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A Materials-science Approach to Understanding Limestone-tempered Pottery from the Midwestern United States

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Limestone temper is common in Late Woodland period pottery from the central United States. Calcium carbonate in the mineral form of calcite, the predominant component of limestone, makes sticky clay more workable, but it also causes pots to spall if fired over 600°C, the low end of the open-firing temperature range. A test of performance characteristics of limestone-tempered prehistoric pottery replicates demonstrates that they are significantly more resistant to mechanical stress than are grit- or grog-tempered replicates fired to 600°C. Limestone-tempered pottery also appears to be more resistant to thermal shock because the calcium carbonate allows for thinner vessels to be made and possibly because the expansion rate of calcite is very similar to that of clay. Some effort must have been taken by prehistoric potters to overcome the spalling produced by limestone temper, suggesting that the inclusion of limestone was a technological shift requiring refinement of pottery-manufacturing techniques.

Keywords: POTTERY, MATERIALS-SCIENCE, THERMAL STRESS, MECHANICAL STRESS, LIMESTONE TEMPER.

Introduction

Limestone as a tempering agent has received little attention from archaeologists. With few exceptions (e.g. Morgan, 1985), when limestone temper is considered in any depth it is as part of a ware or type description (e.g. Chapman, 1980). This comes as a surprise, as limestone was widely, though sporadically, used in the midwestern United States* and was the predominant temper type in most areas of Missouri during the Late Woodland period (AD 400–900) (Chapman, 1980: 80–81) and in the American Bottom of western Illinois during the Emergent Mississippian period (AD 750–1000) (Kelly *et al.*, 1984: 129–157). In the Midwest, the shift to limestone was rapid, occurring between AD 600 and 700 (Hoard, 1992: 90–96; O'Brien & Hoard, in press). Evidence of abrupt technological change often tempts archaeologists to invoke population intrusions as a cause (e.g. Emerson & Fortier, 1986: 511; Munson, 1986), though we propose it is more productive to investigate the functional attributes of different temper types to determine if shifts are indicative of technological changes in pottery.

In this study, test specimens made of naturally occurring clay and crushed granite (grit),† fired clay (grog), or limestone that replicate the paste of prehistoric midwestern cooking vessels are compared using materials-science methods. Using this approach, the functional properties of these different clay mixtures can be isolated and quantified. Tests of the replicated prehistoric pottery demonstrate that limestone temper can produce stronger vessels than can grit or grog temper, both commonly used in the Midwest. Also,

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*The midwestern United States includes areas drained by the upper and central Mississippi River and the lower Ohio and Missouri rivers. Hereafter, this region will be referred to as the Midwest.

†While crushed granite is used in this study, grit typically refers to crushed igneous or metamorphic rock, most commonly granite or schist.

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measurements of wall thickness of limestone-tempered vessels show that they are thinner than those of contemporaneous grog-, grit-, or sand-tempered vessels. These qualities are significant, as they increase the longevity and heat conduction of cooking vessels. We also demonstrate that for limestone temper to be used successfully, potters must take special care in manufacturing their wares.

Limestone-tempered Pottery from Missouri and Illinois

Prehistoric people used many kinds of temper; among the most common are sand, grit, grog, limestone, and shell. Limestone temper was used in many areas of North America, including the central peninsular Gulf Coast (Widmer, 1988: 68–70);* north-central Alabama and Tennessee (Sears & Griffin, 1953); southern Indiana (Kellar, 1979; Muller, 1986); southeastern Ohio (Murphy, 1975); western and west-central Illinois (Griffin, 1952; Taylor, 1958; Struever, 1960; Kelly *et al.*, 1984; Farnsworth & Koski, 1985; Morgan, 1985, 1986); eastern, central, southwestern, and western Missouri (Wedel, 1943; Vehik, 1978; Chapman, 1980; O'Brien, 1987; Fox, 1992; Hoard, 1992); southeastern and east-central Kansas (Witty, 1982; Williams, 1988); and northeastern Oklahoma (Baerreis, 1953).

In Missouri and Illinois, limestone temper rarely was used in Early Woodland pottery (1000–250 BC) (Farnsworth & Asch, 1986: 356, 362, 415) and appears only sporadically in Middle Woodland (250 BC–AD 400) pottery (Griffin, 1952; Morgan, 1985; 1986: 374). Middle Woodland Hopewellian ware from western Illinois was an important exception, where limestone was the preferred temper type. In the central and lower Illinois Valley, much of the Hopewell ware was limestone tempered (Griffin, 1952: 115; Braun, 1985: 117; Farnsworth & Koski, 1985: 124; Morgan, 1986: 200). These small, very thin, well-executed vessels were among the finest wares produced during the Middle Woodland period, unmatched in their artistry until shell-tempered Mississippian wares appeared as early as AD 800 in some areas of the Midwest. However, Hopewell pots were used primarily as mortuary vessels and rarely if ever used for domestic activities such as cooking. This important case will be discussed in more depth later in this paper.

After AD 700, limestone temper became the predominant temper type in parts of Missouri and Illinois. In central and northeastern Missouri, Late Woodland-period sherds (Boone, Moreau, and Meramec Spring phases) were typically limestone tempered. Limestone-tempered wares also are documented from the western and southeastern part of the state (Wedel, 1943; Vehik, 1978). Grog-, sand-, or grit-tempered wares were most common in southeastern Missouri (Chapman, 1980: 80–81). Kelly *et al.* (1984: 128–157) describe Emergent Mississippian wares (*c.* AD 750–950) from the southern portion of the American Bottom of Illinois. The Dohack (AD 750–850), Range (AD 850–900), and George Reeves (AD 900–950) phases all were represented by limestone-tempered wares. In the northern American Bottom, Merrell phase (AD 900–950) pottery was tempered with grit or grog; limestone and shell occurred in less than 10% of the sherds. In the following Edelhardt phase (AD 950–1000), limestone or shell were the predominant temper types.

The shift to limestone temper was coincident with changes in vessel shape. The addition of calcium-rich limestone makes sticky clays more workable; thus the use of limestone may have facilitated the manufacture of a wider range of vessel shapes. In Missouri, limestone-tempered vessels were more globular than the straight-sided, grit-tempered jars that preceded them (Chapman, 1980: 276, 286–287). Dohack phase vessels in western Illinois were more globular than in earlier times, and shoulders and flared rims were more common. During the Range phase, large bowls, pans, and vessels with handles first appeared, and vessels with constricted orifices, possibly bottles, appeared in the following George Reeves phase (Kelly *et al.*, 1984: 142–143). In all regions of Missouri and Illinois where limestone-tempered wares occurred, they were followed by shell-tempered Mississippian pottery, which had an even greater range of vessel shapes.

Shell-tempered pottery has received substantial attention in the archaeological literature (Rye, 1976; Bronitsky & Hamer, 1986; Stimmell, Heimann & Hancock, 1982; Steponaitis, 1983; Feathers & Scott, 1989; Feathers, 1989), primarily because of its near ubiquity in post-AD 900 sites in the Midwest and Southeast. A brief review of studies of shell-tempered pottery is germane, because both shell and limestone are composed primarily of mineral forms of calcium carbonate.

One reason for analytical interest in shell temper is that it has long been known that calcium carbonate can cause severe problems when it is included in pottery. Calcium carbonate is found in the mineral forms of calcite and aragonite in shell and calcite in limestone. When aragonite is heated to 200–400°C, its crystal form changes to calcite (Nesse, 1986: 144). Aragonite fractures into blocks, whereas calcite fractures into plate-like particles. The plate-like structure of shell in prehistoric Midwestern pottery is an indication that it was burned to facilitate crushing (Feathers, 1989: 580–581). When calcite is heated above 600°C† it is

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*Mitchem (1986: 71) warns that some temper identified as limestone may be Fuller's Earth.

†There is considerable variation in cited examples of this threshold temperature: Feathers (1993)—537°C; Stimmell *et al.* (1982: 219)—600°C; Rye (1976: 120)—620°C; Dunnell & Feathers (1991: 30)—700°C; Rice (1987: 98)—870°C. All these authors note that the reaction takes place within a temperature range rather than at a specific temperature and that the intensity and speed of the reaction is dependent upon the firing atmosphere (oxidizing or reducing) and rate of temperature increase. We expect the composition of the clay body also is a significant factor. In any case, spalling can occur within typical prehistoric open-firing temperatures.

converted to calcium oxide and carbon dioxide ($CaO+CO_2$). Upon exposure to moisture, calcium oxide reacts with water to form calcium hydroxide, $Ca(OH)_2$. The calcium-hydroxide crystal has a larger volume than does the calcite crystal, causing cracking and spalling that can destroy a pot (Rye, 1976).

Studies of limestone inclusions in bricks (Butterworth, 1956; Laird & Worcester, 1956) make it clear that "lime blowing," or spalling, is a serious problem in fired clay containing visible pieces of limestone. Therefore, the intentional addition of shell or limestone in prehistoric pottery comes as a surprise. Why would potters use a tempering agent that can ruin a pot at such a low temperature?

There are benefits to be gained from using tempering materials high in calcium carbonate. First, calcium ions released in the wet clay composite increase flocculation, making soft, sticky clay stiffer and thus easier to work (Lawrence, 1972: 77–80; Million, 1975; Stimmell *et al.*, 1982: 220, 227; Feathers, 1990). We see this, for example, in the ceramic assemblage from the Woodall site in the Malden Plain of southeastern Missouri, where a shift to slightly larger and more globular vessels was coincident with the shift from sand- to shell-tempered wares (Dunnell & Feathers, 1991: 29). Furthermore, two minor forms, funnels and bottles, both exclusively shell tempered, appeared at that time. Although the increase in flocculation provided by the addition of calcium carbonate to clay is often discussed, this proposition has not been tested using quantitative techniques. Still, the assertion is supported by the increased diversity of vessel forms and decrease in vessel-wall thickness in areas of Missouri and Illinois coincident with the use of both limestone and shell temper.

A second benefit of calcium-carbonate tempers is that the thermal-expansion rate of calcite is very similar to those of the raw clays used by prehistoric potters (Rye, 1976: 117). In contrast, when quartz, a predominant constituent of sand and grit temper, is heated, it undergoes an α–β crystal conversion at 573 ± 5°C, which increases the crystal volume by roughly 2% (Lawrence, 1972: 112, 125–126; Rye, 1976: 117; Rice, 1987: 95). During firing, or in a fired pot that is reheated, the expansion of a quartz crystal has the potential to create cracks in the more slowly expanding clay body. Large cracks that form during the initial firing and cooling of a pot can destroy the vessel before it can be used. Smaller cracks can (a) become compounded during use, causing vessel failure from thermal stress alone, or (b) weaken the pot, making it more likely to break on impact. The use of tempering agents such as grog or calcite (when conversion to calcium oxide is controlled in the latter), both of which exhibit expansion curves similar to that of the clay, should not cause cracking because of the differential expansion of the temper and the clay body, though this proposition has yet to be tested.

A third benefit of calcium-carbonate temper, at least when it is in the form of shell, is that it creates a strong ceramic composite (especially when large particles of shell are used) that resists cracking (Steponaitis, 1983; Feathers, 1989; Feathers & Scott, 1989). Feathers & Scott (1989: 556) state that it is the plate-like structure of burned shell that arrests crack propagation.

Note that these studies of calcium-carbonate-based tempers have focused on shell. Although limestone and shell are both calcium-carbonate based, it cannot be assumed a priori that limestone will have the same benefits and problems as shell. Because limestone is predominantly calcite, it should increase flocculation and should not cause expansion cracks if it is not heated above 600°C. But when limestone is crushed, its shape is subrectangular, not platy like crushed, burned shell. If it is the fibrous nature of calcite in plates of burned shell that makes the ceramic composite tougher, we would not expect the blocky crushed limestone particles to be as likely to increase the composite's resistance to crack propagation. Still, the sudden and pervasive presence of limestone in pots from parts of Missouri and Illinois is evidence that limestone temper may have properties that make it, in some cases, more desirable than grog, crushed igneous rock, or sand tempers, all of which are widely available in the Midwest.

Morgan's (1985) analysis of pottery from the Middle Woodland Smiling Dan site in western Illinois shows differences in wall thickness for cooking pots tempered with grit, sand, or limestone. Grit-tempered pots are thickest (mean = 7·9 mm), sand-tempered pots are thinner (mean = 7·7 mm), and limestone-tempered pots are thinnest (mean = 5·9 mm). No significant difference in thickness is shown by *t*-tests between grit- and sand-tempered sherds, but limestone-tempered sherds are significantly thinner (Morgan, 1985: 217). Limestone-tempered sherds from the Late Woodland component of the Burkemper site in eastern Missouri also are thinner than grog- or grit-tempered sherds (Hoard, 1992: 96; O'Brien & Hoard, in press).

This evidence suggests two reasons why pots tempered with limestone may be more resistant to thermal shock than those tempered with crushed igneous rock, sand, or grog. First, the thermal-expansion properties of limestone (composed predominantly of calcite) should make it less likely to cause microcracks from the differential expansion of temper and clay than will quartz-rich sand or granite. Second, because limestone makes sticky clay more workable, it may allow for more flexibility in vessel form and for pots to be made thinner. More globular pots are less likely to break from differential thermal expansion during heating than those with conical bases or abrupt angles (Braun, 1983: 118; Rice, 1987: 369). Similarly, thin-wall cooking pots are more resistant to thermal shock because they conduct heat more efficiently, thus decreasing expansion differentials in the vessel (Lawrence, 1972: 182; Braun, 1983, 1985, 1987). However, thinner vessels are less resistant to breaking from impact, so unless the addition of limestone also makes the pot more impact resistant, its usefulness seems limited. This raises a question: does the addition of limestone allow for thinner, stronger pottery? Analysis of the physical properties of limestone-tempered ceramics, when compared to those tempered with grit or grog, shows that limestone is indeed beneficial for creating strong, thin vessels.

Table 1. Measurements of limestone temper in sherds

Sample number	Weight before soaking in HCl (g)	Weight after soaking in HCl (g)	Percent weight of limestone
1	4·64	2·49	46·4
2	8·04	4·57	43·2
3	9·28	6·16	33·6
4	4·00	3·18	20·5

A Test of Mechanical Properties: the Three-point Bend Test

A three-point static bend test was used to examine the mechanical strength, deformation, and energy absorption characteristics of fired-clay test specimens to which crushed granite, grog, or limestone was added. This test (a) offers a quantitative means of assessing the performance characteristics of different clay-temper composites commonly used in the prehistoric Midwest and (b) assists in determining if limestone-tempered wares are more durable than their grit- or grog-tempered counterparts when subjected to the stresses of cooking and impact. Further, the bend test allows analysis of crack propagation after initial failure. This is important because different types of temper may be more efficient in preventing the spread of cracks that have developed.

Procedure

The clay used for this experiment came from the bank of an intermittent stream in Boone County, Missouri. It is reddish-brown with gray mottles. X-ray diffraction of the clay showed that it is composed primarily of kaolinite (41%), montmorillonite (39%), and quartz (9%). The clay was dried for several weeks, crushed, and passed through a 0·50 mm sieve to ensure the homogeneity of the clay and to remove large organic and mineral inclusions. Sieving is important because the objective of the experiment is to test the effect of inclusions on the clay body. If naturally occurring temper-sized mineral particles are present in large amounts, they can mask the effects of the temper types under study.

Test specimens of this clay, mixed with crushed limestone, granite, or grog, were made to examine the effect of the different temper types on the functional properties of fired pottery. Limestone was taken from an outcrop of the Burlington Formation in Boone County, Missouri (Howe, 1961: 64); granite was obtained from surface exposures of Late Precambrian deposits near the town of Graniteville, Iron County, Missouri (Howe, 1961: 157). Grog was made by crushing tiles of the same clay as was used for the test specimens. These tiles were fired for one hour at 600°C. All temper types were crushed in a hydraulic press to particles approximately 3 cm in their longest dimension, then further crushed by pounding with a basalt cobble. The crushed temper was passed through 1·41 mm and 0·50 mm sieves, yielding particles that approximate the size of temper particles in prehistoric sherds from the Midwest (Braun, 1982; Hoard, 1992; O'Brien & Hoard, in press). Temper particles between those minimum and maximum sizes were used in the study.

An additional particle-size range was used for limestone, based on observations of Late Woodland sherds from Burkemper feature 76, which has a corrected (Damon *et al.*, 1974) radiocarbon age of 1240 ± 60 years BP [AD 710 (β 12116)]. Those sherds exhibit very few limestone particles, all of which are under 1 mm in diameter. Sherds from feature 76 also are among the thinnest (5–6 mm) of all the hundreds of sherds of Burkemper sherds examined, the great majority of which are predominantly grit or sand tempered. These observations suggest that the limestone was thoroughly crushed, almost to a powder, before being added to the clay, with the only visual indication of limestone being a small number of larger particles that were included. Informal experimentation with limestone temper showed that when limestone powder was added to a clay body, the body was noticeably more workable than when coarse limestone, grog, or grit was added. Also, coils could be made thinner without cracking. Therefore, an additional set of test specimens was made using limestone particles <1·0 mm in diameter, with 65% of the particles being <0·5 mm.

To determine the amount of limestone temper to be used, four limestone-tempered sherds from 23CP40, a Late Woodland-period Boone phase site in central Missouri (Fox, 1992), were dried in an oven at 105°C for 4 h, crushed, weighed, then placed in a 10% solution of HCl until the limestone had been dissolved (approximately 2 h). The substrate was poured through filter paper, dried in an oven for 4 h at 105°C, and weighed to determine the amount of limestone dissolved. The results of this test are given in Table 1.

Based on this experiment, temper of all three types was added as 35% by dry weight to all test specimens. After they were fired, some of the test specimens were cut with a rock saw. The size and volume of temper evident in the sawed edge of the test specimens compared favorably to those seen in sawed edges of prehistoric grog- or limestone-tempered sherds from central Missouri. The amount of grit temper, however,

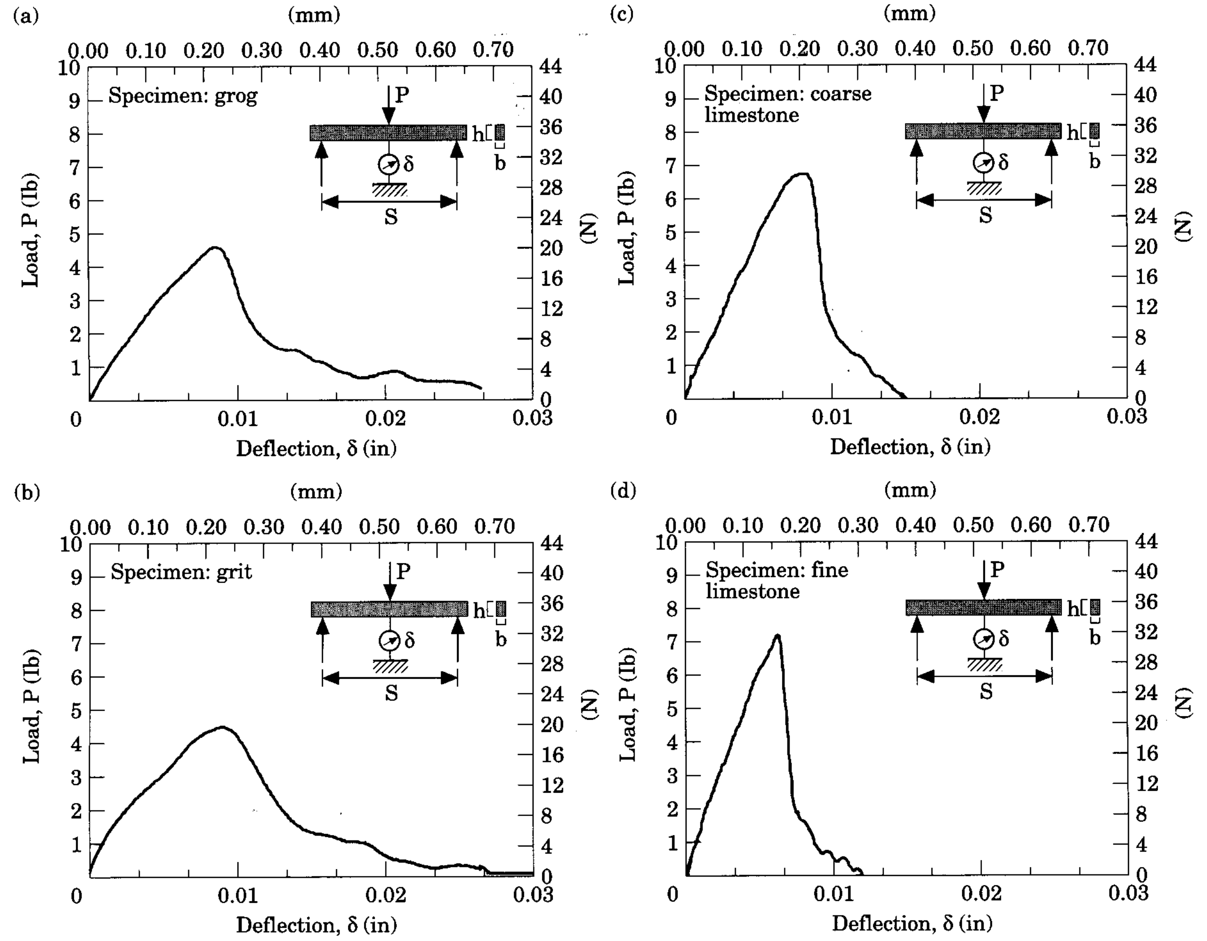

Figure 1. Typical load-deflection response from one test of each composite type tested. Elastic modulus, flexural tensile strength (F_{mor}), energy absorbed up to peak load, and work of fracture can be computed from these responses. Although limestone-tempered composites are stronger than grit- or grog-tempered composites, their post-peak energy absorption capacity is smaller, as indicated by the steeper descending response.

is problematic. Typically, grit temper is present in prehistoric wares in amounts less than 35%. For example, petrographic analysis of 11 Late Woodland sherds from the Sperry site in west-central Missouri showed that they had, on average, 17% temper (Brown, 1985: 236–237, 242), though this value ranged from 3% to 45%. Further, Carr's (1993) radiographic analysis of temper volume of 14 grit-tempered Middle Woodland sherds from Ohio shows that estimated temper volume averaged around 31%, with a range of 27–32% (Carr, 1993: 99). Thus, while prehistoric grit-tempered sherds often contain less temper than the grit-tempered test specimens, 35% temper volume is within the range of prehistoric grit-tempered sherds.

To make the test specimens, dry temper and clay were mixed, wetted until plastic and workable, wedged by hand to mix the composite and to remove air bubbles, and rolled into coils, which were then pressed into moulds 126 × 13 × 10 mm. The test specimens were air dried for 14 h and further dried in an oven for 24 h at 105°C. The dry tiles were fired in a programmable furnace in an oxidizing atmosphere at a rate of 10°C min^{-1} until reaching 600°C, at which temperature they were fired for 1 h to ensure the core of the bar reached the peak temperature, then cooled to room temperature in the oven (approximately 5 h). The test specimens shrank during drying and firing, approximately 1 mm in each dimension. Also, all specimens warped during drying. Those that warped more than 2 mm were not used in the study, and all specimens used for the test were sanded to create straight, even-sided bars that were 124 × 12 × 9 mm. Five test specimens of each temper type were tested under displacement control using a closed-loop electro-hydraulic testing machine that directly measured deflection during the experiment. A cross-head displacement rate of 0·1 mm^{-1} in a three-point configuration (span of 95·25 mm) was used (see Figure 1).

Results of the Three-Point Bend Test

Each specimen was placed on the testing frame and was loaded in the three-point configuration until

Table 2. Results of the three-point bend test

	Peak load (P_{max}) (N)	Modulus of rupture (F_{mor}) (g cm^{-2})	Energy absorbed up to peak load (Nm)	Work of fracture (Nm)	Modulus of elasticity (g cm^{-2} × 10^6)
Grog					
1	18·4	23,208	0·167	0·316	19·0
2	19·3	24,385	0·202	0·342	15·2
3	23·2	29,358	0·228	0·473	20·8
4	22·8	28,804	0·193	0·377	20·8
5	17·4	22,032	0·131	0·280	19·1
Mean	20·9	25,557	0·184	0·359	19·0
Grit					
1	22·2	28,045	0·167	0·289	19·9
2	22·6	28,599	0·193	0·491	20·8
3	20·1	25,496	0·272	0·500	16·3
4	20·1	25,419	0·158	0·289	21·6
5	21·0	26,522	0·237	0·412	16·2
Mean	21·2	26,817	0·202	0·394	19·0
Coarse limestone					
1	26·3	33,295	0·210	0·351	30·9
2	33·3	42,137	0·289	0·456	34·2
3	29·2	36,888	0·324	0·394	25·3
4	29·8	37,716	0·245	0·403	30·6
5	30·4	38,476	0·280	0·517	29·2
Mean	29·8	37,702	0·272	0·421	30·0
Fine limestone					
1	36·0	45,499	0·219	0·570	45·4
2	34·8	44,002	0·210	0·377	37·0
3	35·6	45,313	0·263	0·429	36·5
4	35·6	45,107	0·289	0·447	29·7
5	33·8	42,828	0·245	0·333	40·4
Mean	35·2	44,550	0·263	0·429	37·8

complete fracture. A PC-based automated data acquisition system was used for continuously recording the load and displacement response during the test (four examples are shown in Figure 1). The vertical axis is the applied load, the horizontal axis is the amount of midpoint deflection of the test specimen. The initial slope of the load-deflection response was used to calculate the modulus of elasticity. The amount of energy absorbed up to the maximum load is denoted as the energy absorbed up to peak load. The amount of energy absorbed to the point when the specimen was completely fractured is the work of fracture, an indication of the toughness of the material. Energy absorbed up to peak load and work of fracture is computed as the area under the load-deflection curve up to the peak load and under the complete response, respectively. A shallow descending response after peak load (grog and grit, Figure 1(a) and (b)) indicates that the material dissipates energy during further crack growth; an abrupt drop after peak load indicates a relatively brittle material with lower resistance to crack propagation (both limestone examples, in Figure 1(c) and (d)). Although limestone-clay composites have higher strength (larger peak-load capacity), they exhibit a more brittle post-peak response.

Values of peak load and corresponding values of modulus of rupture, shown in Table 2, are substantially higher for test specimens tempered with coarse or fine limestone than for test specimens tempered with grit or grog. The elastic equation for the modulus of rupture (F_{mor}), or bending tensile strength, is

$$F_{mor}=1{\cdot}5(P_{max}S)/b\,h^2)$$

where $P_{max}S$=peak load, S=span, b=specimen width, and h=specimen depth.

Table 3. Difference in modulus-of-rupture values by t-tests

	Grit	Grog	Fine limestone
Grog			$t=15{\cdot}258$ $P=0{\cdot}00$
Grit		$t=0{\cdot}630$ $P=0{\cdot}563$	$t=20{\cdot}012$ $P=0{\cdot}00$
Coarse limestone	$t=7{\cdot}496$ $P=0{\cdot}002$	$t=5{\cdot}860$ $P=0{\cdot}004$	$t=3{\cdot}872$ $P=0{\cdot}018$

In this study, peak-load values for grit and grog are very similar to each other, indicating that there is no substantial difference in strength between them. The difference in amount of stress needed to cause failure in the test specimens is shown by t-tests to be insignificant at the $P\leqslant{\cdot}05$ level between grog and grit but that it is significant at the same level between grit or grog and both size grades of limestone (Table 3).

Similarly, energy absorbed up to peak load and work of fracture are higher for both size grades of

Table 4. Difference in energy absorbed up to peak load by t-tests

	Grit	Grog	Fine limestone
Grog			$t=3{\cdot}129$ $P=0{\cdot}035$
Grit		$t=0{\cdot}857$ $P=0{\cdot}440$	$t=1{\cdot}600$ $P=0{\cdot}185$
Coarse limestone	$t=5{\cdot}654$ $P=0{\cdot}005$	$t=4{\cdot}451$ $P=0{\cdot}010$	$t=1{\cdot}079$ $P=0{\cdot}341$

Table 5. Difference in modulus-of-elasticity values for test specimens by t-tests

	Grit	Grog	Fine limestone
Grog			$t=6{\cdot}254$ $P=0{\cdot}003$
Grit		$t=0{\cdot}009$ $P=0{\cdot}993$	$t=6{\cdot}025$ $P=0{\cdot}004$
Coarse limestone	$t=11{\cdot}686$ $P=0{\cdot}000$	$t=4{\cdot}716$ $P=0{\cdot}009$	$t=2{\cdot}663$ $P=0{\cdot}056$

limestone than for grog or grit (Table 2), indicating that more energy is needed to fracture the limestone-tempered specimens completely. Comparing the energy absorbed up to peak load for the different composites is shown by *t*-tests to have a significant difference between grog and fine limestone, grog and coarse limestone, and coarse limestone and grit (Table 4).

However, the relatively abrupt failure of the limestone-tempered specimens after peak load indicates a low post-peak toughness. This gives support to Feathers & Scott's (1989: 556) statement that it is the plate-like structure of the calcite from burned shell that toughens shell-tempered sherds, because it if were a *chemical* reaction between clay and calcite that toughens the composite, the limestone-tempered test specimens should demonstrate significant resistance to final fracture, similar to that demonstrated by shell-tempered test specimens in Feathers' (1989) study. Photomicrographs in Feathers & Scott's study (1989: 556–557) show that the clay adheres to the shell particles and that cracks travelling through the shell particles are deflected along the preferred cleavage orientation of the shell. The deflection of cracks allows the shell-tempered composite to absorb more energy before breaking completely.

The curves in Figure 1 and the values of peak load and modulus of rupture (Table 2) demonstrate that limestone-tempered composites are substantially stronger than those tempered with either grit or grog, requiring more applied force to cause the test specimens to fail. But both grog- and grit-tempered composites are shown to have a greater capacity for post-peak deformation and ductility.

Deformation capacity in the elastic region, as indicated by values of modulus of elasticity (Table 2), is one of the important properties in determining the ability of a composite to withstand thermal shock prior to cracking. Jones & Berard (1972: 159) give a general rule for thermal-shock resistance: resistance improves with increasing strength *and* with decreasing thermal expansion and modulus of elasticity. When the outside of a vessel expands more rapidly than the inside, a vessel with a greater capacity for deformation will bend and warp rather than break. Low values of modulus of elasticity are desirable for resisting cracking from thermal shock because the composite is able to bend, rather than crack, when experiencing differential expansion caused by heating one side of a material (Lawrence, 1972: 175). A measure of deformation capacity (Young's E, modulus of elasticity) was calculated for the bend-test samples. The equation for modulus of elasticity (E) is:

$$E=P_{max}S^3/4\,b\,h^3d_0$$

where P_{max}=peak load, S=span, b=specimen width, h=specimen depth, and d_0=midpoint deflection at P_{max}.

Limestone-tempered test specimens had much higher values of modulus of elasticity than did grit- or grog-tempered specimens (Table 2), showing that limestone-tempered specimens exhibit smaller elastic deformation capacity. Significant differences are shown by *t*-tests (Table 5) between both types of limestone-tempered test specimens and the grit- and grog-tempered specimens, but not between coarse and fine limestone or between grit and grog.

Thermal-Shock Resistance

The low deformation capacity of limestone-tempered specimens might suggest that they are less resistant to thermal shock. But thermal-shock cracks can be caused by the differential expansion of the interior and exterior of the pot upon heating *or* by differential expansion of the clay and the temper particles. Although limestone-tempered composites may not appear to be resistant to thermal shock in the first case because of their low capacity for deformation, they are better in the second case because the thermal-expansion coefficient of limestone is very similar to that of clay. Furthermore, because it takes more stress to *initiate* cracks in the limestone-tempered specimens, cracking is less likely to occur. These factors, along with the tendencies for limestone-tempered vessels to be thinner and more globular, suggest that limestone-tempered vessels are more resistant to thermal shock than grit-, or sand-tempered wares. Further, fine limestone exhibits a smaller deformation capacity than coarse limestone. Thus, coarse limestone should perform better under repeated episodes of heating and cooling. Strength tests of specimens subjected to thermal shock are needed to better understand thermal-shock resistant characteristics of such composites.

Discussion

Results of this experimental study show that limestone-tempered pottery has properties that make it more resistant to mechanical and possibly thermal stresses than those exhibited by grit- or grog-tempered wares. Ideally, prehistoric sherds could be compared using the techniques employed in this study to determine the relative strength of different temper/clay composites. Archaeologists have done this in the past (Shepard, 1963: 133–136; Steponaitis, 1983: 35–45, 197–298), but we believe that it is invalid to test archaeological sherds using these techniques because the sherds have undergone structural changes during use and throughout their depositional history that cannot be controlled or corrected. Also, the sherds are variable in thickness and curvature, making comparison between sherds difficult.

Any replicative study such as the one discussed here will have problems related to concordance with the archaeological record. For instance, the problem of the amount of grit temper used has been addressed. While 35% is not typical of the volume of grit temper found in midwestern prehistoric wares, standardizing temper volume does allow rigorous comparison of all samples to measure the net effect of different tempering materials. The firing temperature of 600°C is also problematic. While 600°C is within the range of prehistoric firing temperatures, it is at the low end of the range, particularly for the grit- and grog-tempered wares, which, because they are not subject to spalling, can be fired for higher temperatures and for longer duration (Feathers, 1993).

Test specimens were fired at 600°C because higher firing temperatures caused spalling in limestone-tempered specimens. While limited surface spalling might not significantly affect the strength of the clay body, most prehistoric limestone-tempered wares exhibit little spalling, suggesting that other means were used to prevent its occurrence. The presence of the mineral gehlenite in shell-tempered pottery from the midwestern sites of Cahokia, Angel, Aztalan, Armstrong, and Mero indicates that these pots were fired at temperatures above 800°C, the lowest temperature at which the mineral can form (Stimmell *et al.*, 1982: 222). This further suggests that something was done to reduce the effects of lime spalling at temperatures over 600°C.

Laird & Worcester's (1956: 555) experimental studies of bricks containing limestone particles show that a reducing atmosphere inhibits lime spalling. Reduction is apparent in some limestone-tempered sherds from the central Missouri site 23CP40 (Fox, 1992), but it is not consistent. While some sherds exhibit redness (an indication of oxidation) from interior to exterior, others have black centers (caused by reduction), redness only on the exterior, or are black throughout. However, a reduction firing of limestone-tempered specimens for this study showed that spalling was a problem even at 700°C. These specimens were placed in a bed of wood shavings, wrapped tightly in three layers of aluminum foil, and fired in a muffle furnace for four hours, with the temperature peak of 700°C being reached after 2·5 hours. It may be that a *rapid* firing in a reducing atmosphere inhibits or eliminates spalling (Feathers, 1993). Ethnographic accounts of open-air firings (Rye, 1981: 102–103) indicate that firings can be successfully accomplished very quickly, often less than 30 min from lighting the fire to removing the pots. Studies by Feathers (1993) have confirmed that rapid firing at temperatures above the degradation level of calcium carbonate effectively reduce spalling, even in a partially oxidizing environment.

Laird & Worcester (1956: 555) also found that quenching fired clay bodies with cold water immediately after firing (a process termed docking) eliminates spalling in limestone-tempered bricks. The size of the particles also is a factor: use of smaller, rather than larger, limestone particles retards spalling. Finally, the addition of NaCl—as little as 0·3% by weight when 5% limestone is added or 0·5% when 10% limestone is added (Laird & Worcester, 1956: 555)—inhibits the transition of calcite to calcium oxide and eliminates spalling (Rye, 1976: 123; Stimmell, 1978; Stimmell *et al.*, 1982).

The clay used in the experiment we conducted had an average sodium content of 0·47%, as determined by neutron-activation analysis of five unfired clay samples from the same batch used to make the test specimens. Even if we can assume that all of this sodium was present as NaCl in the clay,* this amount of NaCl would not be expected to retard spalling, given that limestone temper was added as 35% by weight to test specimens. Other midwestern clays may have considerably higher or lower amounts of sodium. It is possible that (a) salty clays were intentionally used to prevent lime spalling, a procedure documented ethnographically (Arnold, 1971: 29–30), or that (b) salt was added to prevent spalling. Still, the addition of salt has never been demonstrated for prehistoric pottery from the Midwest. Provenience studies based on compositional analysis of clay sources and prehistoric sherds could be used to determine if salty clays were selected by potters using limestone or shell temper.

Earlier in this paper we mentioned that Middle Woodland-period potters used limestone to make very thin, well-decorated "Hopewell" pots. As we have stated, Laird & Worcester's (1956) experiments showed

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*None of the compositional analysis techniques we used was able to detect NaCl in the clay with the accuracy that would be useful here. NAA detects only elements, not compounds, and thus we cannot be certain that the sodium detected was from NaCl; X-ray diffraction can detect NaCl but not in amounts <1%.

that the use of small limestone particles prevented spalling. Finely crushed limestone would presumably increase flocculation, but one of the functions of temper is to support the vessel as it is being made, while the clay is still wet. Larger pots require larger temper particles to support the walls (Rye, 1981: 27). This may explain why limestone temper was not typically used for other Middle Woodland vessels: fine limestone temper could be used in small Hopewell vessels that would not need large temper particles to prevent slumping during forming, but larger vessels, such as domestic cooking vessels, required larger limestone particles, which would produce spalls unless measures were taken to prevent this reaction. It may be that until other techniques were used to inhibit spalling, limestone was not used for larger vessels.

Conclusion

Once spalling is controlled, the potential benefits of limestone temper for large cooking pots are three-fold: (a) it produces a more workable clay body, which allows for (2) a thinner and more globular vessel and (3) a fired clay body that is more resistant to mechanical and possibly thermal stresses. These conclusions are based on the materials-science approach used here, which allows for a quantitative assessment of the properties of different ceramic wares. These types of analytical methods have demonstrated similar qualities for the shell-tempered pottery that succeeded limestone-tempered wares in the regions discussed in this study (Rye, 1976; Bronitsky & Hammer, 1976; Steponaitis, 1983; Feathers, 1989; Feathers & Scott, 1989). The results of materials-science-based investigations provide an explanation for why potters would choose to use a "risky" temper. The manufacture of limestone-tempered pots requires particular care to prevent spalling, and this explains why such a widely available temper material with several desirable qualities was used only sporadically before AD 700. It has yet to be determined how spalling was controlled, but compositional and structural analysis may provide the answer.

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