ARCHAEOLOGICAL STARCH PRESERVATION AND METHODOLOGICAL PARAMETERS: WHERE DOES QARAQARA FIT?

Thesis
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By
Nicole Marie Hernandez, B.A.

Department of Anthropology
The Ohio State University
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Master’s Committee:
Professor Julie S. Field, Advisor
Professor Kristen J. Gremillion
Professor Robert Cook
ABSTRACT

Starch granule analysis is a relatively new methodology that can aid in various areas of archaeological research, including the determination of subsistence patterns and transitions, mobility, tool use, and ecology. This study examines undecorated ceramic fragments recovered from Qaraqara, Fiji for the presence of adhered starch. Although the three starch extraction methods used were not successful, the research process provides an opportunity to explain the lack of success by investigating ideal archaeological starch preservation parameters and extraction methodologies. Many factors contribute to the rarity of preserved starch granules in archaeological settings, including its bio-chemical structure, the decomposition of microorganisms in soil, chemical interactions, and environmental conditions. To determine ideal environmental conditions for starch preservation, successful extraction methods, and the effect of archaeological materials on success rates for extraction, a meta-analysis was conducted on a sample of archaeological studies that also sought to extract preserved starch. Key environmental variables that support the preservation of starch were identified, including a specific range of average temperatures, rainfall amounts, and altitudes. Using chi-square statistical tests, this study determined that an average temperature of 19-22° C significantly contributes to the preservation of archaeological starch. Similar analyses determined that extremes in aridity are detrimental for starch preservation, but extremes in altitude were not. Since
the environmental conditions of Qaraqara fall within the acceptable parameters for starch preservation, further analyses conclude that extraction methodology may explain the failure to extract starch from the samples. Additional chi-square tests were conducted using the archaeological case studies to determine the frequency of successful extraction when using different extraction methods on a variety of artifact materials. The results allow for the conclusion that there is a significant failure rate for ceramic artifacts when a liquid extraction with pipette and scalpel is used. These results are put forward as the explanation for the failure to extract starch from the Qaraqara samples. In addition, these results provide an extensive analysis and review of methods, and materials to act as a guideline for future researchers conducting starch granule analysis.
For Alexis and Kristina.
You can do anything.
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Last, but certainly not least, I am forever grateful to my wonderful circle of family and friends. Thank you to Madre, my sisters Alexis and Kristina, my brother Dudeman, and Abuela.
VITA

2009…………………………………………………………Farmingdale Senior High School

2012…………………………………………………………B.A., Anthropology,
Adelphi University

2015…………………………………………………………Graduate Teaching Associate, Department of Anthropology, The Ohio State University

FIELDS OF STUDY

Major Field: Anthropology
Specialization: Archaeology
TABLE OF CONTENTS

Abstract................................................................................................................ii
Dedication............................................................................................................iv
Acknowledgements..............................................................................................v
Vita........................................................................................................................iv
List of Figures......................................................................................................ix
List of Tables.......................................................................................................x

Chapter 1: Introduction........................................................................................1
  Content of this Thesis...........................................................................................2
  Applications of Starch Granule Analysis............................................................3
  Prehistoric Subsistence Patterns....................................................................3
  Environmental Reconstruction..........................................................................3
  Tool Use.............................................................................................................4

Chapter 2: Background.........................................................................................5
  What is Starch?..................................................................................................5
  Methods of Starch Extraction.........................................................................6
  Observations of Preserved and Extracted Starch.............................................7
  Contexts for Archaeologically Preserved Starch: Sediments and Tools.........9
  Starch Preservation in Archaeological Contexts..............................................9
  The Fiji Context...............................................................................................10
  Research Questions........................................................................................12

Chapter 3: Materials and Methods.................................................................13
Meta-Analysis of Case Studies

Materials and Methods for Extraction of Archaeological Starch from the Qaraqara Ceramics

Generation of Modern Starch Reference Collection

Chapter 4: Results

The Effect of Environmental Parameters on Starch Preservation and Extraction

The Effect of Artifact Material on Starch Preservation and Extraction

The Effect of Starch Extraction Protocols on Successful Starch Extraction

Explaining the Failed Extraction of Preserved Starch from the Qaraqara Ceramic Sample

Chapter 5: Discussion

Starch Preservation or Extraction?

Suggested Extraction Methods Based on Artifact Material

Suggestions for Detecting Starch at Qaraqara

Other Limitations to Assessing Starch Extraction Protocols

The Benefits of Comparative Research

Implications of Results for Detecting Agriculture in Prehistoric Fiji

Chapter 6: Conclusion

References

Appendix of Tables
LIST OF FIGURES

Figure 1: Two taxa of archaeological starch..................................................8
Figure 2: Map of Fiji showing Qaraqara....................................................11
Figure 3: Temperature category frequencies from case studies.....................13
Figure 4: Rainfall category frequencies from case studies............................14
Figure 5: Altitude category frequencies from case studies.............................16
Figure 6: Artifact type frequencies from case studies..................................16
Figure 7: Starch extraction success frequencies and rainfall categories..........22
Figure 8: Starch extraction success frequencies and temperature categories.....23
Figure 9: Starch extraction success frequencies and altitude categories.........27
Figure 10: Starch extraction success frequencies and artifact type categories...27
LIST OF TABLES

Table 1: Chi-Square results based on environmental variables…………………………21
Table 2: Pairwise comparison based on rainfall categories…………………………….22
Table 3: Pairwise comparison based on temperature categories………………………24
Table 4: Pairwise comparison based on altitude categories……………………………26
Table 5: Pairwise comparison based on artifact type…………………………………….28
Table 6: Success frequencies for artifact material and extraction protocol………………29
Table 7: Environmental variables for case studies……………………………………….48
Table 8: Chi-Square results for artifact material and extraction protocol………………51
CHAPTER 1: INTRODUCTION

Despite over five decades of intensive study, food production in antiquity remains a vital field of archaeological research. A major focus is the origins of food production, and reasons for shifts in diet. Understanding food production is important as food provides the fundamental measurement for the study of demographic changes, environmental change, technological innovation, settlement patterns, and resource scarcity. An important method in the study of food production, ancient starch granule analysis allows for the construction of extended inferences regarding past environments and human behaviors. In particular starch analysis has value in detecting subsistence patterns, tool use, and environmental reconstruction (Torrence 2006a, Horrocks and Wozniak 2008).

Fiji serves as an excellent case study for understanding first food production for many reasons. The semi-isolated nature of the archipelago allows Fiji as a whole to be considered an “island laboratory” allowing for the testing of various research questions. Answers to these questions can then be applied to other regions of the world as a model. Viti Levu, the largest island in Fiji, has an archaeological record that can be used to address questions of food production. The island is large and has geological deposits that favor agricultural production, and it was originally colonized by a small population of maritime foragers (Szabo 2000). How these foragers became farmers, and how they employed food production to change both the island and their society, is the topic of recent research programs (Field et al. 2014).
Content of this Thesis

Research for this thesis originally sought to obtain starch granules from ceramic samples from the archaeological site of Qaraqara, located in the Sigatoka Valley of Viti Levu. The larger goal of research at Qaraqara is to detect subsistence change from marine foraging to starchy food cultivation via the analysis of ceramics, fire history, and agricultural features. These analyses include the detection of starch on the interiors of ceramic vessels used to process starch-rich tubers. However, despite a series of attempts, no starch was extracted from the Qaraqara ceramic samples. In order to explain this result, this thesis further examines the environmental parameters, starch extraction methods, and materials that can affect the preservation and extraction of archaeological starch. Via a series of statistical analyses, it establishes a set of preferences for matching materials and methodologies, which are more likely to lead to the successful extraction of archaeological starch.

In the following chapters, this thesis describes starch as an energy storage unit, its preservation in archaeological contexts, and prominent extraction protocols that generated the research questions (Chapter 2). In Chapter 3, the materials and methods used in starch granule analysis of the Qaraqara ceramic samples are described, as are those for archaeological starch meta-analysis. Chapter 4 focuses on the results of the meta-analysis. These results are further discussed in Chapter 5, where suggestions for starch extraction protocol are made. Limitations of this study and future research directions are included in the concluding chapter (Chapter 6).
Applications of Starch Granule Analysis

Prehistoric Subsistence Patterns

Interaction between humans and their environments is an integral part of past societies that can be better understood through starch granule analysis. The use of plants for consumption or other necessities is often difficult to infer due to limited organic material preservation in the archaeological record. While macro-remains may not be preserved, micro-botanical remains, such as starch, can be found in archaeological deposits. Since starch residue is often preserved on artifacts, it is possible to combine various artifact analyses with starch analysis to infer aspects of food production in the past (Harris 2006). In the Fiji case, the presence of starch on ceramics has been hypothesized to indicate the processing and consumption of starch-rich tubers (Horrocks and Nunn 2007). The ceramics are evidence for the colonization of the remote interior of the island, and also the reliance on food production (rather than coastal marine resources) in the last 2000 years.

Environmental Reconstruction

Reconstruction of paleoenvironments can be determined with a combination of different methodologies, including starch granule analysis. Starch grains can be preserved in sediments and can therefore be extracted and identified by researchers (Torrence 2006b, Lentfer et al. 2002). In the Fiji case, the identification of these grains in soils could identify land that was used for the production of starchy tubers. At a broader scale, the detection of starch from dated deposits could be used to trace
the temporal transition of forests to gardens. Starch analysis can also be used in combination with phytolith analysis to add understanding about specific site function and ecology (Lentfer et al. 2002).

Tool Use

Artifact function can be determined through a combination of methods, especially morphological and use-wear analyses. Starch residue is often also found on artifacts, particularly stone tools, indicating that these tools were used to process starch-rich materials (Fullagar 2006, Fullagar et al. 2006, Piperno et al. 2000, 2004). Thus, starch analysis can also determine potential tool function that could be overlooked by use-wear analysis alone. By proxy, diet can also be reconstructed via the detection of starch on tools used to prepare food. In the Fiji case, the presence of starch on lithics and ceramics is inferred to be related to processing two domesticated plants, taro (Colocasia esculenta) and Asian yam (Dioscorea sp.) (Horrocks and Nunn 2007). Both of these plants produced roots and corms that required cooking and peeling prior to consumption. This process had the potential to leave starch residues on tools used to handle and produce the prepared food. In addition, starch becomes modified when there is a disruption of the physio-chemical or morphological structure, which often occurs when the food is roasted or boiled. Analysis of modified starch can also be used to aid in understanding the variations in cuisine between different social groups. While this type of analysis is valuable, preservation of modified starch is varied and still not completely understood.
CHAPTER 2: BACKGROUND

A relatively new methodology, starch granule analysis has great potential for archaeological research. This section will outline the implications for starch found in archaeological contexts, and discuss the various methods for its extraction. Many factors also affect the differential preservation of starch, and these will be outlined and discussed. Lastly, the context of the site of Qaraqara, and the implications for the recovery of starch in that locale, will be addressed. This information will provide the necessary background information for a series of research questions that structure the remainder of this thesis.

What is starch?

During the process of photosynthesis, plants convert the sun’s energy into glucose. When not needed for growth, this glucose is then stored in the body of the plant in the form of starch granules. These deposits of starch can be especially dense in tubers or roots, which are used as reserves of energy for the next growing season (Gott et al. 2006). As mentioned in Chapter 1, when preserved, starch granules can provide a direct indicator of prehistoric cultivation and agricultural activities. Such archaeologically invisible events become evident when starches are observed within
sediments. Starch analyses will especially help determine first use of starchy foods requiring intentional cultivation and also aid in the determination of consumption of starch as part of the prehistoric diet. However, starchy foods (taro, yam, cassava, potatoes, others) often require cooking before being edible (Gott et al. 2006). The heat of cooking modifies starch, altering its physio-chemical and morphological structure in a particular way (Samuel 2006). For this reason, starches that are produced and consumed as a cooked food can be difficult to detect.

Methods of Starch Extraction

There are three prominent starch extraction protocols employed in archaeological research. These include pipette extraction, scalpel extraction, and sonic bath extraction. Heavy liquid separation (HLS) is also employed, but is a complicated and expensive process, and therefore less common. These protocols are described below.

**Pipette Extraction:** The pipette extraction protocol involves applying a small amount of distilled water to artifacts that appear to have starch residue. The artifact is agitated with a disposable pipette tip. After a few minutes, water and starch granules are extracted with the pipette and transferred to a microscope slide. Slides are left overnight for water evaporation to occur, leaving only the residue behind. A cover is then placed over the slide.
**Scalpel Extraction:** The scalpel extraction protocol uses a sterile scalpel to scrape a small area of residue from an artifact’s surface. This residue is placed directly onto a microscope slide. A cover is then placed on the slide.

**Sonic Bath Extraction:** The sonic bath extraction protocol requires a sonic bath, distilled water, and Ziploc bags. The artifact to be tested is placed in the Ziploc bag, submerged with distilled water, and sealed. Sealed bags are placed in a sonic bath and sonicated until the artifact appears clean. The water in the bag and the floating starch granules are extracted using a sterile pipette. The liquid is then placed on a microscope slide in the same manner as described for pipette extraction.

*Observation of Preserved and Extracted Starch*

The largest starch granules are generally smaller than 100 microns, and therefore must be viewed using a microscope (Barton and Fullagar 2006:47). The nature of the material will dictate the type of microscopy and techniques used. Generally, starch granules are best observed using a compound microscope with various light conditions and multiple levels of magnification. Low level magnification is useful for detecting patterns of starch residue on artifacts to infer tool use. High magnification is used to identify individual grains and groups of starch granules to the genera and species level. Starch identification is based on granule morphology and the presence of extinction crosses (Figure 1). Starch granules are formed beginning at the hilum (the core or staring point of the granule) with additional layers successively being added on top. Generally, starch granules of each
plant species feature unique shapes and sizes, which are under both genetic and environmental control. Features of starch granules that are genetically controlled include the morphology of the hilum and its location on the grain, the presence or absence of interior vacuoles and their density, and the thickness of accretion layers

Figure 1: Two taxa (A-B: *Ipomoea batatas* [sweet potato], C-D: *Dioscorea cf. alata* [greater yam]) of archaeological starch extracted from shell tools. From Allen and Ussher 2013:2810.

(Loy 1994:90). Other grains can be pressed together in groups of single grains, taking shapes that conform to adjacent grains. It is important to understand the possible morphological changes of individual grains with the formation of these grain
groups, which are called compounds. These morphological characteristics are used to identify archaeological starch granules with assistance from a reference collection.

**Contexts for Archaeologically Preserved Starch: Sediments and Tools**

Sediments routinely have starch granules as a fraction of the matrix. Heavy liquid separation is an extraction protocol that is traditionally used to extract starch from sediment, although its use is being expanded to artifacts. Sediment samples are prepared by drying the sample in a dessicating oven, and then the samples are disaggregated and deflocculated with chemicals, such as cesium chloride (CsCl), to break up the sample into single particles (Piperno et al. 2009). Next, undesired macro-particles are mechanically removed and the remaining sample is mounted on a microscope slide (Coil et al. 2003:996-1001).

All four of the extraction protocols described above are used to extract starch from tools. Multiple protocols can be combined to increase the likelihood of extracting starch.

**Starch Preservation in Archaeological Contexts**

Many taphonomic processes affect the preservation of starch granules in archaeological settings. Microorganisms are the major factor in starch decomposition, accounting for 95 to 99 percent of molecular breakdown of organic
material in the soil (Andrews et al. 1974, Jorgensen 1983). Earthworms and ants degrade the reminder. Starch hydrolysis rates depend on the enzyme activity of microbes, the physical condition of starch, and the physical and chemical properties of local soil (Barton and Matthews 2006:82). Enzymatic activity is reduced with the absence of water (Eglinton and Logan 1991:320). However, excess water may also add to archaeological starch degradation. Water in soils can also affect soil pH and soil components that interact with starch enzymes and microorganisms. To that end, extremely dry soils can inhibit enzyme-catalyzed hydrolysis, which allows the starch granule to retain its shape and form (Barton and Matthews 2006:83). This can also have a negative effect on starch preservation as extraction of sugars will be prevented, shriveling the starch granule. These conditions indicate that extremes in moisture can have a dramatic impact on the preservation of starch in archaeological contexts.

Less research has been conducted on the effects of temperature on starch preservation. In laboratory settings, starch from some taxa begin to gelatinize with water with extreme high temperatures (above 50°C). While this is unlikely to occur at archaeological sites, sudden dramatic increases in temperature could allow for premature starch gelatinization, resulting in the dissolving of the cellular biochemical structure of the granule (Barton and Matthews 2006:83).

The Fiji Context

Qaraqara is an archaeological site located in the Sigatoka Valley on the island of Viti Levu, Fiji (Figure 2). Food production during Fijian prehistory is not
understood. Human colonizers of Fiji, circa 2800 BP, existed on a diet composed of marine resources (fish, shellfish), and terrestrial fauna (bats, birds, tortoises) (Szabo 2000). In contrast, 1,000 years later, most Fijians had transitioned to a fully agricultural diet (Field et al. 2009). A major focus of research in Fiji is on the development of food production following island colonization. Archaeologically preserved starch has been found in sediments in early Lapita-era sites (Horrocks and Nunn 2007), but it is uncertain to what extent these starches represent a reliance upon root-crops as a dietary staple. The investigation of Qaraqara, a site that is far from the coast but which contains deposits immediately after the Lapita colonization era, provides a context for the testing of the scale and focus of food production. Identification of starch at Qaraqara will aid in the understanding of how food
production was organized, what foods were produced, and the scale at which starchy foods dominated the diet immediately following colonization.

**Research Questions**

1) Can starch be retrieved and identified from artifacts found from the surface, and within the deeper deposits of Qaraqara?

   a) If not, what are the reasons for this failure? More specifically, what are environmental constraints on archaeological preservation of starch?

   b) Are some archaeological materials more amenable to the preservation and extraction of starch?

   c) Are there ideal extraction methods for specific artifact materials?

2) Are any environmental or methodological parameters that could result in successful starch preservation and extraction present at Qaraqara?

3) Based upon these results, how might future research design more successfully and accurately detect starch, and by proxy the production of starchy foods, in antiquity?
CHAPTER 3: MATERIALS AND METHODS

This thesis reports on attempts to extract and identify starch granules from the surface of ceramics recovered from the archaeological site of Qaraqara, Viti Levu, Fiji. The methods used to extract starch will be described and discussed below. Ultimately, no starch was successfully extracted from the ceramic samples. The reasons for these negative results are further assessed in a meta-analysis of published

![Average Temperature Category Frequencies](image)

Figure 3: Temperature category frequencies (°C) correlated with individual artifacts from the sites listed in the case studies.
studies. Meta-analysis is used to compare the environmental parameters that may affect starch preservation in archaeological deposits, and to assess differential results of starch extraction and identification as a result of particular extraction methodologies.

Figure 4: Average Rainfall category (mm) frequencies correlated with individual artifacts from the sites listed in the case studies.
Meta-Analysis of Case Studies

Twenty eight archaeological publications referencing starch granule analysis were obtained, dating from the years 1992-2015. The key variables identified from each publication include local average temperature, local average rainfall, and altitude (as a proxy for humidity)(Table 7). These variables were placed into frequency categories (Figures 3,4,5). If not listed in the publication, the provenance of the archaeological samples were determined using Google Earth. Individual analyses (chi square, pairwise comparisons) were conducted on each variable obtained from the case studies.

The protocols employed to extract starch from archaeological materials were recorded for each case study. These included pipette, scalpel, sonic bath, and Heavy Liquid Separation (HLS). Combinations of extraction methods were often utilized. In addition, the material that was tested for embedded starch was recorded, and placed in the categories marine shell, ceramic, lithic, wood, and sediment (Figure 6). Based upon the success rates noted in the publications, the success or failure of starch extraction was noted for all methods and materials. While rare, published negative results on archaeological starch analysis were sought.
Figure 5: Altitudes ranged from -212 meters to 4050 meters, in 49 sites in the case studies.

Figure 6: Artifact material frequencies from the sites listed in the case studies.
Multiple chi-square analyses were used to determine significant patterns between starch extraction success frequencies, environmental variables, and artifact material. When significant patterns existed, many pairwise comparisons between categories of each variable were compared. These pairwise comparisons were conducted for all categories of temperature, rainfall, altitude, and artifact material.

Chi-square analyses were also conducted to determine if there are significant differences in starch extraction success based on artifact material and extraction method. Pairwise comparisons were utilized between each material/method combination to reveal significant differences. Patterns identified for each variable suggest ideal parameters for successful extraction of archaeologically preserved starch and starch extraction methods. Outliers are included in establishing success parameters. These results are discussed in Chapter 4.

**Materials and Methods for Extraction of Archaeological Starch from the Qaraqara Ceramics**

Ten ceramic fragments from Qaraqara were analyzed for preserved starch granules. The fragments varied in size. Seven were body sherds, and three were rim sherds. Two of the ten fragments were carbonized on one side as a result of being exposed to heat and combustion. All were recovered from the surface of the site of Qaraqara, and thus were likely a maximum of 500 years in age (Field et al. 2014). After starch extraction failure, five additional ceramic samples from the 2014 excavation at Qaraqara were tested. Attempts to retrieve preserved starch granules
from the surface of each sherd were conducted using the previously described
(Chapter 2: Background) methods of scalpel, pipette, and sonic bath.

All attempts at extraction were performed in the Pacific Islands
Archaeological Research Laboratory on The Ohio State University campus. This is
not a sterilized laboratory space, so attempts were made to reduce the risk of
contamination of the samples by introduced starches. Powder-free latex rubber gloves
were used at all times when handling the ceramics, and microscope slides affixed
with double-sided tape were placed around the extraction area to ‘catch’ any stray
starches (from the ceramics, or from the laboratory itself) that may have been present
or produced during extraction. These slides would be later tested for starch presence.

The first method employed stainless steel disposable scalpels to scrape the
surface of the ceramics. A new scalpel was used on each ceramic fragment to avoid
cross-contamination. Scrapings were relocated onto pre-cleaned #1 microscope slides
and covered with a #1 cover glass, and a small amount of clear nail polish was used to
adhere the cover glass to the microscope slide. Each ceramic fragment was sampled
using this method three times.

The second extraction method used pipettes and sterile water to effectively
‘wash’ the starch granules off of the surface of the ceramics. Approximately 50
microliters of Milli Q distilled water was applied to each ceramic sample using a
micro-volume pipette with disposable plastic pipette tips. A new, unused pipette tip
was used for each sample to avoid any contamination. After agitation with the pipette
tip, the water was retracted from the ceramic fragments through the micro-volume
pipette and placed on individual microscope slides. Samples were covered with a new, unused cover glass and adhered to the microscope slide using clear nail polish.

The last extraction method used was a modified sonic bath method, which sought also to ‘wash’ the starch granules off of the surface of the ceramics. Due to the frailty of the ceramic samples, a low-powered (< 100 Hz) sonication system was used. Each ceramic sample was placed in a Ziploc plastic bag with a small amount of distilled water. Each bag was sonicated at 100 Hz for one minute, following by two minutes of sonication on 50 Hz. The artifact was then removed from the sonicator and the water that surrounded it was removed with the micro-volume pipette and transferred to microscope slides. The slides were then covered with cover glass and adhered.

All slides from every extraction methodology were analyzed using Leica lightfield microscopy in the Near Eastern Archaeology and Archaeobotany Laboratory. Microscope slides affixed with double-sided tape were placed in this lab two week prior to microscopic analysis of Qaraqara samples to determine what (if any) starches were naturally airborne in the room. All precautionary slides produced a negative result for starch. In addition, no starch was detected on any of the slides derived from the Qaraqara ceramic samples. The only items detected microscopically was ceramic dust and micro-fragments.
Generation of Modern Starch Reference Collection

In the event that starch was retrieved from the Qaraqara samples, a modern reference collection of starches from botanical sources was established for this study. Samples of fruits, seeds, tubers, and corms were acquired at local supermarkets in Columbus, Ohio. These included sweet potato (*Ipomoea* sp.), taro (*Colocasia* sp.), coconut (*Cocos nucifera*), red sweet potato (*Ipomeoa* sp.), plantain (*Musa* sp.), and banana (*Musa* sp). Additional fruit samples were desired, including breadfruit (*Artocarpus altilis*) and cassava (*Manioc esculenta*), but these could not be obtained in Columbus. The reference sample was created from each of the purchased plants, and each sample was processed in the Pacific Islands Archaeological Research Laboratory (PIARL) three weeks prior to the extraction of starches from the archaeological ceramics. Each sample was sliced using new, sterile disposable scalpels. Samples were then placed on microscope slides and covered with a cover glass. Three microscope slides were created for each sample. Additionally, one slide was devoted to the skin or peel of each referenced sample. The established collection is held in the PIARL for future use.
CHAPTER 4: RESULTS

Results from chi-square analyses for the three environmental parameters (Table 1) obtained from the case studies suggest that annual average temperature, annual average rainfall, and altitude are significant contributors to archaeological starch preservation. Additionally, the chi-square analyses of artifact material and extraction protocol indicate that there is a significant and positive relationship for successful extraction of starch under certain conditions. These results indicate that both environmental and laboratory protocols influence the likelihood of successful archaeological starch extraction.

<table>
<thead>
<tr>
<th></th>
<th>P value</th>
<th>Significant?</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Average Temperature</strong></td>
<td>&lt;0</td>
<td>Yes</td>
</tr>
<tr>
<td><strong>Average Rainfall</strong></td>
<td>&lt;0</td>
<td>Yes</td>
</tr>
<tr>
<td><strong>Altitude</strong></td>
<td>.006</td>
<td>Yes</td>
</tr>
<tr>
<td><strong>Artifact Material</strong></td>
<td>&lt;0</td>
<td>Yes</td>
</tr>
</tbody>
</table>

Table 1: Chi-Square of Success frequency based on environmental variables and artifact material from the case studies. P values less than 0.05 are significant.
Table 2: Pairwise comparisons of starch extraction success/failure frequencies (all protocols) between average rainfall categories for sites in the case studies. P values less than 0.05 are significant.

<table>
<thead>
<tr>
<th></th>
<th>0-250mm</th>
<th>250-500mm</th>
<th>500-1000mm</th>
<th>1000-1700mm</th>
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<tr>
<td>250-500mm</td>
<td>&lt;0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>500-1000mm</td>
<td>&lt;0</td>
<td>0.549</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1000-1700mm</td>
<td>&lt;0</td>
<td>0.141</td>
<td>0.156</td>
<td></td>
</tr>
<tr>
<td>Over 1700mm</td>
<td>0.379</td>
<td>&lt;0</td>
<td>&lt;0</td>
<td>&lt;0</td>
</tr>
</tbody>
</table>

Figure 7: Starch extraction (all protocols) success and failure frequencies and annual average rainfall categories for sites in the case studies.
The Effect of Environmental Parameters on Starch Preservation and Extraction

A wide range of annual rainfall values were represented in the case studies, and the three categories (250-500 mm, 500-1000 mm and 1000-1700 mm were equally represented (Figure 3). Chi square analyses indicated that there are significant differences in successes and failures in starch extraction in the two extremes of the range, categories “0-250 mm” and “Over 1700 mm” (Table 2). These two rainfall ranges both feature a higher number of starch extraction failures than successes (Figure 7). These results indicate that extremes in aridity and rainfall both effect archaeological starch preservation and extraction. They also suggest that extremes in rainfall result in more failures.

![Success and Failure Frequencies for Temperature Categories](image)

Figure 8: Starch extraction success and failure frequencies (all protocols) based on annual average temperature for sites in the case studies.
The case studies provide an uneven representation of annual average temperature categories. The “19-22° C” category dominates the sample (N = 147), while the “3-12° C” and “23-32° C” (N = 83 and N = 84) are moderately represented. Category “13-18° C” (N = 14) occurred in the fewest cases (Figure 4). Despite representing a relatively low proportion of the case studies, the “13-18° C” category had a 100% success rate for starch extraction (Figure 8). The low frequency and high success rate of the “13-18° C” category may skew results in favor of this category. Significant differences exist in success and failure frequencies between all categories except “23-32° C” with the “13-18° C” range, and the “3-12° C” and “19-22° C” range (Table 3). This later combination features similar ratios of starch extraction failures to successes. These results indicate that it is likely that temperature has a substantial impact on starch preservation, given the statistically significant differences between temperature categories.

<table>
<thead>
<tr>
<th></th>
<th>3-12°C</th>
<th>13-18°C</th>
<th>19-22°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>13-18°C</td>
<td>0.003</td>
<td></td>
<td></td>
</tr>
<tr>
<td>19-22°C</td>
<td>0.472</td>
<td>0.002</td>
<td></td>
</tr>
<tr>
<td>23-32°C</td>
<td>0.001</td>
<td>0.139</td>
<td>&lt;0</td>
</tr>
</tbody>
</table>

Table 3: Pairwise comparisons of starch extraction success and failure frequencies (all protocols) for temperature categories (°C) for sites in the case studies. P values less than 0.05 are significant.
Archaeological sites from the case studies range in altitude from 212 meters below sea level to 4050 meters above sea level. The “500-1000”, “2500-3000”, and “3000-3500” meters above sea level categories are not represented in the case studies. There are significant differences in success and failure frequencies of extracted starch between all altitude categories except “-250-0” and “2000-2500” categories and the “2000-2500” and “Over 4000” category (Table 4). There is a similarity in the number of successful starch extraction cases between the first two categories and a similarity in the number of failed starch extraction cases between the two later categories (Figure 9). All of these mentioned categories are underrepresented in this sample from the case studies, which may have statistical implications for the results. In combination with other environmental factors, altitude, (and, by proxy, humidity) may influence preservation of archaeological starch.

The Effect of Artifact Material on Starch Preservation and Extraction

The successful extraction of archaeological starches from lithics, ceramic, marine shell, sediment, and wood were all evaluated for the case studies. Lithic artifacts had the highest frequency of successful starch extraction (Figure 10), although this may be due to the abundance of tested lithic materials (N = 210) in the case studies. The durable nature of lithics, and their excellent preservation compared to marine shell or ceramic, may also add to the higher rates of successful extraction. Marine shell, sediment, and wood were all marginally represented in the literature
review sample (N = 23, N = 19, N= 13). There are only significant differences in starch extraction success/failure frequencies

Table 4: Pairwise comparison of starch extraction success and failure frequencies (all protocols) for altitude categories (m) for sites in the case studies. P values less than 0.05 are significant.

<table>
<thead>
<tr>
<th></th>
<th>-250-0m</th>
<th>0-500m</th>
<th>1000-1500m</th>
<th>1500-2000m</th>
<th>2000-2500m</th>
<th>3500-4000m</th>
</tr>
</thead>
<tbody>
<tr>
<td>0-500m</td>
<td>.002</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1000-1500m</td>
<td>.014</td>
<td>&lt;0</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1500-2000m</td>
<td>.007</td>
<td>&lt;0</td>
<td>&lt;0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2000-2500m</td>
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<td>&lt;0</td>
<td>&lt;0</td>
<td>&lt;0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3500-4000m</td>
<td>.032</td>
<td>&lt;0</td>
<td>&lt;0</td>
<td>&lt;0</td>
<td>.012</td>
<td></td>
</tr>
<tr>
<td>Over 4000m</td>
<td>N/A</td>
<td>.002</td>
<td>.014</td>
<td>.007</td>
<td>.333</td>
<td>.032</td>
</tr>
</tbody>
</table>

when comparing ceramic to all of other artifact materials (Table 5). Although ceramics are well represented in the sample of case studies (N=106), the success rate is 46%. Compared to the success rates for marine shell (74%), sediment (92%), and wood (71%), starch extraction from ceramic artifacts is relatively unsuccessful.
Figure 9: Starch extraction success and failure frequencies (all protocols) based on altitude categories from the case studies.

Figure 10: Starch extraction success and failure frequencies (all protocols) for artifact material categories from sites in the case studies.
The Effect of Starch Extraction Protocols on Successful Starch Extraction

Chi-square analyses indicate that the success of an extraction protocol is directly related to artifact material (Table 5). Significant differences exist for extraction success frequency between every material/method combination, especially when compared to the scalpel/pipette protocol used on ceramic materials. No successes were recorded for this combination. Thus, the scalpel/pipette protocol is significantly less successful than all other combinations, including those that had a minimum of one successful instance of extraction. Similarly, there are significant differences in extraction success frequencies between the ceramic and scalpel combination (success N=21, 43%) and most combinations, except sonic bath and heavy liquid separation used on lithics (success N=17, 94%). This earlier combination features about equal frequencies of successes and failures (Table 6). There are very few significant differences between success frequencies of material and method combinations on lithic artifacts. All methodologies conducted on lithic artifacts are generally successful in archaeological starch extraction.

<table>
<thead>
<tr>
<th></th>
<th>Ceramic Shell</th>
<th>Marine Shell</th>
<th>Lithic</th>
<th>Sediment</th>
</tr>
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<tbody>
<tr>
<td>Marine Shell</td>
<td>0.043</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lithic</td>
<td>&lt;0</td>
<td>0.307</td>
<td></td>
<td></td>
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<tr>
<td>Sediment</td>
<td>0.002</td>
<td>0.19</td>
<td>0.268</td>
<td></td>
</tr>
<tr>
<td>Wood</td>
<td>0.023</td>
<td>0.382</td>
<td>0.526</td>
<td>0.542</td>
</tr>
</tbody>
</table>

Table 5: Pairwise comparisons of starch extraction success and failure frequencies (all protocols) between artifact materials from sites in the case studies. P values less than 0.05 are significant.
Based on the case studies, extraction methodologies involving distilled water (pipette and sonic bath methods) were the least successful when performed on porous artifact materials, such as undecorated and decorated ceramics. Overall, extraction protocols used on ceramics generally have a higher frequency of failures than other artifact materials (Table 6). However, scalpel–based extraction on smooth surface artifacts (such as lithic and shell) similarly had low success rates for extraction. The limited number of case studies employing these combinations of materials and protocols may further accentuate this relatively low success rate.

<table>
<thead>
<tr>
<th>Method/Material</th>
<th>Success</th>
<th>Failure</th>
<th>Total</th>
<th>Success %</th>
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</thead>
<tbody>
<tr>
<td>Ceramic/HLS</td>
<td>1</td>
<td>0</td>
<td>1</td>
<td>100%</td>
</tr>
<tr>
<td>Lithic/HLS</td>
<td>25</td>
<td>1</td>
<td>26</td>
<td>96%</td>
</tr>
<tr>
<td>Lithic/Pipette</td>
<td>28</td>
<td>2</td>
<td>30</td>
<td>93%</td>
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<tr>
<td>Marine Shell/Pipette/SonicBath</td>
<td>17</td>
<td>6</td>
<td>23</td>
<td>74%</td>
</tr>
<tr>
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</tr>
<tr>
<td>Lithic/Scalpel</td>
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<td>1</td>
<td>9</td>
<td>89%</td>
</tr>
<tr>
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<td>20</td>
<td>0%</td>
</tr>
<tr>
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<td>7</td>
<td>19</td>
<td>63%</td>
</tr>
<tr>
<td>Lithic/SonicBath</td>
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<td>0</td>
<td>24</td>
<td>100%</td>
</tr>
<tr>
<td>Lithic/SonicBath/HLS</td>
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<td>1</td>
<td>18</td>
<td>94%</td>
</tr>
<tr>
<td>Ceramic/Scalpel</td>
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<td>28</td>
<td>49</td>
<td>43%</td>
</tr>
<tr>
<td>Ceramic/SonicBath/HLS</td>
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<td>3</td>
<td>18</td>
<td>83%</td>
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<tr>
<td>Ceramic/SonicBath</td>
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<td>0</td>
<td>6</td>
<td>100%</td>
</tr>
<tr>
<td>Lithic/Pipette/HLS</td>
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</tr>
<tr>
<td><strong>Total</strong></td>
<td>189</td>
<td>63</td>
<td>252</td>
<td>75%</td>
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</table>

Table 6: Success/failure frequencies for artifact material and extraction protocol combinations from case studies, with success percent rate. P values less than 0.05 are significant.
Explaining the Failed Extraction of Preserved Starch from the Qaraqara Ceramic Sample

The Sigatoka Valley, located in the western portion of the Island of Viti Levu, Fiji, enjoys an annual average temperature of 31° C. The average annual precipitation is 1875 mm, most of which falls during the wet season between December and February. The altitude at the site of Qaraqara is 29 meters above sea level. With these conditions, Qaraqara fits within the ideal parameters for starch preservation. However, none of the attempts to obtain starch from the Qaraqara ceramic assemblage was successful. How can these results be explained?

Based upon the analysis of the materials, methods, and environmental parameters in the case studies, the most parsimonious conclusion is that the Qaraqara sample, being composed of surface ceramics, was a poor material for the preservation of archaeological starch, or, the materials themselves, due to their porosity, make the removal of starch exceedingly difficult. Since the local environment (annual rainfall, temperature, and altitude) of Qaraqara meets most requirements for good archaeological starch preservation, failure to extract starch is likely related to interactions between extraction protocols and artifact material. When compared to the success rates for the previously described extraction protocols and their application with other undecorated ceramics (Table 6), the failure in extraction for the Qaraqara sample is consistent with the failure to extract starch in other samples that were composed of ceramic. These results indicate that since the Qaraqara samples are porous, neither the pipette nor sonic bath methods would be very successful for the extraction of starch. The porosity of the material simply absorbs the liquid, and prohibits the recollection of any adhered starch.
CHAPTER 5: DISCUSSION

*Starch Preservation or Extraction?*

This research sought to extract starch from the ceramic samples obtained from Qaraqara Fiji. It also attempted to determine a plausible explanation for the failed extraction of starch from the samples. Based upon the survey of the case studies, the site of Qaraqara was not extreme in terms of aridity, but higher in rainfall, and therefore would have experienced conditions that favored the preservation of starch in archaeological deposits. However, average temperature and altitude may or may not have been detrimental; the case studies indicate that both successes and failures are associated with conditions similar to those of Qaraqara. As an additional factor, ceramics were shown to be less amenable to starch extraction, especially when liquid and pipette methods were employed. Therefore it should be noted that although no starch was extracted from the Qaraqara ceramics, it does not necessarily mean that there is no starch on these samples. The ceramic material may simply have inhibited the removal of starch when the liquid and pipette extraction methods were applied. Other extraction methods may have been more successful.

This same conundrum applies to the case studies assessed in this research: it is difficult to discern if the environment degraded the starch granules, the material may have prohibited their extraction, or the extraction methodology was not well suited.
for the particular material. Based upon the result of the case studies, certain environments, materials, and methods produce significantly better results for the preservation and extraction of starch. But when multiple variables are present, which combination is more likely to produce a success?

Possible solutions to this problem can be tested by comparing different artifact materials from the same site using that were analyzed using one extraction protocol. Two research studies from the case studies use the same extraction protocol on multiple artifact types from archaeological sites of the same region that feature identical environmental conditions. A sandstone grinding stone and a ceramic fragment from two archaeological sites in Argentina were tested for starch using the pipette protocol by Bonomo et al. (2011). Both artifacts yielded starch. Similarly, ceramic artifacts from Salvatierra and lithic artifacts from Granja del Padre (both in Bolivia with similar environmental conditions) were tested using both the sonic bath and heavy liquid separation protocols (Dickau et al. 2012). Eleven ceramic artifacts and nine lithics were tested. There is a 100% extraction success rate for the ceramic artifacts, and an 89% success rate for lithics. These two case studies show similar extraction success rates between lithics and ceramics for two protocol combinations. Since there are similar extraction success rates between environmental similar sites, it can be concluded that the extraction protocol may be responsible for successful starch extraction.

Conversely, the results from testing identical artifact materials at one site using different extraction protocols can provide an indication of the more successful combination. Although rare, a single example of this kind of cross-analysis is seen in Piperno et al. 2009. Twenty six lithic artifacts recovered from the Xihuatoxtla Shelter
site in Mexico were tested using three different protocols. The scalpel protocol was used on nine grinding stones and had an 89% success rate of extraction. The heavily liquid separation was used on twelve grinding stones, featuring a 92% success rate of extraction. Five chipped stoned tools were sonicated in a sonic bath and had a 60% success rate. Since all the lithics were from the same archaeological site, with the same environmental conditions, it can be concluded that all experienced the same level of starch degradation. As such, it is the difference in applied extraction protocols that account for extraction failures and successes, not the environment or the materials.

*Suggested Extraction Methods Based on Artifact Material*

From the chi-square analysis of the published case studies, starch extraction success was dependent on artifact material, and lithics were the material that produced the most success (Table 8). Failures were significantly more common when the extraction method coupled distilled water with porous materials. This was expected as porous artifacts absorb water as well as starch granules. Previous research suggests that dry extraction methods for porous artifact materials, specifically scalpel extraction, is preferable. The pairing of this method and material results in higher rates of success (Barton 2006, Torrence et al. 2004).

Although lithic artifacts in the case studies generally yielded archaeological starch, some protocols are more successful than others. Three protocol combinations used on lithic artifacts feature a 100% extraction success rate: pipette and sonic bath, pipette and heavy liquid separation, and sonic bath alone. The least successful protocol
combination for lithic artifacts is scalpel and heavy liquid separation (63%), followed by scalpel alone (89%). This suggests that the scalpel methodology is not best suited for lithics, especially smooth artifacts that do not have crevices where starch can adhere. Extraction protocols featuring distilled water or other liquid chemicals are best suited for lithic artifacts.

Only one case study tested for archaeological starch on marine shell. Similarly, only one extraction protocol combination was utilized: pipette and sonic bath. This protocol combination was not highly successful (74%). Protocols using distilled water may be at fault for the relatively low success rate. While not an ideal success rate, this protocol combination should not be completely disregarded when attempting to extraction archaeological starch from marine shell artifacts.

**Suggestions for Detecting Starch at Qaraqara**

Exploring extraction methodologies advances archaeological science. These results suggest limitations to current protocols for starch extraction based on artifact material, but also suggest ideal starch extraction methods based on artifact material. Based upon the analysis of the case studies, the reason for the failed extraction of starches from the Qaraqara can be attributed to either the artifact materials (ceramic) or the extraction methodology. Various extraction protocol combinations were used on ceramic artifacts in the case studies. While there was generally poor success in starch extraction on ceramic artifacts, some extraction protocol combinations were more successful than others. Heavy liquid separation used on ceramics in the case studies had a perfect
success rate (although it should be noted that this protocol was used on only one artifact). Disregarding that small sample, the most successful extraction protocol combination used on ceramic artifacts was sonic bath and heavy liquid separation (83%). The scalpel and pipette protocol combination, which was used with the Qaraqara samples, had no successes in these case studies.

Ethnographic data, as well as the presence of carbonized materials on pots, indicate that the ceramics vessels of Qaraqara were probably used to cook starches. The antiquity of ceramics as a cooking technology is under investigation in Fiji; other techniques, such as bamboo-section steamers or earth ovens, are also probable methods for processing roots. Based upon the research presented in this thesis, future research that seeks to accurately test ceramics for starch must use alternative methods. Possible additional protocols to be used includes heavy liquid separation, and the sonic bath protocol in combination with heavy liquid separation.

There are several other ways the presence of archaeological starch could be tested for the site of Qaraqara. Sediment at different levels of excavation could be tested for starch. While the Qaraqara site is currently a family farm that cultivates starchy foods, sediment from beneath the plow zone and past the root’s reach could be tested for starch. Additionally, although most artifacts recovered from Qaraqara are ceramic fragments, some lithic artifacts are available. Lithics compose a substantial portion of the artifact materials from the case studies and generally have a high starch extraction success rate (Table 6). If there is archaeologically persevered starch on the Qaraqara lithic artifacts, a variety of extraction protocols and combination of protocols can be implemented. These include pipette, scalpel, sonic bath, and heavy liquid separation protocols. Due to the
non-porous nature of lithics, the best protocols are those including distilled water. Regardless of environmental conditions, when the sonic bath protocol was used on lithic artifacts in the case studies, there was a 100% success rate. Although all extraction protocols are generally successful on lithic artifacts, the least successful protocol combination was scalpel and heavily liquid separation (63% success rate).

Other limitations to assessing starch extraction protocols

The limited number of reported negative results retrieved from the archaeological literature suggests that failed extraction attempts are only minimally reported. This is problematic, as it leaves the causes for the failures unassessed. Additional studies that report the successes and failures associated with starch preservation, extraction method, and artifact material type is needed to substantially extend understanding of the ideal parameters suggested here.

The Benefits of Comparative Research

This research evaluates the environmental parameters, extraction methodology, and artifact material that effects archaeological starch detection. These results will benefit archaeologists and archaeobotanists by provided a set of expectations for the conditions of starch preservation, and making suggestions for the materials and methods most likely to result in the successful extraction of starch. Specific extraction methodologies are more successful on certain artifact types. Providing an extensive classification of methods and materials will save valuable time, money, and energy when extraction starch from different types of settings.
Implications of Results for Detecting Agriculture in Prehistoric Fiji

Although it is difficult to detect starch on ceramic artifacts from Fiji, starch granule analysis can be successfully used on other materials recovered to study subsistence change and agriculture. The described starch extraction protocols can be used on other artifact types with greater success, including lithics and marine shell. Additionally, starch granule analysis can be conducted on dental calculus retrieved from archaeological human remains. This methodology has been successful in Fiji (Valentin et al. 2006) as well as other regions of the world (Hardy et al. 2009).

Other methodologies can be used to study subsistence change and agriculture in Fiji. Stable isotope analysis has been used to identify diet and dietary transition on archaeological human remains (Kusaka et al. 2010, Larsen 1997, Lubell et al. 1994). Plant type can be determined isotopically in bone collagen based on stable isotope analysis of carbon. Additionally, it is possible to discriminate between marine and terrestrial food sources using nitrogen stable isotope analysis. While stable isotope analysis can be used on human bones, applying this methodology to human teeth will show diet change through time in an individual. Although stable isotope analysis is an invaluable methodology to identify variation and transition in past dietary reconstructions, limited analyses of this sort have been conducted at Fijian archaeological sites (Field et al. 2009, Jones and Quinn 2009, 2010, Valentin et al. 2006).
CHAPTER 6: CONCLUSION

Starch granule research can aid in determining various aspects of life and behavior in antiquity, including food production. Certain environmental conditions play a larger role than others in the preservation of archaeological starch, both in sediment and adhered to artifacts. Furthermore, there are some artifact material and extraction protocol combinations that are more successful than others. As such, these combinations should be considered before engaging in archaeological starch granule analysis.

Qaraqara is an important archaeological site in Fiji that can contribute to archaeological research questions regarding food production. Although the application of starch granule analysis on the surface ceramics from Qaraqara was unsuccessful, additional samples will be tested using other extraction protocols. Starch granule analysis can also be utilized on sediments from Qaraqara. Regardless of the extraction protocol, starch analysis will continue on artifacts from this site. Additional archaeological research, such as isotope analysis and zooarchaeological analyses, will be employed.

Starch granule analysis is an invaluable methodology in archaeology for addressing questions regarding food production, tool use, and environmental reconstruction. As such, it should be implemented in archaeological studies around the world, complimenting other methodologies. Although a relatively new method of
analysis, starch granule analysis will likely continue to increase in popularity among archaeologists and archaeobotanists.
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Barton, Huw, and Richard Fullagar

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Clark, Geoffrey and Atholl Anderson (Eds.)

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Crowther, Alison


Crowther, Alison, Michael Haslam, Nikki Oakden, Dale Walde, and Julio Mercader

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Zarrillo, Sonia, Deborah M. Pearsall, J. Scott Raymond, Mary Ann Tisdale, and Dugane J. Quon
APPENDIX OF TABLES
Table 7: Environmental variables for sites examined in this study

<table>
<thead>
<tr>
<th>Excavation Location</th>
<th>Site</th>
<th>Average Temperature (°C)</th>
<th>Average Rainfall (mm)</th>
<th>Altitude (M)</th>
<th>Publication</th>
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<td>New Ireland</td>
<td>Anir</td>
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<td>Over 1700</td>
<td>12</td>
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</tr>
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<td>Marquesas Islands</td>
<td>Anaho: Teavau’ua</td>
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<td>1000-1700</td>
<td>11</td>
<td>Allen and Ussher 2013</td>
</tr>
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<td>Ecuador</td>
<td>Real Alto</td>
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<td>0-250</td>
<td>25</td>
<td>Pearsall et al. 2004</td>
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<td>250-500</td>
<td>-212</td>
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<tr>
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<td>23</td>
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<td>640</td>
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<td>Over 1700</td>
<td>750</td>
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</tr>
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Table 8: Chi-Square p-values for success frequency based on every artifact material and extraction method combination from case studies. * = 0% success rate. **Bold combination** = statistically significant. P values less than 0.05 are significant.

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