UNLOCKING ANCIENT DIET: USING STARCH GRANULES IN FOOD RESIDUE FROM COOKING CERAMICS TO ANALYZE PRE-COLUMBIAN ERA CADDO DIET

A Senior Scholars Thesis

by

AMY LEE SKRLA

Submitted to the Office of Undergraduate Research
Texas A&M University
in partial fulfillment of the requirements for the designation as

UNDERGRADUATE RESEARCH SCHOLAR

April 2011

Major: Anthropology
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ABSTRACT

Unlocking Ancient Diet: Using Starch Granules in Food Residue from Cooking Ceramics to Analyze Pre-Columbian Era Caddo Diet. (April 2011)

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This thesis examines the nature of food residues on sherds of ancient Caddoan ceramic cooking vessels from East Texas, which was the homeland of Caddoan peoples for more than 2,000 years. Interior surfaces of some ceramic cooking vessels retain small areas of encrusted residue representative of what was cooked therein. Food plant microfossils, starch granules, and phytoliths are sometimes embedded in this residue. Extraction and analysis of the residue is a reliable pathway to the study of Caddoan diets and these same analytical techniques are applicable to ceramic vessels in general. Diet- related information gained from residue analysis is compared to data about Caddoan diets derived from the archaeological and ethnographic records. Reference slides are created by extracting and mounting starch granules from fresh samples of key Caddoan foods, including maize (i.e. corn), beans, squash and other food resources. This study affirms that microscopic analysis of food residue provides an independent assessment of ancient diet and is applicable to a variety of settings.

DEDICATION

For my family
and Kit

ACKNOWLEDGMENTS

I thank my colleagues for their contributions towards my research project. Special thanks must be given to my research advisor, Dr. Alston V. Thoms, for presenting the opportunity for me to undertake this project and allowing me to utilize the resources available within the Archaeological Ecology Laboratory. In addition, I would like to thank Andrew Laurence, a PhD student at the Archaeological Ecology Laboratory for his guidance and instruction in laboratory techniques. Dr. Tim Riley, of the Archaeobotany Laboratory, provided additional assistance in creating references slides. Without these people, my project would have been nearly impossible to complete.

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CHAPTER I

INTRODUCTION: A REVIEW OF STARCH RESEARCH AND CADDO HISTORY

This research explores the extraction and identification of starch granules on ceramic sherds and is carried out with the ultimate goal of providing a reliable pathway to studying ancient diet as a means of supplementing previous archaeological research and ethnographic sources. Combining laboratory analysis with a review of the literature on the topic provides different methods of studying ancient diet. This research explores the utility of ceramic residue analysis using a sample of pottery sherds from East Texas, the Pre-Columbian homeland of numerous Caddoan groups. It begins with an overview of past research, followed by a discussion of the analytical methods in the study.

Prior research of microfossils

Starch research has been conducted in many locations, including in Texas. However, there has been little research in Texas pertaining to the analysis of microfossils on ceramic residue. As a result, my pilot study involving analysis of Caddoan diet is one of the first in Texas to focus on the analysis of starch granules extracted from cooking vessels.

This thesis follows the style and format of *Texas Journal of Science.*

While there has not been a great deal research of this nature conducted in Texas, that is not to say that studies have not been undertake elsewhere. A primary example of prior starch granule research is that of Boyd et al. 2007. This study focused on the analysis of extracted starch residue to understand the prehistoric role of maize (*Zea mays*) in the diets of smaller groups across Manitoba, Canada (Boyd et al. 2007)

The methods that Boyd et al. 2007 utilized when extracting residue are as follows. After excavation, each sherd was washed to remove any excess materials such as soil. Each sherd was viewed under a microscope to ascertain whether or not all materials that would obscure residue had been removed. To extract the residue, a dental probe and dissection knife were used to remove the three materials the researchers were sampling for: plant microfossils, stable isotopes, and various trace elements. By analyzing starch and microfossil residue extracted from archaeological food residue, Boyd et al. (2007) contended that the consumption of maize and other domesticated crops within these groups was spreading throughout the continent, and especially within the eastern Canadian prairies (Boyd et al. 2007).

Another important study focusing on the analysis of starch granules recovered from ceramics is that of Xiao Yan Yang and LePing Jiang (2009). These two researchers focused their geographic scope of starch research to the Zhejiang Province in China; the two researchers relied upon extraction and analysis methods very similar to Boyd et al. Prior to any extraction, all implements that were to be used in extraction and analysis were sonicated and boiled for 15 minutes to remove any potential sources of contamination. They analyzed three samples, and extracted residues by gently scraping some of the surface residue onto aluminum foil. The starch granules would then be separated by using a solution of CsCl that would cause the starch granules to separate and float to the top, where they could then be extracted and place on slides for viewing. Upon examination and analysis, Yang and Jiang found a wide variety of starch granules, suggesting that there was a great diversity of ancient plant foods cooked and consumed in the Zhejiang Province.

The importance of starch research

Very rarely are the charred remnants of cooked food discovered in the archaeological record; there are certain materials that tend to preserve rather poorly. As a result, much of the evidence archaeologists have regarding ancient diet are ethnographic accounts from European explorers who encountered these groups. While these accounts can be an important tool in the reconstruction of ancient diet, the analysis of starch granules can provide more solid information on the topic. Ancient starch research is well established around the world in the academic community and has been applied to food residue on ceramics in a number of places. In Texas, work has been done recently on residue extracted from fire cracked rock (FCR), and has yielded a solid foundation for reconstructing ancient diet (Perry 2009).

The Caddo homeland

The agricultural-based group that utilized the sherds included in this study is the Caddo.

The Caddo lived in areas of present-day East Texas, an area known as the Pineywoods region.

Caddo diet and cooking

The Caddo people were among the best known of ancient Texas farmers who grew several varieties of corn, beans, and squash in addition to other crops. They also relied upon wild plant foods but the extent to which they were used is unknown (La Vere 2004). The Caddo relied upon a diverse range of wild plant food resources in addition to domesticated crops that they cooked in a variety of ways. (Texas Beyond History 2003.

Several beans, corn and wild fruits such as plant foods such as pumpkins, squash, and blackberries would often be dried and stored away. These foods could then be eaten in their dry preserved state or boiled them in ceramic vessels. Some foods, such as sunflower, squash and other seed crops are typically bland and as a result they would be prepared in stews or gruels (Texas Beyond History 2003).

Perhaps the most important plant food resource being prepared for consumption was corn. Archaeologically, corn appears at most sites associated with the Caddo around 800 A.D. At that time, the Caddo had integrated the farming of corn heavily into their agricultural repertoire. While corn did require greater effort to farm due to the fact that it could be difficult to clear the land, fend off animals, and keep the area around the crop weed-free, corn did have a large payoff for the Caddo. It was relatively easy to harvest because entire stalks could be broken off and corn requires little cleaning or processing prior to cooking and consuming. Corn was useful to the Caddo for a number of reasons. For one, it tasted more sweet and pleasant than other plants foods, and could be cooked in ceramic vessels more ways than other crops could be: raw, roasted, steamed, boiled, parched, and ground into a flour that could be cooked and eaten as a bread or corn mush (Texas Beyond History 2003).

Caddoan cooking ceramics

It was around 1,200 years ago, or around 800 A.D., that the Caddo began creating forms of pottery that were remarkably distinct due to the combinations of materials used, designs, and the manner in which they were created (Texas Beyond History 2003). However, there is archaeological evidence that the Caddo may have been creating pottery as early as 500 B.C. (Perttula 2004).There are two types of pottery that the Caddo created, fine ware and utilitarian ware, but the utilitarian ware was the type that the Caddo used as cooking vessels. These were more course ceramics that had a very plain and basic design and were sturdier than their fine ware counterparts due to the fact that these ceramics needed to withstand being placed directly on a fire when cooking. These course wares were frequently adorned with incisions or engravings, but were not as nicely finished as those on fine wares (Perttula 1995).

Collection history

The collection analyzed is remnants of an estate sale. The pottery sherds used in particular were in the possession of a private collector in Bryan, Texas until his death. The sherds analyzed for this study were not sold at auction because they were generally associated with human skeletal remains. They were loaned to Alston Thoms by the Bryan Police Department to help determine whether they may have been made and used by ancestors of the Caddo Nation, presently located in Oklahoma. The pottery tradition that this collection is most likely associated with is that of the Goose Creek pottery tradition. This type of pottery has been found in Southeast Texas, and is characterized by its composition of sandy paste and plain design. Typically, these types of ceramics were undecorated (Goose Creek Plain) but were sometimes decorated with incisions (Goose Creek Incised). There is a great deal of charred residue found on many Goose Creek ceramics, providing evidence that they were used for cooking (Aten 1983).

CHAPTER II

METHOD: LABORATORY MICROFOSSIL ANALYSIS

By extracting residues from pottery sherds and processing within the laboratory, one will be able to analyze the residues in order to determine if there are phytoliths, microfossils and starches present. These residues left behind are remnants of charred food that was cooked within that vessel. Sherds analyzed in this study are representative of Pre-Columbian Caddo groups from the greater East Texas area, a group of people whose history has been well-documented. As such, there is a solid foundation for again to which to compare and assess the utility of the microfossil analysis technique.

Reference slide creation

Perhaps the most important part of starch granule analysis for this thesis is the preparation of reference slides. Toward that end, a list of the key Caddoan food resources was compiled. Samples of the following foods were prepared: *Zea mays* (corn), *Rubus fruticosus* (blackberry), *Cucubita pepo* (yellow squash and pumpkin skin and seeds), *Phaseollus vulgaris* (beans), *Helianthus annus* (sunflower seeds), and *Disophyos virginiana* (persimmons). Each seed was separately ground with a mortar and pestle and the particles were placed in a small vial with water and EtOH (ethanol). Several drops of the liquid mixture are then placed on a slide and examined microscopically. Starch granules from each sample were photographed using a mounted digital camera and thoroughly documented. Starch granules on the reference slides were described according to size, shape, position of the hilum, and the manner of grouping. The resulting descriptions, reference slides, and photographic images proved the necessary “type collection” for comparisons to starch granules recovered from residue on Pre- Columbian ceramic sherds.

Pre-extraction preparation

Prior to extraction of residue, the sherd must be cleaned to remove excess dirt or other kinds of potential contamination. This is done by gently scrubbing the sherd with a toothbrush and rinsing it in distilled water. To prevent cross contamination between artifacts, fresh distilled water is used for the rinse each time, and the toothbrush used is rinsed and soaked in bleach. The sherd is allowed to fully dry before any residue is extracted.

Residue extraction

Each sherd is carefully examined microscopically to determine if residue is visible. Black residue is a sign that food was cooked within that vessel. Sherds with black residue are most likely to yield microfossils, and, hence, are preferred for analysis. A note on the location and density of residue is taken and a sketch is drawn of the sherd to show the location(s) of the visible residue.

There are two methods of extracting microfossils. The first method entails removing the visible residue area (ca. 1 cm^2) with a dental pick and depositing the material obtained in a beaker. Distilled water is added and the solution is pipetted into a 15 ml test tube. It is labeled with the sherd's catalog number and stored for follow-up processing. The dental pick is rinsed in bleach to avoid cross-contamination between sherds. The first method of extraction is problematic because pieces of the sehrd were removed along with the residue and removing it from the sample was problematic.

The second method entails using a Sonicare electric toothbrush to loosen and detach residue from a given area. A few drops of distilled water are applied with a pipette to the targeted area. After allowing the water to penetrate the area for three minutes, the remaining water is pipetted and placed in a clean beaker. The residue area is then gently scrubbed with a sonicating toothbrush. A few drops of distilled water are placed on the area and extracted by pipette into the same beaker. The same process is repeated two or three times, and the solution in the beaker is transferred to a 15 ml test tube and labeled appropriately. A clean toothbrush head is used for each sherd, and in between use they are soaked in bleach to sanitize.

In cases where the sample cannot be processed immediately, it is centrifuged for one minute, a few milliliters of water are decanted, and a small amount of EtOH is added to prevent degradation of the sample.

Heavy density separation

In some instances, portions of the sherd would be removed during the extraction process. When this occurred, the sample could not be properly analyzed and it was necessary to carry out heavy density separation (HDS), which begins by centrifuging the solution for three minutes and decanting the supernate. Next, 4 ml of $ZnBr_2$ (Zinc Bromide) is added to the remaining sample and thoroughly vortexed before adding enough EtOH to bring the total amount of material in each test tube up to 5 ml so that the centrifuge will remain balanced. Following this, the samples are centrifuged, beginning at a low setting around 25% capacity and increasing it slowly over the course of three minutes to 100%. Samples are then centrifuged at full speed for five minutes.

In centrifuging the samples, starch granules and other microfossils rise to the top layer and the heavier materials sink to the bottom. The microfossils are extracted and transferred to a sterilized separate test tube and labeled accordingly. Distilled water is added to both samples and vortexed to loosen the materials that have collected at the base of the test tube.

These samples must be centrifuged again for one minute to remove any excess $ZnBr_2$. The supernate must be decanted into a waste container insofar as $ZnBr_2$ is a toxic heavy metal. Once the supernate is decanted, distilled water is added to the test tubes and they are centrifuged until all of the $ZnBr_2$ has been separated from the samples, which is indicated when the samples no longer look "oily" or cloudy. After all of the $ZnBr_2$ has been removed from the set of samples containing the starch granules and microfossils, the material is placed on a slide, covered with a petri dish, and allowed to dehydrate. The $ZnBr_2$ waste is poured into a special container and the beaker that held the waste is rinsed in the waste-sink located in the fume hood. If the sediments need to be processed for further analysis, they are centrifuged, decanted, and stored for further use. If they do not need to be retained, they are allowed to dehydrate and then discarded.

Microscope analysis

After the slides containing the samples have sufficiently dehydrated, they are examined under a high-powered microscope at 200x magnification to detect microfossils. If any are found, the magnification is increased to 400x and the slide is rotated to make sure the objects are indeed microfossils, and not air bubbles, hair, or other materials. If it is a starch granule, it is photographed with a mounted digital camera at 400x magnification. The X and Y coordinates for each starch granule are recorded. The photographs are downloaded and labeled appropriately.

CHAPTER III

RESULTS

Archaeological sample (sherds)

A total of 20 ceramic sherds were processed for this project. Of these 20, only six yielded residue that contained starch granules. Identified starch granules were limited to those representative of *Rubus fruticosus* (Figure 3.1) and *Zea mays* (Figure 3.2).

TABLE 3.1
REFERENCE SLIDES

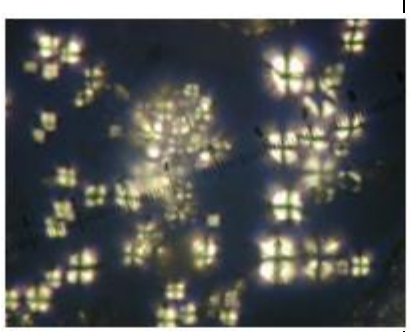
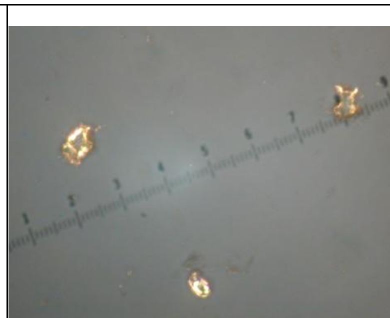
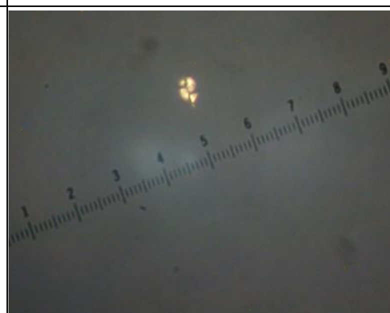
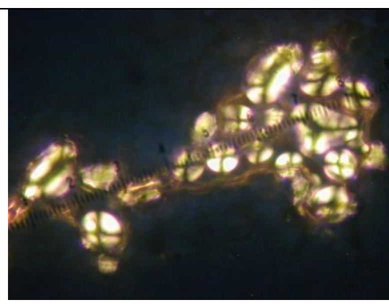
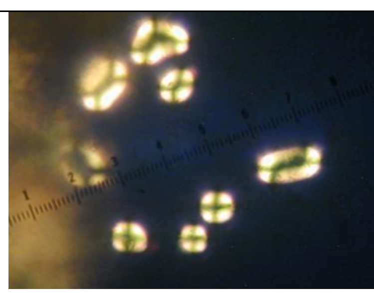
Taxonomic Grouping	**Reference Photo**	**Simple Shape Category**	**Granule Form**	**Size Range**	**Hilum Position**
***Zea mays* (Hopi)**		spherical	single, clustered	10-22.5 µm	centric
Zea mays		spherical	single, clustered	7.5-22.5 µm	centric

TABLE 3.1 CONTINUED

Rubus fruticosus		bean, kidney shaped	single	20 µm	elliptical
Cucurbitae **(yellow, rind)**		oval	single	12.5 µm	elliptical
Phaseollus vulgaris **(Mitla Black)**		Oval, kidney shaped	single	17.5-35 µm	Centric, eccentric
Phaseollus vulgaris **(San Juan Pinto)**		Oval, kidney shaped	single	17.5-40 µm	Centric, eccentric

The reference slides were created by sampling the most common cooked Caddoan plant foods, and organized by analyzing several characteristics.

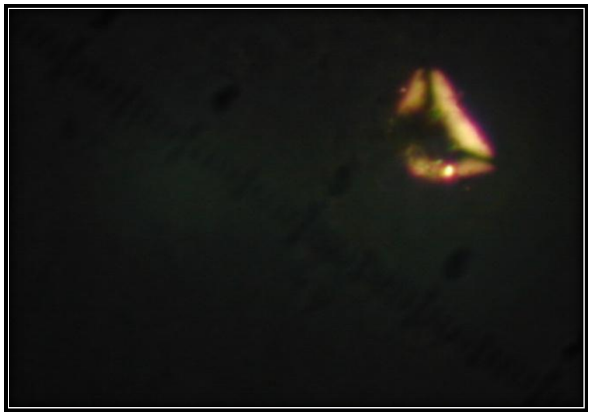


Fig. 3.1 Starch granule of *Rubus fruticosus*. Taken at 400x magnification and located on sample C8.

Fig 3.2 Starch granule of *Zea mays*. Taken at 400x magnification; obtained from sample C16.

Both recovered starch granules are from foods that are well-documented as being a part of the Caddoan diet.

Other materials of interest

Other microscopic materials were also recovered and identified. The most frequently recovered substance on the sherds was burned residue (Figure 3.3), which was an indicator that ceramic sherds had once been used for cooking. On the sherds with starch granules present, the starch granules were found in juxtaposition with these burned residues.

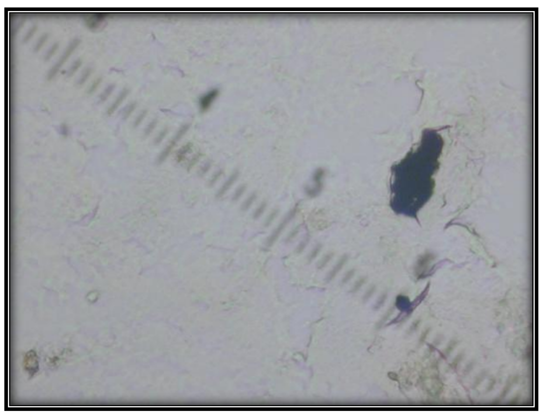


Fig. 3.3 Charred food residue at 400x magnification. Present on the surface of sample C16

On one sherd, a pollen grain and a piece of charcoal (Figure 3.4) were isolated and identified. While the pollen is most likely present due to contamination, the charcoal is a potential indicator that the ceramics from the collection were used for cooking. Another explanation is that the charcoal could have been deposited after the pot or sherd had been discarded.

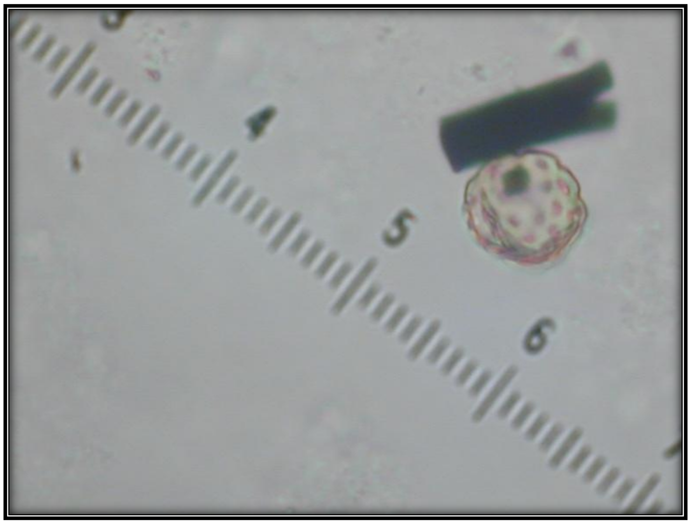


Fig. 3.4 Pollen and charcoal at 400x magnification. While discovering charcoal in juxtaposition with a pollen grain is an interesting find, it is most likely the source of contamination and has no bearing for this project in particular.

CHAPTER IV

SUMMARY AND CONCLUSIONS

This is a pilot study that assessed the utility of analysis of residue on sherds for food starch granules. The analysis was completed by creating reference slides of key Caddoan cooked foods, extracting the residue from the sherd surface which then yielded starch granules representative of *Rubus fruticosus* (blackberry) and *Zea mays* (corn). That only a few sherds yielded identifiable starch granules is consistent with results from other studies. That identifiable starch granules were found is encouraging, especially give that the types were consistent with evidence for archaeological and ethnographic records. With larger samples and more reference slides of more plant foods, results are likely to be more productive and informative.

Using ceramic cooking vessels as a means of reconstructing ancient diet is a solid and dependable method of doing so. It can be used in two ways: if we know where the ceramics came from, it can be used to ascertain more information about the diet of the people. This can go in the other direction—reconstructing the diet can give us a better idea about where the ceramics came from and who was using them.

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