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ABSTRACT

Groups living on Cedar Mesa, SE Utah in the late Basketmaker II period (Grand Gulch phase, AD 200–400) were heavily maize-dependent, but lacked beans as a supplemental plant protein, and pottery vessels for cooking. Common occurrence of limestone fragments in their household middens suggests 1) limestone may have been used as the heating element for stone-boiling maize and 2) this practice might have made some maize proteins more available for human nutrition. Experiments examined these possibilities; results indicate that stone-boiling with Cedar Mesa limestone creates an alkaline cooking environment suitable for nixtamalization of maize kernels, and that maize cooked in this fashion shows significant increases in availability of lysine, tryptophan, and methionine. Archaeological limestone fragments from a Grand Gulch phase site show amounts of fragmentation and changes in density consistent with repeated heating. While not conclusive, these data indicate that further research (e.g., examination of archaeological limestone fragments for maize starch grains or phytoliths) is warranted. It is suggested that greater attention be paid to archaeological indications of stone-boiling with limestone among maize-dependent but pre-pottery societies.

1. Introduction

Fresh maize ears can be roasted, and the dried kernels parched or popped, but cross-culturally, the predominant mode of cooking stored (and hence dried) maize involves boiling. Prior to the advent of pottery vessels, this must ordinarily have involved stone-boiling, one of the techniques of "hot rock cooking" widespread in both the Old and New Worlds (Thoms, 2008, 2009; Nakazawa et al., 2009; Clark et al., 2007; Atalay and Hastorf, 2006).

In the American Southwest, maize was introduced to the Colorado Plateau by about 2100 BC (Huber, 2005; Merrill et al., 2009), but pottery did not become common in that area until between AD 200 and 500 (Cordell, 1997: pp. 227–228). On Cedar Mesa, southeastern Utah, the late Basketmaker II period (Grand Gulch Phase, ca. AD 200–400) lacks pottery, but had a population heavily dependent on maize, as indicated by multiple lines of evidence, including stable carbon isotope analysis of human bones (Matson and Chisholm, 1991; Chisholm and Matson, 1994; Matson, 2006: pp. 155–156).

By the 1970s, it had become known that cooking maize in an alkaline environment enhanced some of the maize proteins and made them more available for human consumption (Katz et al., 1974). Maize is notably low in availability for human nutrition of the amino acids lysine and tryptophan. The process of thermoalkaline treatment is commonly called nixtamalization (Warinner and Tuross, 2009) after the Aztec term for it (Lovis et al., 2011).

During archaeological surveys in the 1970s, Lipe and Matson noted the regular occurrence of burned limestone fragments on habitation site middens of the Grand Gulch phase (Matson et al., 1988; Matson, 1991). They hypothesized that the Cedar Mesa people might have used pieces of local limestone as heating elements for stone-boiling maize, possibly creating alkaline cooking environments. Matson and Lipe also thought (Matson, 1991: p. 7) that this might have been nutritionally important because Cedar Mesa Basketmakers were heavily dependent on maize, and because beans had not yet been incorporated in the diet as a supplemental source of plant protein.

Below, we present the results of experimental studies designed to examine whether 1) local Cedar Mesa limestone can be used effectively as heating elements for boiling maize; 2) if so, whether this creates an alkaline cooking environment; and 3) whether

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cooking maize in this way enhances the availability of some of its proteins. The experiments indicate an affirmative answer to all three questions. We also briefly review evidence that the limestone fragments recovered archaeologically from a Grand Gulch phase habitation site show evidence of repeated heating, although we cannot claim that this is sufficient to show that they were in fact used in stone-boiling maize.

2. Archaeological background

Cedar Mesa is a prominent landform located north of the San Juan River in southeastern Utah (Fig. 1). Extensive surveys carried out by Lipe and Matson in the 1970s documented several episodes of intensive occupation, including in the Classic Basketmaker II period (ca. BC 500–AD 500). The Grand Gulch phase (ca. AD 200–400) represents the latter part of the Basketmaker II occupation, and is characterized by shallow pithouses, and an artifact assemblage that included atlatl dart points, one-hand manos, and metates ranging from basin to semi-troughed forms, but no pottery (Matson et al., 1988, 1990; Matson, 1991, 2006).

Stable carbon isotope analysis by Matson and Chisholm (1991) indicates Grand Gulch phase people were getting between 80 and 90 percent of their subsistence from C_4 plant sources, which are likely to have been predominantly maize (Matson and Chisholm, 1991; Chisholm and Matson, 1994; also see Coltrain et al., 2006, 2007). Analysis of Basketmaker II period human coprolites from a dry shelter in Grand Gulch, a Cedar Mesa canyon, shows that pollen and macrobotanical remains of maize were ubiquitous in the specimens sampled (Aasen, 1984). Settlement pattern data also indicate that Grand Gulch phase habitation sites are associated with the best dry-farming locations, very similar to patterns seen in later Pueblo II and III periods on Cedar Mesa (Matson et al., 1988; Matson, 1991; Matson and Chisholm, 1991).

Grand Gulch phase habitation sites typically have a single pithouse with a south-oriented entryway, and a sheet-trash midden extending south of the house (see Pollock, 2001). The midden deposits are usually ash-stained, and include lithic debitage, other artifacts, and numerous chunks of local sandstone and limestone. Limestone occurs widely in small lenses within the Cedar Mesa Sandstone that caps the entire mesa, and is exposed in its canyons and canyon rims. Such small limestone sources commonly occur within a few hundred to a few thousand meters of Basketmaker II habitation sites. Other types of hard stone suitable for use in stone-boiling are somewhat less widely distributed (e.g., igneous cobbles occur in some of the drainages), but were by no means unavailable.

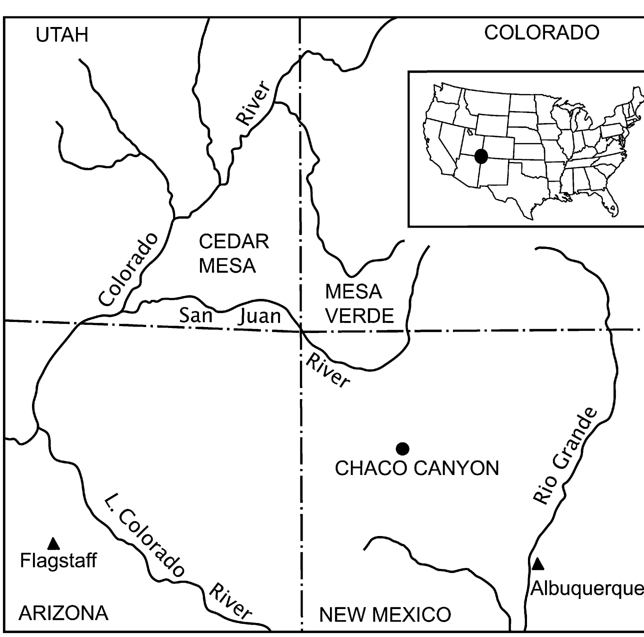

Fig. 1. "Four Corners" area of the American Southwest.

Matson and Lipe found that of 122 surveyed and surface-collected sites classed as definitely or probably being a Basketmaker II single component site, 88 had limestone fragments present. Of the 34 that did not, 13 were very small lithic scatters (Matson et al., 1990: pp. V-22–26, and Table V-1). Because limestone fragments were almost never found on later Basketmaker III and Pueblo period sites in the area, Matson and Lipe considered the occurrence of limestone to be a useful marker of the Basketmaker II period (Matson et al., 1988). In later work on Cedar Mesa, Dohm (1994) also documented the occurrence of limestone on Grand Gulch phase pithouse sites.

As noted above, pottery appears about AD 500 in southeastern Utah, at approximately the same time that evidence of beans (*Phaseolus vulgaris*) begins to be found north of the San Juan River. Because beans may take up to several hours to cook (Skibo and Blinman, 1999: p. 183), it would have been much more efficient to boil them in pottery containers rather than to use stone-boiling, which would have required constant monitoring and continued addition of heated stones to maintain cooking temperatures. This greater convenience would also have applied to boiling maize, even though it does not take nearly as long to cook as do beans. We speculate that the precipitate decline in occurrence of limestone on Cedar Mesa sites post-dating AD 500 reflects both the availability of pottery and of supplemental vegetable protein from beans, which are a near-complete source of protein, including abundant lysine and tryptophan (Mora-Aviles et al., 2007). The availability of the full spectrum of essential amino acids through consumption of both beans and maize would have made the nutritional contribution of stone-boiling maize with limestone relatively less important, as would the appearance of pottery, which was a more efficient cooking technology (Myers, 1989; Clark et al., 2007).

The desirability of further research on hot stone cooking in the northern Southwest is indicated by the near absence of information on this topic in archaeological publications and reports dealing with sites in this area. The presence of fire-cracked rock at late preceramic sites is occasionally mentioned (e.g., Hovezak et al., 2003: pp. 7–24) but without analysis.

An exception is Phil R. Geib's report on excavations at Basketmaker II sites in the Kayenta region, south of the San Juan River, which documents fire-cracked rock at several sites, including Panorama House, where 67% of the FCR was limestone, with the remainder sandstone (Geib, 2011: p. 245). He suggests that "much of the rock was used for stone boiling, both because no roasting pits were found in or around the structure, and because most of the burned limestone had been reduced into small chunks" (Geib, 2011: p. 245). He also observes (Phil R. Geib email, January 27, 2011) that limestone fragments are commonly present on Basketmaker II sites in several regions of northeastern Arizona.

Another exception to the prevailing lack of attention to evidence for hot rock cooking is a paper by Sullivan et al. (2001), which reports on fire-cracked rock piles at sites of varying ages in northern Arizona. Evidence is presented that these result primarily from cooking plant foods in some manner. No evidence of maize was found in association with the FCR concentrations, and whether or not limestone was present is not reported.

3. The benefits of lime treatment

Although uncooked maize, like all living things, contains 20 major amino acids, only 10 of these are necessary to human nutrition, and are thus known as 'essential' amino acids (Bressani et al., 1963; Mertz, 1970). Most of these nutrients cannot be metabolized by human consumers until they are released through cooking or chemical treatment. In human beings, to prevent malnutrition or dietary deficiency diseases such as pellagra (Wacher, 2003; Katz et al., 1974), larger percentages of these amino acids must be made biologically available to populations that are getting the majority of their calories from maize. Typically, if maize is a primary dietary staple, this deficiency is addressed by supplementation with other sources of protein, or by treating the maize in a manner that releases more of its proteins (Scrimshaw et al., 1958: pp. 485–486).

Alkaline treatment of maize can make several of the most important amino acids—including lysine, tryptophan, and methionine—more available for human nutrition. Even then, maize lacks other essential amino acids required to constitute a complete protein, so these must be supplied by additional foods. Improving the availability of key amino acids through alkaline treatment does, however, enhance the ability of human populations to rely on maize as a dietary staple. It allows for improved digestive breakdown and subsequent absorption of these nutrients to take place (Pearson et al., 1957: p. 451; Trejo-Gonzalez et al., 1982: p. 245). Thermoalkaline treatment of maize is most commonly accomplished by cooking it in a slaked lime (calcium hydroxide) and water mixture, but wood ash, lye, or burned shells can be used as well. This process of alkaline treatment is frequently referred to by the term "nixtamalization", which is derived from Aztec terms for ash (nextli) and maize flour (tamal) (Lovis et al., 2011).

Contemporary studies of *Zea* show that gross chemical composition among modern varieties is variable. However, for yellow and white dent corns grown in the United States, Rooney et al. (2004: p. 288) report that the kernel typically contains about 8–10% protein and 65–70% starch, with the rest consisting of ash, oil, sugars, water, and fiber. About 70% of the protein can be found within the endosperm, with the remaining 30% in the germ.

Thermoalkaline treatment causes several key shifts in the structural as well as the nutritional quality of the kernel (Bressani et al., 2002, 2004; Wacher, 2003). Cooking maize kernels in a slaked lime and water solution results in a softening of the pericarp and breaks down starch granules within the endosperm. The remaining starch in the inner structure of the endosperm redistributes the organized proteins (Wacher, 2003: p. 738). The starch then becomes gelatinized (Robles et al., 1988: p. 91) and the resultant protein composition allows for easier grinding as well as enhanced digestibility. Thermoalkalinization processes continue to be used for contemporary mass-produced, maize-based products such as tortillas and corn chips (e.g., Carrera et al., 2011; Gutierrez-Dorado et al., 2008; Oerthoefer and Eastman, 2004). Thus, the benefits of this system for nutrition and ease of processing have been thoroughly tested.

Some form of thermoalkaline treatment is widely recognized as essential if *Zea* is to be consumed on a large scale by people with few or no supplementary protein sources. Although it is clearly nutritionally beneficial, there is disagreement in the literature as to the levels of protein shifts and their timing during alkaline cooking and steeping. For example, Milan-Carrillo et al. (2004: p. 43) found that optimal quality protein availability was achieved through cooking at 85 °C followed by a steep of 8 h. Another study reported a significant increase in protein availability in lime-treated maize, with an overall drop in digestible protein only after 11 h of soaking, and noted "the changes in the lysine and tryptophan content were not proportional to the steeping time" (Rojas-Molina et al., 2008: p. 409).

Other analyses have found that lime treatment improves the availability of only a few amino acids, with little to no effect on others. For example, in some cases, lysine levels increased through treatment, while results showed a drop in tryptophan (Bressani et al., 1963; Trejo-Gonzalez et al., 1982; Wacher, 2003). Yet, lysine alone would provide little advantage without the presence of tryptophan because "it is now well recognized that the relative lack of one or more of the essential amino acids reduces the biological value of food protein" (Scrimshaw et al., 1958: p. 485). An increase in niacin is also nutritionally beneficial (Gutierrez-Dorado et al., 2008); this important B-vitamin is derived from tryptophan, so levels of that amino acid also affect availability of niacin.

Despite some lack of agreement in the detailed analysis of the effects of nixtamalization on individual amino acids, the merits of nixtamalized proteins are markedly clear. Comparative analyses of swine bone collagen have found that feeding animals lime-treated maize products significantly enriches stable carbon and oxygen isotope ratios when compared against those of animals fed only raw maize (Warinner and Tuross, 2009: p. 1690). Studies conducted with rats as well as human children have also found that the overall growth rates are improved when their diets included corn treated with lime (Bressani et al., 1963; Gutierrez-Dorado et al., 2008; Harper et al., 1958; Robles-Ramirez et al., 2011). These patterns suggest that some essential amino acids are being released in lime-treated maize, perhaps along with specific vitamins and minerals vital to bone and tissue growth and development.

In fact, although the existence and size of increases in availability of various proteins are arguable, the availability of calcium and dietary fiber has consistently been shown to markedly improve through nixtamalization with lime (Bressani et al., 2002: p. 81). This is likely due to the absorption of calcined water during the cooking period, as the overall moisture content in grains treated with lime is generally much higher than in those that have simply been boiled (Sefa-Dedeh et al., 2003: p. 319). Though heat is the likely catalyst for initiating this process, studies have shown that calcium may continue to be taken into the grains for up to 135 min after heating has ceased (Trejo-Gonzalez et al., 1982: p. 254). Following an extended steep in an alkaline solution, calcium levels may exceed those in raw maize by around 400 to 750%; there also are increases in the availability of dietary fiber of between 10.3 and 11.7% (Bressani et al., 2002: p. 81; Wacher, 2003: p. 740).

The up-take of high levels of calcined water into the maize grains also produces a product that is ultimately easier to grind into dough or flour due to the fact that the pericarp has been softened (Nations, 1979: p. 569). Thermoalkalinization also extends the time during which maize grains may be consumed following cooking; aflatoxins and their resultant mycotoxins, or toxic molds such as *Aspergillus*, *Fusarium*, and *Penicillium* that infiltrate the granular structure, are all significantly reduced (FAO, 1992b; Rooney et al., 2004: pp. 290–291; Sefa-Dedeh et al., 2003: p. 317).

Maize was consumed on a grand scale among such groups as the ancient Maya (Abrams and Freter, 1996: p. 424), and if the grain had remained untreated prior to consumption, the likely result would have been widespread occurrences of diseases such as pellagra and osteoporosis (Wacher, 2003: p. 740). However, evidence of these diet-based ailments is relatively rare in the New World archaeological record (Katz et al., 1974; Myers, 2006; Wacher, 2003). Because specific forms of malnutrition (such as pellagra's niacin deficiency, and osteoporosis, which results from an insufficient intake of calcium) do not seem to be present prehistorically, scholars have suggested that thermoalkaline treatment of the grain must have co-evolved as dietary reliance on the crop grew (Katz et al., 1974: p. 766).

Historic and ethnographic evidence tends to support these claims, as ethnographic records report the widespread establishment and continued usage of maize alkaline treatment on a large scale across North and Central American native groups (Katz et al., 1974; Myers, 2006; Stross, 2006). The application of alkaline cooking technology (using slaked lime, ash, lye, or in a few cases, burned shell) has nearly always coincided with a relatively high dependency on the crop (Katz et al., 1974: p. 770). Historically, the technology was clearly well-established among groups producing and utilizing moderate to high quantities of maize (Roys, 1934: 97).

Lime treatment of maize has been described ethnographically, for example, among the Mesoamerican Tenejapa Tzeltal linguistic groups, who produce hominy, or paynil as well as the treated maize dough matz' for further preparations into tortillas (wah) or tamales (patz') (Stross, 2006: pp. 581–582).). Lime treatment is characteristic of Mesoamerican groups and is found as far north as the Tepehuan of Chihuahua in northern Mexico (Pennington, 1969:102).

Alternatives to limestone-derived calcium hydroxide as the chemical agent used to treat maize have been noted in Mesoamerica, but especially elsewhere in North America, e.g., use of ashes, lye, or crushed burned mollusk shells (Stross, 2006; Ulery et al., 1993; Nations, 1979; Katz et al., 1974).

Among Southwestern groups, such as the Rio Grande and Western Pueblos, and the Navajo, the production of corn-based products has been described ethnographically. Pueblo preparation of stored maize has typically relied on grinding the kernels dry to produce cornmeal, whereas in Mesoamerica, it was common for the lime-treated kernels (nixtamal) to be ground wet into masa, from which tortillas or other food types could be made. In the Southwest, ashes were employed in some cases during the production of such maize foodstuffs as paper bread, cakes, hominy, and dumplings (Mills, 1999: p. 101). The Hopi as well as the Navajo have been noted to use ashes to alter the color of dough (Katz et al., 1974; Mills, 1999; Whiting, 1939). Among the Navajo, ashes from cedar wood or bean vines were mixed with cornmeal in order to give color to baked cornbread (Hill, 1938: p. 47). Whiting (1939: p. 15) describes how ashes were mixed with water and finely-ground cornmeal to form a gruel which was spread on a hot griddle stone to produce piki or paper bread.

If the Grand Gulch phase Basketmakers were stone-boiling maize, they likely were cooking whole kernels, instead of cornmeal, which would have been much more difficult to extract from the mix of stone fragments and water in the cooking container. The Grand Gulch archaeological complex does not include griddle stones, suggesting that tortillas were not a standard part of the diet. Stone-boiled maize kernels could have been used in stews or ground into dough for other dishes. Grand Gulch phase grinding tools consist predominantly of large basin grinding slabs and one-hand manos, but some semi-troughed metates also occur, as well as occasional manos in the two-hand size range. In the subsequent Basketmaker III period in SE Utah, two-hand manos and troughed metates – thought to be specialized for the production of cornmeal (Woodbury, 1954) – become predominant. Perhaps this indicates a shift to the type of dependence on dry-ground cornmeal that characterizes the later Pueblo archaeological and ethnographic record.

Today, alkaline treatment of corn products continues to be the preferred method of processing worldwide not just by native groups, but also commercially, with respect to tortillas and corn-based snacks (Oerthoefer and Eastman, 2004). Current research shows that the application of historic and prehistoric nixtamalization is, in fact, essential to optimize nutritional benefits of the crop (FAO, 1992a, 1992b, 1992c). Modern treatments differ from the more variable historic practices in that steeping technologies and standards have been formalized for nearly all commercial maize products that are cooked and steeped in an alkaline solution.

4. Experiments in stone-boiling maize with Cedar Mesa limestone

On the basis of the archaeological data reviewed above, we hypothesized that Cedar Mesa people of the Grand Gulch phase were using local limestone as a heating element to stone-boil maize, and that this would have enhanced the availability of certain essential amino acids from the maize. The experiments described below were designed to examine whether this would have been possible. These hypotheses would be falsified if use of limestone in stone-boiling did not create a suitable thermoalkaline environment, or if maize cooked with limestone did not show a significant increase in the availability of key amino acids.

4.1. Effects of heated limestone on pH of water

The first set of experiments addressed the temperatures at which Cedar Mesa limestone would begin to calcine – that is, to change from calcium carbonate to calcium oxide, which would then produce calcium hydroxide (slaked lime) with the addition of water. Stones heated to various temperatures were dropped into water, and changes in the alkalinity of the water were measured (Holstad, 2010). To do this, limestone fragments collected from a geologic source on Cedar Mesa were heated to a pre-designated temperature, at intervals from 300 °C to 900 °C and then placed into room-temperature distilled water, where they were allowed to soak for 24 h. Measurements of pH were made periodically throughout this steeping period to determine whether the mesa's natural limestone possessed the ability to create high pH ranges similar to those resulting from adding slaked lime to water, or around 11.4–11.6.

These tests revealed that between 600 °C and 700 °C (Table 1), the natural limestone began to experience calcination, or the transformation from calcium carbonate to calcium oxide (quicklime). These temperatures are relatively low for this reaction, indicating that the quality of the Cedar Mesa limestone is quite high (Boynton, 1980: p. 178).

Stones heated to 900 °C produced very high pH values of around 13.00. However, this level of heating also promoted the dissolution of the limestone's mineral structure, so they would not have been well-suited to hot stone boiling. Stones heated to 800 °C proved much more effective for cooking, as they not only yielded high pH values, but also managed to retain and continuously release significant heat, with water temperatures beginning at around 75 °C and dipping to 50 °C after approximately 30 min (Table 2). Although several of the other tested temperatures yielded high pH ranges, only those heated to 700 °C or 800 °C released enough sustained heat to cook the maize kernels (Bressani et al., 2002).

Table 1
pH readings during limestone steeping period.

Tone temperature	Control (Dist. H_2O)	30 min	1 h	2 h	5 h	10 h	24 h
300 °C	6.00	7.20	7.45	7.55	7.56	7.55	7.55
400 °C	7.42	7.68	7.76	7.83	7.89	7.93	8.13
500 °C	7.38	7.65	7.72	7.92	8.00	8.40	9.32
600 °C	7.22	7.90	7.95	9.07	9.25	10.00	9.26
700 °C	7.34	11.70	12.15	12.36	12.38	12.50	12.55
750 °C	7.20	12.70	12.77	12.82	12.82	12.84	12.87
800 °C	7.53	12.89	12.96	12.94	12.95	12.97	12.96
900 °C	7.47	12.92	13.01	12.99	12.99	12.99	12.99

Table 2
Water temperature readings taken during the limestone steeping period.

Stone temp.	Water temperature									
	0 min	15 min	20 min	25 min	30 min	60 min	2 h	5 h	10 h	24 h
300 °C	25 °C	25 °C	25 °C	25 °C	20 °C	20 °C	18 °C	16.9 °C	16.9 °C	16.9 °C
400 °C	40 °C	30 °C	25 °C	25 °C	20 °C	20 °C	20 °C	18.3 °C	18.3 °C	18.3 °C
500 °C	50 °C	30 °C	30 °C	25 °C	25 °C	25 °C	25 °C	20.1 °C	20.1 °C	20.1 °C
600 °C	50 °C	28 °C	25 °C	25 °C	25 °C	25 °C	25 °C	16.9 °C	16.9 °C	16.9 °C
700 °C	50 °C	55 °C	50 °C	50 °C	48 °C	30 °C	20 °C	18.3 °C	18.3 °C	18.3 °C
800 °C	75 °C	60 °C	60 °C	50 °C	50 °C	30 °C	20 °C	15.7 °C	15.7 °C	15.7 °C
900 °C	75 °C	48 °C	30 °C	30 °C	28 °C	25 °C	16.9 °C	16.9 °C	16.9 °C	16.9 °C

4.2. Stone-boiling maize with heated limestone

Here, we determined whether dried maize kernels could be cooked in water heated through the application of hot pieces of Cedar Mesa limestone. With the recommendation of Dr. Karen Adams of the Crow Canyon Archaeological Center, 400 g of three traditional maize varieties were used for testing, based on their probable similarity to varieties of maize utilized by Basketmaker II groups (Karen Adams, personal communication, September 2009; Matson, 1991). Ancestral Zea varieties of the three types used in this study may have been available in Utah during the Basketmaker II period (Adams, 1994: p. 294). In particular, Reventador, a Popcorn variety, was likely similar to Chapalote; characteristics of Chapalote have been noted for maize remains recovered in southwestern Colorado from the Basketmaker II site of Falls Creek North Rockshelter (Adams and Patersen, 2011). Two others, Navajo White—a Flour, and Tarahumara Chomo—a Flint variety, were also selected to include a wide range of maize varieties for nutritional assessment. The samples of these traditional varieties were purchased from Native Seeds Search of Tucson, Arizona.

The easy removal of the pericarp tends to be the marker of adequately cooked and steeped maize (Trejo-Gonzalez et al., 1982: p. 249). Using this criterion, a series of early, informal tests were performed on commercial popcorn, along with a Yellow Dent variety purchased from a feed store prior to testing of the traditional varieties. These analyses were based on cooking and steeping standards established by contemporary recipes as well as within common nixtamal literature (Table 3); they were performed to determine optimal cooking and steeping conditions for the three archaeological maize analogs.

The 'hard' external pericarp possessed by the popcorn is relatively thick, while the outer shell of the Yellow Dent is thinner. However, the results of two separate cooking and steeping times—a 15-minute cook with a 10-hour steep versus a 30-minute cook followed by an 8-hour steep—showed that the pericarp of both maize types was most easily removed with a cooking period of approximately 20 min at around 85 °C, followed by a 10-hour steep.

These popcorn/Yellow Dent tests revealed that for uniform results, the limestone needed to be dried and then baked for 3 h at 800 °C. The long baking period was to ensure that the stones had been uniformly heated. As noted above, experimental heat applications to natural Cedar Mesa limestone (Tables 1 and 2 above) found that heating the stones to 800 °C was most likely to produce the 11.0–12.0 pH range as well as the initial cooking temperatures necessary for nixtamalization to take place (Gutierrez-Dorado et al., 2008; Milan-Carrillo et al., 2004; Trejo-Gonzalez et al., 1982).

Analyses were initiated by selecting pieces of the natural limestone collected from a single geologic source location on Cedar Mesa. After the limestone sample was selected, the limestone pieces were placed into a Thermolyne Furatrol 133 oven to heat for 24 h at 105 °C to facilitate the removal of residual water vapor trapped in the stone. Once this heating period was complete, they were then baked for 3 h at 800 °C (this does not imply that we think that cooking stones had to have been heated this long in prehistoric contexts).

An initial ratio by weight (g) of one part limestone to two parts water was used, and the ratio of maize to water was one part maize kernels to three parts water. Immediately following the heating period, the limestone was transferred from the oven into a container of distilled water and maize kernels. Typically, three heated stones of similar weight were initially added to the water and maize mixture. Two more were then added as needed (if the temperature of the water dropped below 75 °C) during the approximately 20-minute cooking period to maintain a temperature range between 75° and 85 °C (Table 4). However, the two extra limestone pieces were removed from the solution before steeping to prevent inflated pH values. Readings of the pH of the water were taken immediately following the 20-minute period of maize cooking as well as periodically during the steeping session to monitor alkalinity and to track the liquid's alkalinity fluctuations during the steeping portion of the treatment. This procedure was followed for each of the three varieties of maize. Control samples were cooked in water heated on a hot plate, without use of stones as heating elements.

Table 3
Common nixtamalization cooking and steeping standards.

Reference	Cook time (min)	Maize to dist. water (:)	Cook temps (C)	Steep time (hours)
Bressani et al. (2002)	75	Unknown	96°	10
Gutierrez-Dorado et al. (2008)	31	1:3 part	85°	8
Milan-Carrillo et al. (2004)	20–85	1:3 part	85°	8–16
Robles et al. (1988)	5–10	1:2 part	85°	15
Trejo-Gonzalez et al. (1982)	"Until pericarp loosened"	1:3 part	Boil	8–12
Secondary sources:				
www.bigoven.com	15	1:2 part	Boil	"Several hrs"
http://mexicanfood.about.com	15	1:2 part	Boil	"Overnight"
www.mexconnect.com	40–50	1:2 part	Boil	"Until pericarp loosened"
www.gourmetsleuth.com/masa.htm	2–15	1:4 part	Boil	24

Table 4
Cooking temperatures taken during the maize cooking period.

Cook time	Tarahumara Chomo (Limestone)	Tarahumara Chomo (Control)	Reventador (Limestone)	Reventador (Control)	Navajo white (Limestone)	Navajo white (Control)
0 min	75 °C	80 °C	75 °C	80 °C	82 °C	88 °C
5 min	86 °C	83 °C	86 °C	86 °C	82 °C	86 °C
10 min	82 °C	84 °C	86 °C	82 °C	79 °C	89 °C
15 min	77 °C	82 °C	75 °C	85 °C	74 °C	82 °C

During the steeping period of each of the three maize varieties, multiple samples of approximately 10 g (dry weight) of maize kernels were collected and rinsed with distilled water until the rinse liquid provided a pH reading of around 8 to 8.5 (Trejo-Gonzalez et al., 1982: p. 249). Samples of this size were removed at the end of the cooking period and then every hour throughout the steeping period up to a total of 10 h. Each was then ground with a small food processor, clearly labeled, and dried at room temperature in a forced air oven for 24 h. At this point, the maize was ground a final time and then immediately refrigerated until all maize samples were similarly processed (Table 5).

4.3. Amino acid measurements

Eleven nixtamalized maize samples were prepared for each of the three maize varieties: Reventador, Navajo White, and Tarahumara Chomo (Table 5). A matching set of flours was prepared from maize cooked without lime, and a third set of three samples consisted of flour prepared from raw (uncooked) maize. This last group of flours was analyzed to provide baselines for any nutritional shifts incurred from simply boiling the grain. The second, or control sample set—maize cooked without limestone—was treated the same way as the first (nixtamalized) set had been, except that a hot plate instead of limestone was used to heat the water. Samples of the three varieties of maize were cooked in a 1:3 parts grain to water mixture. The control samples were processed in a similar manner to the nixtamalized flours. The final result was 23 flour samples for each maize variety—11 steeped samples, 11 control samples, and one raw sample. Thus, a total of 69 samples were prepared for protein analysis.

Both the limestone-treated samples as well as the control sets were comparably analyzed by subjecting the flours to a hydrolysis method that utilized pepsin as the primary digestive component. Under two separate conditions, porcine pepsin with a pH level of 2 was applied to the samples for 3 h at 0.075 mg/ml, or 24 h at 0.2 mg/ml. This was done in order to obtain partial as well as full digestions of the samples. Amino acids released by a partial digestion should provide an indication of the digestibility of the sample, whereas a complete digestion would reflect the total amino acid content of the sample.

Following hydrolysis, extracts were added to minimal media which was inoculated with cultures of *E. coli* that were auxotrophic for either lysine, methionine or tryptophan. Under these conditions, the extent of culture growth was limited by the level of lysine, methionine or tryptophan contained in the hydrolyzed extract. Following incubation, the 595 nm light scattered by the culture was measured in a microplate reader to give an optical density value reflecting the level of amino acid per mass of culture (cf. Scott et al., 2004). Thus, culture turbidity measurements were proportional to the amount of limiting amino acid released by hydrolysis. In effect, these treatments reproduced the conditions under which maize proteins would be digested and absorbed by the human body, and provided results as to the effect of cooking with limestone on the biological, or digestible component of essential amino acids. Table 6 provides the mean optical density values for the three corn types under the three treatment conditions: raw (uncooked); control (cooked on a hot plate); and limestone (stone-boiled with pieces of hot limestone). Because the "limestone × pepsin digest effect" (Table 8) was not significant, the values in Table 6 are averages of the 3 h and 24 h digest conditions.

To facilitate comparison of the changes in amino acid content as a result of cooking with and without limestone, Table 7 and Fig. 2 express the measured values for the control and limestone-cooked samples as proportions of the values obtained for the raw samples.

The analysis produced a number of statistically significant results through the application of an Analysis of Variance (ANOVA) to the resultant data (Table 8). First, patterns of significance were detected among the three differing corn varieties selected for this analysis, or the 'Corn type' effect. This is consistent with the general variation in amino acid content that has been well-documented among different types of maize.

The effect of pepsin digestion time was significant for all amino acids (Table 8), suggesting that the milder treatment was indeed a partial digestion and may provide information about the digestibility of the samples. (As noted above, however, the "Limestone × pepsin effect" was not significant). Among the three

Table 5
Sample set of maize cooked with limestone.

Tarahumara Chomo (sample no.)	Steep time	Reventador (sample no.)	Steep time	Navajo white (sample no.)	Steep time
TS 1	0 steep	RS 1	0 steep	NS 1	0 steep
TS 2	1 h	RS 2	1 h	NS 2	1 h
TS 3	2 h	RS 3	2 h	NS 3	2 h
TS 4	3 h	RS 4	3 h	NS 4	3 h
TS 5	4 h	RS 5	4 h	NS 5	4 h
TS 6	5 h	RS 6	5 h	NS 6	5 h
TS 7	6 h	RS 7	6 h	NS 7	6 h
TS 8	7 h	RS 8	7 h	NS 8	7 h
TS 9	8 h	RS 9	8 h	NS 9	8 h
TS 10	9 h	RS 10	9 h	NS 10	9 h
TS 11	10 h	RS 11	10 h	NS 11	10 h
Total: 11 samples		Total: 11 samples		Total: 11 samples	

Table 6
Mean optical density values from pepsin hydrolysis and culture growth of *E. coli* auxotrophic for Met, Lys or Trp for uncooked samples (Raw); samples cooked without limestone (Control); and samples cooked with limestone (Limestone) for three corn varieties (T. Chomo, Revent., and N. White).

Amino acids	Raw			Control			Limestone		
	T. Chomo	Revent.	N. White	T. Chomo	Revent.	N. White	T. Chomo	Revent.	N. White
MET	0.105	0.111	0.115	0.099	0.105	0.108	0.098	0.102	0.111
LYS	0.486	0.490	0.494	0.423	0.410	0.431	0.436	0.415	0.469
TRP	0.161	0.196	0.180	0.171	0.183	0.179	0.168	0.194	0.182

essential amino acids tested, total levels were found to increase significantly as a result of limestone boiling, as indicated by the significance of the 'Limestone effect' (Table 8). The magnitudes of the Limestone effect in the ANOVA indicate that maize flours subjected to thermoalkalinization gave average increases in our assay of 1.9, 4.3, and 6.1% for methionine, tryptophan, and lysine, respectively, over maize that was simply cooked without limestone.

The significance of the "Limestone × Corn Type" effect indicates whether the degree of the limestone effect differs among the varieties tested. This effect is only significant for methionine (Table 8). Consequently, beneficial increases in the availability of lysine and tryptophan in the presence of limestone were unlikely to be differentially affected by maize variety. Steep time, found in some studies to be a factor in key physical as well as nutritional shifts, at least for lysine and tryptophan (Rojas-Molina et al., 2008), proved relatively insignificant in this experiment.

Analysis of the maize zeins further elucidated why lime-treated samples furnished the observed differences in amino acid content as compared with the control samples. Maize prolamins, or zeins—a particular family of alcohol-soluble proteins—are the most abundant storage proteins within the grain. Together, they comprise around 40 percent of the total protein available within a maize kernel. Their components determine to a great extent the amounts of essential amino acids such as lysine, tryptophan and methionine available in the three maize varieties selected for the limestone treatment analyses.

Zein levels of the same flour samples used for protein tests were subsequently analyzed to assess differential responses to lime treatment. Zeins were classified as alpha and non-alpha zeins and each class was quantified. The former group contains little tryptophan, lysine and methionine, and the latter non-alpha zeins possess high levels of methionine. Non-alpha zeins were enriched due to limestone treatment by 14 percent, as opposed to the control samples cooked without a heated limestone source. Alpha zeins were also around 4.7 percent higher than those found in the non-limestone maize controls.

5. Evidence of prior heating of archaeologically recovered limestone

Experiments were also done on native Cedar Mesa limestone in order to identify physical signatures of repeated heating. Samples of the limestone were subjected to three episodes each of heating at temperatures of 600°, 700°, and 800 °C. Reddening, cracking, and fragmenting of the stones were observed more or less in proportion to heat levels and frequency of reheating. Stones heated at 800 °C began to show crumbling at the second and third heating, and some of them broke down completely through dissolution. Modest increases in density were observed as a result of heating, except for samples heated a third time at 800 °C.

Samples of limestone fragments were collected in the field from a transect of the surface of a midden at the Veres Site (42SA 7406) on Cedar Mesa. This is an excavated and well-dated single

Table 7
Mean optical density values from pepsin hydrolysis and culture growth in maize samples cooked with and without limestone shown as proportions of values for raw (uncooked) samples for three corn varieties. Based on measured values shown in Table 6.

Amino acids	Without limestone			With limestone		
	T. Chomo	Revent.	N. White	T. Chomo	Revent.	N. White
MET	0.944	0.946	0.939	0.933	0.926	0.968
LYS	0.872	0.836	0.871	0.898	0.848	0.949
TRP	1.066	0.937	0.999	1.047	0.993	1.013

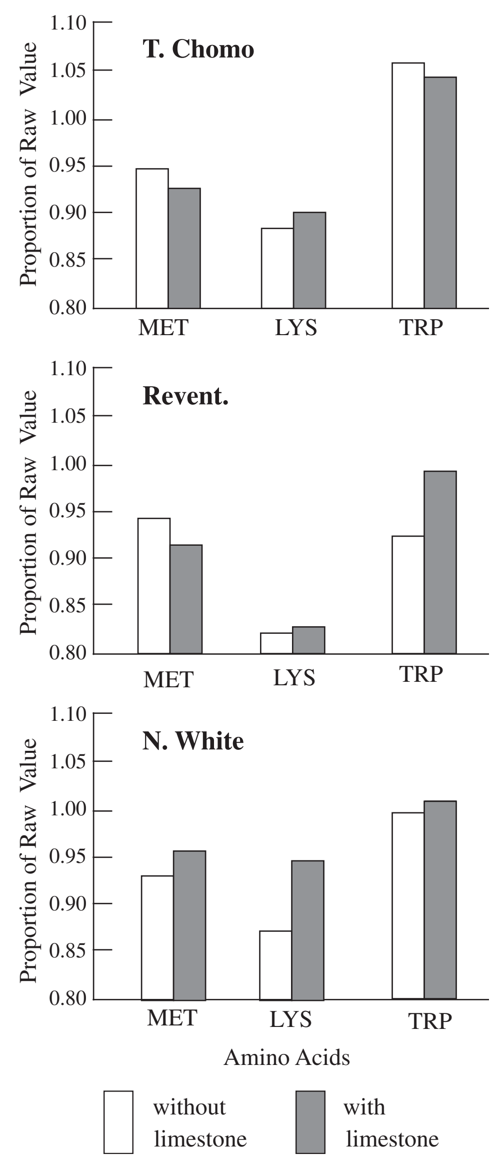

Fig. 2. Graphical presentation of data from Table 7.

Table 8
ANOVA of microbial digestibility assay for three essential amino acids.

Source of variation	Degrees of freedom	Met	Trp	Lys
Corn type	2	**[c]	**	**
Limestone	1	*	*	**
Pepsin digest	1	**	**	**
Steep time[a]	1	n.s.	n.s.	n.s.
Row	7	*	n.s.	n.s.
Column	8	*	n.s.	**
Plate# [Pepsin digest]	4	*	**	**
Limestone × corn type	2	*	n.s.	n.s.
Steep time × corn type	2	n.s.	n.s.	n.s.
Steep time × limestone	1	n.s.	n.s.	n.s.
Steep time × Pepsin digest	1	n.s.	n.s.	n.s.
Pepsin digest × corn type	2	**	**	**
Pepsin digest × limestone	1	n.s.	n.s.	n.s.
Error d.f.[b]		350	353	359
Model R[b]		0.94	0.84	0.56

[a] Steep time was fit as a co-variate in the model.
[b] Error degrees of freedom varies slightly among the amino acids because of differences in the number of outliers removed. Fewer than 4% of the observations were removed in each analysis.
[c] *significant at $\alpha = 0.05$; **significant at $\alpha = 0.01$; n.s., not significant.

component Basketmaker II period habitation site occupied during the Grand Gulch phase (Pollock, 2001). All observed fragments larger than approximately 1-cm diameter were collected from the midden transect. The fragments displayed staining from iron oxide prevalent in the sandy sediments at the site, so color was not helpful as an indicator of past heating. However, the size distribution of the sample was consistent with fragmentation resulting from repeated heating. Of the 84 fragments assessed, nearly 40 percent weighed between one and 5 g, and the second most common interval (31 percent) was between six and 10 g. Since the limestone had been brought to the site from some distance, it seems likely that the fragmentation that produced the smaller pieces would have taken place at the site. Although some fragmentation could have been induced by natural weathering, repeated heating seems a plausible cause for much if not most of it. Future work is needed, however (e.g., analysis of thin sections) to determine if the archaeological limestone shows other physical changes consistent with repeated heating.

The densities of the limestone fragments from the Veres site sample were also measured. Although there were some exceptions, most of the archaeological fragments showed slightly higher densities than examples of geologic Cedar Mesa limestone that had not been heated. The higher density would be consistent with past heating of the samples (F.F. Foit, personal communication, 2010). More work would also be beneficial here, especially to characterize the natural variability in density of Cedar Mesa limestone.

6. Summary and discussion

Stone boiling with limestone as both the purveyor of heat and the source of chemical lime is clearly beneficial in increasing the availability of digestible proteins lysine, tryptophan and methionine. Under the laboratory conditions outlined here, contact of the heated stones with the distilled water and corn mixture resulted in a significant increase in the availability for human digestion of these key amino acids. Thus, this processing technique may have proved beneficial to prehistoric groups that used limestone as a heating element for boiling maize, including the Grand Gulch phase inhabitants of the Cedar Mesa, Utah region.

Studies have shown that not only were Grand Gulch phase people consuming very high levels of maize (Chisholm and Matson, 1994; Matson and Chisholm, 1991; Aasen, 1984; Rylander, 1994), but also that the necessary resources needed to nixtamalize the maize were locally available. Limestone from lenses located within Cedar Mesa Sandstone was found to retain high levels of heat sufficient to both cook and chemically treat maize. Similarly, open fire temperatures using locally abundant fuel would likely have been high enough to heat the stones to a sufficient level (Ermigiotti, 1997; Pierce, 2005). Although they are not hardwoods, locally available pinyon and juniper, at a rate of 274,000 to 289,000 BTUs per cubic foot, produce more convertible energy during burning than most other western American wood species and so possess the potential to reach high heat levels (Barger and Folliott, 1972). Replications of prehistoric Anasazi open-pit pottery kilns (Blinman and Swink, 1997; Brisbin, 1999; Ermigiotti, 1997), primarily using juniper wood, all show that 700–900 °C was well within reach in open fires after relatively short but intense burning periods. Studies have noted that temperatures of around 800 °C can be attained after only 30 min (Ermigiotti, 1997) or 80 min (Pierce, 2005) of consistent burning.

Turning to the archaeological record, preliminary examination of limestone from a Grand Gulch phase habitation site on Cedar Mesa (Pollock, 2001) provided indications it had been heated prehistorically, although additional follow-up studies are needed. Limestone collected from a midden at the site displays fragmentation and weight-to-mass ratios consistent with having been heated at 600–800 °C (Holstad, 2010). This is consistent with its use in stone-boiling, but does not demonstrate definitively that it was so used.

Although the precise kinds of cooking implements used by the Grand Gulch phase people are unknown, a variety of cooking containers would have been available. Leather bags, baskets, gourds, and shallow earth pits have all been reported in ethnographic studies of stone boiling techniques (Voorhies and Gose, 2007: p. 43). Archaeological evidence from the U.S. Southwest indicates that well-made coiled baskets were used by Basketmaker groups in prehistoric times, and would have been suitable for cooking, as well as for water storage (Morris and Burgh, 1941; Weltfish, 1930: p. 480). Gourd containers are also known archaeologically and ethnographically in the Southwest, and presumably leather from deer or bighorn sheep would have been available for bags or to line pits.

This study did not undertake tests of the extent to which stone-boiling maize with Cedar Mesa limestone increased uptake of calcium by maize kernels, the availability of dietary fiber from cooked maize, or the amount and rate of softening of the pericarp of maize kernels. Also, color or taste shifts in the grain—significant drivers of lime or ash steeping, as reported in the ethnographic record—were also not accounted for in these tests.

If creating alkaline cooking environments through the use of limestone heating elements was widespread prior to the advent of pottery and/or beans, then the occurrence of burned limestone at sites used by preceramic maize farmers might be expected in areas where limestone is readily available. Because limestone sources may vary in the temperatures at which they begin to calcine, further experiments with different sources are warranted. In addition, further examination of archaeologically recovered limestone is needed, e.g., thin section analysis to look for structural changes consistent with repeated heating. Archaeological limestone fragments might also be inspected for maize starch granules and/or phytoliths (Pearsall, 2000; Piperno, 2006; Torrence and Barton, 2006). Such studies would need to carefully employ controls to reduce the chance that starch or phytolith evidence had been introduced from modern sources or from the sediments with which the fragments had been associated (e.g., Hart, 2011).

The evidence presented here indicates that limestone could have been used for stone-boiling maize on Cedar Mesa, and if so, it likely would have had beneficial effects on the availability of important maize proteins. Maize became an important subsistence item in Mesoamerica and parts of North America well before pottery vessels became widely used for cooking, and usually before beans became available as a supplemental plant source of protein. Whether the use of limestone as a heating element for stone-boiling maize was a widespread practice is worthy of further research that will involve much closer attention by archaeologists to evidence left by hot stone cooking than has typically occurred, at least in the US Southwest.

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