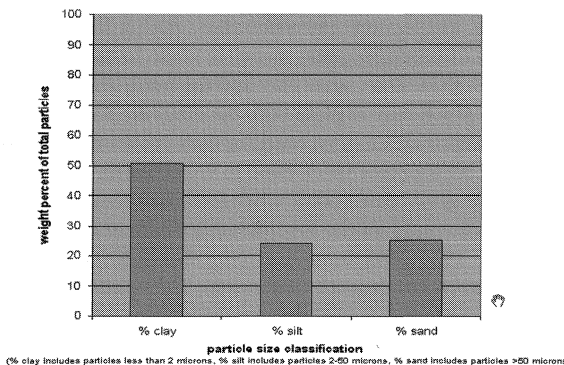


Figure 2. Breakdown of Carter Clay by Particle Size

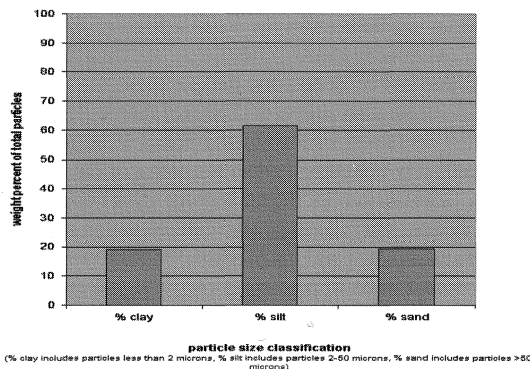


Table 1 summarizes the chemical analysis of the two clays.

Table 1. Chemical Analysis Results for Clays Used.

	pH	Organic Matter %	Fe % (oxalate extraction)	Exchangeable Ca (mg/kg)	Exchangeable Mg (mg/kg)	Exchangeable K (mg/kg)	Exchangeable Na (mg/kg)
Carter clay	7.2	1.4	0.23	1670	520	71.0	30.6
north clay	6.7	2.1	0.24	3360	1336	198.7	83.5

Typically clays with a pH in the neutral level (6 to 8) are more plastic than alkaline clays (Rice 1987:63). Both the Carter and north clays have pHs which fall within this range. The size amount and distribution of impurities in clay, especially iron and organic matter as well as the time, temperature and atmosphere during firing determine the color of the fired clay. Clay free of iron and organic matter is white in its raw form and white or cream when fired (Rice 1987:333). The Carter clay and the north clay contain iron and organic matter in small quantities which may explain why the clays are similar colors when fired. Organic matter in raw clay contributes a gray, black or dark brown color. Iron in raw clay contributes a red, yellow, or brown color when fired in an oxidizing atmosphere and a gray, black, bluish, greenish, or gray-brown color when fired in a reducing atmosphere. Iron doesn't determine the fired color until all the organic matter carbon in the raw clay is eliminated from the clay body (Rice 1987:334). In an oxidizing atmosphere carbon is burned off quickly beginning at approximately 200 degrees Centigrade. The volatilization of organics can contribute significantly to weight loss and shrinkage during firing. As the carbon oxidizes it is off-gassed as carbon monoxide and carbon dioxide. Rice indicates that as carbon moves from the interior of the clay object to the surface, the surface can blacken (Rice 1987:88). Blackening was not observed when firing the tiles. This may be due to a low organic content of the clay. The carbon present in any organic matter in the clay will not be completely eliminated until the firing temperature exceeds 600 degrees C. Additionally, oxygen must be available in the firing atmosphere for oxidation of the carbon to occur (Rice 1987:88). When firing the first batch of tiles in an oxidizing atmosphere the maximum firing temperature reached 590 degrees Centigrade. At this temperature it is unlikely that the organic matter in the clay was completely eliminated. An indication of incomplete removal of organics is the presence of a dark core that can be seen in the cross section of the object. One of the tiles in batch one broke into two pieces shortly after firing and showed a very slight dark core in its cross section. This provides evidence that the carbon in the clay was not completely eliminated as the firing temperature also suggests. The fact that the core was very slight, supports the fact that the raw clay contained a only small amount of organic matter.

The north clay contains a greater number of the exchangeable ions calcium (Ca^{2+}), magnesium (Mg^{2+}), potassium (K^+), and sodium (Na^+) than the Carter clay. This is as would be expected as finer clays have greater ion exchange capacity (Rice 1987:76). Divalent ions such as calcium (Ca^{2+}) and magnesium (Mg^{2+}) tend to increase the plasticity of clay while monovalent ions such as potassium (K^+) and sodium (Na^+) tend to decrease plasticity (Rice 1987:77).

Shell. Shells were prepared for use as temper by first burning and then crushing them. Large, indigenous mussel shells from the Upper Mississippi River in the La Crosse area were used. The shells ranged in size from about five to seven inches in length and three to four inches in width and were historically widespread and abundant in the Mississippi River adja-

cent to La Crosse. A small open fire was started using miscellaneous hardwood two to four inches in diameter. The fire was started and allowed to burn until a layer of hot glowing coals developed. The fire was approximately three feet in diameter with an open, tepee-like structure, and was smoke free throughout the shell burning process indicating an oxidizing atmosphere. The shells were placed directly on the hot coals in the center of the fire. The shells were allowed to burn for up to 24 minutes and the fire reached a maximum temperature of approximately 420 degrees Centigrade. The temperature of the fire was measured with an analog pyrometer. With the shell only reaching a maximum temperature of 420 degrees C, the chemical composition of the calcium carbonate was not changed to calcium oxide. After approximately six minutes in the fire the shells began to flake and burn. After nine minutes on the fire the shells were hot enough that they glowed red in the dark. At this time the shells became very brittle and began delaminating into thin sheets. After 14 minutes and up to 24 minutes the shells were removed from the fire. There was a burning, ashy smell associated with the burned shells. The shells were gray in color after burning and had delaminated into sheets separating along the growth rings. After cooling the burned shells were easily crushed between the fingers and were crushed into a fine powder which contained larger pieces of shell. This crushed shell was mixed with clay as temper prior to the addition of water. Much of the crushed, burned shell was a fine gray powder that when mixed with the clay and water was no longer visibly distinguishable from the clay mixture. There were larger pieces of crushed shell that were easily visible in the clay mixture and prepared tiles.

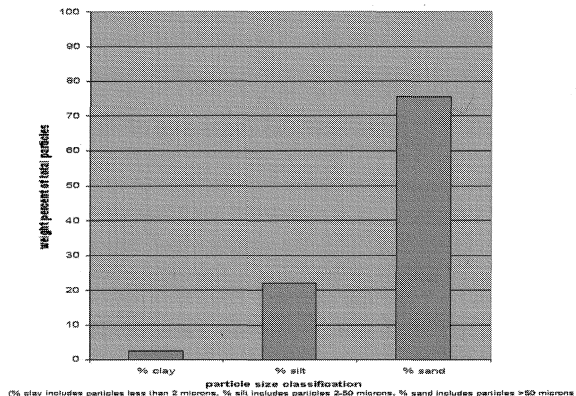
A sample of burned and crushed shell was submitted to the Milwaukee Soils Laboratory for particle size analysis. Figure 3 gives a breakdown of the particle size of the burned and crushed shell into clay, silt, and sand particle size ranges.

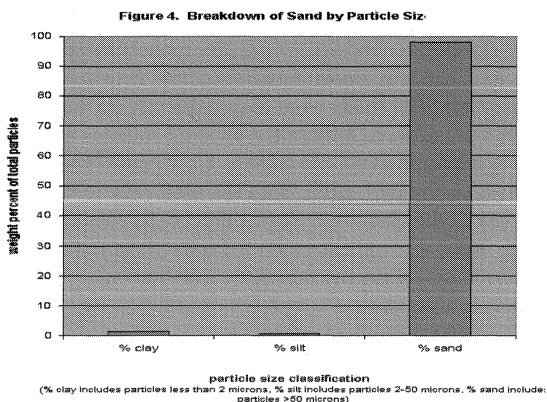
Sand. The grit used for tempering the clay was silica sand obtained from a sandbar on the Upper Mississippi in the La Crosse Area. Large pieces of organic material were removed prior to use as temper.

A sample of the sand was submitted to the Milwaukee Soils Laboratory for particle size analysis. Figure 4 gives a breakdown of the particle size of the sand into clay, silt, and sand particle size ranges.

Water. To avoid the introduction of ions such as iron and chlorine that could potentially have a large effect on the properties of clay, water from the Mississippi was used in the clay mixtures, to more closely approximate the water used by the Oneota. (See Rice 1987 pp 59-60 for a discussion of the impact that ions introduced in the water can have on clay properties.)

Figure 3. Breakdown of Shell by Particle Size





Summary. Table 2 summarizes the physical properties of the raw materials

Table 2. Physical Properties of the Raw Materials Used.

	density (gm/cc)	color	Munsell classification	soil classification ¹
Carter clay	0.96	yellowish brown	10YR 5/4	silty loam
north clay	1.10	pale brown	10YR 6/3	clay
shell temper	0.97	light gray	GLE Y 7/N	sandy loam
sand temper	1.52	light gray	2.5Y 7/2	sand
water	1.00	not applicable	not applicable	not applicable

Clay Mixture and Tile Preparation

Four batches of tiles were prepared from clay mixtures varying in the type of clay used and the type and amount of temper added. Three batches of tiles were made using clay from the researcher's property. A fourth batch of tiles was prepared using clay obtained from the source on the north side of La Crosse. Tiles were made from raw clay, clay tempered with sand, and clay tempered with shell. An identification number was etched on the back of each wet tile (e.g. CN-01A-S, CG-01A-L, CS-01A-S, and NN-04A-S). In this alpha-numeric identification the first letter identifies the source of the clay used. The letter C represents clay from the researcher's (Carter's) property and N represents clay from the north side of La Crosse. The second letter identifies the type of temper added. N represents no temper, G represents grit temper, and S represents shell temper. The following two digit number indicates the batch of tiles (e.g. 01 is the first batch of tiles made). The letters A through E identify the individual tile and the letters S or L represent whether the tile is a small tile (approximately 4 centimeters by 7 centimeters) or a large tile (approximately 6 centimeters by 10 centimeters).

A Tile Data Sheet was prepared for each tile. This data sheet lists the ambient conditions at the time the tile was prepared, the clay type, a description of how the clay was prepared, the temper type, a description of how the temper was prepared, and the amount of clay, water, and temper added to the mix. Additionally, notes and observations on the way the materials were mixed and the workability of the clay mixture were recorded on the back side of the data sheet.

When preparing the clay mixture, the amount of the clay and temper used was weighed with a scale to one tenth of a gram. These dry materials were mixed together by hand in a small plastic mixing bowl. Water was then added to the dry materials in one milliliter increments from a 5 milliliter syringe until the clay mixture was wetted to the point of plasticity. The wet clay mixture was mixed and kneaded by hand for approximately two minutes before being pressed into a tile mold. Tile molds were made from one centimeter strips of aluminum bent into the desired rectangular tile size. After pressing the wet clay mixture into the tile molds and scraping off excess clay with a steel scraper, each side of the tile was lightly wetted with a finger to smooth out surface imperfections. The tile was removed from the mold and the identification number was etched into one side. On the other side two parallel lines were etched into one of the corners of the tile. These lines were etched at a known distance apart and were used to measure the linear shrinkage of the tile throughout the drying process. Additionally the length, width and thickness of the tile was measured in reference to these lines throughout the drying process to record volumetric shrinkage. The bowl used to mix the clay bowl was washed and dried between each batch of clay mixed. The wet tile was weighed and measured and the Munsell color classification recorded. The handling of the wet tiles was initially difficult as they would bend easily. After about one hour of drying the tiles reached a leather-hard state and could be handled with ease.

Amount of Shell Temper. Various literature sources indicate that pottery can be tempered with anywhere from 10% to 60% shell by volume (Betts 2000:137). One of the batches of tiles was prepared to evaluate various concentrations of burned and crushed shell. The first tile was prepared from raw Carter clay with no temper as a control for comparison with the other tiles. Additional tiles were prepared with Carter clay and 20 percent shell temper by dry weight, 30 percent shell temper by dry weight, 40 percent shell temper by dry weight and 50 percent shell temper by dry weight. Adding 20 weight percent shell made the clay mixture less sticky than the untempered clay and was from that standpoint easier to work with. At 30 percent shell the workability and plasticity of the mixture was somewhat diminished compared to that of the 20 percent shell mixture. At 40 and 50 percent shell temper the plasticity decreased dramatically and it was difficult to keep the clay in a cohesive mass. The clay mixture had a tendency to crumble as it was handled and the finished tile was weak and difficult to handle. Based on this it is unlikely that a clay mixture with this amount of shell could be used to form a vessel.

Comparison of Clays Used. Another batch of tiles was prepared to compare the clay from the researcher's property with clay obtained from the source on the north side of La Crosse. Tiles made from each clay were prepared using similar methods and were fired at the same time to ensure similar firing conditions for the comparison. The plasticity of the clay from the north side of La Crosse was considerably better than the clay from the Carter property. The clay was less sticky and easier to work. The tiles, once formed were stiffer and easier to handle immediately after they were prepared in contrast to the tiles made from Carter property clay which were less stiff, bent easily, and were difficult to handle until they dried to a leather hard state. The plasticity of clay increases with decreasing particle size and an increasing number of small particles (Rice 1987:38). The particle size data which shows the north clay contains 50.7 percent clay-size particles by weight in comparison to 19.0 percent by weight for the Carter clay supports the fact that the north clay was observed to be more plastic and easier to work.

As with the tiles prepared from Carter clay to evaluate the amount of shell temper used, a series of tiles with varying concentrations of shell temper were prepared using the north clay.

The first tile was prepared from raw clay with no temper as a control for comparison with the other tiles. The second tile was prepared from a mix of 80 percent clay and 20 percent shell temper. The plasticity of the clay mixed with 20 percent shell temper was similar to that of untempered clay. The third tile was prepared from a mix of 70 percent clay and 30 percent shell temper. With 30 percent shell temper the plasticity and workability of the clay decreased somewhat. This is similar to the workability of the Carter property clay when tempered with shell in similar quantities.

Tile Air Drying

The tiles were allowed to air dry from one to four weeks prior to firing. The tiles were prepared during the summer and allowed to dry in an outdoor shed which protected the tiles from rain but allowed them to dry at nearly ambient temperature and humidity. Daytime temperatures ranged in the 70s and 80s (degrees Fahrenheit). The relative humidity was typically from 60 to 80 percent. Nighttime temperatures ranged in the 60s and 70s (degrees Fahrenheit). The tiles were weighed to ensure that they had reached a constant weight prior to firing. It typically took from 50 to 67 hours for the tiles to reach a constant weight. A Tile Drying Log was completed for each tile. This log records observations throughout the period that the tile was air dried. The observations recorded include: the date, time, wet bulb temperature, dry bulb temperature, and percent humidity, the weight of the tile, the dimensions of the tile, the linear shrinkage distance, the Munsell Color Classification and observations.

There are two mechanisms for water loss and shrinkage for the clay tiles. The first is the loss of water that is mechanically combined with the clay. The second is the loss of water that is chemically combined in the clay. There are four kinds of water in the wet clay tile. They are:

- 1) shrinkage water
- 2) pore water
- 3) surface absorbed water
- 4) interlayer and crystal lattice water (also known as water of hydration)

The first type of water that is lost as the clay tile dries is shrinkage water. Shrinkage water surrounds and separates the clay particles. As the shrinkage water is removed the clay tile will shrink until the clay particles are touching each other. The shrinkage water evaporates from the clay at a constant rate and the time period when shrinkage water is evaporating is known as the constant rate period of drying. When all the shrinkage water is evaporated the clay tile will no longer shrink during the air drying process even though the clay still contains water. The point when all the shrinkage is evaporated is called the critical point. The clay tile is rigid at this point and is referred to as leather hard. The moisture remaining in the tile at the critical point is called the critical moisture content (Rice 1987: 64).

The constant rate of drying can be seen by plotting the weight of the clay tile over time as it air dries. Figure 5 is an example of the weight loss for an untempered tile plotted over time. The constant rate period of drying is seen on the left-hand side of the plot and occurs until the critical point is reached at a weight of 53.3 grams and 577 minutes after which the tile no longer dries at the same rate as can be seen in Figure 5. At the critical point the shrinkage water has evaporated and the remaining water in the tile is pore water, surface absorbed water and crystal lattice water.

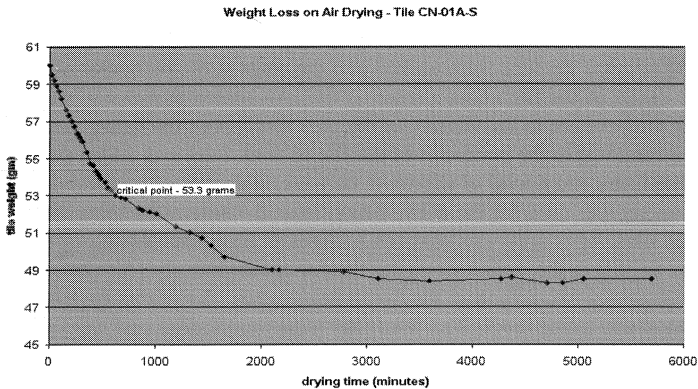


Figure 5. An example of the water weight lost during air drying of a clay tile.

The next type of water to evaporate from the clay tile is the pore water. The pore water is the water left between the clay particles that are now touching after the loss of the shrinkage water. The pore water is lost more slowly than the shrinkage water because it takes longer for the water to move from the interior of the clay tile to the surface where it evaporates (Rice 1987:65). The porosity of the clay tile can be determined by subtracting the dry weight of the tile from the weight of the tile at the critical point. This gives the weight of the pore water. Because the density of water is one gram per cubic centimeter, the weight of the pore water is equivalent to the volumetric porosity of the tile in cubic centimeters. The percent porosity can then be determined by dividing the volumetric porosity of the tile by the total tile volume. (The total tile volume can be calculated by adding the volumetric porosity to the clay body bulk volume which is the volume of the dry materials (i.e. clay and temper) in the tile.)

After the shrinkage water and pore water have evaporated, surface absorbed and interlayer water remain. The surface absorbed water is water that is absorbed by the air dried or drying tile. Even though the tile appears dry it holds a microscopic film of water on its surface. This film is a result of the equilibrium between the vapor pressure of the water in the clay and the vapor pressure of the water in the atmosphere. Changes in humidity change the amount of surface absorbed water as is evident in the slight fluctuation of the air dried tile weight with fluctuations in humidity. The interlayer water is chemically combined in the internal structure of the clay molecules. This water is the last water lost and is not driven off until the tile is fired (Rice 1987:65).

Firing

The tiles were fired during four different firing events. The following describes the general procedures used for firing the tiles. The tiles were fired when the weather was calm and mild (ambient temperature 16 to 20 degrees Centigrade). A small fire was constructed using miscellaneous hardwoods of approximately one to five inches in diameter. The fire was allowed to burn and additional fuel was added for 20 minutes to one hour until a bed of coals was established. With the total fuel used up to this point ranging from approximately two to three cubic feet. The fire was approximately two to three feet in diameter with approximately two to three foot flames. The fire had an open, tepee-like structure and a slight amount of smoke was visible (less than 10% opacity) indicative of an oxidizing atmosphere. After the

fire was established the tiles were preheated by setting them approximately 2 feet from the fire, periodically rotating them, and gradually moving them closer. After preheating for 15 minutes to 30 minutes the tiles were moved into the coals at the perimeter of the fire and then into the center of the fire under a tepee of fuel. An analog pyrometer was used to measure the firing temperature in the vicinity of the tiles throughout the firing. After firing when the tiles were removed to the perimeter of the fire to cool. Fuel was added periodically throughout the firing and a total of approximately six cubic feet of hardwood, one to five inches in diameter was used for the entire firing, from the time of building the fire to the end of cooling the tiles. After approximately ten minutes of firing the tiles glowed red. The tiles were allowed to cool at the perimeter of the fire for approximately 15 minutes and then were moved approximately two feet from the fire. After another 15 minutes the tiles could be handled and were warm to the touch. The fired tiles were fire clouded and had white ash specks that could be wiped off. The fire clouding is indicative of a uneven firing atmosphere that is not entirely oxidizing.

One set of tiles prepared from both clay types was fired along with Oneota pottery sherds. Refiring sherds at the same conditions as tiles prepared from local clay sources allows for a better comparison of the clays used in the test tiles and the clay used by the Oneota by bringing out the original post firing color of the pottery that may have been altered by surrounding soil conditions. After refiring the tiles and sherds were compared in terms of hardness, color and porosity.

One group of tiles was fired in a reducing atmosphere which was approximated by smothering the coals and heating tiles with sawdust. The tiles that resulted from firing in this type of reducing atmosphere were very dark brown to black in color and were radically different than actual Oneota sherds. It is unlikely that the Oneota smothered their fires with organic material in this manner.

Tile Parameter Testing

Each tile and the Oneota sherds were tested to measure a number of parameters.

Hardness. The hardness of each tile and sherd was determined using a Mohs Scale of Hardness Mineral Test Kit before and after firing.

Color. Each tile and sherd was classified using Munsell Soil Color Charts before and after firing.

Shelf Life Testing. The fired tiles were stored in a shed so they remained at ambient temperature and humidity, but sheltered from rain. This testing was conducted during the months of July through November. Each fired tile was periodically weighed to determine if changes were occurring as the fired tile aged. The change of most concern was the absorption of water from the atmosphere that might cause the tiles to crack, spall, or exfoliate. This is a concern in shell tempered ceramics fired at temperatures high enough to change calcium carbonate in the shell into calcium oxide. Calcium oxide is hygroscopic, meaning it absorbs moisture from the atmosphere. The absorption of moisture by the calcium oxide causes fired pottery to crack and spall.

While the weight of the tiles fluctuated a few tenths of a gram depending on the humidity, none of the tiles fired during the course of this research showed a significant increase in weight. The tiles that were fired at temperature exceeding 800 degrees Centigrade however did exhibit exfoliation and spalling after 16 days of shelf life testing. Some of the tiles were fired at temperatures as high as 850 degrees Centigrade. Based on the fact that spalling did

not occur in tiles fired at or below 800 degrees Centigrade it can be assumed that these tiles did not reach the critical temperature where calcium carbonate is converted to calcium oxide.

Water Resistance and Apparent Porosity Testing. Each of the fired tiles and refired Oneota sherds underwent testing to determine apparent porosity. The tiles and sherds were dried in a 110 degree Centigrade oven for two hours. After drying each tile and sherd was weighed. The tiles and sherds were then submerged with boiling water and returned to 120 degree Centigrade oven which was turned off. The tiles and sherds were allowed to return to room temperature and soak in the water for eight hours and ten minutes. The surface of each tile and sherd was dried and the soaked tiles were reweighed. The difference in weight of the soaked tile and the oven dried tile equals the apparent porosity of the tile. Apparent porosity includes the larger open pores connecting to the surface of the tile but does not include closed pores which do not open on the tile surface or micropores. Apparent porosity is related to absorption (Rice 1987:351). During the porosity testing the water resistance of each tile was determined by exposing each tile to water. All tiles and sherds withstood exposure to water and are water resistant.

RESULTS

Amount of Shell Temper

Based on observations made while preparing clay tiles containing various amounts of shell temper, clay mixtures containing 20 percent shell by dry weight was less sticky and easier to work than untempered clay. The workability and plasticity of a clay mixture containing 30 percent shell by dry weight was diminished compared to clay mixtures containing 20 percent shell by dry weight or no temper. At 40 and 50 percent shell temper the plasticity decreased dramatically and it was difficult to keep the clay in a cohesive mass. The clay mixture had a tendency to crumble as it was handled and the finished tile was weak and difficult to handle. Based on this it is unlikely that a clay mixture with shell concentrations greater than 30 percent by dry weight could be used to form a vessel.

I visually compared the test tiles prepared with varying amounts of shell temper with Oneota sherds from the Mississippi Valley Archaeological Center and found that the tiles tempered with 20 percent shell most closely resembled the amount of shell visible in the Oneota sherds.

Stoltman performed thin-section analyses of locally made, shell tempered pottery sherds recovered at the Fred Edwards site near Prairie du Chien, Wisconsin and found them to contain shell temper ranging from 5 to 18 percent by volume (Stoltman 1991:112). (Note: In this case percentage based on dry weight is nearly the same as volume percent as the density of the shell and clays used in the test tiles is approximately 1 gram per cubic centimeter. See Table 2.) In his analysis Stoltman indicated that clay sized particles could not be discriminated individually under the petrographic microscope (Stoltman 1991:110) and assumed all silt-size particles to be natural inclusions except for distinctive tempers such as shell, where it is sometimes possible to identify reliably some temper grains in the silt size range. In cases where silt-size shell particles were identifiable, Stoltman assigned them a value of 1 in an ordinal size scale based on maximum grain diameter where 1 = fine (0.0625-0.249 mm); 2 = medium (0.25-0.499 mm); 3 = coarse (0.50-0.99 mm); 4 = very coarse (1.0-1.99 mm); and 5 = gravel (>2.0 mm) (Stoltman 1991:108). Stoltman gave a Temper Size Index of 3.5 for the local shell tempered sherds analyzed from the Fred Edwards site (Stoltman 1991:112). This indicates that clay and silt-size shell particles are not included in the volume percentage range given for the local shell tempered sherds.

The particle size analysis of the burned and crushed shell used for temper in tiles prepared as part of this research, indicate that 27.4 percent of the shell temper is in the clay and silt size range (0.0625 mm and smaller) and would not have been accounted for in the type of thin section analysis performed by Stoltman. The particle size of the shell temper is a factor of the way in which it was prepared, however if one assumes that the shell temper preparation techniques used by the Oneota was similar to the techniques used in this research, the results of Stoltman's thin section analysis should be adjusted by approximately 25 percent to account for the shell temper included in the clay and silt size range. Making this adjustment, the percent shell temper by volume in the local Fred Edward's sherds would range from 7 to 24 percent. Therefore the observation that clay containing greater than 30 percent shell temper by dry weight has decreased workability and the results of the visible comparison of test tiles with Oneota sherds which indicate approximately 20 percent shell temper by weight are consistent with Stoltman's findings using thin-section analysis of shell-tempered pottery sherds found at the Fred Edwards site near Prairie du Chien, Wisconsin. In conclusion, based on the workability of the clay/shell mixtures and the visible comparison with Oneota sherds, the Oneota were likely using about 20 percent shell by dry weight in their clay mixtures.

Advantages of Shell Temper During the Drying Process

Cracking during the drying process occurs if water evaporates faster from the surface of the clay than it can be resupplied by the interior. As a result the surface shrinks more rapidly than the interior of the clay body. The resulting cracks may not be visible during drying but will show up upon firing. Therefore drying the clay slowly is important (Rice 1987:67). Minimizing the shrinkage water and increasing the porosity of the clay body will help avoid cracking during the drying and firing process. Figure 6 shows the average percent porosity and the amount of shrinkage water contained in untempered, grit tempered and shell tempered tiles from batch one. This analysis shows that the shell tempered tiles have a greater porosity and therefore shrink less during the drying process than either the grit tempered or untempered tiles. Figure 7 also illustrates this by showing the shell tempered tile has a shorter constant rate of drying period during which shrinkage water is lost than either the grit tempered or untempered tiles.

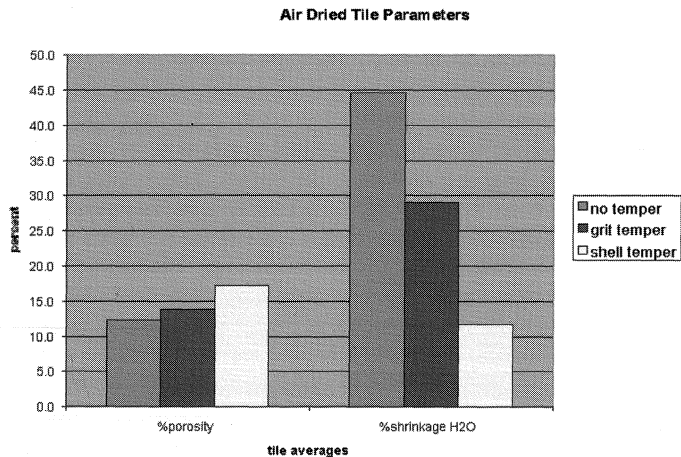


Figure 6. The percent porosity and percent shrinkage water contained in untempered, grit tempered, and shell tempered tiles.

Variation in Air Drying Weight Loss

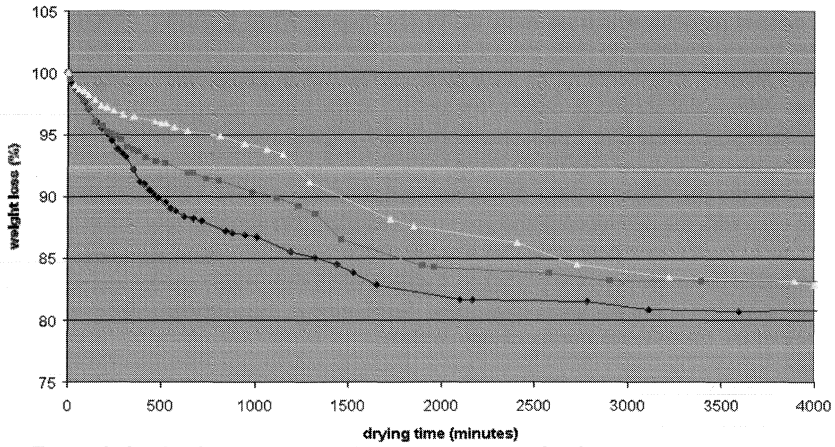


Figure 7. The variation in air drying weight loss for untempered, grit-tempered, and shell-tempered

Variation in Air Drying Weight Loss

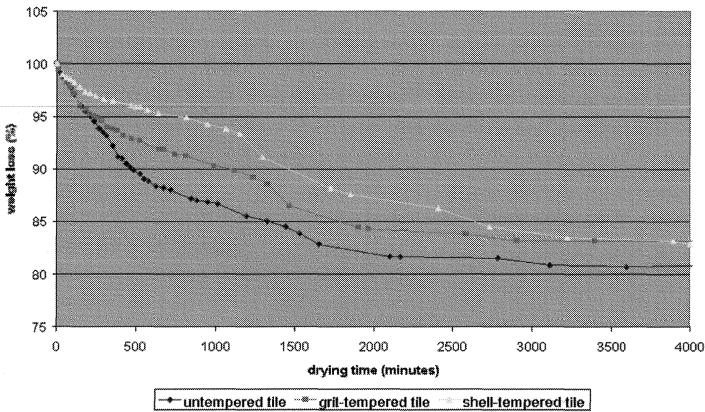


Figure 8. Comparison of the amount of linear drying shrinkage (LDS), volumetric drying shrinkage (VDS) and thickness shrinkage for untempered, grit tempered and shell tempered tiles.

Another interesting thing that occurs with shell temper is that air drying shrinkage is not uniformly the same in all directions. The shell particles are flat and plate-like in comparison to grit or sand particles which are more uniformly shaped. When preparing the tiles, the shell particles have a tendency to align perpendicular to the direction of pressure rather than being randomly aligned. Alignment of the shell particles resulted in differential drying rates and therefore differential shrinkage rates when looking at tile length compared to tile thickness. During air drying of the tiles I measured linear drying shrinkage (LDS) in the longest direction of the tile and volume drying shrinkage (VDS) by measuring the length width and thickness of the tile. With uniform drying and shrinkage the ratio of VDS to LDS is 3 to 1, as was the case with the untempered and grit tempered tiles. The shell tempered tile had a ratio of VDS to LDS of 6 to 1 indicating that less shrinkage was occurring in the linear direction (or perpendicular to the pressure placed on the tiles when they were made) and more shrinkage was occurring in the thickness of the tiles. See Figure 8. This further reduces the tendency of the tile to crack during drying or firing.

Firing Conditions

One of batch of shell tempered tiles was fired in an oxidizing atmosphere in what is known as a Draw Trial. The tiles were removed to the perimeter of the fire throughout the firing at 100 degree Centigrade intervals starting with 500 degrees Centigrade. The first tile was removed when the fire surrounding the tiles reached 500 degrees Centigrade after four minutes of firing. The second tile was removed when the fire reached 600 degrees Centigrade after five minutes of firing. A red glow was visible at the corners of this tile at the time it was removed. The third tile was removed when the fire reached 700 degrees Centigrade after six minutes of firing. A red glow was visible at the corners of this tile at the time it was removed. The fourth tile was removed when the fire reached 800 degrees Centigrade after nine minutes of firing. The entire tile was glowing red when it was removed from the fire. The fire reached a maximum temperature of 850 degrees Centigrade after seventeen minutes of firing after which the temperature began to drop. The fifth tile was

removed after 25 minutes of firing when the temperature began falling to 840 degrees Centigrade. The entire tile was glowing red when it was removed from the fire. The sixth tile was removed after 31 minutes of firing when the temperature had fallen to 720 degrees Centigrade after

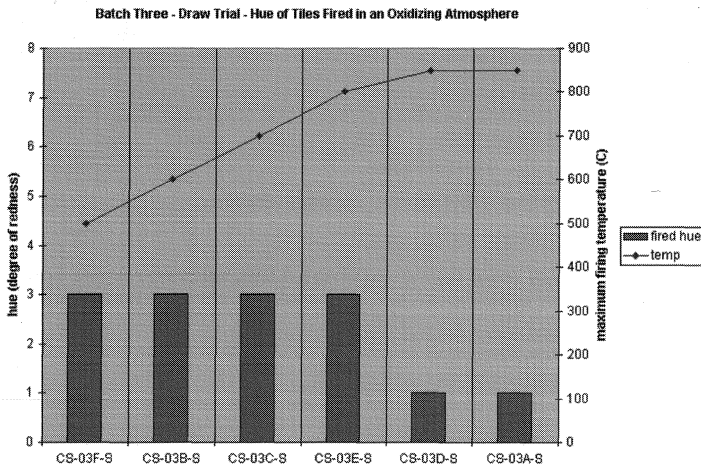


Figure 9. The change in hue with increased firing temperature.

peaking at 850 degrees Centigrade. The entire tile was glowing red when it was removed from the fire. When a tile was removed from the fire it was moved to the perimeter of the fire so that the tile would cool slowly. This cooling took place in an oxidizing atmosphere. The tiles were gradually moved back from the fire and cooled to the point they could be handled over 36 to 41 minute periods. Fuel was added periodically throughout the firing and a total of approximately six cubic feet of hardwood, one to five inches in diameter was used for the entire firing, from the time of building the fire to the end of cooling the tiles. It should be noted that in order for the fire to reach 850 degrees Centigrade additional fuel had to be aggressively added to the fire and even then it was hard to maintain the fire at this high of a temperature.

The tiles that reached a maximum temperature greater than 800 degrees Centigrade had a very different appearance than those that did not exceed 800 degrees. The tiles fired at greater than 800 degrees were a light olive brown color in comparison to the more reddish hue of the tiles fired at lower temperatures. See Figure 9. Additionally the color of the shell temper in the tiles fired at greater than 800 degrees Centigrade changed to a chalky white color in comparison to the gray color of the shell in the tiles fired at lower temperatures. Furthermore, after 16 days of shelf life testing the tiles fired at greater than 800 degrees Centigrade began to chip and flake indicating that the calcium carbonate in the shell had converted to less stable calcium oxide. Interestingly all tiles, including the tile fired at 500 degrees Centigrade for 4 minutes were adequately fired to be resistant to water. Table 3 summarizes the firing conditions and other parameters of the tiles fired during the Draw Trial.

Table 3. Draw Trial - Shell-Tempered Tile Firing at Various Temperatures

Tile Number	Firing Time (minutes)	Max. Firing Temp. (C)	Heated to Visible Glow?	Mohs Hardness	% wt loss on firing	Munsell Color Class	Shell Color	Tile Flaking	Water Resistant?
CS-03F-S	4	500	no	1	2.8	7.5YR 5/4.5	gray	none	yes
CS-03B-S	5	600	corners only	1	3.1	7.5YR 5.5/4	gray	none	yes
CS-03C-S	6	700	corners only	2	3.1	7.5YR 5.5/6	gray	none	yes
CS-03E-S	9	800	entire tile	2	3.9	7.5YR 5.5/6	gray	none	yes
CS-03D-S	25	850	entire tile	2.5	5.7	2.5Y 5.5/3	chalky white	after 16 days	yes
CS-03A-S	31	850	entire tile	2.5	5.8	2.5Y 5.5/3	chalky white	after 16 days	yes

Test Tile and Oneota Sherd Comparison

One batch of tiles prepared from both clay types was fired along with Oneota pottery sherds. Refiring sherds at the same conditions as tiles prepared from local clay sources allows for a better comparison of the clays used in the test tiles and the clay used by the Oneota. After refiring the tiles and sherds were compared in terms of hardness, color and porosity. The results of the comparison are summarized in Table 4. The hardness of the test tiles and the refired sherd are comparable at 2 to 2.5 on the Mohs Hardness Scale. The color of the tiles are close in similarity, yet the clays used to prepare the test tiles have a redder hue which may be indicative of a higher iron content in those clays as compared to the clay used in the Oneota sherds. The apparent porosity of the Oneota sherds is slightly lower than the apparent porosity of the test tile clays. This may be due to the fact that the clay used for the

sherds is of a finer particle size than the test tile clays.

Table 4. Comparison of Shell Tempered Tiles and Refired Oneota Sherds

	Mohs Hardness	Munsell Color Classification	Apparent Porosity
Carter Property Clay Tiles	2 to 2.5	7.5YR 5.5/6	15.3
North Clay Tiles	2.5	7.5YR 5/4.5	14.1
Unprov. Sherd - Large	2	10YR 6/4	12.3
Unprov. Sherd - Small	2.5	10YR 6.5/4	11.9

Comparison of Clays Used

The plasticity of the clay from the north side of La Crosse was considerably better than the clay from the Carter property. The clay was less sticky and easier to work. The tiles, once formed were stiffer and easier to handle immediately after they were prepared in contrast to the tiles made from Carter property clay which were less stiff, bent easily, and were difficult to handle until they dried to a leather hard state. Research indicates that the plasticity of a clay increases with decreasing particle size and an increasing number of small particles (Rice 1987:38). Therefore the particle size data which indicates that the clay from the north side of La Crosse has a higher percentage of clay-size particles (less than 2 microns) than the clay from the Carter property (50.7 percent by weight versus 19.0 percent by weight respectively) supports the fact that the north clay was observed to be more plastic and easier to work.

Tiles made from clay from the north side of La Crosse experienced a greater amount of shrinkage during the air drying process than tiles made from the Carter property clay. See Table 5. An untempered tile made from the north side clay experienced so much shrinkage that it likely experienced microscopic cracking during air drying which resulted in the tile breaking apart during the firing process. This increase in shrinkage is due to the finer particle size of the north side clay as compared to the Carter property clay.

Table 5. Comparison of Clays - Shrinkage Upon Air Drying

Clay Type	Tile Number	% Shell by Dry Weight	% Linear Drying Shrinkage	% Volume Drying Shrinkage
Carter Clay	CN-02A-S	0	5.7	9.4
	CS-02A-S	20	5.0	5.1
	CS-02B-S	30	1.7	6.5
	CS-02C-S	40	1.7	3.0
	CS-02D-S	50	0	3.1
North Side Clay	NN-04A-S	0	8.3	14.1
	NS-04A-S	20	6.7	13.5
	NS-04C-S	20	6.7	13.2
	NS-04B-S	30	5.0	10.7

CONCLUSIONS/DISCUSSION

The Oneota were able to successfully produce large shell tempered ceramic vessels despite the challenges that arose from using shell as temper. Shell was burned prior to crushing. While Bronitsky and Hamer and Feathers suggest that burning the shell prior to crushing and using as temper may help to reduce the cracking and spalling problem, they agree that this benefit was likely a secondary and unknown benefit and the primary purpose of burning the shell appears to have been to make it more friable and easier to crush (Bronitsky and Hamer 1986:97, Feathers 1989:581 and Bronitsky 1989:590). Based on experimentation with burning and crushing the shell this definitely seems to be the case. Unburned shell is nearly impossible to crush into pieces in the size range found in Oneota pottery and burning the shell for a relatively short duration of 20 to 30 minutes makes them friable and easy to crush with the fingers.

Based on the workability of the clay/shell mixtures and the visible comparison with La Crosse Locality Oneota sherds, the Oneota were likely using about 20 percent shell by dry weight in their clay mixtures. They could be using less than 20 percent but it is unlikely that they used more than 30 percent as the workability of the clay diminishes significantly at this concentration of shell. At greater than 30 percent shell temper the plasticity decreased dramatically and it was difficult to keep the clay in a cohesive mass. The clay mixture had a tendency to crumble as it was handled and the finished tile was weak and difficult to handle. Based on this it is unlikely that a clay mixture with more than 30 percent shell could be used to form a vessel.

Based on observations made while firing the test tiles, the Oneota likely started the firing process by burning enough fuel to establish a bed of hot coals, preheating their pots at the perimeter of the fire and then moving them into the center of the fire. They may have added fuel during the firing process. However, they would not have done so in a rapid or aggressive manner. Adding too much fuel at a time during the firing process would have raised the temperature above 800 degrees Centigrade and resulted in pots that cracked and spalled. Feathers indicated that having the ability to prevent over firing was a necessary technical advance before shell tempering could be adopted (Feathers 1989:587). It seems that this could have been done by moderating the amount and rate at which fuel was added to the fire. Based on the observations made from firing the test tiles, firing the pots to a hard, water resistant state would not have required a long firing time. One challenge that would have arose with the large Oneota vessels would have been getting all portions of the pot heated. To do this they may have constructed an open tepee of fuel over the pots to fire the portions of the pot that were not sitting in the coals or may have rotated the pots during the firing process. There is some evidence of stacking broken pieces of pottery around whole pots during the firing process as a way to more evenly distribute the heat around the large pots. This is evident on Oneota pots that show clearly delineated areas of oxidized firing and clearly delineated areas of fire clouding. Firing pottery using the oxidizing, open-air firing techniques described in this paper at around 700 degrees Centigrade but not greater than 800 degrees Centigrade would result in pottery that has similar properties to those of La Crosse Locality Oneota sherds examined.

The use of shell temper increases the porosity and reduces shrinkage allowing the use of clays with greater plasticity and finer particle size. This would have allowed the Oneota to use finer grades of clay that would have enabled the manufacture of their large vessels. Coarser clays and clays tempered with grit may not have exhibited the workability properties necessary to produce such large vessels.

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¹ Soil classification is from the Ternary diagram of soils classification by particle size. (Rice 1987:39, figure 2.3)