University of Évora

ARCHMAT
(ERASMUS MUNDUS MASTER IN ARCHaeological MATerials Science)

Mestrado em Arqueologia e Ambiente (Erasmus Mundus – ARCHMAT)

Understanding the Tibes ceremonial center in its regional context:
Ceramic material culture characterization from the Río Portugués drainage in Puerto Rico

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(Supervisor - Universidade de Évora)

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Évora, October 2018

A tese não inclui as criticas e sugestões do Júri

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Compreendendo o centro cerimonial de Tibes em seu contexto regional:
Caracterização da cultura de materiais cerâmicos da drenagem do rio Portugués em Porto Rico

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Abstract

This thesis employs a multi-analytical, materials science approach to study ceramics from Tibes and three other sites in the region (PO-42, PO-43, and PO-48). The main goal is to gain insight on (1) raw material provenance, (2) interpretation of function, and (3) manufacturing technology in the early Ostionoid Caribbean (600-1200 AD), to understand ceramic variability. Mineralogical (Petrography, X-Ray Diffraction) and chemical (Energy Dispersive X-Ray Fluorescence, Inductively Coupled Plasma Mass Spectrometry, and Scanning Electron Microscopy) analysis were conducted on 50 samples from south central Puerto Rico, and the results were integrated within the archaeological context. Employing the theoretical framework of 'technological choices' (Sillar and Tite 2000), this research discerned four different raw material sources, and consequently, four distinct paste recipes. This thesis discusses the anthropological implications of intersite and intrasite ceramic variability in south central Puerto Rico, and its relationship to the concomitant rise of chiefdoms in the region.

Key words: Tibes ceremonial site; Pottery analysis; Caribbean archaeology; Ceramic provenance; Ceramic technology

Resumo

Esta dissertação emprega uma abordagem multi-analítica à luz da ciência dos materiais para o estudo de cerâmicas de Tibes e três outros locais na região (PO-42, PO-43 e PO-48). O principal objetivo é obter informações sobre (1) a proveniência da matéria-prima, (2) interpretação da função das cerâmicas e (3) tecnologia de fabrico no início do Caribe Ostionóide (600-1200 dC), para compreensão da variabilidade cerâmica. A análise mineralógica (Petrografia, Difração de Raios-X) e química (Fluorescência de raios-X por energia dispersiva Espectrometria de Massa com Plasma Indutivamente Acoplado e Microscopia Eletrónica de Varrimento) foram conduzidas em 50 amostras do centro-sul de Porto Rico, e os resultados foram integrados dentro do seu contexto arqueológico. Aplicando o quadro teórico das "escolhas tecnológicas" (Sillar e Tite 2000), esta pesquisa permitiu distinguir quatro diferentes fontes de matérias-primas e, consequentemente, quatro diferentes receitas de pastas. Esta dissertação discute as implicações antropológicas da variabilidade cerâmica entre sites e intra sites no centro-sul de Porto Rico, e sua relação com o aumento concomitante de chefes na região.

Palavras-chave: Sítio Cerimonial de Tibes; Análise de cerâmica; Arqueologia caribenha; procedência cerâmica; tecnologia cerâmica

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I would like to recognize my father's constant availability for correcting earlier drafts of this thesis. Also, I would like to thank my mother for always encouraging me to travel and to grasp bold opportunities (even if it keeps me away from home for 2 years). Finally, I would like to say a heartfelt thank you to my two older brothers, José Ciro and José Darío, who have led by example through their hard work, dedication and passion for their fields. I am eternally grateful for both of you coming to visit me in the small town of Évora, and for bringing with you a piece of home on each of your visits. This thesis is dedicated to them.

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1 Introduction

Ceramics are frequently one of the most abundant materials found at archaeological excavations and the technological choices behind production are poorly understood in the Caribbean. In depth archaeometric studies on pre-Columbian Caribbean material culture have emerged in the past twenty years (Conrad et al., 2008; Crock et al., 2008; Curet, 1997; Descantes et al., 2008; Hofman et al., 2007; Isendoorn et al., 2008, 2008; Siegel et al., 2008; Ting et al., 2016). This thesis aims to help understand the potter's choices behind ceramic manufacturing technology during the early Ostionoid period (600-1200 AD) in Puerto Rico, and to frame these insights within the regional context. The core of this study is the site of Tibes, which is located in south central Puerto Rico in the municipality of Ponce. In addition, ceramics from three neighboring sites in the region along the Portugués and Chiquito rivers were studied, including the site of La Mineral (PO-42), Los Gongolones (PO-43), and Escuela Río Chiquito (PO-48) (Figure 1). By employing a multi-analytical approach, this work intends to gain insight on (1) raw material provenance, (2) interpretation of function, and (3) manufacturing technology to ultimately understand intersite and intrasite ceramic variability.

Tibes is the earliest known civic-ceremonial center in the Greater Antilles, and contains valuable information about ritual and burial practices, and social stratification (Curet and Stringer, 2010). The site was occupied between 400 BC until 1200 AD, and to date, it is the earliest ceremonial center in the Caribbean. Tibes's regional significance stems from its liminal position in a cultural transition zone. Between the western and eastern Puerto Rican ancient cultural spheres, Tibes is unique in bringing together two different types of iconography, ceramic styles and cultural practices (Curet and Stringer, 2010). The site, commonly visited as a sightseeing attraction for native Puerto Ricans and tourists, has been an active archaeological park open to visitors since April 30, 1984. Most recently, the Tibes Archaeological Project was established by Prof. L. Antonio Curet in 1995 to study the rise of social complexity in Puerto Rico. This project differs from previous excavations throughout the site's history because of its focus on a multidisciplinary and multi-stage approach (Curet and Stringer, 2010, p. 2). Since its inception, the project has integrated expertise from a multiplicity of disciplines to understand different aspects of ancient life. This will be the first contribution to the study of ceramic technology through a materials science approach in south central Puerto Rico.

This thesis employs a synchronic approach to gain an in depth understanding of ceramic technology at Tibes. By studying ceramics that stem from stratigraphically excavated layers dated by radiocarbon analysis to the same time period, we intend to understand the interregional variation of provenance, use, and production technology. The highest incidence of cultural remains, including ceramics, monumental architecture, bones, shells, and burials, are found during the early Ostionoid period, which suggests a higher frequency of activity at the site. With nine *plazas* and *bateys* during this period, Tibes is the largest ceremonial center in south central Puerto Rico (Curet and Stringer, 2010) (See Figure 2). This period is not only characterized by rich ceremonial architecture, but also by a rise in the density of sites, a decrease in house size and a lessening in the "quality" and the decorative intricacy of ceramics (Curet, 1997). This differs from the earlier Saladoid ceramics that stem from influence from the Orinoco Valley in present day Venezuela. The later ceramics from the early Ostionoid period, which are the focus of this study, are hypothesized to be a product of an organically developed local manufacturing. In the literature, many archaeologists have discussed this variance in ceramic "quality" as a side effect of the development of these local Ostionoid ceramics (Curet, 1997; Siegel et al., 2008). Although there is a decline in aesthetic decoration, Curet argues that the general quality and durability of the ceramics increases due to a decrease in overall porosity (Curet, 1997). In addition, the near absence of adornments has been an indicator of the rise of hierarchy and complex societies around the world (Siegel, 1999). Therefore, the change in ceramic technology between the Saladoid and Ostionoid periods is not only fundamental to understanding how manufacturing technology developed or changed, but also can give insights or be an indicator of the prominent socio-political organizational alterations and the concomitant development of chiefdoms in the region.

The Tibes Archaeological Project began in 1995 and continues to this day with its main goal of "studying the changes in social organization in Ancient Puerto Rico from a lower level and smaller scale of analysis" (Curet and Stringer, 2010, p. 2). Following this approach, Curet's project focuses on the local unit of analysis. Most studies in the Caribbean have focused on cultural spheres that are based on ceramic stylistic analysis, and thus, might not correspond directly to social processes happening on a local level.

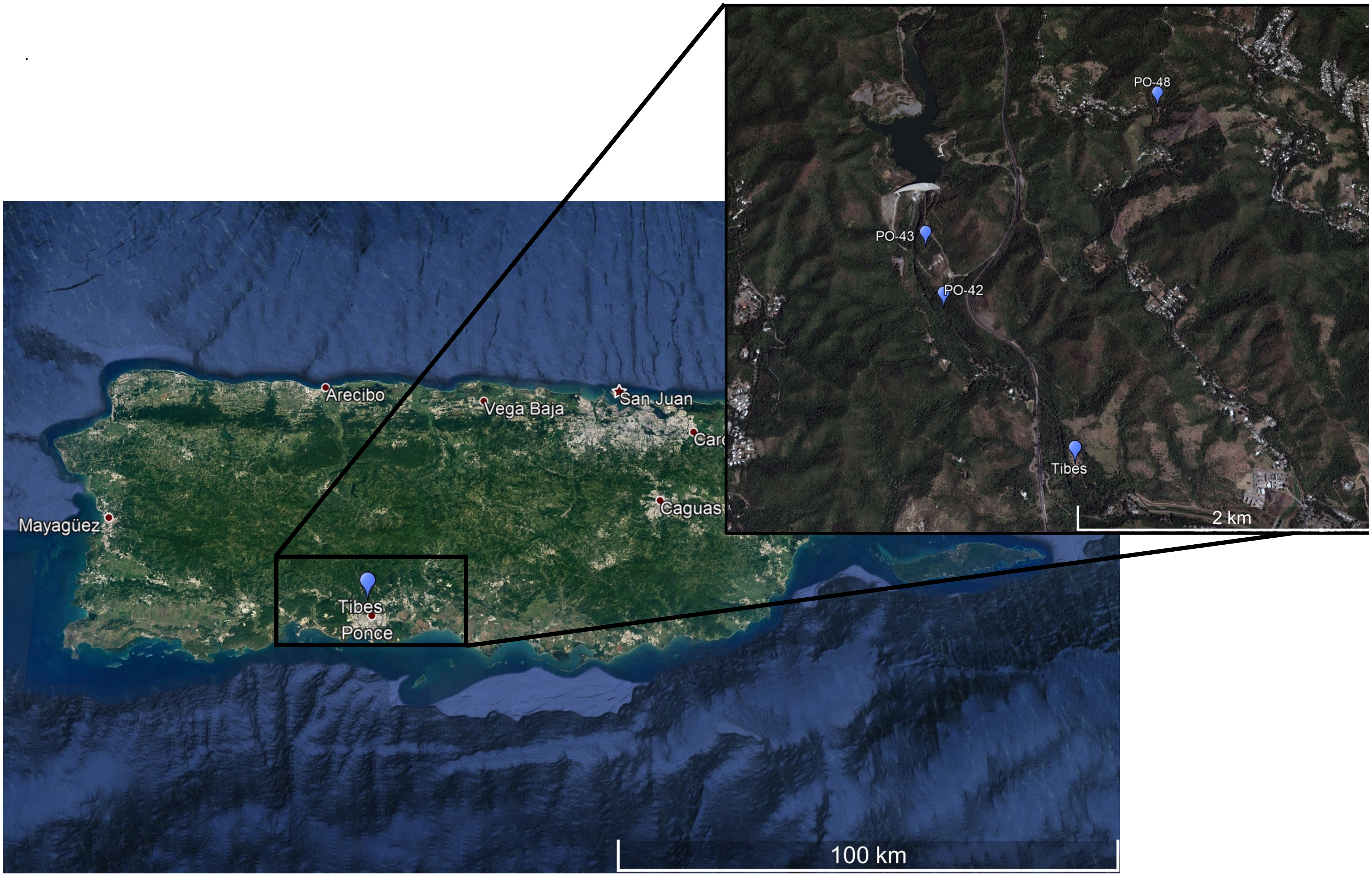

Figure 1 Map of Puerto Rico showing the sites of Tibes, PO-42, PO-43, and PO-48 (Images from Google Earth Landsat Images).

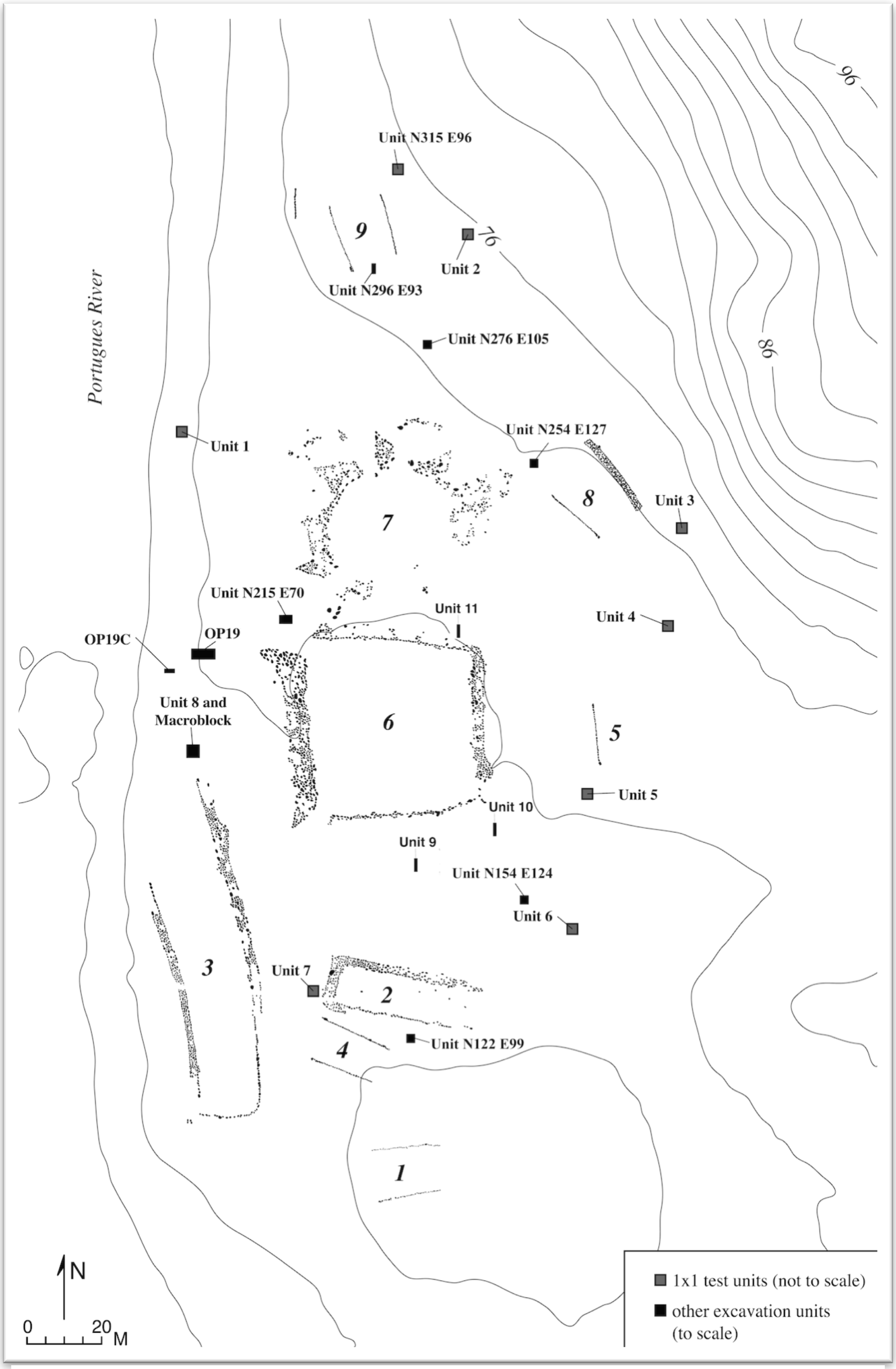

Figure 2 Drawing of the excavation units at Tibes by Jill Seagard.

The materials from the other three sites stem from the Tibes Archaeological Survey Project (TASP). This project is the result of the doctoral dissertation of Joshua M. Torres and was mainly conducted in the summer of 2008. Using the information obtained from survey, test excavations, and settlement pattern analysis, Torres's main goal was to understand the regional context surrounding Tibes between 600-1200 AD and to comprehend how *cazicazgos* or chiefdoms evolved in the region through the social construction of community (Torres, 2012). Torres explores the use of plazas and *bateys* in the region, and analyzes how material culture evolved through time. As noted in Torres's conclusion,

> "it is necessary to expand investigations to consider comparative intersite analyses between proximally related sites to establish variability in particular artifact assemblages that would give clues to interrelationships between sites and indicators of power and identity through material culture" (Torres, 2012, p. 436).

This thesis aims to contribute to these efforts by conducting an exploratory study on ceramics from sites in the region and observing how the material properties of the ceramics can contribute to answering the questions posed by Torres. In addition, Curet and Torres, (2010) propose important questions to guide future research at Tibes. Most importantly, they question whether Tibes was occupied or vacant at its height of power and religious significance. Although this question has been tackled at many sites with ball court plazas in Mesoamerica, it still remains largely unanswered in Puerto Rico and the Caribbean basin. This thesis will not attempt to answer whether Tibes was occupied or vacant, but aims to understand the ceramic variability at Tibes and other sites, and subsequently hypothesize about the dynamics of pottery production in the region.

The true value of importing materials science approaches to archaeological materials relies on the archaeological interpretations which can be attained through this information. In essence, what questions can be asked to the materials and how can researchers answer them? The contemporary debates on the theoretical frameworks employed in materials science approaches to archaeology are reviewed in Chapter 2. Due to Tibes's wealth of archaeological background and context, the materials from the site are well suited to initiate well grounded, archaeologically driven archaeometric studies. Although Tibes was occupied since 400 AD, this study focuses on the last phase of occupation, when the ceremonial structures were constructed and in use. The early Ostionoid period (600-1200 AD) is known in Puerto Rico for an increase in the number of sites, the simplification of material culture, and a centralization of power due to the institutionalization of social inequality and chiefdoms. The intricacies of this archaeological period in the Caribbean are presented in detail in Chapter 3. In addition, the role of ball courts and plazas, ceremonial sites and previous studies at the site will be outlined.

Although there is not an abundance of previous archaeometric studies done in the Caribbean, Chapter 3 discusses the available literature in terms of the materials studied and the methods employed. Ceramic characterization through knowledge of the local geology is only the first step in archaoemetric studies. In conjunction with archaeological background, it will be paramount to understand the local geology in the region. To distinguish between what is local and non-local, a solid grasp on the geological formations in proximity to the sites is key. The region is mainly characterized by a combination of volcanic rocks in the north close to the Cordillera Central mountain range and limestone formations towards the coast. Due to the wide-ranging geological formations summarized in Chapter 4, it is plausible to associate the ceramics with possible production centers of raw clay sources.

To characterize the ceramics from south central Puerto Rico, a variety of methodologies that stem from materials science and geology are applied. The current debates on which methodologies are best suited for cultural heritage are discussed in Chapter 4. Methodological considerations from archaeologists and scientists will be incorporated to demonstrate why each methodological approach was chosen, a brief overview of the theory of the techniques, and a justification for choosing them for this study.

Ceramics are the main remnant of ancient civilizations found in the Caribbean, and throughout the ancient world. They can contribute a myriad of information about society, aesthetic valuation and the technological choices by the potters. This study has at its disposition 50 ceramic samples from four different sites in south central Puerto Rico including, Tibes, La Mineral (PO-42), Los Gongolones (PO-43) and Escuela Río Chiquito (PO-48). These ceramics were chosen because they are coeval and are from sites with *bateys* or small ball court plazas.[1] Due the availability of information about the archaeological contexts, this study will assess Tibes within its regional context. To understand past technological choices used in the production of ceramics,

[1] Escuela Río Chiquito site (PO-48) does not appear to have a ball court plaza, but has not been able to be studied in full.

a set of mineralogical and chemical approaches will be engaged. This thesis will employ a multi-analytical approach to provenance, function, and manufacturing technology.

Multiple analytical techniques and microscopy were conducted on all 50 samples to better understand the raw material composition and nature of the inclusions, the firing environment, and overall production technology. Petrographic analysis was used to obtain information on the temper's mineralogical composition and the textural features of the clay matrix. X-Ray Diffraction (XRD) was employed on the 50 powdered samples to identify the different mineralogical compositions of the pottery. Quantitative Energy Dispersive X-Ray Fluorescence (ED-XRF) analysis was conducted on all samples in glass pearl form to identify the major and minor chemical elements. Inductively Coupled Plasma Mass Spectrometry (ICP-MS) analysis was used on the powder samples after an acidic digestion protocol to quantify and identify trace element patterns to distinguish different raw material sources. Point analysis on aplastic inclusions was conducted via Scanning Electron Microscopy coupled with Electron Dispersive X-ray Spectroscopy (SEM-EDS). The data obtained from these different techniques was treated and integrated with the archaeological data. The scientific reasoning behind these choices in methods and the specific parameters used are discussed in Chapter 5.

The results of these methods are discussed in Chapter 6 and interpreted within the archaeological and geological contexts in Chapter 7. In addition, this thesis will demonstrate the importance of using a multi-analytical approach with cutting-edge technology to understand these complex materials. Concluding remarks, limitations and future projects will be expounded on in the conclusions section. This project is an exploratory study on a representative assemblage of ceramic sample from the site of Tibes to gain insight into the socio-cultural and political dynamics in the region. Ultimately, it aims to contribute to the understanding of provenance, function and manufacturing technology in the early Ostionoid period and aid in future studies at the site.

2 Technological Choices: A Theoretical Framework

The interpretation of materials science results in an archaeologically meaningful way is reliant on a defined theoretical framework. Archaeological researchers have developed several approaches to understanding the material properties of ancient artifacts and interpreting their meaning in the past. Since the beginning of New Archaeology, developed in the 1960s, scientific approaches and quantitative results have been integral in the field of archaeology. Following the use of different scientific methods such as radiocarbon dating, New Archaeology stopped concentrating on creating culture histories and relative chronologies and started focusing on anthropologically driven questions about past societies. In addition, because of this new scientific approach, which centered mainly on technical, environmental and functional explanations, several methods and techniques from the physical, chemical, geological and biological sciences were integrated into the archaeologist's toolkit (Albero Santacreu, 2014, p. 127). Arguably, the reliance on quantifiable results and scientific "truths" was taken to an extreme during the heyday of processual archaeology. The reactionary movement of post-processual archaeology, developed in the 1980s, began to focus on human agency in the past. In addition, post-processual archaeology focused on the cultural, ideological and social aspects in antiquity (Albero Santacreu, 2014, p. 127). These two theoretical paradigms were chiefly noticeable in the academic output in the United States and the United Kingdom, and was largely absent in the application of the archaeological sciences in Europe. This can be mainly attributed to the role archaeology plays within anthropology departments in the US and UK, contrary to its close relationship to history and classics in Europe. Although different theoretical backgrounds guided research, similar technological innovations in archaeometry occurred. Today, archaeological science is witnessing the merging of many theoretical approaches with the main goal of understanding and learning about past cultures and peoples. Accordingly, this study hopes to integrate the "representation of people within the whole process of creation, use and deposition of the artifacts," (Albero Santacreu, 2014, p. 125), and to incorporate the analytical results within the anthropological context.

Before discussing the theoretical approach that will be employed in this study, it is paramount to understand the criticisms to current approaches applied in archaeometric ceramic analysis. In archaeometry, many resources are devoted to the pursuit of scientific validation, which contributes sparsely to promoting a more in depth view of ancient societies (Albero Santacreu, 2014, p. 126). Therefore, it is important to understand if specific analytical techniques have the potential to answer archaeological questions or if they are merely a tool to for validation of existing knowledge. Additionally, it is vital to recognize that different theoretical approaches seek to answer different types of question. For example, the ceramic ecology approach hopes to:

> "…understand and regulate the dynamic relationships existing between the ceramics as a cultural product and the natural environment as a way to transcend the objects themselves. From an ecological point of view, the adaption of the pottery to the environment is the primary aspect in determining the physical features of the artifact. Both the environment and the physical dimension of the object condition the human behaviors developed to create the ceramics. (Albero Santacreu, 2014, p. 129)"

The ceramic ecology approach views the environment as a determining factor in the production of ceramics and ignores human agency and cultural contexts which influence the process of ceramic production at every step. In terms of this study, this approach would ignore the synchronic differences that could reflect social organization on a community level in south central Puerto Rico through the exploitation of similar or distinct resources (Albero Santacreu, 2014, p. 143). Furthermore, this approach tends to disregard the inherent inconsistencies which exists within prehistoric societies and seeks to create overarching laws dictating ceramic production. By not focusing solely the environmental restraints, this thesis hopes to integrate environmental variables in terms of cultural sensitivities.

In a different vein, the functionalist framework is based on the fact that "approximations to vessel function can be made through the characterization of the techniques and materials in pottery production" (Albero Santacreu, 2014, p. 147). It is based on the assumption that potters integrate technological requirements to meet certain behaviors, such as choosing coarse-textured pastes for cooking and fine-tempered pastes for serving (Albero Santacreu, 2014, pp. 147–148). This perspective led to the science-based materials approach that seeks to establish universal models to identify specific pottery functions. The main proponents of this approach advocate that materials science can discern the "mental processes related to a particular technology through enquiring the role of each material feature of the artifacts according to the specific functions" (Albero Santacreu, 2014, p. 149). To study aspects of ceramics that are either linked to culture or function, one can identify the properties which are not strictly functional, and therefore must be the product of a cultural choice. Although it is productive and valuable to see which pastes and ceramics were adequate or best suited for certain functions, ceramic technology can be linked intricately to many aspects of social life outside of functional requirements. These include, but are not limited to how production is organized, whether it is done on a household or industrial scale, the local technological traditions, and the possible interaction and contact with different cultural groups (Albero Santacreu, 2014). It is widely accepted that archaeological artifacts are not only the product of choosing the best material properties, but are encompassed and influenced by cultural choices which dictate the available possibilities. Specific to this assemblage, the lack of pottery specialization and the probable polyfunctionality of ceramics makes using this theoretical framework incomplete and misleading. It is probable that several non-functional parameters are determining the characteristics of the ceramics of this study and, therefore, limiting the study to only functional objectives would ignore many of the cultural phenomenon that can be observed through ceramic analysis. Nevertheless, certain concepts and approaches are employed in the present study such as a variation on functional analysis. It is used as a framework of comparison for the mineralogical and chemical groupings, and the possible association of preferential selection of raw material for certain functional roles in society.

To delve deeper into the meaning of ceramics, this thesis hopes to integrate both the social and the symbolic significance of pottery without ignoring their inherent materiality. The following section will discuss the theoretical framework employed for studying the ceramics from south central Puerto Rico. The integration of archaeological and anthropological concepts into the research design of materials science oriented projects is key to understanding the results and to answering the proposed archaeological questions. Due to the complexity of this period in pre-Columbian Puerto Rico, this thesis focuses on how ceramics reflect or reproduce the social and political context of the time. Behind ceramics as ubiquitous daily use objects, there is a cycle which spans from the procurement of raw materials to understanding which market the pottery will sell to and for what purpose. This process is traditionally termed the *chaîne* opératoire (Sillar and Tite, 2000), which spans all the technological and cultural choices made during the production process. By understanding the entirety of this process, or until the archaeological materials allow, we can be witness to the social aspects of pottery production.

To apply the *chaîne opératoire* to ceramic studies, Sillar and Tite (2000) developed the concept of "technological choices" (See Figure 3). This refers to how technologies are an interdependent product of cultural and functional decisions, therefore viewing technology as an outcome of society. The production of technologies is an example of the "active role that material culture plays in the construction and reproduction of cultural values" (Sillar and Tite, 2000, p. 1). The framework employed by this thesis will draw ultimately on these concepts and will try to understand how objects attain their agency, to understand the social relationship people have with the materials (Sillar, 2009).

Sillar and Tite's (2000) technological choices will be the main concept discussed in this thesis due to the combination of both functional and cultural contributions to the final interpretations. To understand variability and production technology, Sillar and Tite (2000) propose following Schiffer and Skibo's (1997) framework where

> "…variability and change in production technology are explained in terms of the constraints imposed by the performance characteristics required for each activity within the overall life cycle, or behavioral chain of an artefact (i.e. procurement of raw materials, manufacture, distribution, use, reuse, discard). The identification of these performance characteristics is achieved by considering how the overall situational context (i.e. environmental, technological, economic, social, political and ideological) impinges on the relevant activities (Sillar and Tite, 2000, pp. 4–5)."

The situational context can impinge on the ceramic activities in a direct or indirect way. Direct influences reflect the material factors such as environment, technological knowledge and the economic system (Sillar and Tite, 2000, p. 5). This mainly refers to technological choices within the *chaîne opératoire* such as raw material procurement, processing, forming, surface treatment, and firing. Therefore not only does this link raw material sources to the availability of clays, but also to the potter's perception of these clays as suitable materials (Sillar and Tite, 2000, p. 7). In contrast, indirect influences consists if the mode of pottery production, the craft specialization, the method of distribution and use of pottery (Sillar and Tite, 2000, p. 7). These factors are not reliant on the environment, but instead on the economic framework and the social organization of society (Sillar and Tite, 2000, p. 7). It is useful to distinguish between contexts of use and the intended or actual function of pottery. Contexts of use refer to the situations in which the ceramics were used, whether in ritual, domestic or funerary contexts (Sillar and Tite, 2000, p. 8). The intended use is for what purpose certain ceramics were used for. Within a domestic context, a certain ceramic has the same context of use, but could perform several different intended uses or functions i.e. cooking versus serving. By only relying on the archaeological context and knowing the context of use, it is incomplete to conclude about the actual use or function. Here is where materials science plays a key role. Manufacturing technology studies must be integrated into the ceramic analysis to have a complete vision of the technological choices made in the past. Notwithstanding, the context of use is also central for understanding the relationship between people and things. As with people, the agency of things is “socially embedded and constrained within wide ranging economic and social structures. One of the primary outcomes of our individual agency is to reproduce these structures” (Sillar, 2009, p. 367). Due to the influence that material culture has had on the development of human actions, consciously or unconsciously, it is important and necessary to study the past through objects.

In addition to technological choices, “technological styles” developed by Lechtman (1977) is a useful term to employ in the study of ceramics. Technological styles reflect “the conscious and unconscious elements that together influence the technological choices…technological style can reflect a cultural function by conveying information on, for example, social status and group identity” (Sillar and Tite, 2000, p. 8). Therefore, choices of temper, forming method, or surface treatment can perform as a social function. In other words, the physical of properties of objects can, and are related to socially significant aspects in the past.

The embedded nature of technological choices must be addressed not only in terms of the physical properties of ceramics but also in terms of the behavioral and cultural factors. This thesis hopes to approach ceramic variability in terms of which social processes can account for these differences, and how these can be identified through materials science techniques. The technological choices perspective will be the main theoretical framework employed in this thesis since it accounts for differences in the material properties of ceramics in terms of the perceptions and intentions of the potters and the social and political climate in which they work in (Sillar and Tite, 2000, p. 11). In addition, it will be paramount to consider how the choices were made, how they were maintained and developed, and what they reflect in the cultural, social and political atmosphere (Sillar and Tite, 2000, p. 11). By understanding how these objects are communicating meaning in the past, we can return agency to objects and partially reconstruct past societies and their socio-political environments.

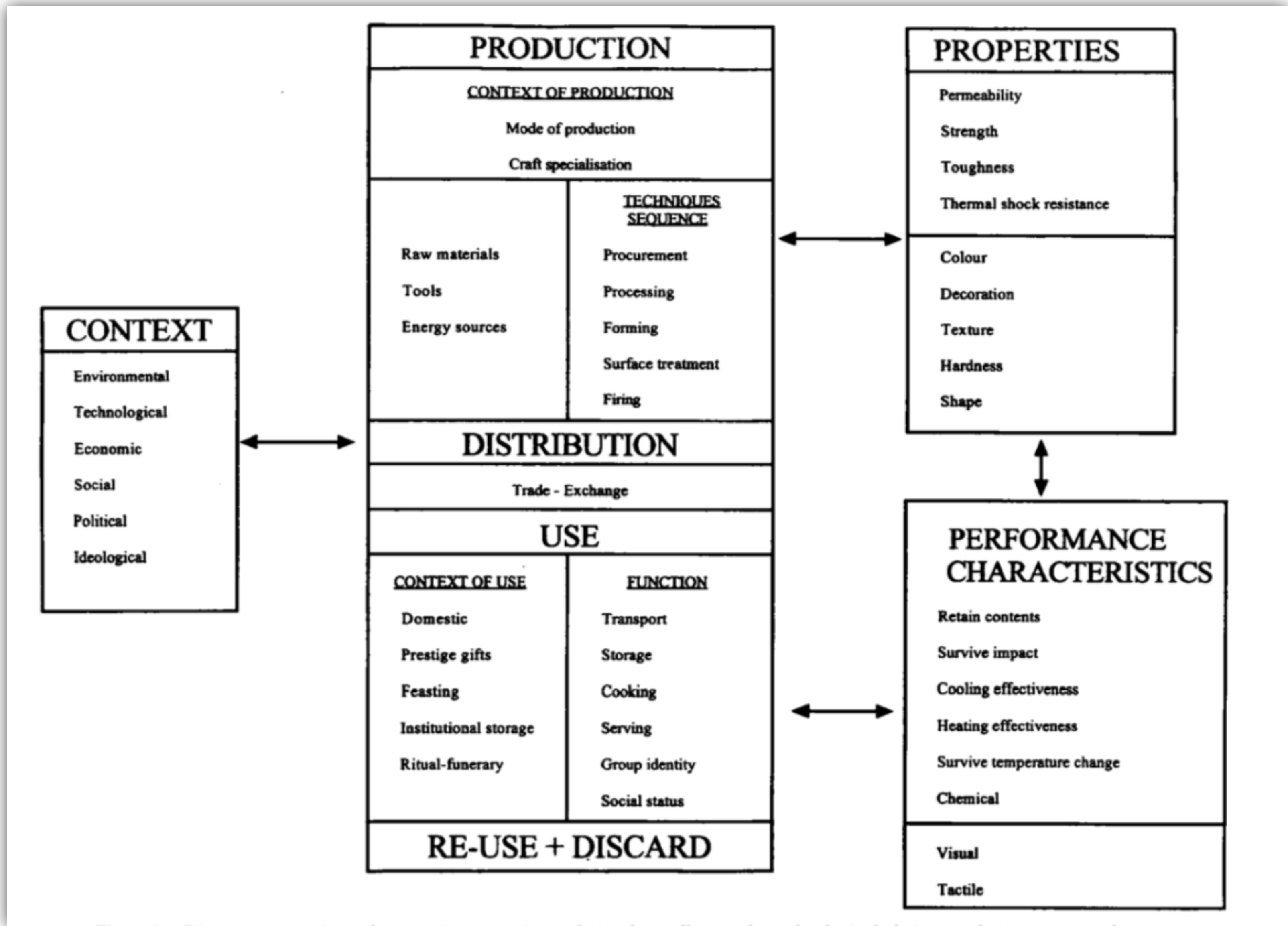

Figure 3 Graphic from Sillar and Tite (2000) explaining the concept of 'technological choices.'

3 The Early Ostionoid Period in Puerto Rico:
The Emergence of *Cazicazgos* (900-1200 AD)

Since antiquity, Puerto Rico has been in a key socio-political geographic location, being the bridge between the Greater and Lesser Antilles. Due to its intermediate position, Puerto Rico's eastern and western sides have been defined as two distinct cultural spheres. The western side influenced by the Greater Antilles and the eastern side by the Lesser Antilles (Curet et al., 2004, p. 60).[2] This thesis focuses on sites which lie within the transition zone of eastern and western Puerto Rico, and which display cultural traits from both areas in a period of grand cultural and political adaptations. After the first migration of peoples, termed the Saladoid series, a new local culture developed and emerged during the early Ostionoid series. Post-Saladoid culture is the main focus of this thesis, mainly the Ostionoid series or Period III in Irving Rouse's chronology (See Figure 4). This period is characterized by important socio-cultural and political shifts reflected in the archaeological record which will be discussed in detail below. It is paramount to understand the importance of the island of Puerto Rico, the significance of the site of Tibes and the implications of Period III to appreciate the driving forces for this study.

Although most archaeologists agree that during Period III there are significant socio-cultural changes reflected in the material culture, ritual architecture, burial practices and use of space in the post-Saladoid period, there is no consensus on how these changes affected socio-political organization on the island and at what scale. Curet (1992, 1996) argues that changes in the material culture and house structure during the post-Saladoid period is evidence for the emergence of chiefdoms on the island. Gradual changes in material culture is used as evidence of the rise of social complexity (Curet, 1996). Period III's shift in ceramic styles and lapidary work, the development of ball courts and plazas, changes in household size, expansion of religious rituals, modifications in mortuary practices, the intensification of agricultural production and different settlement patterns are the most significant changes occurring (Curet, 1996). Specifically,

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[2] This definition of cultural spheres has mainly been done by stylistic ceramic analysis done by Irving Rouse in 1953, and has not been challenged in the literature since.

these changes in material culture are related to "the mechanisms by which elite groups manipulated religious ideology to acquire and maintain power, authority, and prestige over the rest of the population (Curet, 1996, p. 117)." This thesis focuses on the ceramics which are witnesses to these socio-cultural changes.

Considering the sociocultural dimensions of ceramics, including symbolism and ideology, this thesis aspires to not only record the transitions, but to approach material culture as a proxy for socio-cultural change. Therefore, the evident decrease in diversity, quantity, and quality of decorative techniques and designs which takes place between the Saladoid (300 BC- AD 600) and early Ostionoid periods (AD 600-1200) in eastern Puerto Rico could reflect significant socio-

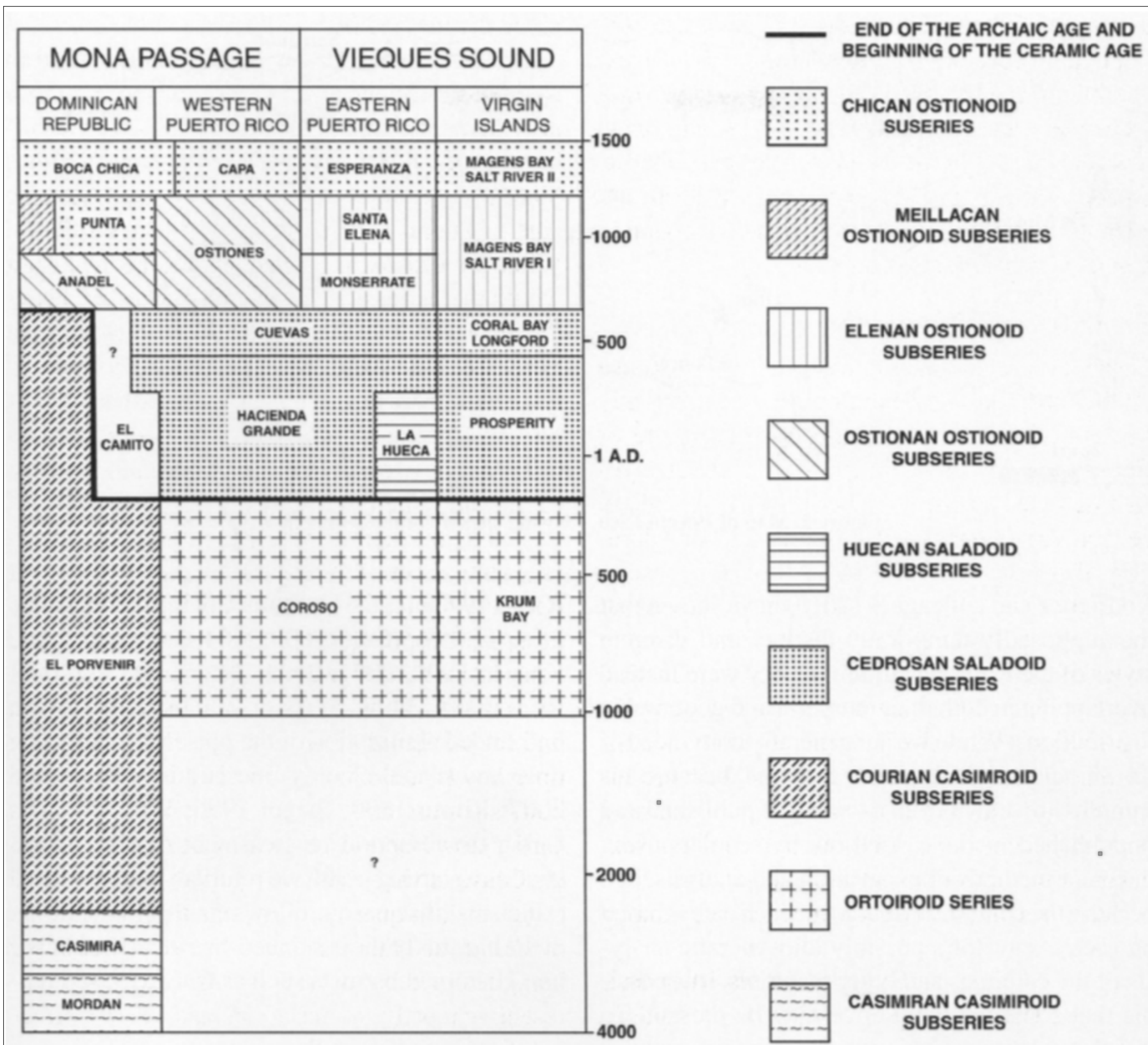

Figure 4 Revised cultural chronology of the Caribbean based on Rouse's culture history chart for Puerto Rico (Pestle et al. 2013).

cultural changes in social processes. In essence, "ceramic changes reflect modifications in the ideological structure of the social groups (Curet, 1996, p. 114)," which in this context is argued to be the development of complex societies or chiefdoms, which are commonly termed *cazicazgos* in the Caribbean.

The previous ceramics on the island from the Saladoid series (300 BC – AD 600) are characterized as high quality ceramics, which are hard, relatively thin, well-fired and of fine paste (Curet, 1996). Decoration mainly consists of white-on-red, white-on-orange and red-on-buff paint, cross-hatched incision, engraving, excision, and modeled zoomorphic and anthropomorphic *adornos*. The Saladoid series includes the Hacienda Grande and Cuevas styles. In the transition to the Elenan Ostionoid subseries in eastern Puerto there is a decrease in decoration but a maintenance of familiar forms and technology in the Monserrate style (AD 600-900). Nevertheless, during the subsequent Santa Elena style (AD 900-1200) paint largely disappears and diagnostic decoration is limited to crude incisions close to the rims of bowls. Notably, ceramics became softer, thicker, coarser and rougher. In this study, there were mostly no diagnostic decorations or identifiable vessel forms, but the ceramics were all attributed to the Santa Elena style or the Modified Ostiones style based on radio carbon dating, other materials found in the same archaeological context, and preliminary modal analysis (Curet and Torres, personal communication). This transition from higher quality to lesser quality ceramics reflects important social processes occurring on the island.

Artifact form and style witness a transition alongside the transformation of household architecture. Although evidence is scarce due to the subtropical and tropical environment on the island, research indicates a transition from large extended family households to a concentration of nuclear families. Change in household size is important for anthropology because it can be a representation of social and economic changes. Households are considered one of the most basic units of analysis for studying people, families and communities. Although house structures are not very well preserved in Puerto Rico due to the environment and perishable materials used for the construction of *bohíos*, the traditional houses in the Caribbean, sparse data has been gathered from the excavations that were able to identify post molds and wall marks at the sites of Maisabel in the north, Playa Blanca 5 in the east and El Bronce in the south (Luis Antonio Curet, 1992). Contrary to the information available in the chronicles, houses during Period III appear to be only for nuclear families instead of the larger house structures which sustained extended households (Luis Antonio

Curet, 1992). This contributes to the notion that socio-cultural and political changes are occurring during this transition period and that they are felt in every aspect of society.

The use of chronicles in archaeological interpretation in Puerto Rico has been highly contested in the literature. Due to the uncritical methodology employed, the incorporation within archaeology has resulted in biased interpretations (Curet, 2006). Traditionally, the Spanish chronicles have been preferred by scholars from several fields to explain socio-cultural processes of indigenous populations instead of looking towards the archaeological record. Consequently, data from archaeological record has been known to be adjusted to fit the assertions made in the chronicles. Therefore, inserting the subjectivity and prejudice which exists in the chronicles within the archaeological record (Curet, 2006, pp. 181–182). This misinterpretation of the archaeological record is evidenced in household structures, since the chronicles only refer to elite housing and fail to describe the housing of the majority of the population.

The transition to nuclear family units is theorized to be an action of power by the elite to dismantle larger community ties (Curet and Oliver, 1998). The change in mortuary practices in the post-Saladoid Caribbean demonstrates another avenue of evidence to suggest the rise of chiefdoms on the island, and the disappearance of larger communal institutions. Curet and Oliver (1998) argue that the change from central burial grounds to domestic graves is evidence of the rise of chiefdoms. Demonstrating a shift in political control, the transition to domestic graves is an indication of the dismantling of communal traditions and institutions. The formalization of social inequality can be seen in the archaeological record in a variety of ways. Although coercive force is a quick way of grasping power, due to the lack of evidence of violence in the osteological record, it appears that subtler methods were employed in the Caribbean (Curet and Oliver, 1998). For instance, legitimizing ideologies which already exist through the control of religious structures such as ball court plazas and symbols such as stone representations of deities known as *cemíes* (Curet and Oliver, 1998). The appropriation of the ancestor cult by the elite demonstrates another aspect which contributes towards understanding the rise of social complexity and chiefdoms in the Caribbean.

Siegel (1992, 1996, 1999) argues that the formalization of ritual spaces such as ball courts and plazas suggests institutionalization of religious ideology. In turn, this ratification of ideology was used to acquire and maintain power and to develop the institution of the *cacicazgo*. His argument focuses on an increase in frequency and size of ball courts and plazas, which appear during Period III. As shaman's accrued power during the Saladoid to Ostionoid transition, the centralization and accumulation of power is represented by the construction of large ritual spaces. By the end of Period IV, there are approximately three to four centers of power known on the island, which Siegel terms "sites of contest." In contrast to Curet and Oliver (1998), Siegel approaches the rise of social complexity on a regional scale, arguing for the consolidation of power throughout the entire island into a few hubs of influence. Although there appears to be a consolidation of power through the institutionalization of religious ideology, which has been evidenced during the Saladoid/Ostionoid transition, it is the regional variances which will shed light on the intricacies of socio-cultural processes happening across the island.

Evidently, significant changes in the socio-political organization and society are occurring in the post-Saladoid Caribbean. However, many criticisms to models such as Siegel's exist due to their unit of analysis. In the three previously mentioned cases, the whole island of Puerto Rico or the eastern part are used as a unit of analysis. Curet argues that this is a homogenizing processes which shrouds the inherent variability which exists within this archaeological record (Curet et al., 2004). In this thesis, it will be impossible to study the material on a local unit level, but it will be studied at a site level conscious of the homogenizing effect this may have on the data acquired. By studying the manufacturing technology and raw material variability of the ceramics, this thesis hopes to contribute to ongoing investigations which are grappling with this key transition period in Caribbean archaeology.

These major changes in socio-political makeup during the Saladoid to Ostionoid periods is the basis of the archaeological context encountered at Tibes and the three sites in the region. The apparent cultural impoverishment of the ceramics from Period III will be investigated and the results will be discussed within the economic, political and religious context. The simplification of ceramic decoration has been previously explained by the emergence of a local ceramic tradition, a Saladoid adaptation to the environment, or a change in subsistence systems which might have produced a decrease in resource availability (Curet, 1996). Although this question has not been fully answered, and might never be, it is important to keep in mind that there were multiple factors that contributed to this clear socio-cultural transition into the Ostionoid period. Curet (1996) points out that these suggestions do not explain the apparent religious complexity which appears in the Ostionoid period. Instead, he postulates that the decrease in decoration is due to "the increased control by the emerging elite groups over symbols and ideology in general (Curet, 1996, p. 121)."

Therefore, by studying these ceramics in detail, we aim to understand if only aesthetic aspects of production have changed or if technology and manufacturing technology was altered during this key Saladoid to Ostionoid transition period in Puerto Rico.

Although this thesis will not be strictly framed on the culture historical chronology developed by Rouse, it will be discussed below due to its ubiquity in Caribbean archaeology.[3] Rouse uses a hierarchical classificatory system, which is divided between series, subseries and styles. The series is a combination of styles, which are sometimes subdivided into subseries. Styles are the lowest division of Rouse's model and consists of pottery assemblages which have the same diagnostic features within the same period and geographic location. Many scholars have criticized Rouse's chronology (Curet et al., 2004; Pestle et al., 2013; Torres, 2012) since it has not survived the test of time due to the changing dates, overlap between periods, and more than one subseries and series living in the same space and time. Another major criticisms towards using Rouse's chronology is the homogenizing effect that occurs (Curet et al., 2004). It tends to hide the possible synchronic variability which can exist in one geographic location. Notwithstanding, the sequence and terminology is still employed in Caribbean archaeology. Since this thesis focuses on synchronic variability within a regional context, it is essential to be conscious that within the series and subseries studied, there can be cultural variability and different social processes can be affecting the material culture being studied. This thesis focuses on Rouse's early Ostionoid Series (AD 600-1200) or Period III. This series previously was classified with two styles, but further research has shown more regional variability. Currently, the common division is between the Elenoid series in eastern Puerto Rico and the Ostionoid series in the western side of the island. This thesis focuses on the Saladoid to Ostionoid transition, which is also known as Period III, and which is the stage where social complexity and chiefdoms begin to develop on the island.

The site of Tibes lies within the eastern and western Puerto Rico culture history transition zone and has many mixed assemblages which include both eastern and western Puerto Rico assemblages. The main differences between the eastern Elenan Ostionoid Series and the western Ostionan Ostionoid series is based on the ceramic styles and on the geographic locations where

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[3] Rouse's chronology is not the only one employed in the Caribbean, although it is the most widespread. Chanlatte Baik calls Period III as Agro III, a group which evolved from the Saladodid and La Hueca Complex[3] groups and the Archaic groups which first inhabited the island

these styles are first seen and where they spread. The Elenan Ostionoid series is first identified by the Monserrate style named after the site of Luquillo Beach in northeastern Puerto Rico and is also associated with earlier Cuevas style pottery (Torres, 2012). The later Santa Elena style from the Elenan Ostionoid subseries was named after the type site of Santa Elena (TB-7) in the municipality of Toa Baja and has been identified across the eastern two-thirds of the island, including at the site of Tibes. The Ostionan Ostionoid style was identified by Irving Rouse from the type site in Cabo Rojo in western Puerto Rico in AD 600 and is believed to have evolved from the earlier Cuevas style pottery, and to have influence from sites in present-day Dominican Republic (Torres, 2012, p. 63)[4]. Although Tibes lies in the eastern Puerto Rico culture sphere, both pottery styles are found at the site and in the south central region in general, proving the continued interaction between east and west occurring on the island during Period III.

3.1 The Ceremonial Center of Tibes

The Tibes site in south central Puerto Rico shares both the Ostionan Ostionoid series and the Elenan Ostionoid series because of its strategic position between the western and eastern spheres of influence. Located just 8 km from the coast, Tibes is situated in a biogeographic and geological transitional zone, as well as a cultural "gray zone" (Curet and Stringer, 2010). Its importance in Caribbean archaeology began in the late twentieth century after the site was re-discovered by a local farmer. Layers of silt were uncovered by a hurricane in the 1970s, and the monumental architecture was quickly recognized. This architecture differentiates Tibes from other sites in the region. The site began to be formally investigated by a local avocational organization.[5] The site began to be formally studied by the avocational organization of La Sociedad Guaynía and was quickly established as one of the iconic ceremonial sites in the Caribbean. Dominated by 10 *plazas*, *bateys* and causeways throughout the site.

[4] Other sites were also used to identify and solidify the identification of the Pure Ostiones style by Rouse including Boquerón, Las Cucharas, Llanos Tuna, Buenos Aires, Cañas, Carmen, and Diego Hernández (Rouse, 1952 as cited in Torres, 2012).
[5] For an in depth discussion on whether the architecture at Tibes should be considered monumental see Torres, 2008.

Due to its importance in Caribbean archaeology, this site has been studied semi-continuously since it was unearthed. The Archaeological Project of the Ceremonial Center of Tibes, run by L. Antonio Curet, beginning in 1995, integrated a multi-disciplinary approach. Although much focus has been given on the individual studies done at the site, the main goal of this project was to create an interdisciplinary approach to help understand how social complexity and chiefdoms developed at Tibes. By integrating data from the artifacts, fauna, botany and spatial organization, archaeologists are one step closer to understanding how and why chiefdoms developed as they did. Most of the ceramics used for this study have been unearthed from controlled excavations of archaeological contexts at Tibes, and for this reason the history of excavation, the methods employed, and overall interpretations from the Tibes project are discussed below.

The site of Tibes has nine restored architectural features which include ceremonial plazas, *bateys* or ball courts, and *calzadas* or causeways (Curet and Stringer, 2010, p. 20). Although there has been a distinction between ball courts and plazas in terms of functional purposes defined by their shape, many different activities might have taken place within these ceremonial or communal spaces (Curet and Stringer, 2010). Relative to this thesis, ball court 8 named "Batey del Murciélago" (bat ball court) and ball court 2 named "Batey de Herradura" (horseshoe ball court) are of most significance since two of the ceramic context are situated in association with these features. These contexts will be discussed in further detail in Chapter 5 in the materials section.

The site of Tibes is located in south-central Puerto Rico on an alluvial terrace of the Portugués River. The site is encircled by physical barriers. On the north and east the site is surrounded by hills and on the west and south by the river. The site is in a geological transition zone because it is situated between the Dry Southern Coastal Lowlands and the Semiarid Southern Foothills or piedmont of the Central Mountain (Curet and Stringer, 2010, p. 10). The Southern Coastal Lowlands are a dry, fertile alluvial plane which suffers from severe droughts between December and April (Curet and Stringer, 2010). This is one of the most arid areas of the island and is characterized by deciduous vegetation of thorny shrubs and trees smaller than 15 m (Curet and Stringer, 2010). In contrast to the arid lowlands, the Central Mountains to the north of Tibes have abundant rainfall and very dense and varied vegetation. Tibes is located between the two abovementioned ecotonal zones in the Semiarid Southern Foothills. This area is considered to be an intermediate area since it receives relatively more rainfall than the Southern Coastal Lowlands.

It is characterized by lush vegetation of grasses, scrub bushes and cacti, and trees up to 20 m tall (Curet and Stringer, 2010). Tibes lies between different climates, geologies and topographies. Other ceremonial sites such as Caguana in the north, also are in a high-diversity ecotonal transition zones (Curet and Stringer, 2010). Curet and Stringer (2010) postulate that these transition zones might have been preferred by indigenous peoples because of the access to resources and/or the symbolically charged spaces in relation to the cosmos (Curet and Stringer, 2010, p. 12). In addition, these transition zones become more populated during the Ostionoid period.

The site of Tibes was discovered in 1975 by a Mr. Luis Hernández, who manufactured charcoal at the site, after Hurricane Eloísa caused the Portugués River to flood and eroded the layers of silt deposited on top of the main architectural features of the site (Alvarado Zayas and Curet, 2010, p. 19). The avocational organization Sociedad Arqueológica del sur-oeste de Puerto Rico (SASOPR), upon Mr. Hernández's tip, visited the area of the site and recognized the archaeological deposits and stone architecture, and contacted another avocational group called the Sociedad Guaynía de Arqueología e Historia de Ponce. Members of this group, Juan González Colón and Pedro Alvarado Zayas, worked on the site for 7 years. They focused on locating, clearing and restoring the architecture of the site and determining the main time periods of occupation. The Sociedad Guaynía also were behind the efforts to create the archaeological park which opened in April 30, 1984 and is visited by over 50,000 every year (Alvarado Zayas and Curet, 2010, p. 20).

Pedro Alvarado Zayas was the first field director under the engineer Juan González who was in charge of the Sociedad Guaynía de Arqueología e Historia to study Tibes through scientific excavation. One hundred and twenty 2 x 2 m units were excavated, with a majority being culturally sterile. In the areas where cultural material was found, sparse ceramics, lithics and food remnants were encountered using artificial excavation layers of 10 cm. Alvarado Zayas deduced from his excavations and ceramic analysis that there were two main phases of occupation: Saladoid or *Igneri* and Ostionoid *or Sub-Taína*. The Saladoid occupation was mainly characterized by the Cuevas style and the later by the Ostionoid series, which could not be discerned to the stylistic level. Alvarado Zayas describes the Ostionoid ceramics at the site as poorly fired fragments with thick walls, incision decoration of lines and dots, some geometric figures done with the "applique" method, zoomorphic adornments, and inward beveled rims (Alvarado Zayas, 1981, pp. 179–180). These fragments were found in association with a rise in agriculture and recollection of marine resources.[6] Most interestingly, Alvarado Zayas argues that the Cuevas style developed slowly into the Ostionoid ceramic style through seriation analysis, but that it did not develop locally at Tibes because the site was not occupied year-round (Alvarado Zayas, 1981, p. 182). His argument is based on the small amount of archaeological materials expected for a site of this size. Few ceramics, lithics and food remnants are found relative to other habitational sites in the south-central region. Therefore, he believes that Tibes was not continuously occupied during the year, but used only for ritual activities. Due to his vacant site theory, he believes that the Saladoid/Ostionoid transition does not occur locally at Tibes, but elsewhere in the region. His main evidence stems from a 5 cm layer of culturally sterile soil between the Saladoid and Ostionoid deposits at the site, which represent a temporary lack of occupation at the site.

L. Antonio Curet continues to add to the resolution of the cultural chronology at Tibes during the subsequent archaeological project. Based on the excavations of the Tibes Archaeological Project, the earliest pottery at Tibes stems from the Hacienda Grande style (300 BC – AD 400), but the assemblage is dominated by the Cuevas style (AD 400 - 600) pottery of the Cedrosan Saladoid subseries (Curet, 2010). Next, the ceramic assemblages are characterized by examples from the Monserrate and Santa Elena style from the Elenan Ostionoid series, which coincide with the radio carbon dates and Rouse's proposed dates (Curet, 2010). In addition to the Elenan Ostionoid series, there is Ostionan Ostionoid ceramics which originate from western Puerto Rico throughout the assemblages. Although Tibes falls within the eastern Puerto Rico cultural sphere, it is common during the Ostionoid series to see an intermingling of the two different cultural areas. As Curet (2010) points out, little attention has been paid to this cultural and social phenomenon. Tibes shows little evidence of occupation during the Chican Ostionoid subseries (AD 1200-1500) since no clear deposits dominated by this style has been identified. Therefore, it appears that Tibes's height of spatial expansion and ritual use was during the Ostionoid period since most of the architectural structures and ceramic assemblages stem from this time.

To understand the site in depth, the pilot phase of the project attempted to discover the boundaries of the site and discover the nature of the archaeological deposits (domestic, ritual,

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[6] The conclusion that agriculture emerged more intensely was mostly the product of speculation (Curet, personal communication).

production site, etc.), understand the community pattern at the site, and begin reconstructing the ancient economic strategies (Curet, 2010, p. 42). Each unit and level were assigned a cultural affiliation, when possible, through the analysis of artifact assemblages (Curet and Stringer, 2010, p. 42). Since most of the ceramics found at the site were fragmented and not decorated, Curet uses a variety of modal analysis adapted from his doctoral dissertation (L. Antonio Curet, 1992) to determine the stylistic affiliation of the ceramic assemblages.

3.2 Tibes Archaeological Survey Project (TASP)

Twenty of the ceramics and sediment samples of this thesis are the product of the Tibes Archaeological Survey Project (TASP) conducted by Joshua M. Torres in 2008. The project consisted in archaeological surface and subsurface survey of 20 km^2 along the Cañas, Chiquito, and Portgués river drainages (Torres, 2012). The main goal was to identify residential settlements in south central Puerto Rico which are associated to the ceremonial site of Tibes, and to understand the community structure in the region in detail, to explain the development of complex societies in the region (Torres, 2012). The main impetus for locating the survey around the ceremonial site of Tibes was to understand if such a large center like Tibes was serving an extremely large population, which would presumably live in the area and be seen in the archaeological record as settlements in the region (Torres, 2012, p. 146). The studies main question is

> "…where are the residential settlements composing the supporting community? Is there more to the local settlement structure than Tibes and PO-29[7]? If so, what does the timing and distribution of these settlements tell us about the composition and organization of the social community associated with Tibes and how do these patterns translate to other contemporaneous localities in the south-central region? "(Torres, 2012, p. 146).

This thesis focuses on three sites, which form part of the supporting communities around Tibes.

[7] PO-29, or Jácana site, has a midden mound and a posible batey.

PO-42 (La Mineral) is located on the eastern side of the Portugués River. The site is oriented northwest to southwest along the river's edge and its extension is approximately 300 x 100 m (Torres, 2012, p. 175) (See Annex 1 for a map of PO-42). There is a small *batey* feature (6 x 15 m), which is composed of two stone rows of local andesitic river cobbles. Eighty shovel tests were excavated and 36 yielded cultural material. PO-43 (Los Gongolones) is on the east bank of the Portugués River and is 400 meters north of PO-42 (Torres, 2012, p. 179). The site consists of a discrete midden deposits surrounding a cleared area, which could potentially be a *batey* feature (Torres, 2012, p. 179) (See annex 2 for a map of PO-43). The feature is surrounded by two disarticulated rows of boulders 25 m apart running from east to west. Sixty-two shovel tests were excavated and 35 tested positive for cultural material. Both sites display materials stemming from Period III and IV and appear to have been part of a community that is characterized by permanent small residential settlement which have specialized activity areas. Conservatively, each of these sites probably held approximately 10 households (Torres, 2012, p. 191). The initial interpretation of the sites based on the size, quantity and diversity of the materials recovered is that they are permanent, long-term small residential settlements, which hosted limited activities (Torres, 2012, pp. 190–191). Exceptionally, PO-42 and PO-43 have evidence of ritual structures, which complicated the interpretation of the ritual landscape in the Portugués river drainage (Torres, 2012, p. 191). This settlement pattern is consistent with the residential settlements found in the north around the ceremonial center of Caguana. These settlement patterns contrast with the previous Saladoid settlements, which were considerably larger and closer to the coast.

In contrast, the site PO-48 (Escuela Río Chiquito) in the Chiquito river drainage shows little evidence for permanent occupation. It is located on the western side of the river and it spans approximately 40 x 60 m (Torres, 2012, p. 184) (See Annex 3 for a map of PO-48). Twelve shovel tests were excavated and seven generated sparse cultural materials. The site was primarily occupied between Period III and Period IV (AD 900-1300) (Torres, 2012, p. 230). Since it shows a lack of marine remnants and waste middens, it appears it was not a permanent residential settlement. Torres (2012) explores questions relating to the settlement patterns, and concludes that these small residential settlements in the foothills and mountainous regions of the area are associated to the Ostionoid period and do not coincide with previous assumptions that this region would be densely occupied to serve such a large-scale ceremonial center such as Tibes.

3.3 Archaeological Science Context

The Caribbean has been relatively underrepresented in chemical characterization and manufacturing technology studies of ceramics (Descantes et al., 2008). In the past twenty years, the main method employed has been instrumental neutron activation analysis (INAA). A large scale project was launched at the University of Missouri Research Reactor Center (MURR) in 2003, which centered on characterizing a wide array of Caribbean ceramics (Descantes et al., 2008). Due to the variability, lack of standardization and the reliance on age-old stylistic analysis, archaeometric ceramic analysis has been relatively absent as a tool for archaeologists in the Caribbean. By being aware of the previous archaoemetric studies done in the region, although sparse, this thesis will be able to build upon the available knowledge and interpretations in the literature to create a more complete assessment of the most suitable techniques to address the archaeological questions.

Although petrographic analysis is not a novel technique and has been used since the early twentieth century, it has not been employed in Caribbean archaeology with much frequency albeit some notable exceptions (Carlson, 2008; Carlson and Torres, 2011; Ting et al., 2016). Ann Cordell conducted detailed petrographic analysis on ceramics from the sites AR-38 and AR-39 in Arecibo to understand the paste variability within sites as part of a larger-scale study in the Tanamá river drainage (Carlson, 2008, p. 259). Cordell identified eight main paste types which can be seen in Table 1, and concluded that the sites tend to share similar pastes and raw material sources. Although some of the pastes were associated to certain styles, there is no definitive association between specific clay sources and specific pastes and there is no discussion of provenance based on local geology. In my opinion, the lack of integration of chemical data within the paste identifications results in the over division of categories, which perhaps are not archaeologically meaningful. Certain pastes can be part of the same raw material source, even though they show slight variation in temper size or slight variations in aplastic inclusion composition. These categories are compared with the categories created during this investigation in the discussion section in Chapter 7. In this way, we can see whether these categories, which were are also applied and adapted by Torres (2012) on his ceramic assemblage, is productive to understand regional ceramic variability in south central Puerto Rico.

Ting et al. (2016) focused their study on pre-contact Chicoid and Meillacoid styles found in Dominican Republic from the late 15th century. In their study, the samples had a defined style and stemmed from two sites in northwestern Hispaniola (Ting et al., 2016, p. 377). Similar to this thesis, their main goal was to

> "explore the compositional, and to some extent the technological, variability that exists within and between assemblages to characterize craft organization, and more importantly, to understand the social and economic contexts under which these ceramics were manufactured (Ting et al., 2016, p. 377)."

Through the petrographic analysis of 32 samples, the authors were able to conclude that a wide variety of raw materials were being used in the production of ceramics at both sites, and that these raw materials were probably local (Ting et al., 2016). This conclusion was reached because there were significant overlaps in the petrofabric groups from both sites studied. Although they believe that similar raw materials were used based on the mineralogy, it is necessary for the authors to conduct chemical analysis to confirm or deny this assumption (Ting et al., 2016, p. 384). Additionally, the authors concluded that there was a low level of standardization regarding choice of raw material and paste preparation method (Ting et al., 2016). One of the main lines of evidence supporting this hypothesis is the variable redox environments evidenced in the sherds. Most interestingly, the authors concluded that ancient potters from both sites shared the same knowledge regarding raw material sources and ceramic production, and due to the overlapping petrofabric groups there was probably exchange between the two sites (Ting et al., 2016, p. 384). Besides discussing the exchange between the sites, Ting et al. (2016) delve into the significance of having the different petrofrabrics define pottery from the same style. Although no final conclusions are made, they propose that if potters are using materials from the same petrofabric group to produce ceramics of different styles in different stratigraphic layers, then there would be a continuation of technical practices such as raw material procurement (Ting et al., 2016, p. 384). In contrast, if ceramics of different styles co-exist within the same stratigraphic layer, then the use of the same petrofabric could suggest a lack of specialization in the types of pottery being produced (Ting et al., 2016, pp. 384–385). Due to sampling and sample size, these hypotheses are not able to be tested in this thesis. Nevertheless, the interpretation is an example of how to integrate the scientific data within the archaeological context to further understand ancient Caribbean society.

Although Ting et al. (2016) discuss briefly aspects of technology observed in the thin section analysis, the petrographic microscope is not very well suited to answer the more functionally inclined questions. Curet (1997) performed an exploratory study to understand technology in a large sample of Ceramic Age ceramics, which had been previously studied petrographically from the Maunabo region in south eastern Puerto Rico. The petrography suggested that local materials were used for tempers, and they were mainly crushed rocks instead of beach or river sands (Curet, 1997, p. 500). This study focused on understanding firing temperature, porosity and density. Although the same methods are not employed in the thesis, the same conclusions hope to be made based on other analytical methods. Curet's main preliminary conclusions are that Saladoid ceramics are more dense and less porous than later Ostionoid and Chicoid Ositonoid ceramics, even though they are harder and have a finer paste (Curet, 1997).

There appears to be a constant firing temperature associated to the entirety of the Ceramic Age Caribbean in Puerto Rico, which within itself has high variability (Curet, 1997). The main differences between the Saladoid and Ostionoid period is the treatment of the paste, instead of the raw material sources or firing technique (Curet, 1997). This conclusion has implications for functional interpretation. Therefore, Ting et al. (2016) and Curet (1997) agree on the continuation of similar technical practices, basing themselves on the procurement of the same raw materials during the Saladoid and Ostionoid periods. Although this theory is plausible, more diachronic studies with samples that have finer chronological resolution must be analyzed to confirm these conclusions.

Chemical characterization via INAA and XRF is another popular study in Caribbean archaeology (Conrad et al., 2008; Crock et al., 2008; Descantes et al., 2008; Isendoorn et al., 2008; Siegel et al., 2008). Siegel (2008) conducted a diachronic study of ceramics from the Maisabel site and HU-7 in northern Puerto Rico to understand compositional variability through time (Siegel et al., 2008). Siegel chose samples which could be definitively assigned to a style, therefore focusing on decorated sherds. These sherds do not represent the compositional variability of coarse ware in the region. The ceramics were studied to understand the control of resources during the institutionalization of inequality in the Ostionoid period. He attributed the "devolution" of ceramics to the "concomitant changes in social organization: egalitarian to institutionalized inequality" (Siegel et al., 2008, p. 28). During this period, the representation of power and prestige changes the material culture. Although Siegel (2008) expects there to be distinctive compositional signatures between periods and clear trade patterns, his samples and the Caribbean as a whole do not appear to follow these patterns. He concludes that compositional differences are not reliant on cultural complexes. Instead geochemical makeup is dependent on site location (Siegel et al., 2008, p. 36). Therefore, although there are apparent socio-cultural changes, indigenous populations were using local materials for pottery production. The lack of interaction between sites could be attributed to the continuity of village autonomy (Siegel et al., 2008, p. 38). There is evidence in northern Puerto Rico to support the hypothesis that socio-cultural change in Puerto Rico did not occur through coercive force, but through the appropriation of religious symbols as proposed by (Curet and Oliver, 1998). Siegel's study acknowledges the limitations of the study. Chiefly, there is a lack of comparison with possible clay sources, the sample is not functionally diverse and they stem from a small number of sites in the region.

The main drawbacks of the previous archaeometric work in the Caribbean is the absence of a multi-analytical approach. Focusing on one analytical technique, the depth and certainty of interpretations is absent in the scientific results, and therefore in the archaeological interpretations. In addition, the sampling strategy has not been geared toward understanding the dynamics within one time period. For this reason, the authors hesitate to make any overarching conclusions based on synchronic variability, and cannot distinguish what is due to synchronic or diachronic variability. This study hopes to elucidate the ceramic variability in the region surrounding the ceremonial center of Tibes, as well as to discern the clay sources pottery production areas used during the early Ostionoid period.

Table 1 Summary of Cordell's petrographic groups.

Fabrics/Pastes	Description	Temper/Matrix ratio	Pottery Style
Coarse Felsic	Granitic rock fragments and felsic constituents	64% matrix 32% aplastics 6% voids	
Medium Felsic	Granitic rock fragments and felsic constituents, quartzite, plagioclase.	62% matrix 38% apalastics 7% voids	
Fine Felsic	Quartz, UID feldspar grains, plagioclase, biotite, mica, mafic amphibole grains.	75% matrix 25% aplastics 4% voids	Cuevas Style
Mixed Felsic	Fine felsic and coarse felsic pastes, two clay sources mixed.	N/A	
Quartz	Fine through coarse quartz particles and other felsic constituents such as UID feldspar grains and quartzite. Volcanic rock fragments occur occasionally.	57% matrix 43% aplastics 8% voids	
Volcanic	Fine to coarse volcanic rock fragments. Plagioclase, mafic pyroxene, biotite, IUF feldspar and occasional grog.	64% matrix 36% aplastics 6% voids	Cuevas Style Boca Chica Style
Limestone	Limestone tempered, matrix is carbonate composition, inclusions are micritized. Coarse volcanic rocks and feldspar grains.	72% matrix 28% aplastics 4% voids	
Vitrified	Frothy, partially vitrified. Volcanic rock fragments and crystalline felsic grains.	76% matrix 24% aplastics 39% voids	

4 Methodological Considerations:
Archaeological Questions and Materials Science Techniques

Similar to understanding the theoretical framework employed, it is vital to comprehend the extension and limitations of the scientific techniques used, and to gear them towards archaeological investigations. Understanding the extent and confidence we can attribute to the analytical results is key to producing accurate and meaningful scientific investigations. This section is an overview of the methodological considerations of the analytical techniques applied to archaeology. Ceramics are approached from a materials science perspective to understand the correlations between mineralogical and chemical compositions with the local geology and the relationship to manufacturing technology. Instead of discussing what information the scientific approaches and methods can provide, the methodological techniques used in this thesis are considered in terms of what they can contribute to studying past societies. The main topics discussed in terms of ceramic variability are provenance studies, functional analysis, and manufacturing technology. Deciphering the relationship between raw materials sources and ceramic production relies on the analytical techniques that can provide mineralogical information, which include petrographic analysis and XRD, and the ones that can afford chemical composition, XRF, ICP-MS and SEM-EDS. The benefits and limitations of each technique is discussed, as well as the justification for using a multi-analytical or "integrated" approach. Incorporating the analytical methods within the research design of studies involving archaeological materials must be justified. This section aims to expound on the reasoning behind the use of specific analytical tools, their benefits and limitations, as well as to set the stage for the methods section in Chapter 5.

4.1 Ceramics as Complex Materials

The definition of ceramics varies between archaeology and materials science. In materials science, ceramics are defined as "…materials manufactured from silicates (usually clays) and hardened by applying heat" (Rice, 1987, p. 4). Furthermore, ceramics are materials made from chemical compounds which combine both metallic and non-metallic elements (Rice, 1987, p. 3). In pre-historic archaeology the term "pottery" is more commonly employed and is defined as "…low-fired, unvitrified objects and/or cooking and storage vessels (Rice, 1987, p. 4)." Therefore, combining these two different views of ceramics or pottery, terms used interchangeably in this study, we will begin to recognize how the material properties contribute to understanding the socio-cultural processes in the Caribbean.

The main constituent of pottery is the clay that was used for its production. Clays are the recent product of the weathering and disintegration of older rocks, and have been categorized with a specific particle-size grade (<2 μm) (Rice, 1987, pp. 36–38). Clays can be either primary (authigenic), overlaying their source rock, or secondary (allogenic), forming elsewhere and transported to their current location (Garrison, 2016, p. 7). In contrast, clay minerals are mineral phases which have the particle-size grade of clays, but also have a specific mineral structure (Velde and Druc, 1999, p. 35). These are commonly the product of the weathering of silicate minerals (Garrison, 2016). Clay minerals by definition are a group of finely crystalline, hydrous silicates with a two-or three-layer crystal structure (McGraw Hill, 2003). Each type of clay mineral has a different chemical composition, which can affect their physical properties of plasticity and during firing (Velde and Druc, 1999, p. 35). The most important properties of the clay material for a potter is that when it is pulverized and mixed with water it has plasticity, becomes rigid when dry, and hardens when heated to high temperatures (Garrison, 2016, p. 7). Therefore, identifying the clay minerals will contribute to understanding other choices made by the potter to adjust the physical properties of the raw materials to accomplish these basic tasks.

When studying clays, it is essential to look at rock forming silicates of aluminum, magnesium, iron, calcium, sodium and potassium. These silicates include feldspars, quartz, clay minerals, micas and ferromagnesian silicates (Rice, 1987, p. 33). Hence, it will key to understand the abundance and composition of these silicates to gain familiarity with the raw materials used for ceramic production.

4.2 Local Geology

In-depth knowledge about the local parent rock formations is essential for determining the approximate area of production of the ceramics. The archaeological sites of Tibes, PO-42, PO-43, and PO-48 fall within the Ponce and Peñuelas quadrangles. The island of Puerto Rico has been the focus of geological studies since the first two decades of the 20th century (Krushensky and Monroe, 1978, 1975). The map of the Ponce and Peñuelas quadrangle, compiled in the 1970s is available in a 1:20,000 scale with a descriptive description of each geological formation (Krushensky and Monroe, 1978, 1975). The archaeological sites are enclosed to the north by a volcanic core, which forms the Cordillera Central, the main mountain range on the island. On the south, the sites are limited by a formation of mid-Tertiary limestone and the Caribbean Sea (Glover, 1971, p. 7). Specifically, the geology of the region is characterized by a series of complex geological formations, which are described below in Table 2. These formations lie in the close vicinity of the archaeological sites as seen in Figure 5, and would influence the composition of the local sediments and clays used to produce ceramics (See Appendix 4 for a complete Geological Map Puerto Rico).

4.3 Methodological Considerations for Assessing Ceramic Variability

Evaluating ceramic variability is one of the most productive archaeometric pursuits. The degree of ceramic variability can shed light on interaction between sites, specialization, functional diversity and other aspects of pottery production. The most common approach towards assessing ceramic variability is to combine a mineralogical and geochemical approach (Braekmans et al., 2011). Ceramic variability can be an indicator of many archaeologically meaningful phenomenon such as different raw materials, function, and manufacturing technology.

4.3.1 Provenance Studies

The provenance postulate is based on basic geochemistry principles that suggest that the chemical composition of ceramics represents the chemical composition of the raw materials (Albero Santacreu, 2014, p. 30). This assumption is dependent on the variability of the geological sources in the area; if the variability between different sources is higher than the variability within a single source (Albero Santacreu, 2014; Rice, 1987, p. 415). This hypothesis is tested through comparing ceramics of unknown origin either to ceramics of known origin or to clay sources in the region.[8] Furthermore, the concentration of different chemical elements in the ceramics can be used as a "compositional fingerprint" of the raw materials to relate the ceramics and their clay with their geologic origin (Albero Santacreu, 2014, p. 30). This approach is suitable for the early Ostionoid ceramics in Puerto Rico because it is believed that there was no intentionally added tempers. Instead, the inclusions stem from naturally occurring clays in the region. Therefore, the chemical signature would not be altered by the addition of tempering agents. Velde and Druc (1999) propose that instead of aspiring to find the exact source, it is more productive to understand the production area. More relevant than assessing the precise source of raw materials, or the geochemical composition, is to understand how chemical composition can reflect technological traditions, recipes, materials and techniques used in the pottery manufacturing (Albero Santacreu, 2014).

4.3.2 Functional Analysis

In addition to provenance, function is one of the most popular archaeological questions that is traditionally answered by archaeometric studies. It is common in the archaeological record to associate different clays or raw materials with different functions when associated with other variables such as vessel form, thickness, resistance to thermal stress, permeability, porosity,

[8] This process can be difficult and unreliable since many clay sources were exhausted in the past, and because the potter alters the clay composition during the manufacturing process (Rice, 1987, p. 417).

density and surface treatments (Rice, 1987). Ethnographically, the main properties potters look for in objects for an intended use are plasticity of the clay, the ability to give shape once dry and the hardness of the pottery as a final product (Velde and Druc, 1999, p. 151). To determine function, this thesis will assess thickness and surface treatments, and attempt to associate these parameters to the mineralogical and chemical groupings to understand weather there are any correlations or preferential selection of certain clays.

Table 2 Description of local geology in south-central Puerto Rico adapted from the USGS Open File Report 98-38.

Geological Unit	Lithology	Description[9]
Granodiorite-quartz diorite of the Utuado batholith	Granodiorite	Granodiorite, locally porphyritic, and subordinate quartz diorite, quartz monazite, and diorite.
Monserrate Formation	Mixed volcanic/clastic rock	Volcaniclastic siltstone and sandstone interbedded with tuff, subordinate conglomerate, calcarenite, and chert, conglomerate composed of augite andesite characteristic of the Lago Garzas Formation.
Lago Garzas Formation	Volcanic breccia	Dark-red volcanic breccia, lava, and subordinate volcanic sandstone-claystone, calcirudite, and pillowed basalt flows are interbedded. Locally interbedded with Anon Formation of Middle Eocene age.
Yauco Formation	Mixed carbonate/clastic rock	Siltstone, claystone; sandstone, limestone, and conglomerate.
Lago Garzas and Yauco Formations interbedded	Mixed carbonate/clastic rock	Characteristic Yauco Formation irregularly interbedded with volcanic sandstone characteristic of the Lago Garzas Formation.
Augite andesite porphyry	Andesite	Augite andesite porphyry.
Quartz diorite-granodiorte	Quartz diorite	Quartz diorite grades into granodiorite, locally proportions are reversed. Locally porphyritic.
Dacite	Dacite	Aphyric vesicular and amygdaloidal dacite.
Juana Díaz Formation	Limestone	Basal gravel to conglomerate overlain by limestone. Maximum estimated thickness is 850 m.

[9] The geological unit descriptions are taken verbatim from the geologic units and faults from USGS Open File Report 98-38, concerning assessment of the mineral resources of Puerto Rico (https://mrdata.usgs.gov/geology/pr/).

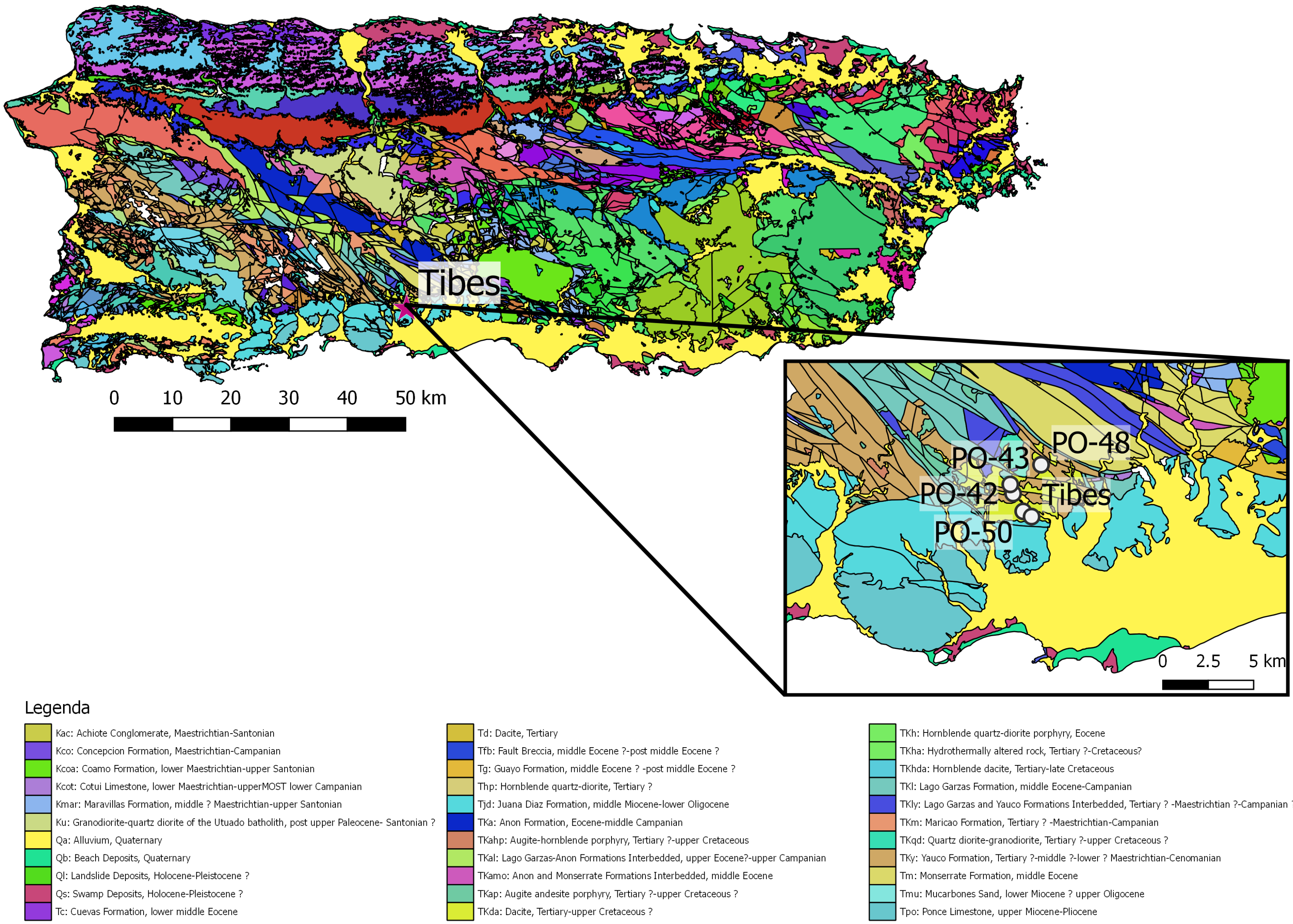

Figure 5 Sediment samples in the Geology Map of the region (the legend corresponds only to images in the image close-up. For a full Geological map see Annex 4).

4.3.3 Manufacturing Technology

Manufacturing technology refers to the decisions made by the potters related to the procurement of raw materials, forming, finishing, decorating, drying, and firing within the *chaîne opératoire*. Not only does manufacturing technology refer to the physical, aesthetic properties of ceramics, but also to the properties of the final product as a material. Understanding technology refers to "the analysis and description of physical (broadly construed), mineralogical, and chemical properties of ceramic materials, both raw and fired, in order to understand their manufacture and use" (Rice, 1987, p. 310). Therefore, by combining knowledge acquired through mineralogical and chemical analysis, the steps of the *chaîne opératoire* can be elucidated.

4.3.4 Multi-Analytical Approach or "Integrated Approach"

Provenance and technology analysis has traditionally been achieved by the so-called "integrated approach," which combines both mineralogical and chemical analysis (Mommsen, 2004; Tite, 2008). Although some authors have argued that chemical and petrographic analysis on their own can be adequate, it is widely accepted that using multiple complimentary methods for provenance, functional analysis and manufacturing technology signifies more scientific legitimacy due to the complimentary nature of the data (Iizuka, 2017; Mommsen, 2004). For example, chemical analysis can provide information on raw materials and technology, but can suffer from unexpected phenomenon such as the "dilution effect" (Frahm, 2018).[10] Therefore, thin section petrography can be used as a predictive method to distinguish between "chemical fingerprints" since the chemical composition is closely related to the mineralogy (Tite, 2008). Thus, by using petrography as a first step to identifying whether the production can be local or not, one can distinguish between certain secondary effects on the chemical data (Tite, 2008). It is essential to combine both mineralogical and chemical techniques to get an accurate picture of both the relationship with the local geology and the chemical "fingerprint" of the ceramics under study.

[10] The "dilution effect" is when the aplastic inclusions within the ceramic matrix affect the chemical signal (Frahm, 2018).

4.4 Analytical Techniques – Mineralogical Approaches

Mineralogical techniques are used to understand the mineral phases present in ceramic analysis to ascertain the composition of parent rocks of the clay sources, and consequently to connect the raw material sources to geological formations in the region. Since it is common practice to use local clays during the early Ostionoid period in south central Puerto Rico, mineral identification was used partially to understand provenance, which was probably from a nearby source, but also to discern if there was preferential selection of clays for pottery manufacturing. Therefore, it is imperative to distinguish petrographic and mineralogical groups to understand the variability of raw material sources in the region.

4.4.1 Thin Section Petrography

Petrographic thin section analysis of geological specimens is an age-old method used by geologists to understand mineralogical phase composition and rock textures. This is achieved through manipulating the optical properties of the minerals under the optical petrographic microscope. The petrographic microscope consists of two main observation modes: plane polarized light (PPL) and cross polarized light (XPL). In PPL mode, the light waves only vibrate in one plane, showing the pleiochroic properties of the mineral phases (Albero Santacreu, 2014, p. 23). Petrography is suitable for studying ceramics since they can be considered a “metamorphosed sedimentary rock” (Orton et al., 1993, p. 132). The main goal of this method is to determine the manufacturing technology, the physical characteristics of the ceramic and, ultimately, the provenance (Orton et al., 1993). With this method, it is possible to identify temper minerals, their abundances, particle orientation, void size, void orientation, and surface treatments. Petrography is used not only to identify mineral grains, but also to identify the size, sorting, shape, and percentage of inclusions in the fabric (Rice, 1987, p. 376). By compiling these elements, petrography can begin to elucidate compositional, and technological groupings.

Although the qualitative observations in petrography can be extremely powerful for distinguishing between groups, textural quantitative analysis allows the results to be comparable to the results of other ceramic technology studies (Velde and Druc, 1999, p. 201). While most studies employ the point counting method to estimate particle size, many automated methods exist which have facilitated and streamlined the procedure. In this study, the temper/matrix ratio and the average percentage area were calculated via quantitative textural analysis. This method has been commonly applied in archaeological ceramic studies (Albero Santacreu, 2017). The main impetus behind conducting textural analysis is its ability to determine or estimate grain size, which is connected to the depositional environment and the erosion of the raw material sources (Albero Santacreu, 2014, p. 12). In addition, the texture can shed light on complex mechanical properties. For example, "…clays improve their plasticity though long periods of exposure to weathering, where variations in temperature and humidity cause physicochemical alterations in the materials and promote the formation of finer fractions"(Albero Santacreu, 2014, p. 12). Therefore, acquiring quantitative information about the texture of the finer fraction of the ceramics can provide useful information on the depositional processes and the degree of alterations (Albero Santacreu, 2014, p. 12). Practically, texture analysis in this study consists in calculating the fine and coarse fractions occurring in the paste (Albero Santacreu, 2014; Orton et al., 1993; Rice, 1987; Velde and Druc, 1999). By distinguishing between unimodal and bimodal grain size distributions, one can establish whether the aplastic inclusions are naturally occurring in the clays or are tempers intentionally added by the potters (Albero Santacreu, 2014). Textural analysis of the ceramic fabric allows the archaeologists to assess technological choices made in the past, and consequently begin to understand some steps within the *chaîne opératoire*.

4.4.2 X-Ray Powder Diffraction (XRPD)

XRPD analysis is an analytical tool which studies the crystalline structures compounds within ceramics. The crystalline structure of a mineral has a characteristic spacing between the planes or the layers of their atom. Since each mineral has a unique atomic lattice arrangement, it is a suitable method for mineral identification (Rice, 1987, p. 382). Although pottery tends to have extremely complex mineralogy, XRD is commonly used to study ceramics semi-quantitatively (González et al., 2018; Sánchez-Climent et al., 2018), and through presence and absence studies (Puglieri et al., 2019; Vieira Ferreira et al., 2018). Due to the semi-quantitative nature of the analysis and the complexity of ceramic diffractograms, this tool is best used to confirm the identifications made during petrographic analysis as a complementary method (Rice, 1987, p. 384).

XRPD cannot always identify clay minerals since they lose their crystalline structure during firing. Clay minerals lose their ability to diffract X-rays after dehydroxylation at temperature of about 500-600°C (Rice, 1987). Since ceramics in the Caribbean were probably fired at relatively low temperatures in open firings, it is expected that most pots would not exceed 400°C. For this reason, the oriented mineral aggregate mounts were developed (Rice, 1987). Essentially, by orienting the sheet structure of the clay minerals and creating a pseudomacrocrystal which diffracts on a larger scale, the clay mineral signal is enhanced, and becomes suitable for identification via XRD (Velde and Druc, 1999, p. 274). Although the clay minerals might not be able to be identified in all ceramic samples, XRPD is a useful tool to study the raw material sources which have not been undergone heat treatments (Shepard, 1985, p. 146).

4.5 Analytical Techniques - Chemical Approaches

Chemical analysis of ceramics is a common method applied to understand the provenance of raw materials, manufacturing technology, and the identification of post depositional alterations (Albero Santacreu, 2014, p. 29). The main benefit of combining chemical techniques with mineralogical analysis is the ability to distinguish between different geochemical origins of pottery that present similar mineralogy. In addition, chemical analysis allows to distinguish between ceramic assemblages which come from similar geologic origins. For example, in south central Puerto Rico it is hypothesized that local raw material sources were employed, and therefore, these would appear mineralogically similar. Nevertheless, due to the different geological processes affecting the raw materials, the geochemistry of these sources might vary and could be an indicator of different raw materials chosen by the potters in antiquity. Since bulk chemical analyses can only provide information regarding the general chemical composition of a ceramic, it is not able to distinguish between the influence on intensities of the clay versus the aplastic inclusions (Rice, 1987, p. 389). Therefore, it is important to couple it with mineralogical analysis. This will aid in the discussion of ceramic variability and raw material exploitation. It is important to keep in mind that different methods vary in accuracy, sensitivity and precision. Sensitivity refers to the detection limit of the technique, precision refers to the reproducibility of the data using the analytical technique and accuracy refers to how close the results it to the true figure (Rice, 1987, p. 390). Therefore, although a lot of information can be gained in bulk chemical composition, it is important to couple it with mineralogical analysis to understand what the intensities of the elements stem from.

4.5.1 Energy Dispersive X-Ray Fluorescence (ED-XRF)

ED-XRF is based on the interaction of X-rays emitted by an X-ray source with the atoms in the sample. This X-ray displaces an electron from the inner orbits, and subsequently the inner orbit is filled with electrons from the outer orbits (Rice, 1987, p. 393). Based on this excitation of the atoms, a secondary X-radiation signature, or fluorescence, is emitted. This emission occurs at a series of specific wavelengths, which allows the chemical species identification. The intensity of the signal is proportional to the number of atoms present in the sample, which is measured in relation to a calibration standard, and allows for quantitative analysis (Velde and Druc, 1999, p. 278). ED-XRF has the ability to analyze up to 80 elements, and can detect from 100% to ppm (Rollinson, 1993, p. 10).

4.5.2 Inductively Couples Plasma Mass Spectrometry (ICP-MS)

ICP-MS is an analytical technique, which has low detection limits (up to ppts) and can analyze trace elements with high precision and accuracy in small amounts of time. It is used in archaeology to determine the sources of raw materials in pottery production (Neff, 2017). The basic principles of ICP-MS consist in the ionization of the sample constituents by the Ar plasma, and the subsequent separation of the generated ions according to their mass to charge ratio (m/z) that is counted by the mass spectrometer (Neff, 2017, p. 433). Although ICP-MS is extremely sensitive and can provide information that can potentially distinguish between raw material sources, it can also be affected by certain interferences during analysis. These consist mainly in isobaric interferences and polyatomic interferences (Neff, 2017, p. 434). Isobaric interferences are when different elements have isotopes with the same m/z ratio, and therefore can be measured erroneously. To remedy this problem, researchers can choose specific isotopes, apply equations for mathematical corrections, or more recently they can use reaction gases such as O_2 (Neff, 2017). Within isobaric interferences, doubly charged ions can occur when the first ionization potential of Ar is higher than the second ionization potential of other elements. Since Ar second ionization potential is lower than most elements this occurs infrequently (Neff, 2017). Polyatomic interferences are also spectral interferences which are characterized by a combination of atoms or a polyatomic molecule, in ionized form, with the same m/z of our analyte. This is mainly Ar based interferences since it is the most abundant species. To remove unwanted interferences, and to improve the limits for detection, researchers can again apply equations for mathematical corrections, or can use collision gases like H_2 or NH_3 and reaction gases such as O_2 (Neff, 2017). ICP-MS is a very powerful technique that has the ability to measure major, minor and trace elements with extremely high accuracy.

4.5.3 Scanning Electron Microscopy Energy Dispersive X-Ray Spectroscopy (SEM-EDS)

SEM-EDS is a powerful tool to study archaeological ceramics. The electron micrographs produced by SEM stem from the interaction of primary electrons with the specimen, which gives rise to the emission of backscattered electrons, secondary electrons and X-rays (Froh, 2004, p. 159). For studying ceramics, secondary electrons provide information on the surface topography and backscattered electron show the differences in elemental compositions. In backscattered electron mode, the electron probe penetrates approximately a few micrometers, and the resolution is inferior to secondary electron images (Froh, 2004). The X-ray spectroscopy provides quantitative information on elemental composition, specifically, multi-elemental maps can be produced showing the differential distribution of elemental influence between the matrix and the aplastic inclusions. Subsequently, the combination of SEM and EDS is most useful when applied to ceramics because of the duality information attained. Since X-rays are produced whenever electrons penetrate a material, the emission of the electron beam is of characteristic X-rays (Froh, 2004). Since in this study EDS was employed, the entire spectrum of X-rays is collected and there was a quick determination of element composition.

The main information obtained by SEM-EDS analysis is about the raw materials used, firing environment, and detailed information on surface decoration. Although SEM has a better resolution than petrography, it is less equipped to identify individual mineral phase

5 Materials and Methods:
A Multi-Analytical Approach

5.1 Materials

Fifty-seven samples of pottery and clay were studied under a multi-analytical approach (See Table 3). Fifty ceramic sherds were chosen from the site of Tibes, PO-42, PO-43, and PO-48 along the Portugués River and Chiquito River in south-central Puerto Rico from scientifically excavated contexts. In addition to pottery, seven sediment samples from various locations in the region collected during the TASP regional survey were studied to understand possible raw material sources for local ceramic production. The sediment samples are associated to several archaeological sites within the regional survey. Below, each group of samples will be discussed in relation to its archaeological context, radio carbon dates and associated materials. Where available, a summary of the context interpretation will be included. The samples have retained the identification number related to their archaeological context for easy recognition during analysis, and have been further identified by a letter to distinguish the samples which stem from the same contexts.

5.1.1 Tibes Pottery

There is a total of thirty sherds from the site of Tibes stemming from three different archaeological contexts shown in Figure 1. The three different contexts have been categorized by artefact collection bag 62, 117, and 294 as indicated in the sample reference numbers. These bags refer to specific 1 x 1 m archaeological units and levels. Each unit is associated with a ceremonial feature or midden deposit, except for the ceramics from bag 64, unit 1.

Bag 64 consists of ten sherds from deposit A, unit 1, level 3. This context consists of a cultural, dark brown silt layer of a trash midden, which contains a high concentration of shells, fauna remains, charcoal and artifacts between 20 and 55 cm below the surface. The ceramics from level three have been characterized as Santa Elena style ceramics (A.D. 900-1200), and have been radio carbon (^{14}C) dated to 1127 cal AD ± 100 (95% confidence interval).[11]

Bag 117 has a total of ten sherds which stem from the north west of the site next to ball court 8 within unit 3, level 3. Unit 3 is located in deposit C, southeast of ball court 8. Levels 3-7 are from 20 to 60-70cm below the surface and consist of dark brown, silt and have a higher concentration of prehistoric materials (Curet, 2003). The archaeologists believe that the top levels of the cultural deposit (level 3), which contain few Santa Elena style sherds are a product of a secondary midden, which could have been produced during the construction of ball court 8 (Curet, 2003). It is common to clear out ceremonial areas, such as ball court 8, during construction. Therefore, this context has been interpreted as being coeval with the construction of monumental architecture at the site and has been radio carbon dated to 1136 cal AD ± 100 (95% confidence interval).

Bag 294 consists of ten sherds, which stem from deposit G, which is about 2 m west of ball court 2. Stratigraphically, this bag stems from unit 7, level 2 which is 20 cm below the surface in a cultural layer of dark brown silt (Curet, 2003). This unit was subdivided into two areas since it was discovered that the western section had been previously excavated. The ceramics from this study stem from the previously unexcavated loci which mainly consisted of Santa Elena and Modified Ostiones Style (AD 900-1200). In context 294, it is notable that both Santa Elena from eastern Puerto Rico and Modified Ostiones style from western Puerto Rico were identified and co-exist. This is one of the examples of archaeological contexts which show Tibes as a cultural transition zone between east and west.

5.1.2 TASP Pottery

There are ten sherds from PO-42, also known as the site of La Mineral. These include samples with reference number FS.75 and FS.76. The two ceramic sherds studied with numbers FS.75 stem from a surface collection, level 1, between 0 and 20 cm below the surface. In this layer, 5 total sherds were found and one lithic sample. FS.76 contains eight sherds and is from level 1,

[11] All radio carbon dates were calculated using CALIB calibration program.

Table 3 Log of the analytical techniques used on each sample by archaeological site.

Site	Sample No.	Petrography	XRD	OMA	SEM-EDS	ED-XRF	ICP-MS
Tibes	117_a	x	x			x	x
	117_b	x	x			x	x
	117_c	x	x			x	x
	117_d	x	x		x	x	x
	117_e	x	x			x	x
	117_f	x	x		x	x	x
	117_g		x			x	x
	117_h	x	x		x	x	x
	117_i	x	x			x	x
	117_j	x	x			x	x
	294_a	x	x			x	x
	294_b	x	x		x	x	x
	294_c	x	x		x	x	x
	294_d	x	x		x	x	x
	294_e	x	x	x	x	x	x
	294_f	x	x		x	x	x
	294_g	x	x			x	x
	294_h	x	x			x	x
	294_i	x	x		x	x	x
	294_j		x			x	x
	62_a	x	x			x	x
	62_b	x	x		x	x	x
	62_c	x	x			x	x
	62_d	x	x	x		x	x
	62_e	x	x			x	x
	62_f	x	x	x	x	x	x
	62_g	x	x			x	x
	62_h	x	x		x	x	x
	62_i	x	x			x	x
	62_j		x	x		x	x
PO-42	FS.75_a	x	x			x	x
	FS.75_b	x	x			x	x
	FS.76_a		x			x	x
	FS.76_b	x	x			x	x
	FS.76_c		x			x	x
	FS.76_d	x	x	x		x	
	FS.76_e	x	x	x	x	x	
	FS.76_f	x	x	x	x	x	x
	FS.76_g	x	x			x	x
	FS.76_h	x	x		x	x	x
PO-43	FS.232_a	x	x	x		x	x
	FS.232_b	x	x			x	
	FS.245_a	x	x		x	x	x
	FS.245_b	x	x			x	x
	FS.246_a		x			x	x
	FS.246_b	x	x			x	x
PO-48	FS.253_a		x			x	x
	FS.255_a	x	x			x	x
	FS.257_a	x	x		x	x	x
	FS.263_a	x	x		x	x	x

0-20 cm below the surface. One hundred and ten sherds were found total, 6 lithics and 4 pieces of bone (Torres, 2012, p. 176). The soils consisted of highly compacted dark brown, clayey soils. These layers have been associated to Santa Elena and Modified Ostiones style.

There are six sherds from PO-43, also known as Los Gongolones site. These sherds are identified as FS.232, FS.245 and FS.246. Each number consists of two sherds. FS.232 is from level 1, 0-20cmbs and is associated with ten ceramic sherds total. FS.245 is also from level 1, 10-20cm and is associated with 27 sherds total and 745 shell pieces. FS.246 is from level 2, 10-20 cmbs and its context consists of 23 sherds, 1 lithic and 729 shells. The soil consists of compacted dark brown clayey soils. The ceramics found here as associated to the Santa Elena and Modified Ostiones style (Torres, 2012, p. 179).

Four sherds were selected from PO-48, the Escuela Río Chiquito site with sample numbers of FS.253, FS.255, FS.257, FS.263. The sample FS.253 is associated with level 1, 0-20 cmbs and was found with 14 sherds total and one lithic sample. FS.255 stems from level 2, 20-50 cmbs, and was the only sherds found in this stratigraphic layer. FS.257 is from level 1, 0-20cmbs and is found with one other sherd in the level. Last, FS.263 is from level 1, 0-20cmbs and is found in association with 9 sherds total and 8 lithics. Soil stratigraphy at the site was mainly formed by dark brown, highly compacted clay and the sherds found were associated to Santa Elena and Modified Ositones style. (Torres, 2012, p. 184). This context is the only one without any type of ritual architecture present as far as could be observed by the archaeologists.

5.1.3 TASP Sediment Samples

The TASP sediment samples stemmed from six different locations associated to five different sites (Table 4). The samples were obtained from below the surface during test excavations and are representative of different types of sediments in south central Puerto Rico. Below, a table with the analysis conducted on each sediment sample.

5.1.4 Sampling strategy

The samples were chosen to understand synchronic ceramic variability in the region. The samples stem from contexts which have been radiocarbon dated to Period III (Ostionoid period). Coarse ware sherds were specifically chosen since they hold more information than fine ware to shed light on the interpretation of the context and on the overall activities occurring at the site. In addition, small amounts of decorated sherds were found at the site of Tibes, and therefore they were not available for destructive analysis. In addition to ceramics, clay samples were analyzed to assess the available raw materials in the region. The samples are a product of the TASP project, and are found in association with several sites surveyed in the region.

Table 4 Analysis log of sediment samples.

Site	Sample Name	XRD	ED-XRF	OMA
PO-42	La_mineral	x	x	x
PO-42	La_mineral_NW	x	x	
PO-48	Rio_chiquito	x	x	x
PO-43	Los_gongolones	x	x	x
PO-50	Horse_pasture	x	x	x
PO-52	Finca	x	x	
N/A	Red_clay	x	x	

5.2 Sample Preparation

The ceramics were sampled sparingly, only destroying what was necessary to perform the analyses. Many of the samples were re-used for various analysis, and can be employed for future studies.

5.2.1 Thin Section Preparation (Optical Microscopy & SEM-EDS)

Thin sections were prepared following standard procedures employed in petrography, which are discussed by Quinn (2013) in greater detail when applied to archaeological ceramics. About 5 g of ceramics were sampled with a minimum dimension of 3 cm. The ceramics were cut perpendicular to the body of the ceramics using a saw (Discoplan TS, Struers). The samples were cleaned with water to remove any remnant soil from burial on the surface. The samples were dried overnight at 40°C and then were embedded in epoxy resin. Epoxy Fix (Struers A/S, Ballerup Denmark) was used with a 7:1 epoxy to hardener ratio and a 24 h hardening time. Next, the cross-sections were polished using fine silicon carbide abrasive paper with different roughness until reaching a 5 µm grain diameter size (Silicon Carbide Paper, SiC FEPA P# 320, 500, 800, 1200,2000, 4000, Struers). The polished cross-sections were glued to a standard glass slide (70 x 50 mm) using Araldite (1:1), left on a hotplate at 100°C for 2 h under pressure, and left to dry during 24 h. Then, the cross-sections were cut off the glass using the saw leaving an approximate 1-2 mm of sample bonded to the glass. Next, the glasses were ground smooth by the saw until there was approximately 50-100 µm of sample on the glass. At this stage the minerals began to become translucent when held up to the light and quartz begins to exhibit first to low second order interference colors (Quinn, 2013, p. 27). The thin sections were consequently polished using carborundum grit (until 5 µm) and water until they reached 30 µm thickness. The desired thickness is confirmed when quartz displays first-order interference colors (gray and white) under XP light, which will allow the identification of the minerals under the polarized microscope. The ceramics were not covered with a permanent glass cover since the thin sections were going to be used for VP-SEM-EDS analysis as well.

5.2.2 Powder Preparation (XRD, XRF, ICP-MS)

The ceramics and clays in powder form were employed for XRD analysis. In addition, the powder was used to create the glass pearls for XRF and to undergo digestion for ICP-MS analysis. The ceramics were sub-sampled for approximately 2.5 mg of powder using a saw (Discoplan TS. Struers). The surfaces were cleaned mechanically using a Dremel 3000 multi-tool with a diamond wheel point of 4.4mm (7105). This was done to assure the samples were devoid of soil residues and to remove any other contaminations, which could influence the chemical analysis. The samples were grinded with a mortar and pestle made of agate, which was cleaned with nitric acid (HNO_3 2%) and milli-Q water between samples. The samples were grinded until a fine powder (approximately less than 10 µm).

5.2.3 Oriented Aggregate Mounts

Oriented aggregate mounts were prepared to identify the clay minerals in the samples. Only samples which displayed peaks of clay minerals were studied. The powder samples were stirred with Milli-Q water during one minute, and the material was pipetted and placed on a glass substrate after settling for fifteen seconds, allowing the heavier fraction of the sample to sink to the bottom. This procedure was performed on all samples in duplicates and were dried at room temperature overnight. After initial XRD analysis was performed, the samples were subjected to three different treatments. The first copy of the samples were submitted to glycolation with ethylene glycol and the other version of the samples were heated to 400°C and 550°C, conducting XRD analysis after each treatment.

The ethylene glycol treatment consisted of placing the oriented aggregate mounts in a desiccator with ethylene glycol on the base in the over at 60°C during 12 hours. The heating treatments were applied to the other copies of the aggregate mounts. The mounts were placed in the oven for 45 minutes, 15 minutes to reach the temperature of 400°C and 30 minutes at the aforementioned temperature. The same procedure was applied at 550°C. XRD analyses was performed between every stage of the sample treatment, to see how the clay minerals reacted. The method was adapted from the Manuar of X-Ray Powder Diffraction by USGS (Poppe et al., n.d.).

5.2.4 Glass Pearls (XRF)

After conducting XRD analysis, the powdered samples were made into glass pearls to attain homogenous samples, and get more accurate measurements of the bulk of the ceramics. The pearls consist of 1.2 g of sample and 12 g of lithium iodide flux (1:10), which are fused at 1065°C. The Claisse Fluxer LeNeo fusion instrument produced the glass pearls employed in the EDXRF analysis.

5.2.5 Sample Digestion (ICP-MS)

Approximately 100 mg of powdered sample was digested for ICP-MS analysis through a hotplate acid digestion procedure adapted from (Ottley et al., 2003). In addition to the 50 ceramic samples, 2 certified reference materials (CRMs) were digested to assure the proper digestion of the samples and the accuracy of the analysis. Andesite, AGV-2 from Guano Valley in Lake County, Oregon (USGS Reference Material) and Diabase, W-2a collected in a quarry near Centerville Virginia (USGS Reference Material) were used since they have the same chemical matrix as ceramics.[12] The powdered samples were digested in three steps. First, the silicates in the samples were digested in PFA closed beakers, using 0.5 mL HNO_3 (65%, Suprapur ® grade, Merck) and 2 mL HF (50% OPTIMA® grade, Fisher Chemicals) for each sample during 48 hours on a hotplate at 150°C. After 48 hours, the samples were dried on the hotplate. Second, 2 mL of *aqua-regia,* freshly prepared (1 HNO_3: 3 HCl), was added. This was left during 24 on a hotplate at 120°C. Third, the samples were dried on the hotplate and 2 mL of HNO_3 were added during 24 hours at 120°C. Lastly, the samples were dried and 3 mL of milliQ H_2O and 1.6 mL of HNO_3 (65%) were added. After, the solutions were transferred to 50 mL PFA volumetric flasks and they were filled up to 50 mL with milliQ H_2O to ensure a final acidic matrix of 2% HNO_3.

The calibration curve was built with 11 levels of calibrations of multi-elemental solutions of High Purity Standards® (ICO-MS-68-A and ICP-MS-68-B) with HNO_3 2% to replicate the same matrix of the samples. The calibration curve consisted of 11 different concentrations (0, 2.5,

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[12] Andesite from AGV-2 (minerology and classification of AGV-2 data is unavailable, but it is believed that it is very similar to AGV-1) is aphanitic, finely porphyritic with a trachytic texture and the certificate values stem from various studies (Gladney 1983; Gladney and Roelandts, 1987; Govindaraju, 1994). Diabase consists of augite, plagioclase, quartz, potassium feldspar, biotite, and opaque minerals and the recommended concentrations are outlined in several publications (Flanagan and Gottfried, 1980; Gladney and Roelandts, 1988; Govindaraju, 1994).

5, 10, 25, 50, 100, 200. 400, 800, 1500 pbbs). A sequence of analysis was constructed with the standards, CRMs, blanks and *washes* (HNO_3 2%) to assure the quality of the analysis throughout.

5.3 Methods and Experimental Conditions

A multi-analytical approach was key in this investigation. Complementary methods to characterize the chemical and mineralogical nature of the ceramics were used. In addition, basic statistical analysis was applied to the chemical analysis to facilitate the evaluation of compositional groups within the large assemblage.

5.3.1 Petrography

Petrographic analysis of thin sections was performed using a Leica DM2500P transmitted light polarizing microscope with a rotating stage in both plane polarized light (PPL) and cross polarized (XP) modes. Images of significant inclusions and of the general fabric were captured with the Leica MC 170HD digital camera attachment. Magnifications of 25 x, 40 x, and 100 x were employed. Specifically, the 25 x magnification was used to to basic grouping and to take images of the fabric. The images of the fabric were further analyzed by textural analysis using the methodology employed by Santacreu (2017). The analysis was performed with the ImageJ software. This study focused on the average temper area and the temper size to focus on identifying different styles and fabrics present at the sites in south central Puerto Rico. The percentage area and the size of inclusions was calculated to determine the temper/matrix ratio and to distinguish between different paste recipes.

5.2.2 Variable Pressure Scanning Electron Microscope – Energy Dispersive X-Ray Spectrometer (VP-SEM-EDS)

To conduct microchemical analysis, a variable pressure SEM-EDS was employed for micro-analysis on the thin sections prepared for petrography. In this study the Variable Pressure Scanning Electron Microscope HITACHI S-3700N (VP-SEM), operating with an accelerating voltage of 20 kV and chamber pressure of 40 Pa. This equipment is well-suited for cultural heritage materials since it was not necessary to coat the samples with conductive material due to the variable pressure setting. A Bruker XFlash 5010 Silicon Drift Detector (SDD) with a resolution of 129 eV at Mn Kα was used for the chemical analysis and the EDS elemental data was acquired by point microanalysis and in the form of elemental distribution maps processed with Espirit 1.9 software. SEM images were captured in backscattering (BSE) mode in a range of magnifications.

5.2.3 X-Ray Diffraction (XRD)

XRD was performed on the Bruker D8 Discover X-Ray Diffractometer with Cu Kα source at 40 kV and 40 mA. Diffractograms were collected at a 2θ angular range of 3° – 75° with 0.05° step size and 1s measuring time. The LYNXEYE linear detector employed provides an increased signal due to presence of 192 individual detectors. The identification of minerals was conducted on the DIFFRAC.SUITE EVA software and using the International Center for Diffraction Data Powder Diffraction File (ICDD PDF-2) as a database for the inorganic materials in the samples.

Semi-quantitative determination of the mineral abundance in the bulk samples was obtained by employing the Reference Intensity Ratio (RIR) method using the reference standard corundum, which is the most popular instrument-independent constant internal standard method (Zhou et al., 2018). The final semi-quantitative values are presented as a percentage relative to presumed 100% matrix of crystalline minerals.

5.2.4 Oriented Mineral Aggregates

Oriented mineral aggregates were run under XRD analysis with the same parameters discussed in section 5.2.3 between each sample treatment. The same analytical conditions were used as for the powdered samples, except the measuring time was increased to 2s per point to accentuate the clay mineral peaks. To identify the clay minerals, the diffractograms after the glycolation and heating treatments were compared and the clay minerals were identified according to the U.S. Geological Survey (USGS) clay mineral ID flow diagram (Poppe et al., n.d.).

5.2.5 XRF

Major and minor elements were analyzed using an Energy Dispersive X-Ray Spectrometer (EDS-XRF, S2 Puma, Bruker) using a methodology based on (Georgiou et al., 2015). Quantification was obtained using an empirical method using 25 reference materials. The software used to process was the Spectra Elements 2.0 developed by Bruker.

5.2.6 ICP-MS

Inductively Coupled Plasma Mass Spectrometry was conducted to study minor and trace elements present in the ceramics. This method not only provides higher accuracy and resolution than XRF analysis, but also allows the measurement of trace elements which are below the detection limit of other chemical analysis conducted during this study. Liquid analysis was performed to attain the bulk composition of the trace elements. The Agilent 8800 ICP-MS Trip Quad system was employed during the study. Prior to commencing the analysis, the equipment was calibrated and the analysis sensitivity was optimized, oxide formation and the double ion formation were tuned and minimized with the Agilent Technologies tuning solution. The oxide formation ($CeO^+/Ce^+<1.35\%$) and the double charged ions ($Ce^{2+}/Ce^+<2\%$) were below the recommended values. The ICP-MS analysis was performed with the MS/MS scan type for the best accuracy, and collision and reactive gases were used in the collision/reaction cell (no gas, He, and O_2) when necessary. The elements selected, the integration times used and the general instrumental conditions are illustrated in Table 5.

Prior to the ICP-MS analysis, limit of detection and limit of quantitation (LOD and LOQ) were experimentally determined, running 11 replicates of a blank solution and 11 replicates of a solution with known concentration. The following equation was employed to calculate the limit of detection (LOD) (See Equation 1). The quantification limit was assumed to be 10 times the value obtained for the LOD for each element (See Annex 5). In order to check for instrumental drift, two certified reference materials (AGV-2 and W-2a) were measured before and after the samples of each batch to confirm also the accuracy of the measurements.

5.2.7 Statistical Analysis

Statistical analysis was performed using SPSS software. Dimension reduction analysis, specifically principal component analysis was employed to study intrasite variability. The specific procedure was adapted from Rousaki et al. (2016).

Table 5 Analytical conditions for ICP-MS.

Acquisition Mode	**Spectrum**
Spectrum Mode Option	Q2 Peak Pattern: 1 Point Replicates: 3 Sweeps/Replicate: 10
Plasma Parameters	
RF Power RF Matching Sample Depth Carrier Gas (Ar) Plasma Gas (Ar) Nebulizer Pump	1550 W 1.70 V 10.0 mm 1.10 L/min 15 L/min 0.10 rps
Collision Cell	
Collision Gas: He Reaction Gas: O_2	Flow: 4.0 mL/min Flow: 0.5 mL/min
Analysis Mode	
No Gas	^{118}Sn, ^{121}Sb, ^{133}Cs, ^{137}Ba, ^{139}La, ^{140}Ce, ^{141}Pr, ^{146}Nd, ^{147}Sm, ^{153}Eu, ^{157}Gd, ^{159}Tb, ^{163}Dy, ^{165}Ho, ^{166}Er, ^{169}Tm, ^{172}Yb, ^{175}Lu, ^{208}Pb, ^{232}Th, ^{238}U
He	^{45}Sc, ^{51}V, ^{55}Mn, ^{9}Co, ^{60}Ni, ^{63}Cu, ^{6}Zn, ^{71}Ga, ^{72}Ge, ^{5}Rb, ^{8}Sr, ^{89}Y, ^{0}Zr, ^{93}Nb
O_2	^{31}P
Dwell time	
0.3s	^{45}Sc, ^{51}V, ^{55}Mn, ^{59}Co, ^{60}Ni, ^{63}Cu, ^{66}Zn, ^{85}Rb, ^{88}Sr,
0.5s	^{31}P, ^{71}Ga, ^{72}Ge, ^{89}Y, ^{90}Zr, ^{93}Nb, ^{118}Sn, ^{121}Sb, ^{133}Cs, ^{137}Ba, ^{139}La, ^{140}Ce, ^{141}Pr. ^{146}Nd, ^{147}Sm, ^{153}Eu, ^{157}Gd, ^{159}Tb, ^{163}Dy, ^{165}Ho, ^{166}Er, ^{169}Tm, ^{172}Yb, ^{175}Lu, ^{208}Pb, ^{232}Th, ^{238}U

$$LOD = \frac{3\sigma \times Conc.(ppb)}{(CPS200ppb - CPSblank)}$$

Equation 1 Limit of Detection (LOD) equation used for ICP-MS analysis.

6 Results

6.1. Petrography

Petrographic analysis was conducted on 43 ceramic samples from south central Puerto Rico. Since all the samples appear to be from the same archaeological styles (Santa Elena or Modified Ostiones), and there is a high likelihood of interaction expected between the sites in this study, petrographic analysis is key for identifying potential differentiation in the technological choices and ceramic variability between and within sites. The petrographic analysis shows three main groups that can be easily differentiated: the volcanic, sedimentary and felsic groups. The distribution of samples (43 out of 50) between sites shows a higher concentration of samples stemming from Tibes (Figure 6a). The petrographic groups are shown in Figure 6b, exhibiting an uneven distribution between the categories. The petrographic groups identified were confirmed through quantitative textural analysis and mineral identification as discussed below.

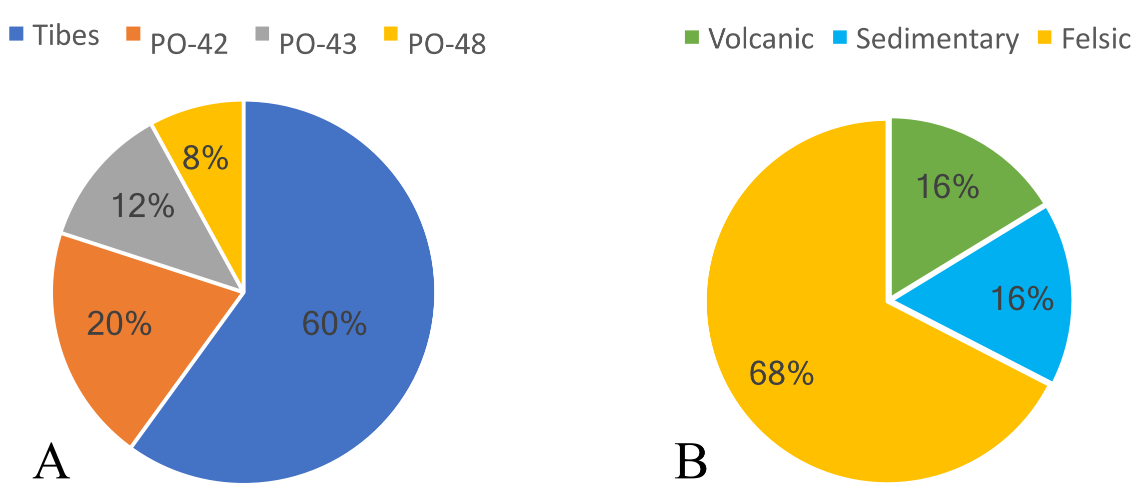


Figure 6 (A) pie chart of sample distribution by site; (B) right a pie chart distribution by petrographic group.

6.1.1 Volcanic Group

The volcanic group is found only at Tibes. It is mainly characterized by large amounts of volcanic lithoclasts and sparse sedimentary rock inclusions (Figure 2a and 2b). This group represents 7 out of 43 samples (16% of the ceramic assemblage). The sedimentary rocks present in the samples appear to be weathered from volcanic rocks due to the occurrence of similar minerals. The volcanic rocks have large plagioclase crystals in the matrix, which have been characterized as albite, oligoclase, bytownite and anorthite through SEM-EDS analysis. In addition to abundant plagioclase, there is ample K-feldspars and quartz throughout the samples. Notably, this petrographic group has no amphiboles, which was confirmed through XRD analysis. In addition, there are pyroxene grains, and weathered pyroxenes in the matrix of the ceramics. Most of the inclusions are between poorly rounded and rounded, and are heavily weathered. The paste appears to be similar to the sedimentary group, containing between 10-20% temper/clay ratio and have between moderately sorted to poorly sorted temper sorting when assessed visually (See Table 6 for all of the petrographic parameters studied).

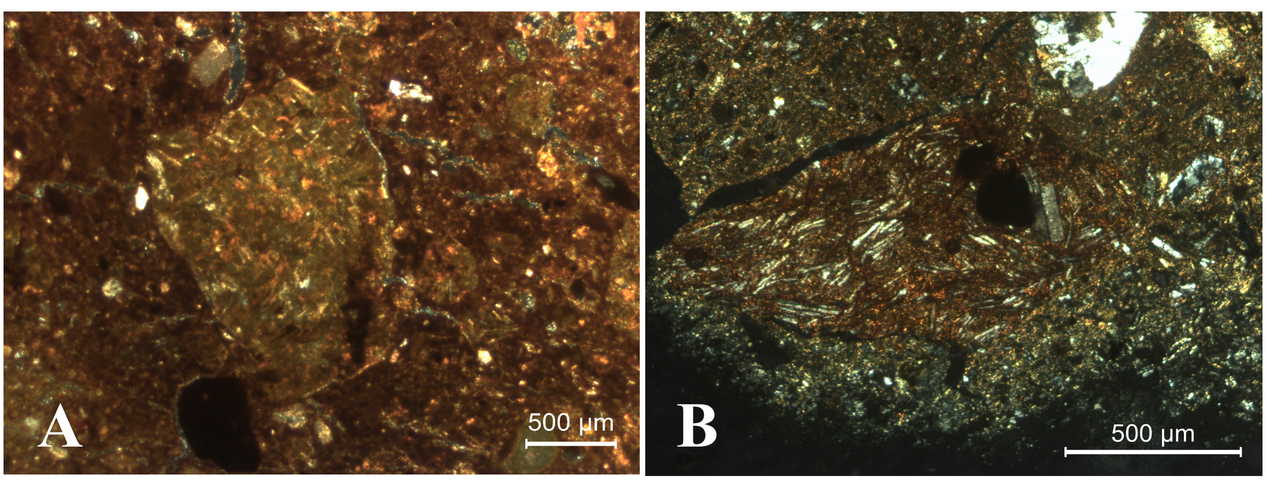


Figure 7 XPL petrographic images of sample (A) 117_c and (B) 294_a from the volcanic group exhibiting characteristic volcanic rock inclusions.

Table 6 Results from petrographic analysis.

Context			Petrographic Analysis[13]																
Sample ID	Site	Group	temper-matrix ratio (%) quan.	temper-matrix ratio (%) qual	sorting	max grain (µm)	sphericity	Qtz	Pl/Kfs	Amp	Ms	Ep	Op. Min.	Cpx	Plut.	Volc.	Met.	Sed.	Grog
117_a	Tibes	sed	15.4%	25%	poorly	1564	r/hs	x	x	x			x					x	
117_b	Tibes	fel	20.6%	25%	moderately	815	a/hs	x	x	x			x	x	x				
117_c	Tibes	volc	13.0%	20%	poorly	2525.4	r/hs	x	x	x			x			x		x	
117_d	Tibes	fel	29.8%	30%	poorly	3435	r/ls	x	x	x	x		x		x				
117_e	Tibes	fel	20.3%	30%	moderately	1463.5	pr/hs	x	x	x	x		x		x		x		
117_f	Tibes	volc	N/A	20%	poorly	1776.7	pr/hs	x	x				x			x		x	
117_h	Tibes	volc	25.4%	20%	very poorly	2205.6	r/ls	x	x			x	x			x		x	
117_i	Tibes	fel	N/A	20%	moderately	503.977	sa/hs	x	x	x			x		x				
117_j	Tibes	fel	27.8%	40%	well	2929.6	a/hs	x	x	x			x			x			
294_a	Tibes	sed	4.6%	20%	poorly	2625.8	r/hs	x	x				x					x	x
294_b	Tibes	sed	9.9%	20%	very poorly	2821.4	s/hs	x	x	x			x					x	
294_c	Tibes	fel	19.7%	20%	well	1131.6	pr/hs	x	x		x		x			x		x	
294_d	Tibes	volc	9.8%	15%	poorly	1397.32	r/hs	x	x				x			x		x	
294_e	Tibes	sed	N/A	20%	poorly	3909.2	pr/hs	x	x				x					x	x
294_f	Tibes	fel	16.2%	25%	well	2182.3	a/hs	x	x				x						
294_g	Tibes	fel	18.9%	20%	poorly	1544.2	sa/hs	x	x	x	x	x	x						
294_h	Tibes	fel	20.2%	20%	moderately	1829.9	a/hs	x	x				x						
294_i	Tibes	volc	4.2%	15%	moderately	2139.3	r/hs	x	x				x			x		x	
62_a	Tibes	fel	18.7%	20%	very poorly	1496.9	sa/hs	x	x	x			x		x		x		
62_b	Tibes	fel	39.3%	30%	well	2123.911	pr/ls	x	x					?					
62_c	Tibes	fel	31.4%	30%	poorly	2274	a/hs	x	x	?			x						
62_d	Tibes	volc	13.1%	20%	poorly	2358.7	sr/hs	x	x				x			x		x	
62_e	Tibes	volc	N/A	25%	well	552.3	sr/hs	x	x				x			x		x	
62_f	Tibes	fel	17.0%	20%	poorly	3180.9	sa/hs	x	x			x	x					x	
62_g	Tibes	fel	14.9%	20%	poorly	1184.331	sa/hr	x	x				x	x					
62_h	Tibes	fel	27.6%	30%	well	944.9	a/ls	x	x			x	x			x			
62_i	Tibes	fel	25.9%	20%	poorly	1340.6	sa/hs	x	x										
FS.232_b	PO-43	fel	14.6%	30%	poorly	1171.1	sa/hs	x	x			x	x					x	
FS.245_a	PO-43	fel	19.8%	40%	poorly	1185.1	a/ls	x	x	x		x	x						
FS.245_b	PO-43	fel	N/A	40%	poorly	1115	a/hs	x	x			x							
FS.246_b	PO-43	fel	33.1%	30%	poorly	674.978	a/hs	x	x	x			x		x				
FS.255_a	PO-48	fel	30.8%	30%	moderately	553.253	sa/hs	x	x	x			x		x				
FS.257_a	PO-48	sed	18.0%	30%	moderately	2262.8	r/hs	x	x				x					x	
FS.263_a	PO-48	fel	20.6%	20%	moderately	967.486	a/hs	x	x	x			x	x	x				
FS.75_a	PO-42	sed	N/A	10%	well	1219.8	r/hs	x	x				x					x	
FS.75_b	PO-42	fel	15.2%	20%	moderately	510.06	sa/hs	x	x	x	x		x		x				
FS.76_b	PO-42	fel	N/A	30%	poorly	491.099	sr/hs	x	x	x			x		x				
FS.76_e	PO-42	fel	26.4%	30%	moderately	489.703	a/hs	x	x	x			x		x				

[13] Sed=sedimentary, fel=felsic, volc=volcanic, quan=quantitative, qual=qualitative, r=rounded, pr=poorly rounded, sa=sub-angular, a=angular, hs=high sphericity, ls=low sphericity, Qtz=quartz, Pl=plagioclase, Kfs=K-feldspar, Amp=Amphibole, Ms=muscovite, Ep=epidote, Op.Min.=opaque mineral, Cpx=clinopyroxene, Plut=plutonic, Volc=volcanic, Met.=metamorphis, Sed=sedimentary.

FS.76_f	PO-42	fel	24.6%	25%	moderately	591.511	a/hs	x	x	x	x	x	
FS.76_g	PO-42	fel	22.8%	15%	moderately	1019.962	a/hs	x	x	x	x	x	
FS.76_h	PO-42	sed	N/A	15%	poorly	3182.4	r/hs	x	x		x		x

6.1.2 Sedimentary Group

The sedimentary group is characterized by a high amount of sedimentary rock inclusions and represents 7 out of 43 samples (16% of the ceramic the assemblage). This group is not limited to Tibes, but is also present sparsely at PO-42 (FS.75_a, FS.76_h) and PO-48 (FS.257_a). The sedimentary rock appears to be the same sedimentary rock present in the volcanic group, which is the product of the weathering of mafic rocks. The mafic composition is confirmed by the low amount of quartz observed in the sedimentary rock inclusions. Petrographic analysis suggests that some of these sedimentary rocks are a sandstone or mudstone (See Figure 8a and 8b). Similar to the volcanic group, there is no evidence of amphiboles and only scant pyroxene grains. In addition, there is quartz, plagioclase and K-feldspars present. Some samples show opaque minerals and epidote in small amounts. Otherwise, this group presents similar mineralogical composition to the volcanic group. The grains are between sub-rounded and rounded, and the temper/matrix ratio is between 10-20%. Similar to the volcanic group, the samples within this subgroup have both the addition of slip and smoothed surfaces, which are not present in large proportion in the felsic group (See Figure 9a and 9b).

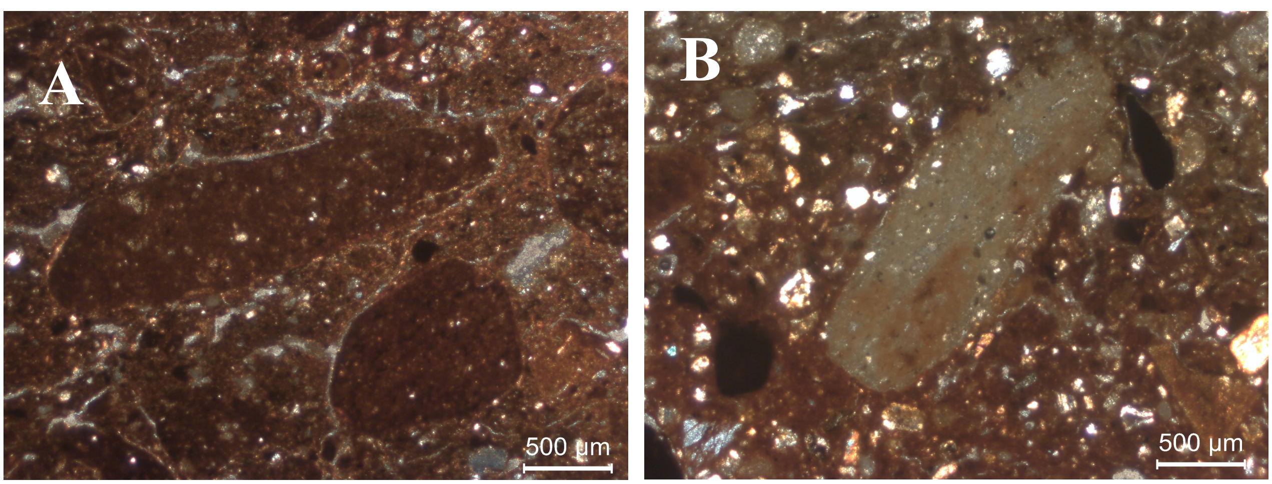


Figure 8 XPL petrographic images of examples of typical sedimentary rocks found in the sedimentary group. (A) 294_a, (B) FS.257_a.

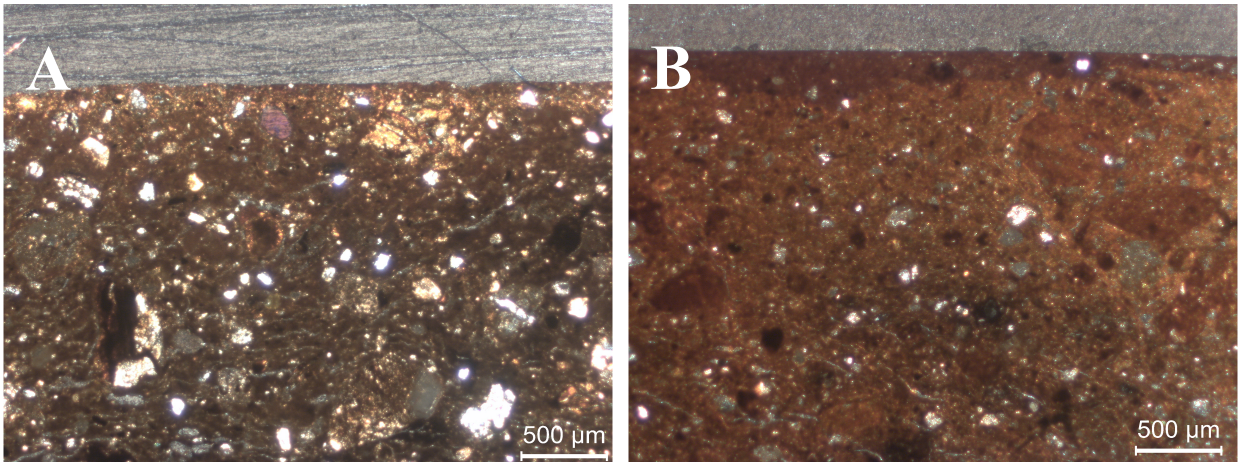


Figure 9 XPL petrographic images of examples of surface treatments in the sedimentary group; (A) 117_a and (B) 294_a.

6.1.3 Felsic Group

This group is the largest group comprising 26 out of the 43 samples (60% of the assemblage). The felsic group is characterized by the abundance of large felsic lithoclasts composed of quartz and feldspar grains. These lithoclasts probably range between granite and granodiorite. Although small variations exist between mineral abundances in this group, there is no significant geological variation which can be discerned regarding differential sources of raw materials. The paste is characterized by a high frequency of amphiboles, and scarce or near absence of pyroxenes. The tempers are categorized as angular and subangular and show minimal weathering. The matrix presents a high temper/clay proportion ranging between 30-40%. The temper sorting is poor due to the apparent lack of sieving (Figure 10).

6.1.4 Group Anomalies

The sample 294_e and 294_a from the sedimentary group from Tibes have grog, pieces of crushed ceramics, added as temper (Figure 11). The large, angular inclusions present are the only examples within the ceramic assemblage studied, although this tempering agent has been evidenced frequently in the Caribbean (Ting et al., 2016). The main visual differences between sedimentary rocks and grog were based on the angularity of the temper.

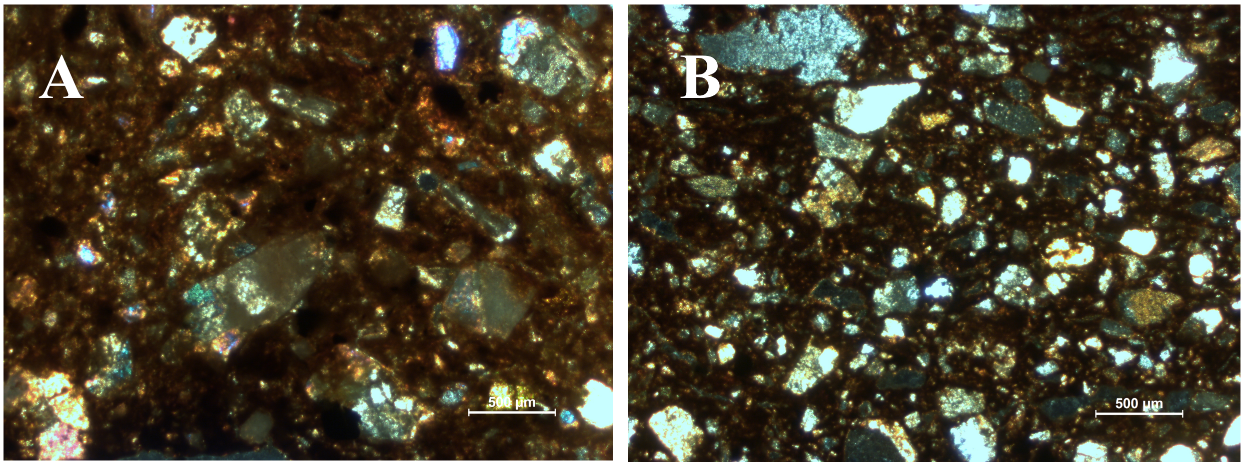


Figure 10 XPL petrographic images of examples of the felsic group, (A) FS.263_a and (B) FS.76_e.

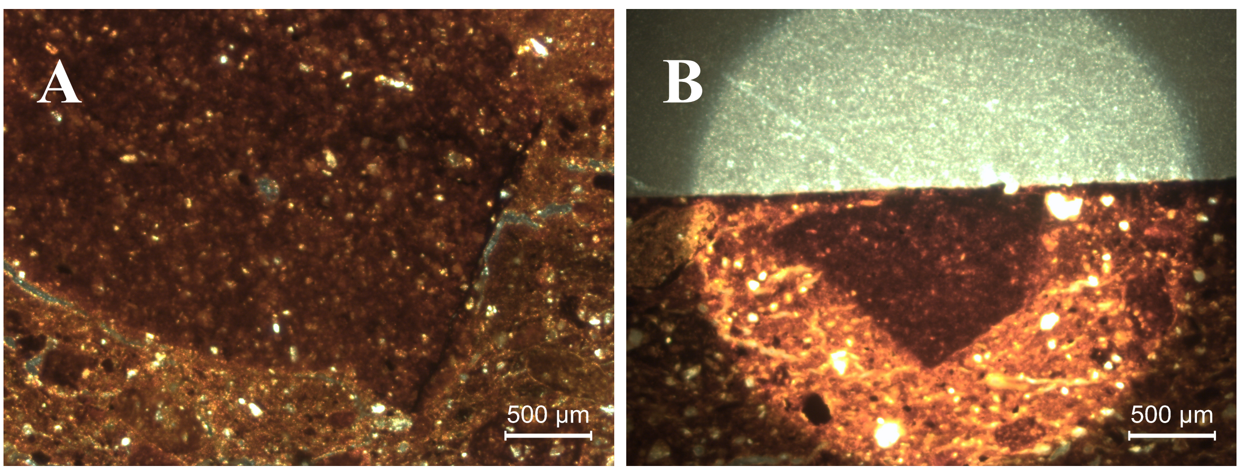


Figure 11 XPL petrographic images of examples of grog temper, (A) 294_e and (B) 294_a.

6.1.5 Textural Analysis

The temper sizes, which ranges from fine (126-250 µm), medium (251-500 µm), medium-coarse (51-1 µm) until coarse (1.1-2µm), were studied using the ImageJ software. The temper size distribution, on the scale developed by Torres (2012), can be seen in Figure 12a and 12b. Temper size helps to distinguish between style and function in the Caribbean and therefore is a productive parameter to compare to mineralogical and chemical data (Torres, 2012). The main temper size differences are between the volcanic/sedimentary groups and the felsic groups. The volcanic and sedimentary groups appear to have more fine tempers, while the felsic group has more medium sized temper.

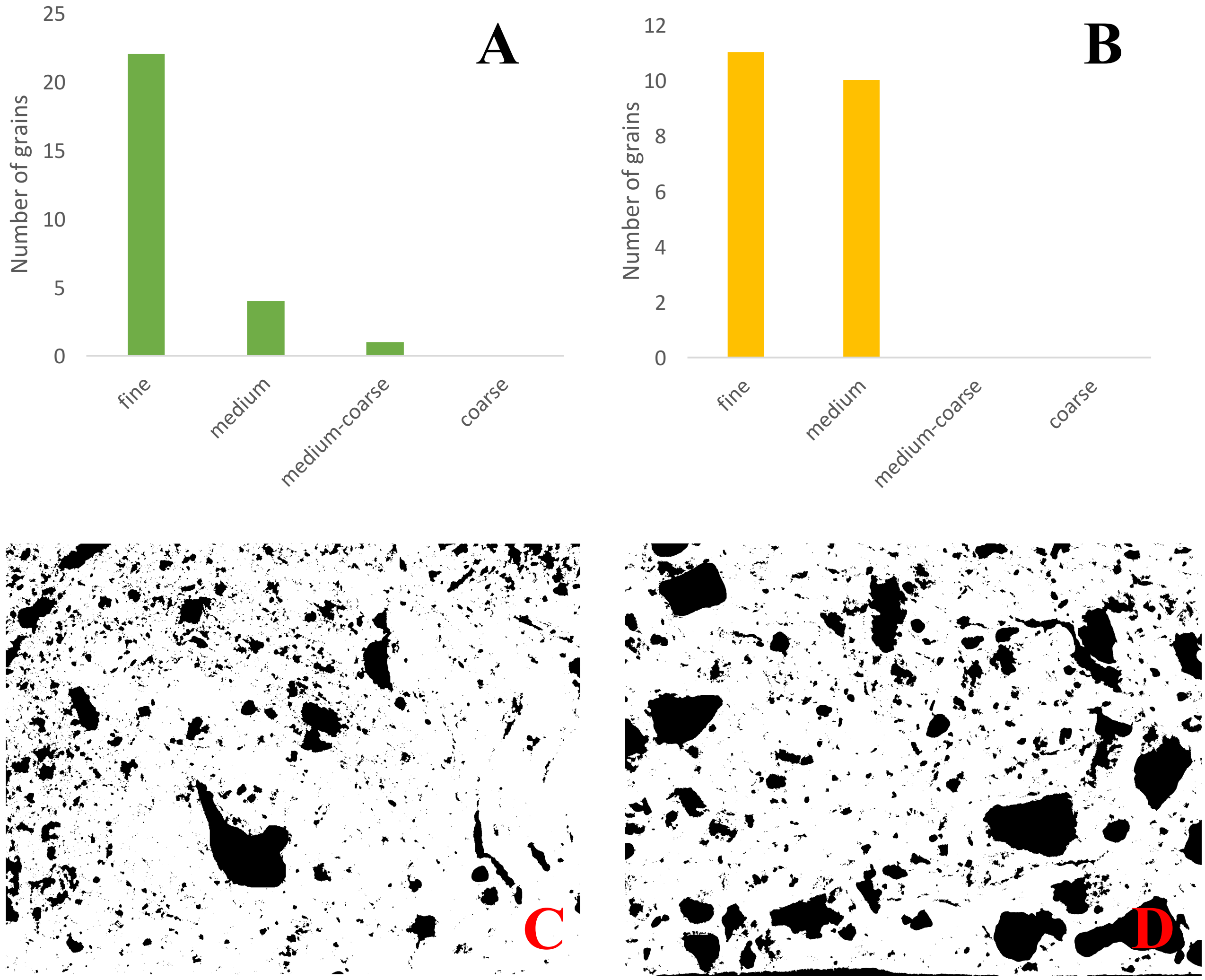


Figure 12 Textural analysis using Image J software on the size scale developed by Torres (2012); (A,C) sample 62_d which is representative of the volcanic group (and a similar texture to the sedimentary group); (B,D) FS.232_a, representative of the felsic group.

6.1.6 Sherd Thickness

Another parameter which contributes to the identification of function and style in the Caribbean is thickness. Specifically, *burens,* a type of cooking vessel found mostly in domestic settings, is distinguished by its signature thickness above 11 mm (Torres, 2012). This thesis follows Torres's (2012) thickness categories to assess the intrasite and intersite variability. The average thickness by petrographic group can be seen in Figure 13. It appears that the volcanic and sedimentary groups are thinner ceramics, while the felsic group has thicker sherds.

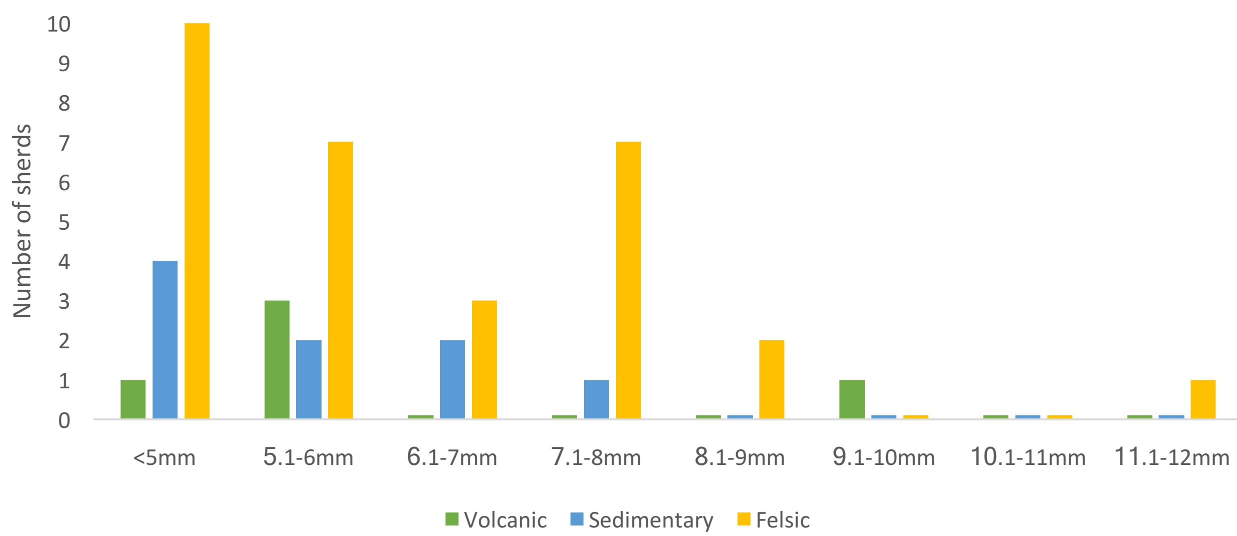


Figure 13 Sherd thickness by petrographic group in terms of number of sherds.

6.2 X-Ray Diffraction (XRD) Results

To determine the phase composition within the ceramics, XRD analysis was conducted on all 50 samples. The mineralogical composition is used to establish provenance groups, and to refine the initial petrographic divisions. The amount of quartz, plagioclases, K-feldspars and amphiboles plays a significant role in distinguishing between mineralogical groups. The results confirm the initial grouping done through petrographic analysis, albeit with some further division within the felsic group. Therefore, to distinguish between the petrographic groups and the groups refined through XRD and further through XRF, new group names are used:

- Group 1a corresponds to the volcanic group;
- Group 1b to the sedimentary group;
- Group 2a and 2b is the felsic group (Table 8).

The reasons behind the division of the felsic group are discussed below. This will aid in understanding the chemical data and the different sources potters were using in antiquity.

This section will compare the averages of each mineralogical group and the sediment samples using the reference intensity ratio (RIR) semi-quantitative analysis of XRD results (Table 9 and Figure 14). The main mineral phases encountered are quartz (SiO_2), feldspars ($KAlSi_3O_8$–$NaAlSi_3O_8$–$CaAl_2Si_2O_8$), muscovite ($KAl_3Si_3O_{10}(OH)_2$), talc ($Mg_3Si_4O_{10}(OH)_2$), amphiboles ($(Ca,Na,K)(Mg,Fe,Al)(Si,Al)O_{20}(OH)_2$), pyroxenes $(Ca,Na,Mg,Fe,Al)_2(Si,Al)_2O_6$) and clay minerals. The specific clay minerals identified using oriented mineral aggregate mounts are discussed below.

Table 7 Equivalence between petrographic and XRD/XRF groups.

Petrographic Group	XRD/XRF Group
Volcanic	1a
Sedimentary	1b
Felsic	2a
	2b

6.2.1 Mineralogical Group 1a

Mineralogical group 1a shows an average of 11.1% of quartz, 51.6% of plagioclase, 15.6% of K-feldspar, 5.8% of muscovite, 2.8% of talc, 0.5% of amphibole, 10.3% of pyroxene, and 1.9% of clay mineral, and corresponds to the volcanic petrographic group. Group 1a is characterized by a relatively high abundance of plagioclase, K-feldspars, talc, pyroxenes and clay minerals (See Figure 15a for a typical diffractogram). The high plagioclase and feldspars are associated with low values of quartz, and amphiboles. This indicates a more mafic composition of the aplastic inclusions within the ceramics.

6.2.2 Mineralogical Group 1b

Group 1b shows an average of 35.5% of quartz, 35.5% of plagioclase, 12.1% of K-feldspar, 6.8% of muscovite, 1.2% of amphibole, 5.6% of pyroxenes and no clay minerals, and corresponds to the sedimentary petrographic group. Group 1b is characterized by a high amount of quartz, talc, muscovite and pyroxenes (Figure 15b for a typical diffractogram). Therefore, although it has a similar matrix as group 1a (volcanic), the values of quartz and feldspars vary greatly.

6.2.3 Mineralogical Group 2a

Group 2a shows 26.2% of quartz, 39.7% of plagioclase, 12.4% of K-feldspar, 4.0% of muscovite, 1.1% of talc, 9.8% of amphibole, 1.5% of clay mineral, and 4.9% of pyroxene, and corresponds to the majority of the samples within the felsic group. Group 2a is characterized by a relatively high amount of amphibole and quartz, and a low amount of plagioclase and K-feldspar (See Figure 15c for a typical diffractogram).

6.2.4 Mineralogical Group 2b

Group 2b shows an average of 12.7% of quartz, 57.5% of plagioclase, 18.2% of K-feldspar, 1.1% of muscovite, 0.8% of talc, 0.9% of amphibole, and 7.2% of pyroxene and corresponds to 4 samples from the felsic group. Group 2b is characterized by a relatively high amount of plagioclase, and K-feldspar (See Figure 15d for a typical diffractogram). Compared to group 2a, there is less quartz, and more plagioclase and K-feldspar

6.2.5 Sediment Samples

The sediment samples have a varying amount of quartz, plagioclase, K-feldspar, muscovite, amphibole, pyroxene and clay mineral (See Table 10). The absence and presence of amphiboles could be an indicator of provenance, and is confirmed via chemical analysis. The amount of quartz ranges between 34.6% and 43.3% with the red clay sample having the lowest amount and the finca feliciana sample having the highest. Plagioclase is between 34.3% (mineral) and 15.3% (río chiquito), muscovite is absent in 4 samples (finca feliciana, horse pasture, gongolones, and red clay), and present in 3 samples (mineral, mineral NW, río chiquito).

6.2.6 Oriented Mineral Aggregates

The clay minerals were identified by XRD via oriented mineral aggregate mounts. The main clay minerals identified is montmorillonite, although some samples have only vermiculite. The clay minerals were identified through the USGS mineral identification flow diagram by comparing the different responses to the treatments discussed in Chapter 5 (Poppe et al., n.d.). As seen in Figure 16, the clay mineral peak appears at approximately at 14Å in the untreated XRD measurement. After the ethylene glycol treatment, the peak shows an expansion to 17Å. The peak collapses to 10Å after the first heat treatment (400°C), and there is no change after the second heat treatment (550°C). This suggests that 62_d (group 1a), 62_f (group 2a), 62_j (group 2a), 294_c (group 2a), FS.76_d (group 2a), FS.76_f (group 2b) have montmorillonite. In contrast, samples FS.76_e and FS.232_a (group 2a) have vermiculite see Figure 17. The clay mineral peak appears at 14 Å when untreated and remains there after the ethylene glycol treatment. Once the first heating treatment was completed, the clay mineral peak collapses to 10 Å. After the second heat treatment, there is no change. The sediment samples were also studied under oriented aggregate mounts. In the 4 samples (horse_pasture, la_mineral, gongolones, and río_chiquito), there is evidence of montmorillonite and possibly of kaolinite. Kaolonite has an untreated peak at 7Å, which remains unchanged after ethylene glycol treatment and during the first heat treatment, but collapses during the second heat treatment (Figure 18).

Table 8 RIR XRD results from all samples organized by mineralogical group and expressed in percentage.

Group	Sample ID	Site	Qtz	Pl	KFs	Mu	Tlc	Amp	Clay	Cpx	Mgt [14]	Total
1a	117_c	Tibes	12.9	45.7	16.7	6.5	2.8	3.5	2.7	9.2	0.0	100
	117_f	Tibes	12.0	54.7	20.3	0.0	0.0	0.0	0.0	11.8	1.2	100
	117_h	Tibes	26.3	39.6	12.6	6.5	2.9	3.8	0.0	8.3	0.0	100
	294_d	Tibes	10.7	47.4	20.4	5.9	2.6	0.0	0.0	13.0	0.0	100
	294_i	Tibes	6.4	52.3	22.1	7.2	4.8	0.0	0.0	7.3	0.0	100
	62_d	Tibes	18.5	45.3	6.0	12.4	5.0	0.0	5.4	7.5	0.0	100
	62_e	Tibes	6.4	62.6	8.3	3.2	2.1	0.0	3.4	13.1	1.0	100
1b	117_a	Tibes	28.3	39.6	15.3	3.4	3.1	0.0	0.0	10.5	0.0	100
	294_a	Tibes	26.5	40.7	13.7	9.9	4.5	0.0	0.0	4.7	0.0	100
	294_b	Tibes	33.9	38.9	12.1	5.2	2.8	2.8	0.0	4.3	0.0	100
	294_e	Tibes	22.2	40.5	14.4	11.0	4.1	0.0	0.0	7.8	0.0	100
	FS.257_a	PO-48	46.7	27.8	12.8	3.2	0.0	3.3	0.0	6.2	0.0	100
	FS.75_a	PO-42	52.7	27.4	8.9	7.0	4.1	0.0	0.0	0.0	0.0	100
	FS.76_h	PO-42	47.7	30.4	7.2	8.2	3.0	0.0	0.0	3.5	0.0	100
2a	117_b	Tibes	17.6	61.2	9.0	0.0	0.0	7.4	0.0	4.9	0.0	100
	117_d	Tibes	25.3	42.6	10.3	0.0	1.1	14.3	0.0	6.4	0.0	100
	117_e	Tibes	24.6	45.1	4.8	5.7	0.0	10.2	0.0	9.6	0.0	100
	117_i	Tibes	22.4	36.3	13.7	6.5	3.7	9.5	0.0	7.9	0.0	100
	294_c	Tibes	39.0	32.0	16.1	8.3	1.4	2.0	1.3	0.0	0.0	100
	294_f	Tibes	33.6	43.8	9.7	7.4	0.8	4.8	0.0	0.0	0.0	100
	294_g	Tibes	23.2	47.3	11.2	6.4	0.7	5.9	1.2	4.1	0.0	100
	294_h	Tibes	33.8	36.8	9.2	11.3	1.9	3.0	1.1	2.9	0.0	100
	294_j	Tibes	29.3	37.3	16.1	3.6	1.6	6.1	3.5	2.4	0.0	100
	62_a	Tibes	40.2	33.6	7.5	0.0	0.0	3.0	5.9	9.8	0.0	100
	62_c	Tibes	42.7	28.1	10.5	8.5	0.0	4.8	2.2	3.2	0.0	100
	62_f	Tibes	34.7	37.9	5.4	8.6	0.0	3.7	2.2	7.5	0.0	100
	62_g	Tibes	12.1	34.8	12.1	5.9	1.5	17.0	1.6	15.0	0.0	100
	62_h	Tibes	23.1	24.2	8.0	0.0	1.3	26.0	7.1	9.7	0.5	100
	62_i	Tibes	30.3	33.9	13.8	0.0	0.0	6.7	4.4	10.9	0.0	100
	62_j	Tibes	30.7	27.1	4.7	0.0	1.3	11.5	14.0	10.9	0.0	100
	FS.232_a	PO-43	30.7	37.0	14.2	7.0	0.9	5.2	1.5	3.6	0.0	100
	FS.232_b	PO-43	25.4	48.4	14.4	0.0	1.7	5.6	2.6	2.1	0.0	100
	FS.245_b	PO-43	21.9	28.7	16.6	4.7	2.2	20.9	1.9	3.2	0.0	100
	FS.246_a	PO-43	34.5	37.5	11.7	4.2	1.9	8.3	0.0	1.9	0.0	100
	FS.246_b	PO-43	24.1	29.4	14.2	4.2	2.0	19.4	2.5	4.2	0.0	100
	FS.253_a	PO-48	19.1	36.3	16.6	6.1	1.2	16.7	0.0	4.1	0.0	100
	FS.255_a	PO-48	31.0	26.5	7.8	3.2	1.7	27.1	0.0	2.7	0.0	100
	FS.263_a	PO-48	6.8	49.7	20.1	3.1	1.4	14.6	1.6	2.8	0.0	100
	FS.75_b	PO-42	17.4	47.7	9.9	4.5	1.1	17.6	0.0	1.7	0.0	100
	FS.76_a	PO-42	35.3	32.3	18.5	5.3	0.9	3.2	0.0	4.4	0.0	100
	FS.76_b	PO-42	27.2	38.6	15.4	0.0	0.0	14.1	0.0	4.6	0.0	100
	FS.76_c	PO-42	35.9	29.5	11.8	4.6	1.0	14.3	1.1	1.9	0.0	100
	FS.76_d	PO-42	9.4	63.2	16.2	4.4	1.8	0.0	1.9	3.2	0.0	100
	FS.76_e	PO-42	40.3	37.0	10.6	0.0	1.7	3.6	3.2	3.7	0.0	100
	FS.76_g	PO-42	28.6	45.6	9.8	3.8	0.0	8.6	0.0	3.6	0.0	100
2b	117_g	Tibes	3.7	51.9	23.8	6.9	4.3	0.0	0.0	9.4	0.0	100
	117_j	Tibes	10.2	45.5	34.1	2.8	1.7	2.4	0.0	3.3	0.0	100
	62_b	Tibes	15.5	63.8	7.5	0.0	0.0	0.0	0.0	12.5	0.8	100
	FS.245_a	PO-43	15.4	51.5	21.0	3.4	2.5	2.7	0.0	3.5	0.0	100
	FS.76_f	PO-42	7.3	57.6	25.6	0.0	0.0	0.0	3.9	5.7	0.0	100

[14] Mgt = Magnetite.

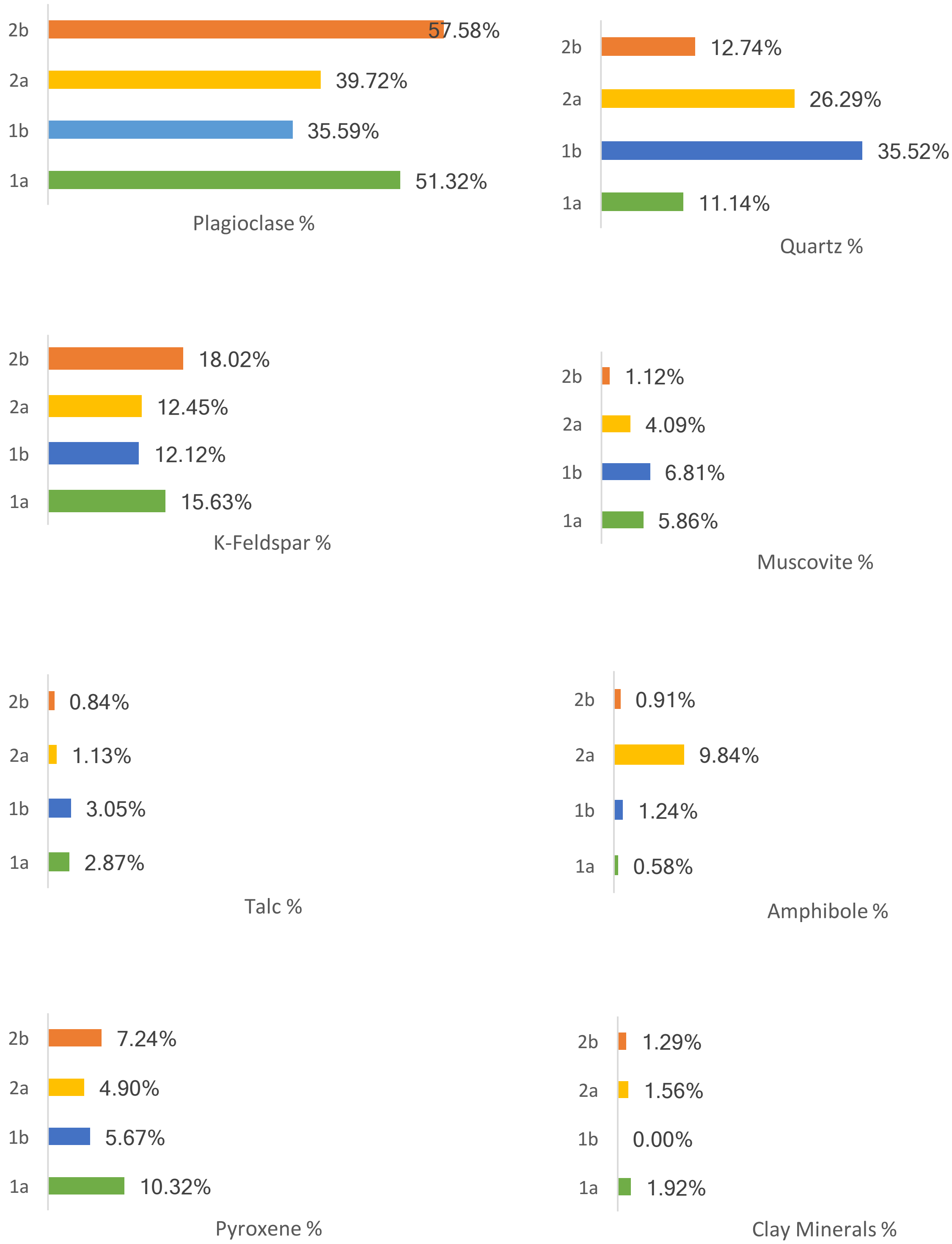


Figure 14 Comparison of RIR XRD semi-quantitative analysis by mineralogical groups.

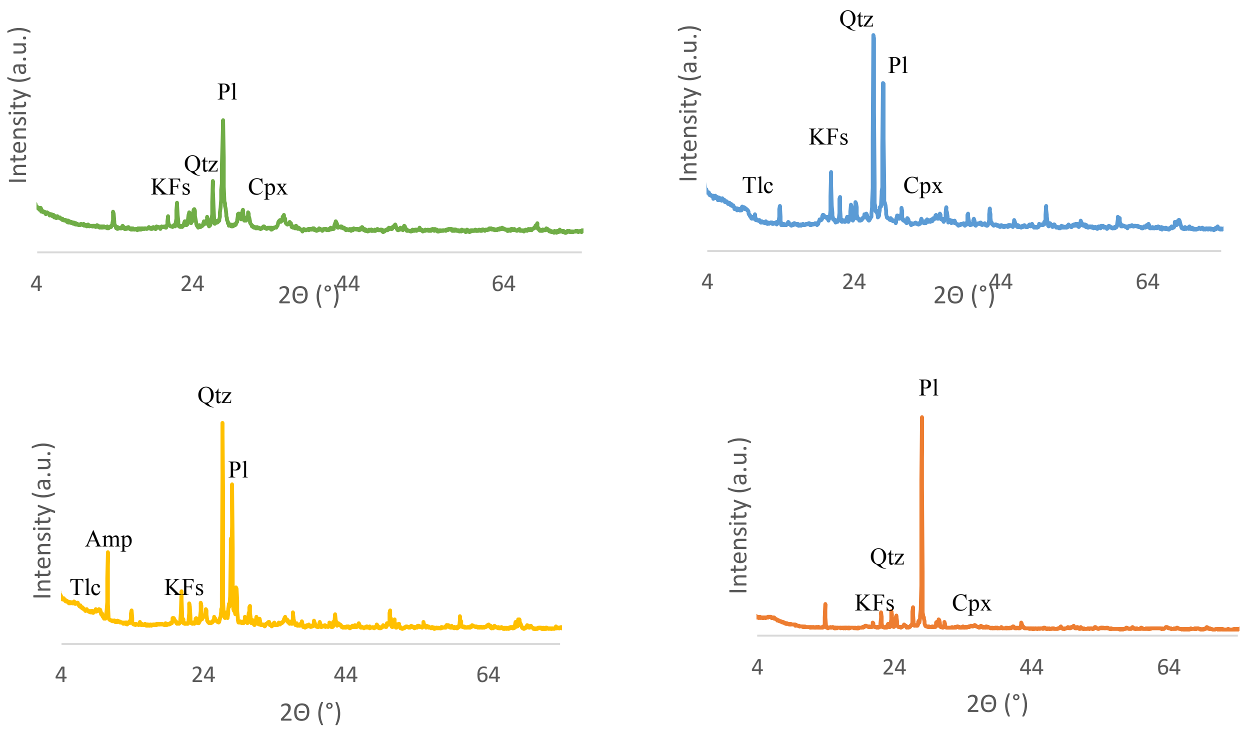


Figure 15 (A) group 1a (sample 117_f) (B) group 1b (sample 294_b) (C) group 2a (sample FS.245_b) (D) group 2b (sample FS.76_f).

Table 9 Sediment samples semi-quantitative analysis XRD expressed in percentage.

Site	Sample ID	Qtz	Pl	Kfs	Mu	Amp	Mnt	Vrm	Cpx	Total
PO-52	Finca	43.4	32.6	9.0	0.0	0.0	5.9	0.0	9.2	100
PO-50	Horse_pasture	38.6	26.6	13.0	0.0	0.0	13.9	7.9	0.0	100
PO-42	La_mineral	36.8	34.4	8.5	7.5	4.5	1.5	0.0	6.9	100
PO-42	La_mineral_NW	40.7	22.1	10.0	17.0	4.5	1.2	0.0	4.4	100
PO-43	Los_gongolones	37.0	27.9	8.5	0.0	0.0	8.6	10.2	7.8	100
N/A	Red_clay	34.7	33.2	4.8	0.0	0.0	22.1	0.0	5.3	100
PO-48	Rio_chiquito	42.5	15.3	5.3	31.4	0.0	2.2	0.0	3.2	100

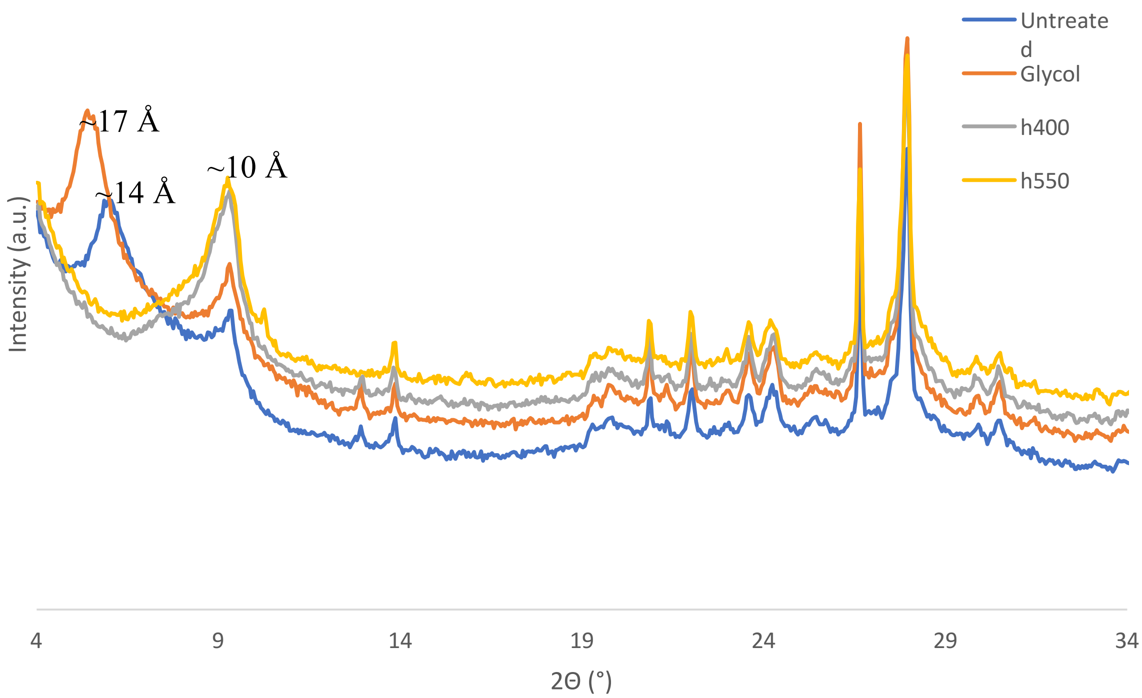


Figure 16 Typical example of a clay mineral diffractogram of montmorillonite (sample 62_d).

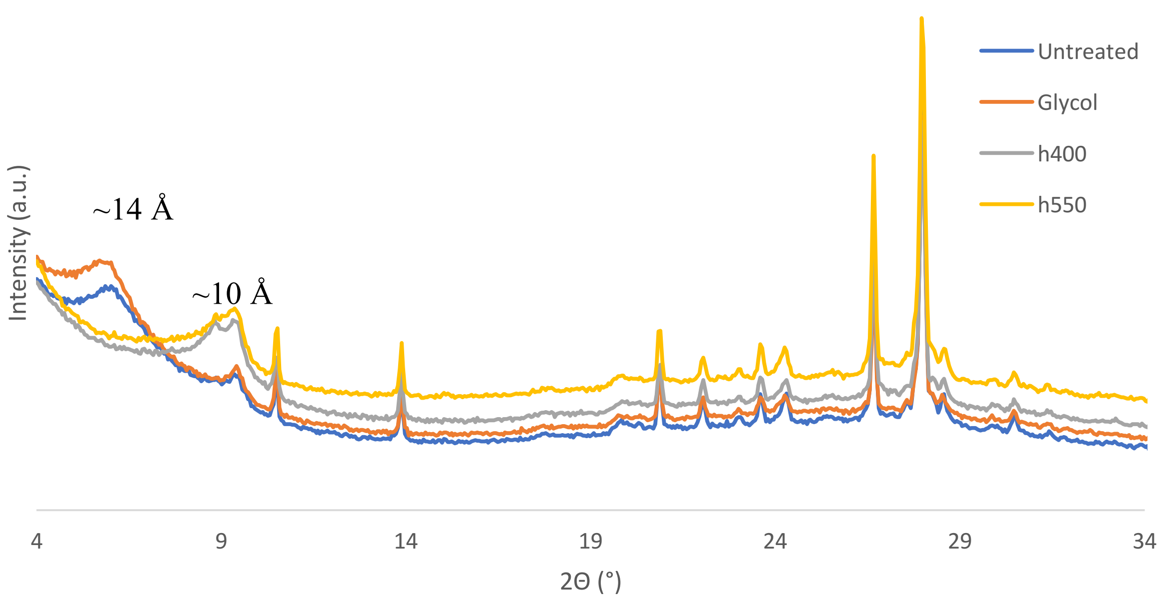


Figure 17 Typical clay mineral diffractogram of vermiculite (sample FS.76_e).

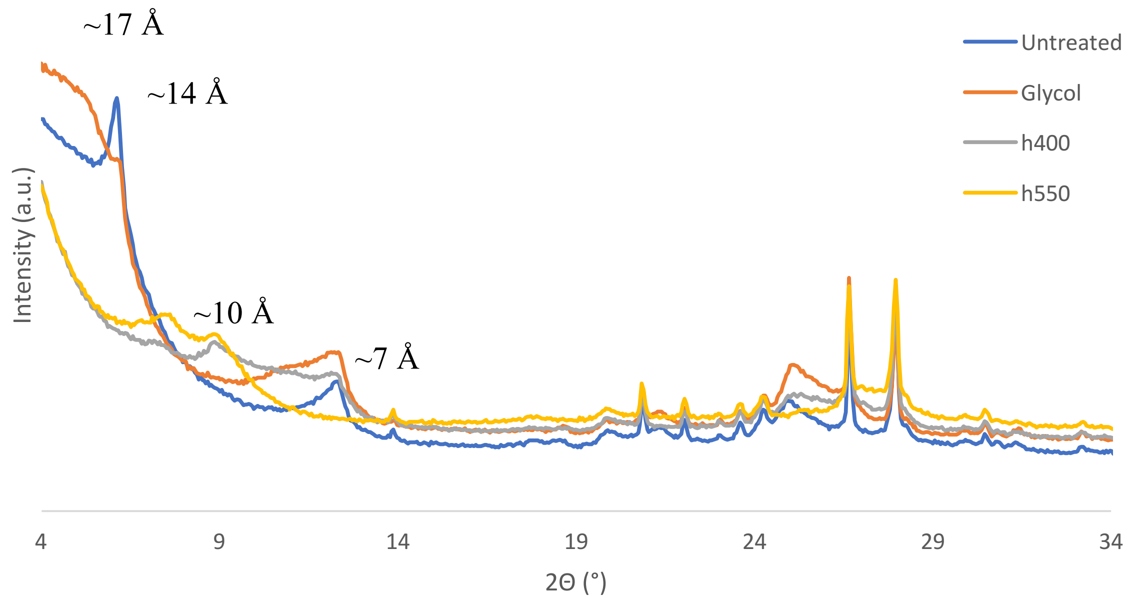


Figure 18 Typical diffractogram of the sediment samples showing vermiculite and kaolinite (Los_gongolones).

6.3 SEM-EDS

SEM-EDS analysis was conducted on 18 representative samples (36% of the assemblage). The samples were chosen based on mineralogical group and archaeological context. The goal of the SEM-EDS analysis is to provide information about the chemical composition of the minerals and the matrix, and to identify opaque minerals which cannot be distinguished through petrographic analysis. The main minerals identified in SEM-EDS analysis are quartz, feldspars, amphiboles, pyroxenes, iron oxides, calcium titanium silicate.

The feldspars, amphiboles and pyroxenes were identified via point microanalysis. The feldspars show high variability. Mineralogical group 1a and 1b are mainly defined by K-feldspars, sodic feldspars (albite) and calcic feldspars (bytownite and anorthite) (See Figure 19 for ternary diagram). Mineralogical group 2a and 2b are mainly defined by K-feldspars, and sodic feldspars (albite and oligoclase), with a notable absence of calcic feldspars (See Figure 20 for relative abundances). Thus, group 1a and 1b are defined by both sodic and calcic plagioclase feldspars, while group 2a and 2b having predominantly sodic plagioclase feldspars. The amphiboles, almost exclusively present in group 2a and 2b, are associated to actinolite ($Ca_2(Mg_{4.5-2.5}Fe^{2+}_{0.5-2.5})Si_8O_{22}(OH)_2$) (Figure 21 for ternary diagram). No significant variation in the composition of amphiboles was observed. The pyroxenes are correlated to hedenbergite ($CaFeSi_2O_6$) and augite $(Ca,Na)(Mg,Fe,Al,Ti)(Si,Al)_2O_6$ (See Figure 22 for ternary diagram). The chemical composition of the matrix shows Si, Al, Fe, Na, Ca, K, and Ti across all samples with little variation.

Below are representative examples from the SEM-EDS for identifying minerals. Sample 294_d from Group 1a (volcanic) shows anorthoclase crystals within a volcanic rock (See Table 11a and Figure 23a and 23b). Sample 62_h (group 2a) exhibits an actinolite grain (See Table 11b and Figure 23c and 23d). Sample 294_i (group 1a) has hedenbergite/augite part of a volcanic rock inclusion (See Table 12a and Figure 24a and 24b). Sample 62_f (group 2a) has sphene, a calcium titanium silicate, part of an igneous rock inclusion (See Table 12b and Figure 24c and 24d). Therefore, by understanding the mineral components of the rock inclusions, we can narrow down the possible rock formations in the local geology.

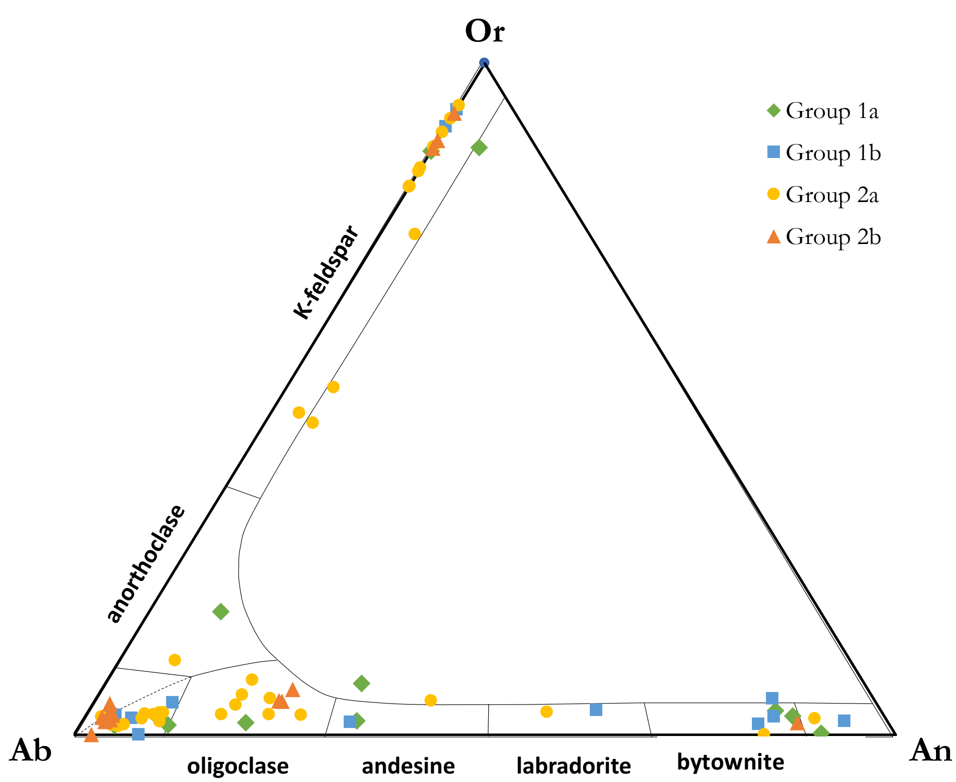


Figure 19 Ternary diagram of feldspars by mineralogical group.

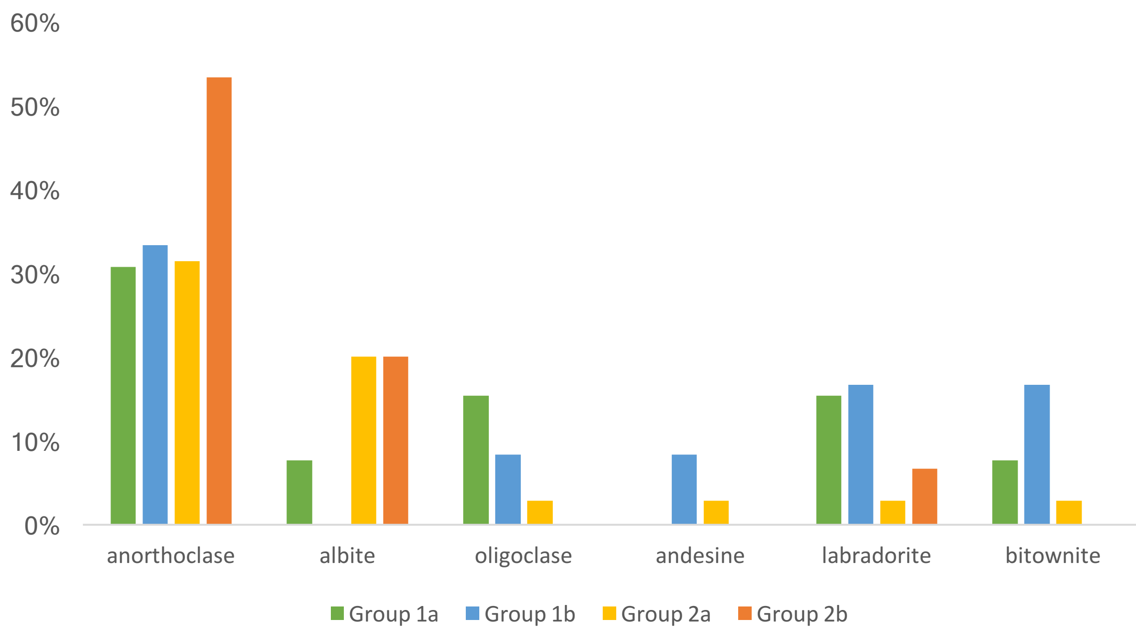


Figure 20 Plagioclase feldspar distribution by mineralogical group.

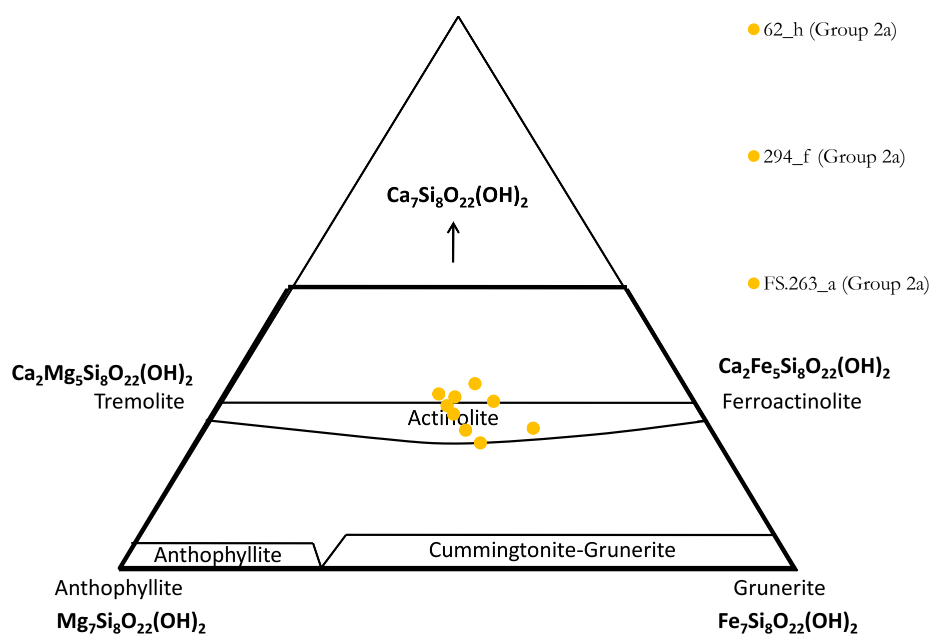


Figure 21 Ternary diagram of amphiboles from group 2a.

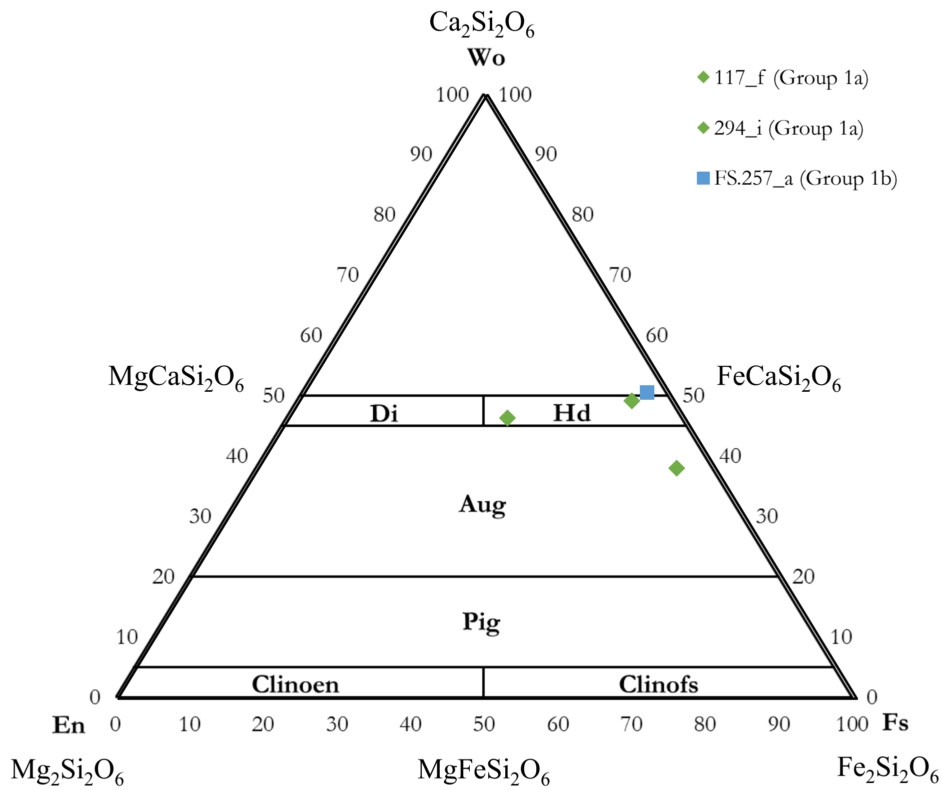


Figure 22 Ternary diagram of pyroxenes from groups 1a and 1b.

6.4 ED-XRF & ICP-MS

The chemical analysis showed a high level of homogeneity across samples. Nevertheless, there is still visible correlations between major, minor and trace elements. These chemical variations will be used to understand any distinctive groups of raw material provenances being employed at Tibes and the sites in the region. Below is a summary of results of both XRF and ICP-MS analysis and the major elemental variations by mineralogical group.

6.4.1 Major Elements (ED-XRF)

The results from the major elements obtained via XRF are expressed in oxides in wt. % (See Table 13 for an average by mineralogical group). Across the major elements (Na_2O, MgO,

Table 10 (A) Results by at.% of point analysis of anorthoclase (sample 294_d). (B) Results by at. % of point analysis of actinolite (62_h).

(A) Anorthoclase

Element	[norm. at.%]
Oxygen	60.1
Sodium	5.6
Magnesium	0.2
Aluminum	8.2
Silica	23.1
Chlorine	0.1
Potassium	1.4
Calcium	0.6
Iron	0.3
Total	100

(B) Actinolite

Element	[norm. at.%]
Oxygen	61.7
Sodium	2.2
Magnesium	7.5
Aluminum	4.5
Silica	13.1
Chlorine	0
Potassium	0.6
Calcium	0.6
Iron	3.8
Total	100

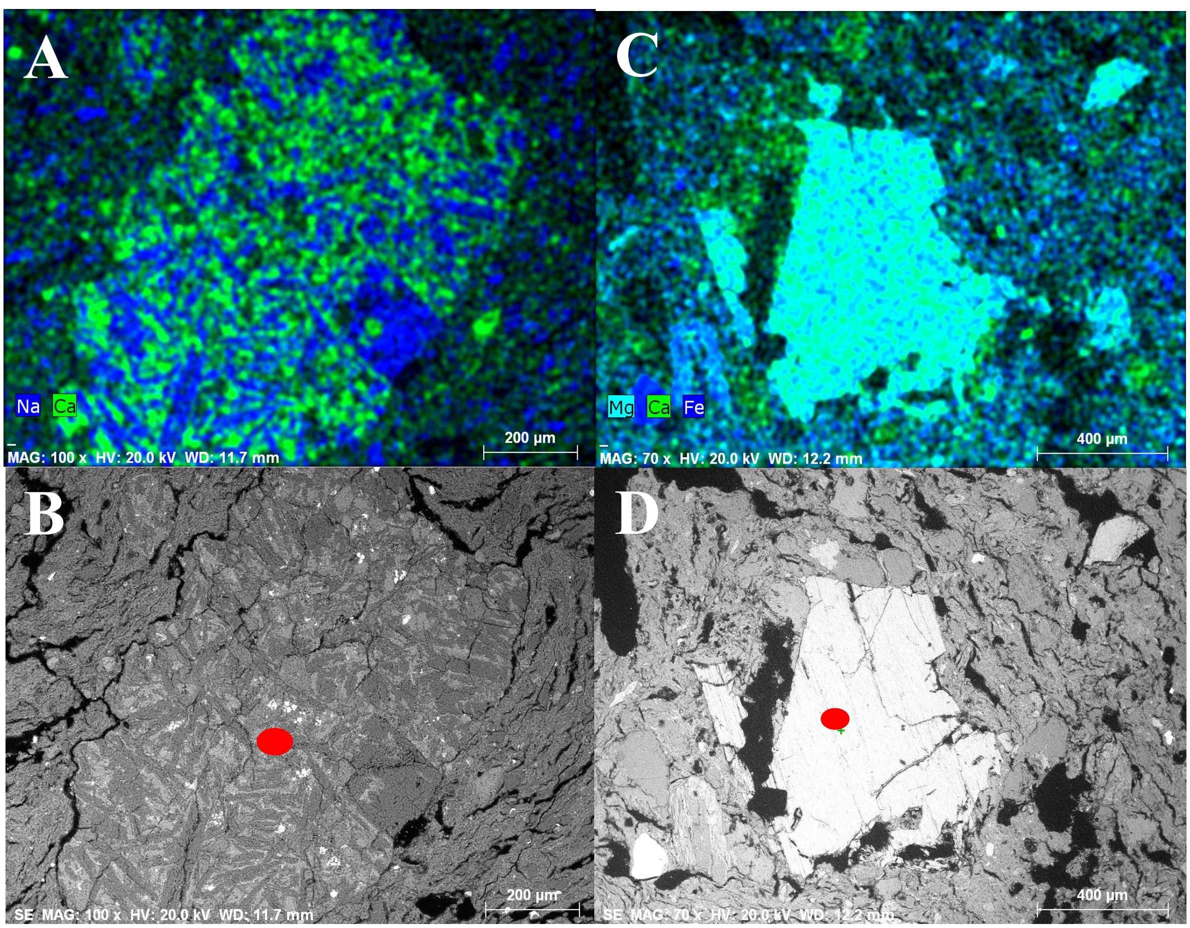


Figure 23 (A) 294_d multi-elemental map of Na (blue) and Ca (green). (B) SE image of anorthoclase point analysis. (C) 62_h multi-elemental map of Mg (turquoise), Ca (green), Fe (blue). (D) SE image of point analysis of actinolite.

Table 11 (A) Results by at.% of point analysis of hedenbergite/augite (sample 294_i). (B) Results by at. % of point analysis of sphene (62_f).

(A) Hedenbergite/Augite

Element	[norm. at.%]
Oxygen	59.
Sodium	0.
Magnesium	1.
Aluminum	7.
Silica	16.
Chlorine	0.0
Potassium	0.0
Calcium	5.9
Titanium	1.4
Iron	6.3
Total	100

(B) Sphene

Element	[norm. at.%]
Oxygen	65.3
Sodium	0.9
Magnesium	0.4
Aluminum	1.2
Silica	10.7
Chlorine	0.0
Potassium	0.0
Calcium	10.3
Titanium	10.5
Iron	0.4
Total	100

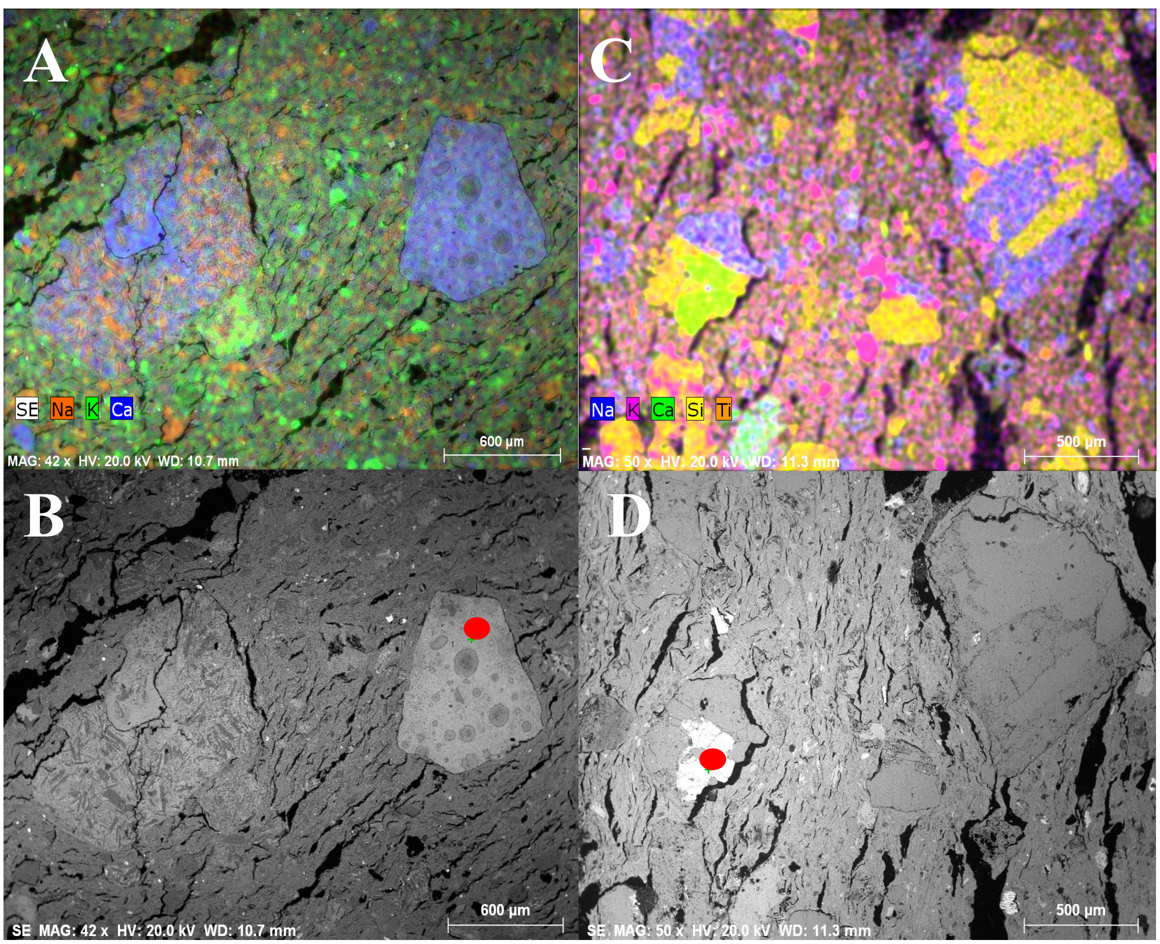


Figure 24 (A) 294_i multi-elemental map of Na (red), K (green), Ca (blue). (B) SE image of hedenbergite/augite point analysis. (C) 62_f multi-elemental map Na (blue), Ca (green), Si (yellow), Ti (orange). (D) SE image of point analysis of sphene.

Al_2O3, SiO_2, K_2O, CaO, TiO_2, Fe_2O_3) there appears to be little variation. Group 1a shows slightly higher values of MgO (3.4%) and CaO (4.4%), and slightly lower values in SiO_2 (51.8%) and Al_2O_3 (18.7%). Group 1b has higher values in SiO_2 (56.4%) and lower values in Na_2O (2.2%). Al_2O_3 (18.3%), and TiO_2 (0.82%). Group 2a shows slightly low values in Fe_2O_3 (7.73%). Group 2b shows high Na_2O (4.9%), TiO_2 (1.6%), and Fe_2O_3 (10.9%), and low values in MgO (1.1%). These minor variations of the major elemental compositions can sometimes be attributed to the vagaries of sampling or can show significant differences in the sample mean populations. Below, the significance results of ANOVA ($p < .05$) will be discussed.

There is a significant difference between the values of Na_2O between group 1a and 2b ($p = 0.00282$), group 1b and 2b ($p = 0.001$) and group 2a and 2b ($p = 0.00119$). Therefore, group 2b shows higher values of Na_2O. In terms of MgO, there are differences between groups 1a and 1b ($p = .00384$), 1a and 2b ($p = 0.001$), and 1a and 2b ($p = 0.001$). This confirms that the group 1a has a higher amount of MgO than the other mineralogical groups. Al_2O_3 shows variances between groups 1a and 2a ($p = 0.00824$), and 1b and 2a ($p = 0.00130$). SiO_2 and K_2O show no variations in the data, and therefore are not useful for identifying provenance groups. CaO shows differences between all mineralogical groups except for 1b and 2b. Specifically, the difference between 1a and 1b ($p=0.001$), 1a and 2a ($p=0.001$), 1a and 2b ($p=0.001$), 1b and 2a ($p=0.0189$), and 2a and 2b ($p=0.0231$). There is no apparent difference between groups 1a and 1b regarding TiO_2. Yet, there is a notable difference between group 1a and 2b ($p = 0.001$), 1b and 2a ($p=.00518$), 1b and 2b ($p=0.001$), and 2a and 2b ($p=0.001$). Fe_2O_3 appears to have less variation, but there are differences between the values of group 1a and 2b ($p=0.04179$), 1b and 2b (0.02856), and 2a and 2b ($p=0.001$). Therefore, 2b is the only group with a noteworthy difference in Fe_2O_3.

Solely based on the major elements, there is already apparent trends and groupings per mineralogical groups. For example, in the CaO and Na_2O there is a linear relationship with the exception of group 2b, which shows elevated Na_2O (See Figure 25). As the amount of CaO increased, the amount of Na_2O also increases somewhat, which in this case is linked to the amount of plagioclase feldspars in the samples and to an increase of Ca in the plagioclases. In the biplot, groups 1a, 1b and 2b cluster differently, but they have the same Na_2O to CaO ratio. Nevertheless, group 2b has low calcium levels confirming the assumption that group 2a is devoid of calcic feldspars. Since these samples are not composed of calcareous clays and do not have limestone inclusions, we can be confident that the CaO is from the aplastic inclusions.

The Fe_2O_3/ TiO_2 biplot, although the variability by samples is small, there is grouping via mineralogical group (See Figure 26). This biplot shows the distinguished felsic and mafic nature of the iron and titanium oxides compositions. As expected, group 1a and 1b cluster jointly since they both show a mafic composition, more enriched in Fe when compared to Ti. The groups 2a and 2b exhibit higher Ti values, and a high amount of Fe-Ti oxides in 2b. Probably, the hematite (or magnetite) would be the more important iron oxide in 1a and 1b while ilmenite ($FeTiO_3$) is more important in 2a and 2b, and more abundant in 2b.

6.4.2 Minor & Trace Elements (ED-XRF and ICP-MS)

The minor (values $<1\%$) and trace elements (values in ppm) aided in distinguishing possible variations on raw material sources, and contributed to identifying possible provenance within the known geological formations (all values are expressed here in ppm for uniformity). Due to the widespread variation, only the large differences across mineralogical groups will be discussed (See Table 14 for averages per mineralogical group). There appears to be large differences in the values of Zr, Nb, and Ni (See Figures 27 and 28). Group 2a has low values of Zr (19 ppm), while group 1a (71 ppm), 1b (108 ppm) and 2b (85 ppm) have higher values. In Nb, Group 2b (11 ppm) has a higher value than group 1a (4 ppm), 1b (4 ppm) and group 2a (7 ppm). These high field strength elements (HFSE) as other (e.g. Hf, rare-earth elements (REE), Th, U, and Ta) are concentrated in erosion resistant minerals and therefore must be an indicator of the raw materials provenance.

Additionally, Ni shows high values in group 1a (75 ppm), and lower values in group 1b (25 ppm), 2a (13 ppm), and 2b (21 ppm). This underlines the mafic character of the mafic rocks.

Table 12 ED-XRF Quantitative values of major elements in oxide wt.%.

Group	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	TiO_2	Fe_2O_3
1a	2.9	3.5	18.7	51.8	1.2	4.4	0.9	8.9
1b	2.3	2.2	18.4	56.4	1.5	1.9	0.8	8.8
2a	2.9	1.5	20.7	55.0	1.1	2.7	1.1	7.7
2b	4.9	1.3	19.3	53.2	1.3	1.5	1.5	10.5

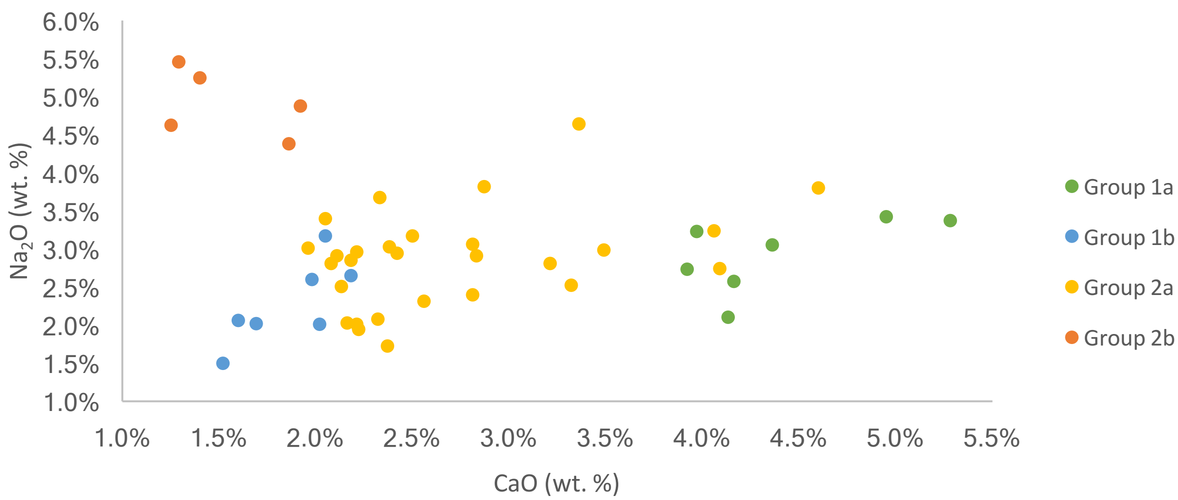


Figure 25 Na_2O/CaO scatter plot by mineralogical group.

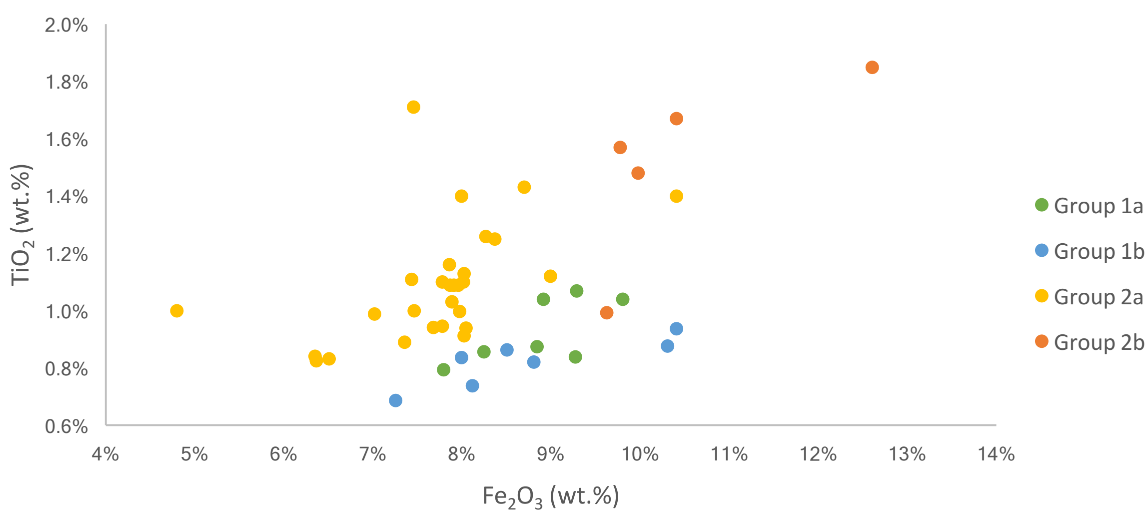


Figure 26 TiO_2/Fe_2O_3 scatter plot by mineralogical group.

Table 13 ED-XRF and ICP-MS quantitative results for minor and trace elements.

Group	Mn	P	S	Rb	Sr	Zr	Nb	Ba	Cr	Co	Ni	Cu	Zn	Ga	As	Sn	Pb	Th	U	V
1a	1042	1430	383	11	248	71	4	473	164	28	75	63	73	15	16	1	3	1	0	207
1b	861	1792	383	14	258	108	4	616	126	18	25	54	75	14	16	1	6	1	1	143
2a	507	1591	397	16	227	19	7	491	71	13	13	28	46	17	16	1	3	2	1	181
2b	871	1848	363	24	237	85	11	665	99	19	21	42	63	17	16	2	3	2	1	237

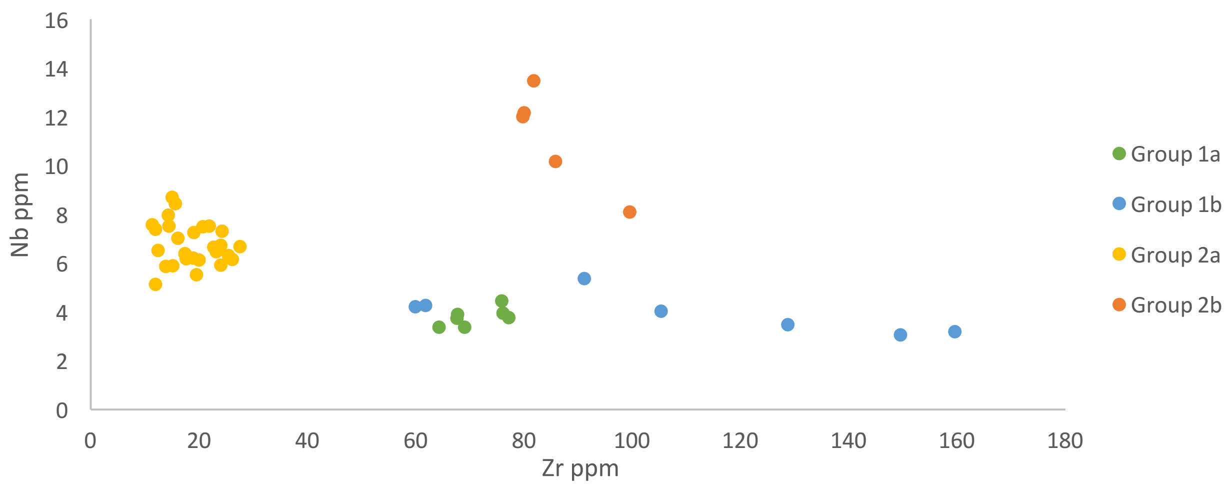


Figure 28 Zr/Nb scatter plot by mineralogical group.

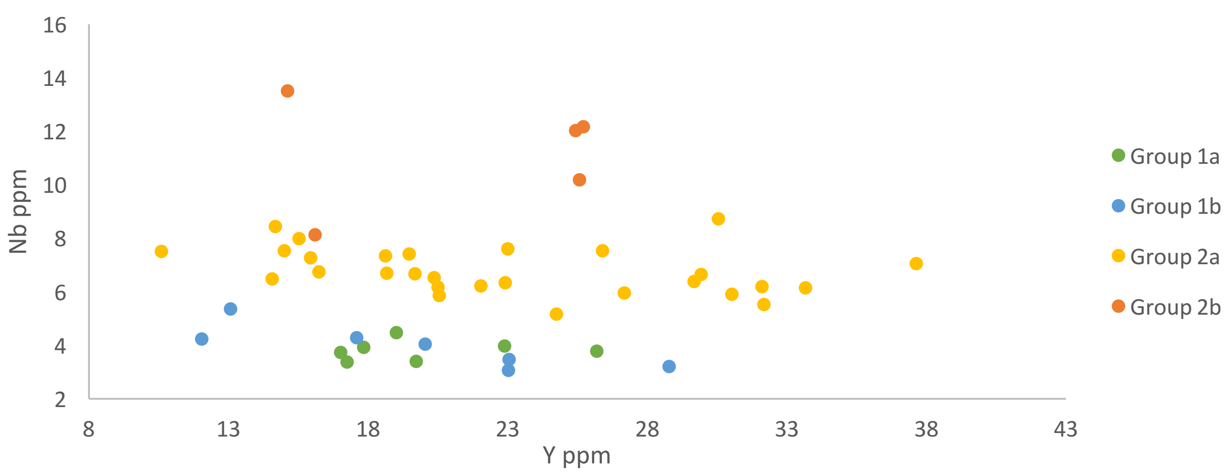


Figure 27 Y/Nb scatter plot by mineralogical group.

6.4.3 REE Spider Diagrams

The REE element spider diagrams were normalized to chondrite (Gromet et al., 1984), and were mainly used to confirm the petrographic and mineralogical groupings. In addition, REE provide higher resolution on differentiation between parent rocks within the groups. Group 1a and 1b show a similar REE element pattern and therefore probably stem from the same parent rock (See Figure 29). The small Ce positive anomaly in 1b is due to its oxidation to Ce^{4+} while the tiny Eu positive anomaly in the group 1a is due to high abundance of Ca-plagioclase that retain Eu^{2+}. Group 2a and 2b appear to have the same pattern, with a small enrichment in LREE in 2b.

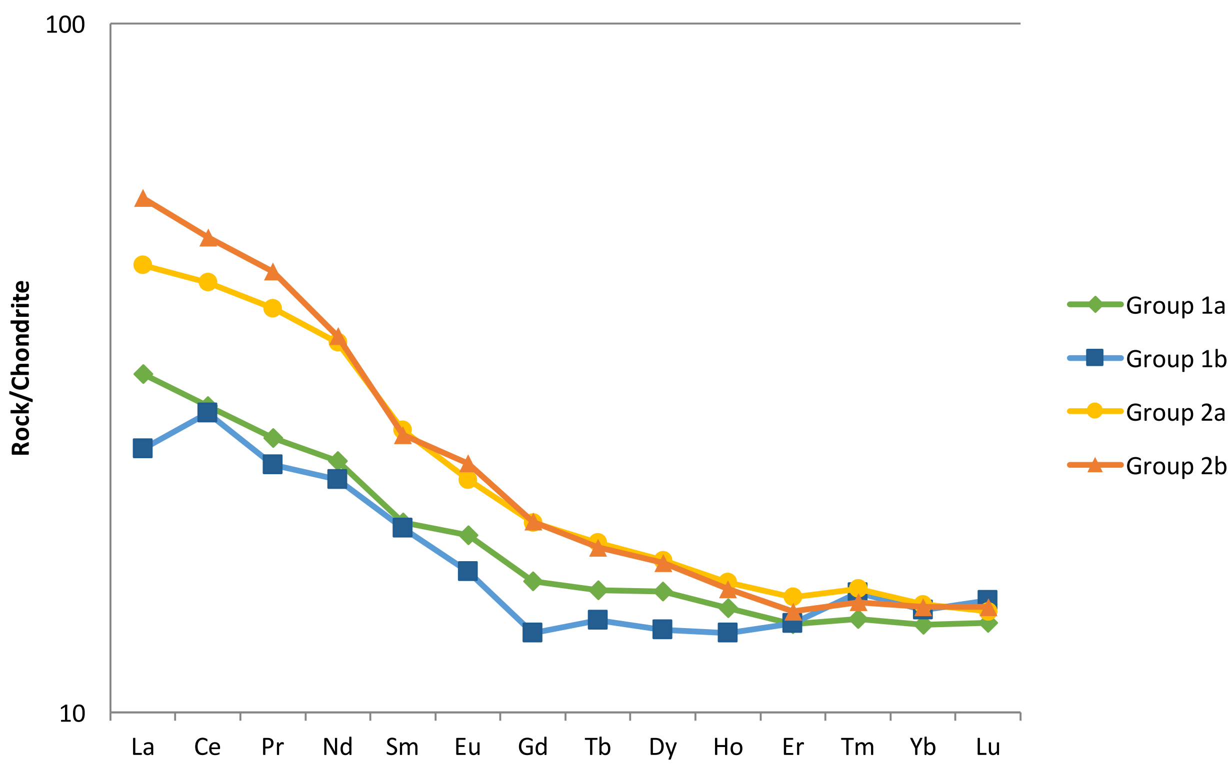


Figure 29 REE spider diagram normalized to chondrite by mineralogical group.

6.4.4 Sediment Samples (ED-XRF)

Samples from sediments of possible raw material sources and artefacts were analyzed via ED-XRF analysis and plot together. The results show that the sediments stem from similar locations as the sources of the raw materials of the ceramics.

The K_2O/Rb ratio shows that for the same amount of K, the Rb concentration is lower in 1a and 1b and higher in the sediments. As these two elements stem mostly from the clays. This means that the sediment clays are more similar to those of group 2a and 2b clays (See Figure 30a). The CaO/Sr are mostly found in the plagioclase. The Figure 30b shows similar ratios of CaO/Sr but a lesser amount of plagioclases in the sediments.

The TiO_2/Zr and TiO_2/Fe_2O_3 plot (Figures 30c and 30d), show the ratios of the elements, which occur mainly in the sands and tempers. It is notable that these plots distinguish between the pairs 1a-1b and 2a-2b. The sediments are plotted more closely to the first pair, 1a-1b.

Although the relationship between the sediments and the ceramics cannot be clearly discerned with these initial analyses, future research is necessary to establish differences between the raw material sources. Minor and trace element analysis would contribute greatly to understanding the natural variations of the sediment samples and to see if there was preferential raw material source selection by the potters. In the following section, the results will be explained in the archaeological context in terms of possible provenance, function, intrasite and intersite variability and manufacturing technology.

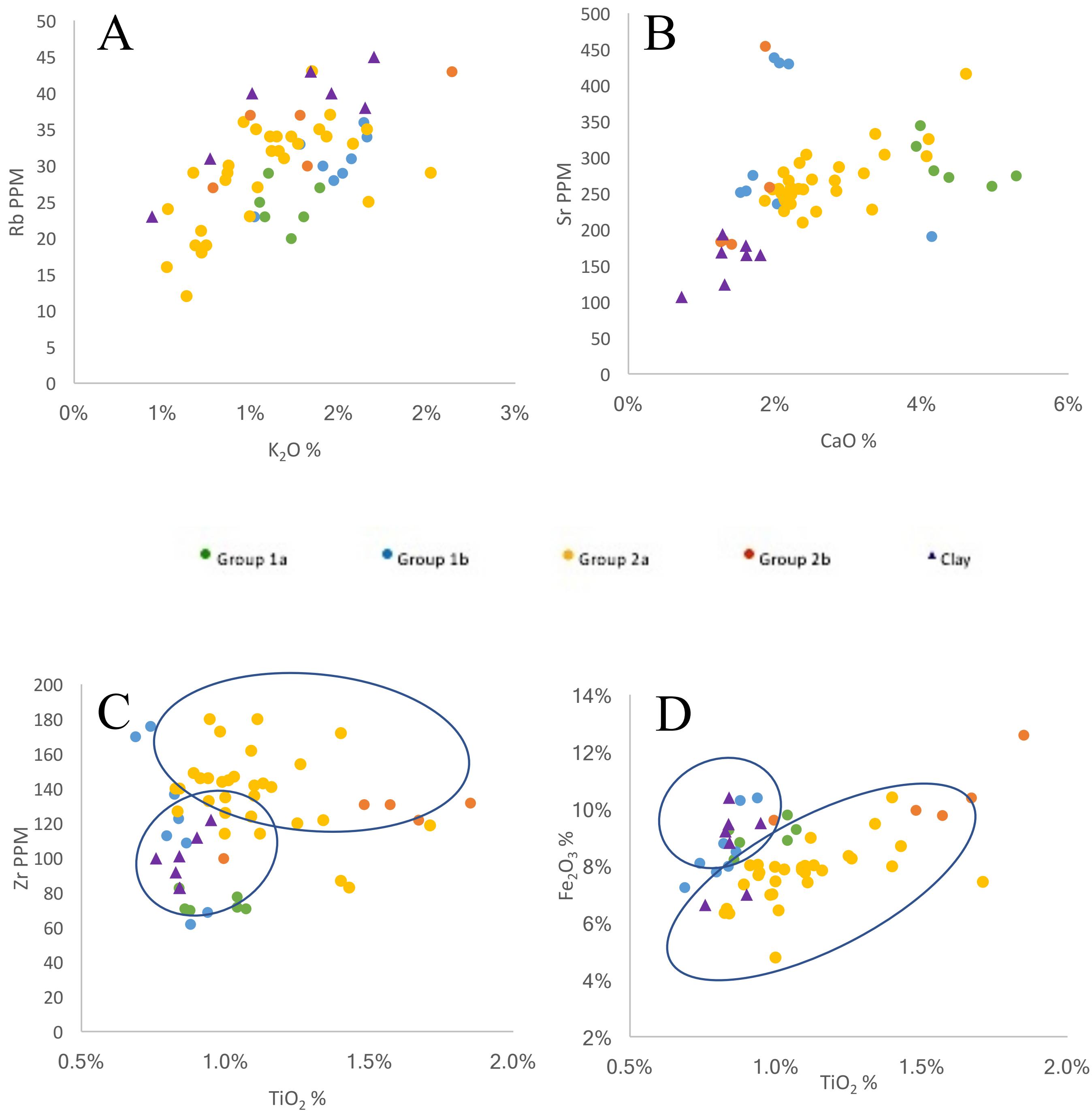


Figure 30 (A) K_2O/Rb; (B) CaO/Sr; (C) TiO_2/Zr (D) TiO_2/Fe_2O_3 scatter plots by mineralogical groups.

7 Discussion of Results in the Archaeological Context

This section is dedicated to integrating the results from the scientific analyses within the archaeological context to answer four basic questions. First, is it possible to determine provenance and pottery production zones from the analytical approaches employed? Second, is the integration of the mineralogical and chemical techniques sufficient to distinguish between function or functional groups? Third, which technological choices are discernable through the study of manufacturing technology? Fourth, what is the extent of ceramic variability at Tibes and its implications within the regional context? Since these questions lie within the scope of the theoretical framework of 'technological choices' discussed in section 2, their dialogue provides a stage to comprehend what possible social functions ceramics play in the early Ostionoid Caribbean.

7.1 Provenance and Raw Material Sources

The mineralogical and chemical groups identified provide a structure to approach the archaeological questions. Through petrographic analysis, three major petrogroups (volcanic, sedimentary and felsic) were discerned. The petrographic categories distinguished in this study have parallels to those described by Ann Cordell (2008) in the Tanamá river drainage in northern Puerto Rico. Cordell's volcanic and felsic groups, discussed in Chapter 3 (Table 1) are likewise evidenced in south central Puerto Rico. In comparison to the Tanamá river drainage, the ceramics from the Tibes volcanic group do not contain pieces of grog and do not exhibit biotite, but instead have grains of muscovite. It is interesting to note that Cordell associates the Tanamá volcanic petrographic group with the Cuevas and Boca Chica styles, which correspond to both the period before and after the early Ostionoid (See Figure 4 from Chapter 3). Cordell's felsic group contains granitic rock inclusions and frequent felsic constituents, such as amphiboles, K-feldspars and plagioclases. The rock inclusions in the Tibes felsic group thin sections were identified as igneous felsic rocks, possibly as granodiorite due to the proximity to the Utuado batholith and a quartz-diorite outcrop (See Figure 5). Although Cordell notes major differences in the coarseness of the Tanamá felsic group, the ceramics in this study show no major disparities in the degree of coarseness. Therefore, it was deemed unnecessary and unproductive to further divide the petrographic groups as done in the Tanamá river study. The most salient discrepancy with Cordell's petrographic groups is the lack of a sedimentary group, and the manifestation of a limestone and vitrified group. Moreover, a limestone tempered group was expected at the site of Tibes due to its proximity to the coast (~ 8 km). Nevertheless, it appears that raw materials were not procured near the coast, but instead within the liminal transition zone of the Semiarid Southern Foothills where Ostionoid sites are concentrated. This could show a break with Saladoid knowledge of raw material procurement from coastal areas, and could represent socio-cultural change in terms of access and control of resources during the Saladoid to Ostionoid transition.

The petrographic groups appear to be local since they are related to the geological formations in the region. The volcanic group is possibly associated with the Monserrate and Lago Garzas formations. The Monserrate formation, consisting mainly of volcanoclastic siltstone and sandstone, has augite pyroxenes. These pyroxenes were identified as hedenbergite and augite in samples 117_f (group 1a), 294_i (group 1a) and FS.257_a (group 1b) through SEM-EDS analysis. The clinopyroxene hedenbergite belong to the overarching category of augite pyroxenes. Since we are studying ceramics, the clay matrix could be influencing the pyroxene composition by "powder" contamination, and therefore this study only has the resolution to identify augite and not a more specific clinopyroxene. Based on the local geology it is possible that these pyroxenes are closely related to augite and the augite-bearing formations. Similarly, the Lago Garzas formation, mainly characterized by dark-red volcanic breccia, could also be a source for the volcanic rock inclusions within the petrogroup. Since both of these formations lie in close proximity to Tibes (less than 10 km away), they could be associated to the clay utilized for pottery production, and possible provenance zones. Due to the relationship between the volcanic and sedimentary groups, it is hypothesized that the major differences are attributed to vertical stratigraphic variances within the same geological formation.

The felsic group originates from a different set of geological formations, possibly the granodiorite of the Utuado batholith. It appears that the ceramics are made from local clays which have weathered from nearby parent rocks, and were deposited at the site via fluvial or terrestrial deposition. The distance of transport of the sediments can be discerned by the degree of weathering and the angularity of the aplastic inclusions. The volcanic and sedimentary group show rounded aplastic inclusions suggesting a longer distance of transport (See figure 31a), while the felsic group shows less weathered more angular inclusions that indicated a smaller distance of transport (See Figure 31b).

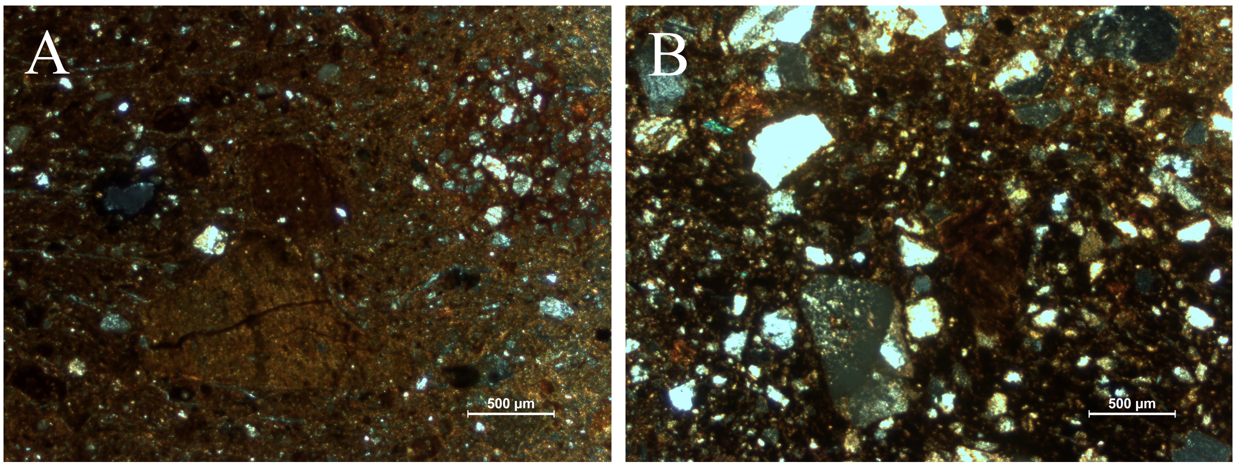


Figure 31 (A) Rounded inclusions (sample 294_a, sedimentary). (B) (A) Rounded inclusions (sample 294_a, sedimentary). (B) Angular inclusions (sample 294_g, felsic).

The descriptions of the geological formations suggest that the volcanic formations are interbedded with the sedimentary formation and vice-versa (Krushensky and Monroe, 1978, 1975). For example, the Monserrate Formation, characterized mainly by volcanoclastic siltstone and sandstone, is also composed of conglomerate of augite andesite which is characteristic of the Lago Garzas Formation (Krushensky and Monroe, 1978, 1975). Although the volcanic, sedimentary and felsic groups are unmistakably from different raw material sources, they probably were gathered in close geographic proximity. In archaeological terms, it is important to not only to establish provenance, but also to determine production zones and what is local or not. Even though the geology of south central Puerto Rico cannot be used to pinpoint exact sites of ceramic production or raw material procurement, potters were choosing specific raw material sources and/or preparing the paste in precise ways. It is important to note that the selection of different raw material sources by the potters depends on the natural variability of the local geology, the resource procurement strategies and the recipes related to paste preparation (Druc, 2013). Therefore, it appears that there was 4 main different production areas or communities of practice in south-central Puerto Rico from the limited sample size studied in this thesis.

Since the island of Puerto Rico measures 170 x 60 km and has a closely packed, diverse geology, it is difficult to determine what should be considered "local." Druc provides a useful framework to understand what should be considered local or not when there is no evidence of production sites, as is the case in Puerto Rico. Arnold (1985, 2006) indicates in the "resource threshold model," through ethnographic and archaeological evidence, that the maximum distance a potter is willing to walk for clay and temper is approximately 1 km for clay and 7 km for temper. This model does not appear to apply in south central Puerto Rico since the sites under study are in very close proximity (approximately less than 5 km), and the people at the different sites appear to have been exploiting the same resources and using the same production technology. Therefore, it appears that what is "local" in Puerto Rico could be influenced by social and political affiliations of land ownership due to the rise of social complexity, which can be discerned through the intensity of production and the acquisition strategies (Druc, 2013, p. 492). Druc (2013:492) rightly points out that the locations of pottery production are not always linked with proximity, availability of resources or abundance of material, but that it tends to be a product of behavioral strategies. Since Tibes is a ceremonial center, and was probably vacant, these models need to be adjusted and re-thought. For the purposes on this study, the raw material sources used in south central Puerto Rico can be considered as local since the resources were available nearby and correlate with the local geology. Nevertheless, this conclusion is tentative since sources with the same composition could have been exploited at a further distance because of the continuance of technical practices from the Saladoid period where sites were located closer to the coast.

7.2 Functional Analysis

Functionality or use is mainly divided between transport, storage, cooking, serving, and even representations of group identity and social status, as discussed in section 2 (Sillar and Tite, 2000). Furthermore, use is limited by the "performance characteristics," which concern how vessels retain contents, survive impact, their cooling effectiveness, heating effectiveness, and their resistance to temperature change (Sillar and Tite, 2000). Although functional studies, can only provide information on what was possible, it is productive framework to understand different properties of different petrographic groups. In this study, thickness and coarseness were studied to understand heating effectiveness and the survival of temperature change. Knowing the function of ceramics within an archaeological context is paramount because it provides information on the distribution of space at archaeological sites. In this case, the location of ceremonial rituals and domestic activities would reveal a myriad of information about Tibes and its relationship to other sites in the region. As discussed in the results section, there is notable inter-site and intra-site variability depending on the petrographic groups. This section is focusing on the discussion of function of intra-site variability at Tibes to discuss the implications for the ceremonial site.

Thickness can be related to function and style in the Caribbean. Thicker walls are likely stronger and more resistant for storage purposes but worse for cooking since the walls conduct heat less effectively, and therefore it takes longer to warm the contents (Rice, 1987, p. 227). Thin walls increase thermal shock resistance (Rice, 1987, p. 228). There is a notable exception in the Caribbean for *burens*, which are known to be used for cooking and have very thick walls of approx. 10 mm. Therefore, it is not as straightforward as anticipated to relate thickness to function, but the trends still contribute information about the pottery technology at the time. Although thickness is a common parameter used to determine certain aspects of function, to determine heating effectiveness, one must use a different set of analytical techniques. As seen in Figure 32, the sherds of different thicknesses are distributed somewhat evenly across the archaeological contexts of Tibes. It is interesting to note that the Unit 7, a trash midden, does not contain the finest type of ceramics, but mostly coarser ceramics, which tend to be associated with domestic contexts.

Coarseness can also be used as a parameter for function. Cooking vessels are usually coarser, and serving vessel are usually finer (Rice, 1987). The divisions of coarse versus not coarse were made based on the results of the quantitative textural analysis (See Figure 33). In essence, the volcanic and sedimentary groups were classified as “not coarse” and the felsic group was classified as “coarse.” The results suggest, that the coarser ceramics are in Unit 1 (Ball Court 8) and the less coarse ceramics are found in Unit 7 (Ball Court 2).

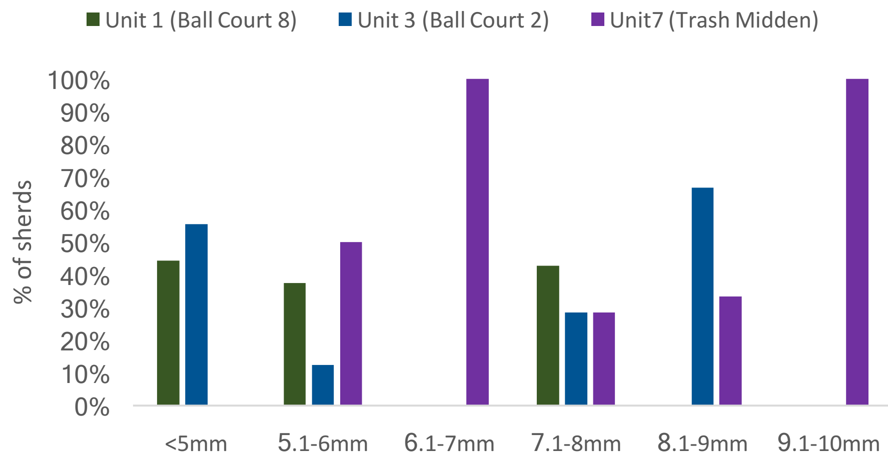


Figure 32 Distribution of thickness by archaeological context at Tibes in terms of % of total sherds by context.

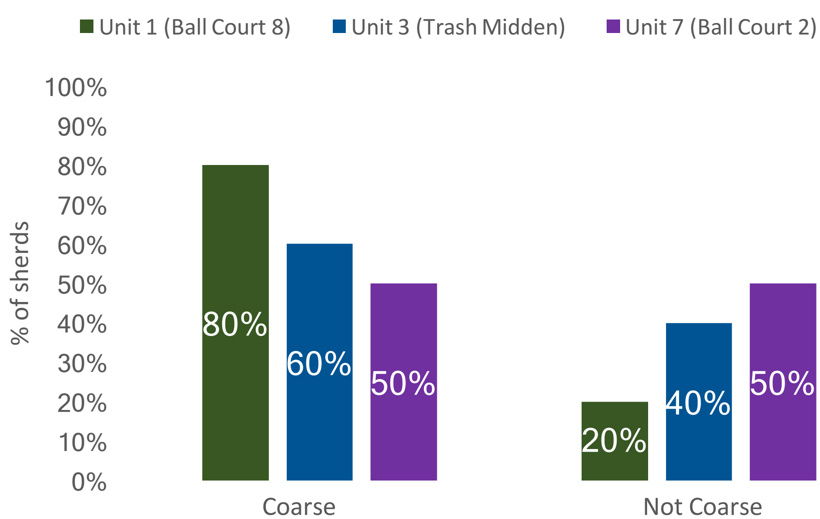
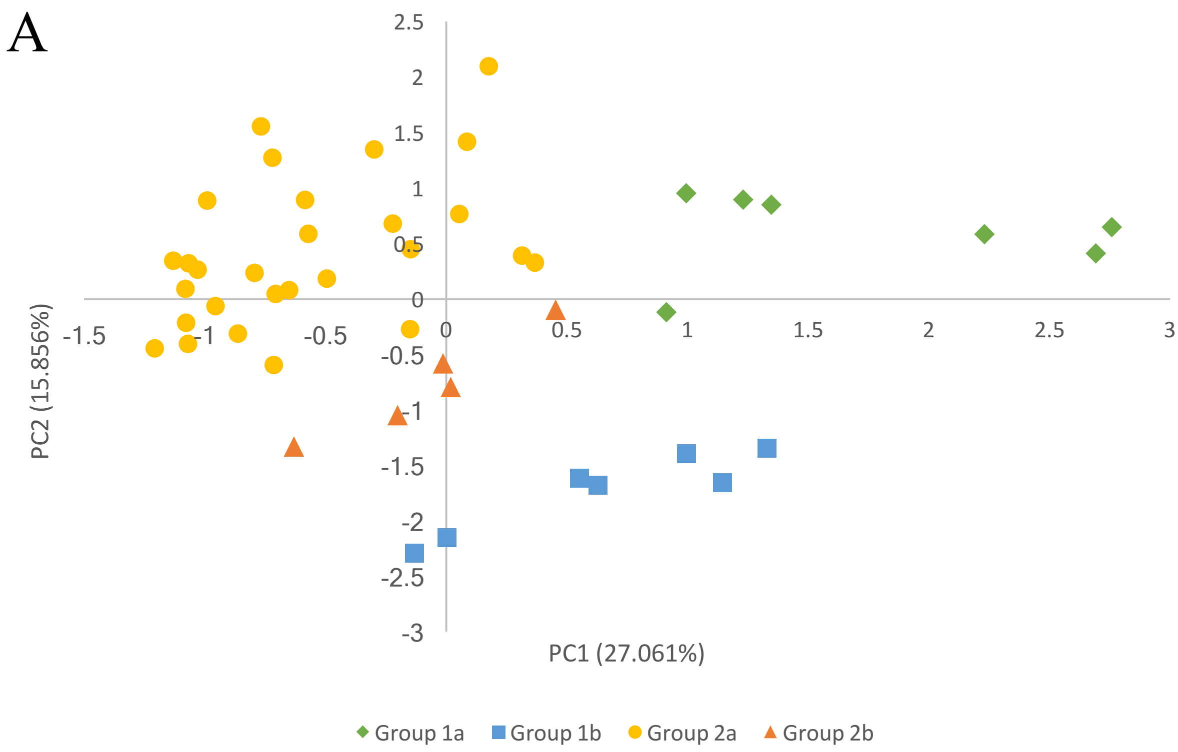


Figure 33 Distribution of coarse and not coarse ceramics by archaeological context at Tibes in terms of % of total sherds by context.

7.3 Manufacturing Technology in early Ostionoid Caribbean

The petrographic and chemical data contributes extensive information on manufacturing technology. The sedimentary and felsic groups show great variability in terms of site, surface decoration and sherd thickness. The volcanic group, only found at Tibes, shows a high proportion of sherds with surface treatments (86%) in comparison to the proportion of sherds in the felsic group (41%). In addition, the volcanic petrographic group is mainly found in the 5.1-5 mm thickness category, while the sedimentary and felsic are more evenly spread across the gamut of thickness categories. Saladoid ceramics tend to be associated with thinner ceramics (Torres, 2012). Contrary to Alvarado Zayas's (1986) theory that Ostionoid culture developed outside of Tibes, this shows that technical practices from the Saladoid period were still thriving in Tibes during Period III. This continuation of manufacturing technology displays the lasting influence of Saladoid culture on Ostionoid ceramics found at Tibes, as well as the possible local development of the Ostionoid culture at the site. Furthermore, the high rate of surface treatments (slip or smoothing) evidenced in the volcanic and sedimentary groups is another indicator of the continuation of expertise deriving from the Saladoid period, since per the literature, it is a diagnostic characteristic of Saladoid ceramics (Torres, 2012).

To further investigate the similarities and differences between the mineralogical groups, the principle components were calculated for Principal Component 1 (PC1) and Principal Component 2 (PC2). The PCA of the mineralogical groups reflects the differences in raw material sources, and contributes further confirmation that 4 different production zones exist within the small community of potters surrounding the site of Tibes (See Figure 34a). For instance, PCA 2 opposed elements as Al-K to Mg-Cr-Co-Ni, in what is well known opposition between felsic versus mafic. As seen in Figure 34b, Group 1a is influenced by Mg, Co, Cr, Cu, Zr, Sr, Ba, Ni, and Mn. Group 1b is related to Na, V, Rb, Pb, Si, Zn, and U. Group 2a is correlated to Th, , K, Al, Ga, and Ti. Lastly, Group 2b is associated to Fe, Nb, and P (For a table of score plot and loading plot see Annex 6 and Annex 7). Therefore, by combining data from major and minor elements (excluding the REE), PCA successfully divides the ceramics along the same petrographic and mineralogical groups. The variability by site and within sites will be key to distinguish possible production areas, but more importantly the relationship between sites as an indicator of socio-cultural processes.



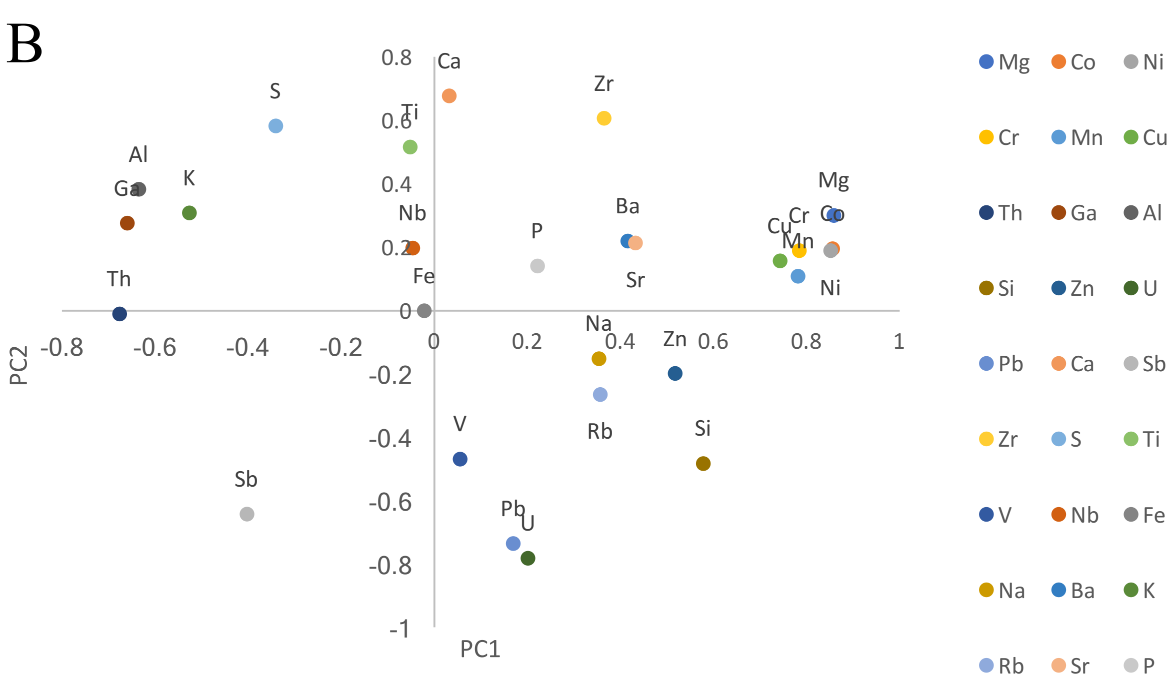


Figure 34 Score plot (a) and loading plot (b) of PC 1 and 2 from the PCA.

7.4 Ceramic Variability at Tibes & Its Implications

Ceramic variability at Tibes sheds light on its role as a ceremonial center in south central Puerto Rico, and contribute to theories regarding its vacant nature, and if ceramics were produced on site.

7.4.1 Intersite Variability

The petrographic groups are not distributed evenly across the sites (See Figure 35). Although this could be an artefact of the sampling size (43 samples studied via petrographic analysis), the volcanic group appears to be only found at Tibes. In addition, PO-42 is only represented by the felsic group. The distribution of sherds with and without surface treatment are not evenly distributed across petrographic groups. The volcanic and sedimentary groups show a higher proportion (85%) of sherds with surface treatment than the felsic group (15%) (See Figure 36). Also, there are notable differences in the surface treatment by archaeological site. Tibes has a higher frequency of surface treatment (80%) and PO-42 has the highest frequency of non-decorated sherds (60%). These results are expected because of the ceremonial nature of Tibes and the association of decorated pottery with non-domestic contexts. In addition, as discussed above, there appears to be variability in terms of the distribution by site depending on the mineral and chemical data, thickness, coarseness, and surface treatment.

7.4.2 Intrasite Variability

It is important to note the value of intrasite variability within the site of Tibes. Depending on the context, whether ceremonial (Unit 1 and 7) or a trash midden (Unit 3), ceramic variability can contribute to understanding these contexts in more depth. Below is the distribution of each petrographic group by archaeological context. First, the volcanic group appears to be more concentrated in Unit 3 (Trash midden) (Figure 38). The sedimentary group is mainly concentrated in the Unit 7 (Figure 39), and the felsic group appears to be quite evenly distributed by context (Figure 40).

Another parameter that shows intrasite variability is surface treatment. Surface treatments in the Caribbean are associated to styles, cultures, and consequently, to technological traditions. At Tibes, there appears to be a majority of sherds with surface treatments, even those part of the coarser felsic group (See Figure 41). Not only can strictly materials science approaches contribute to understanding provenance, function, and manufacturing technology. The combination of these approaches with archaeological methods sheds light on the relationship between material properties, and outward aesthetic properties.

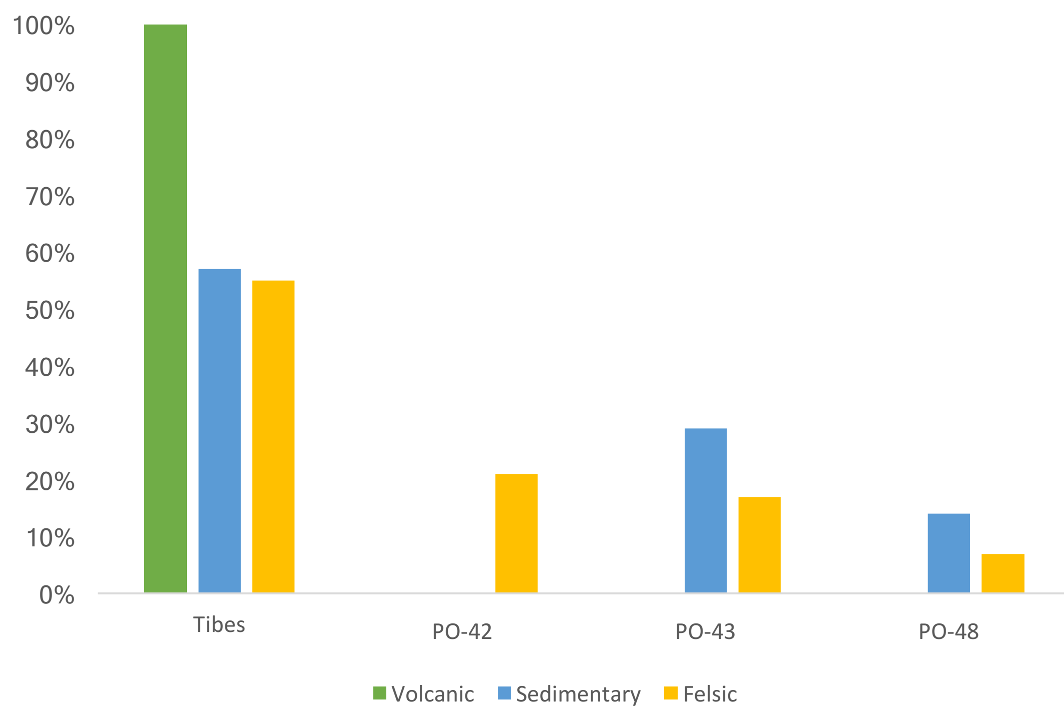


Figure 35 Petrographic group distribution by site expressed as a percentage of total sherds.

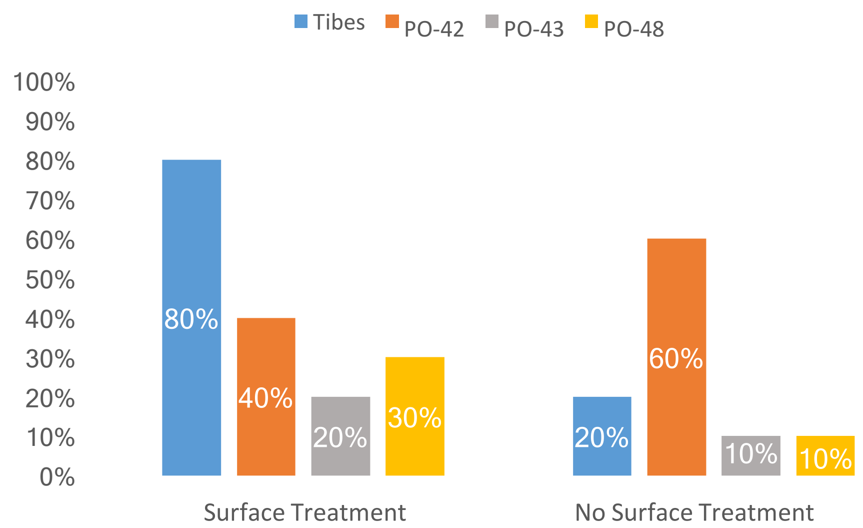


Figure 36 Surface treatment frequency by site.

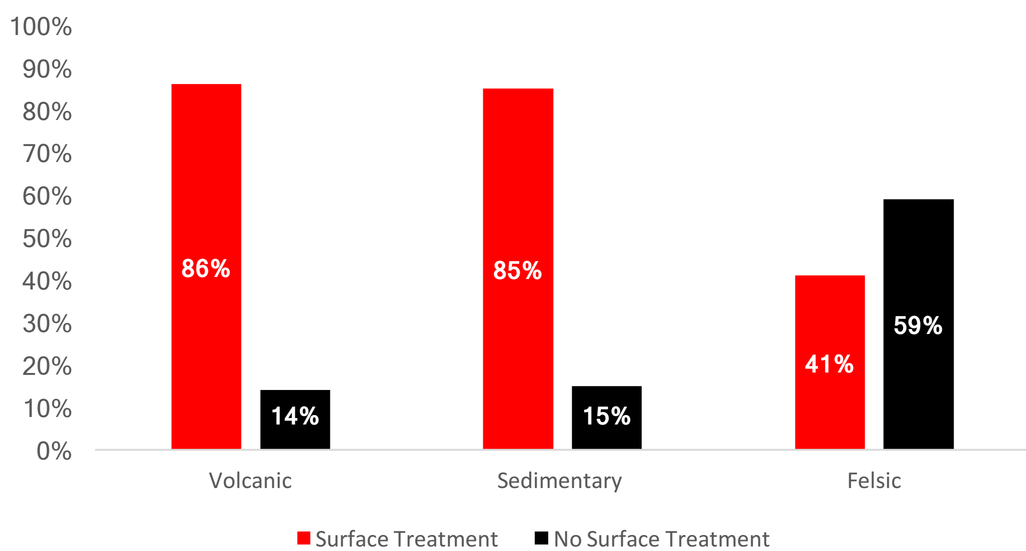


Figure 37 Surface treatment frequency by petrographic groups.

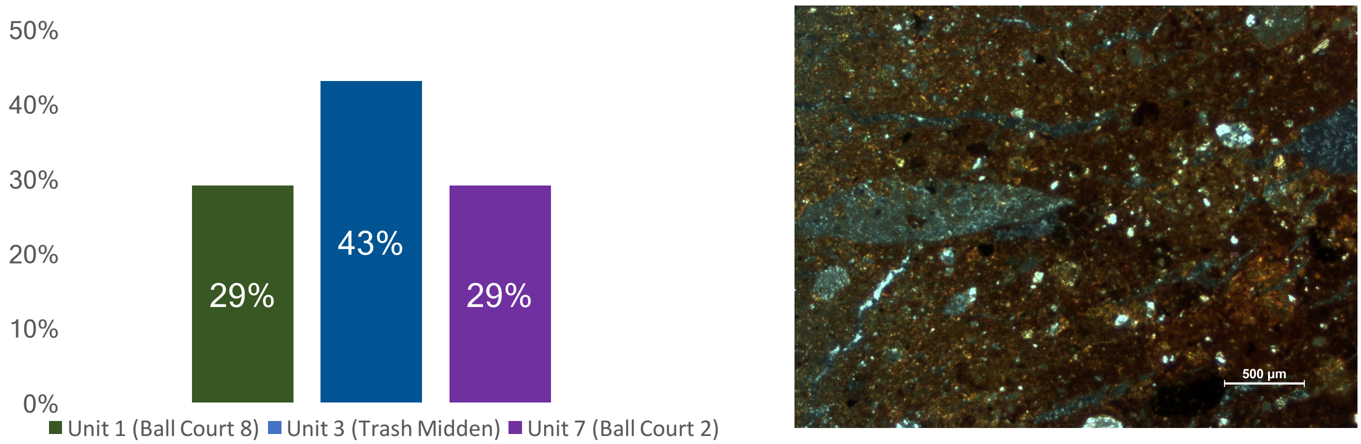


Figure 38 Left - Volcanic group variability by archaeological context at Tibes. Right - Example of Volcanic group (294_d).

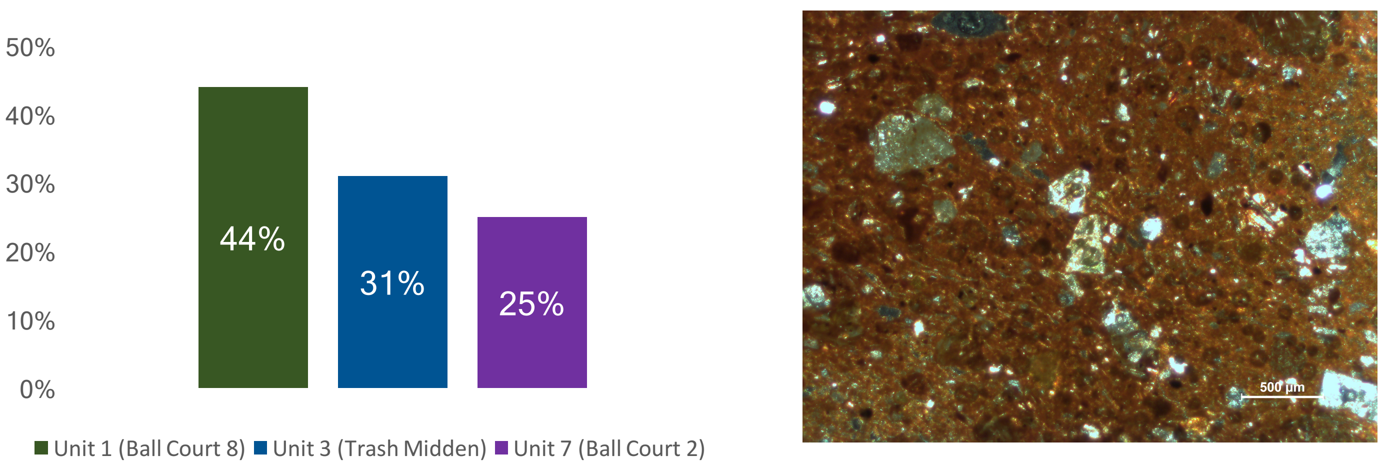


Figure 39 Left – Sedimentary group variability by archaeological context at Tibes. Right - Example of Sedimentary group (294_b).

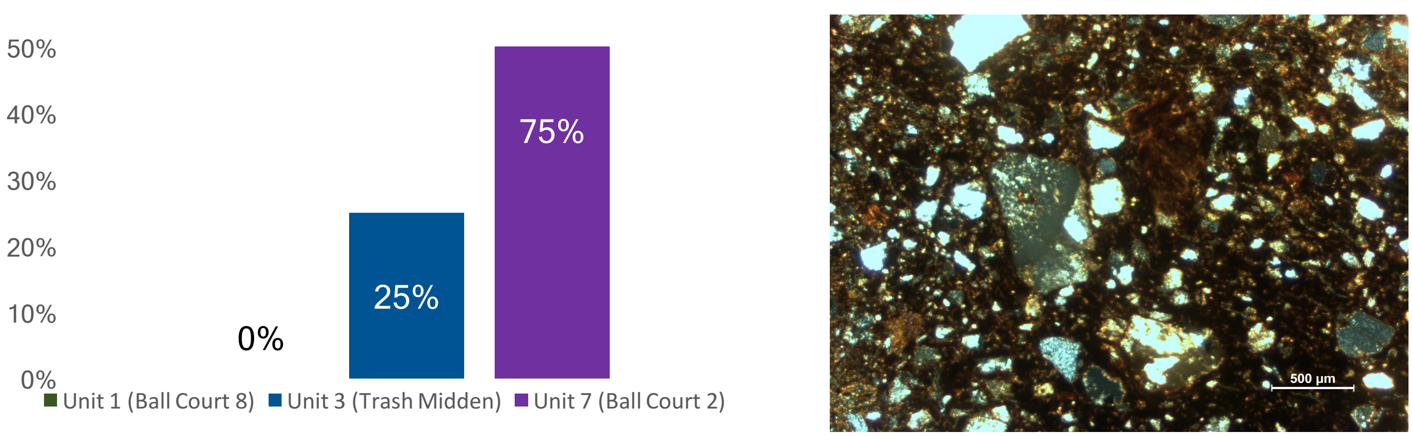


Figure 40 Left - Felsic group variability by archaeological context at Tibes. Right - Example of Felsic group (294_g).

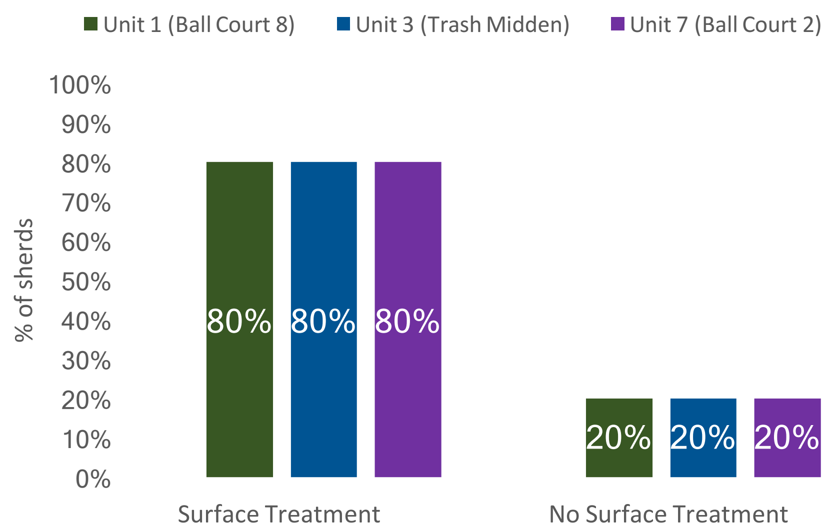


Figure 41 Distribution of the presence of surface treatment an lack thereof by archaeological context at Tibes in terms of % of total sherds by context.

8 Conclusions and Future Directions

Comprehending what dictates technological choice is a fundamental pursuit by researchers investigating material culture. The anthropological motivations behind every step of the *chaîne opératoire* reflects both social and political processes, which can aid in reconstructing past lives. This thesis aims to investigate the provenance, function and manufacturing technology in the early Ostionoid Caribbean to provide a better understanding to the Saladoid to Ostionoid transition, and to shed light on the role of the ceremonial center of Tibes in the development of social complexity in the region. Although this project only had 50 samples at its disposition, certain preliminary conclusions can be made from the multi-analytical data acquired.

Firstly, and most significantly, the results from this thesis exhibit evidence for preferential selection of certain clay resources by a community of potters in south central Puerto Rico. The raw material sources, their procurement and the size and abundance of aplastic inclusions in the paste reflect 4 different paste recipes employed by potters. The paste characteristics can be used as indicators of local production areas, social boundaries and distribution networks since they do not change based on decoration or style (Druc, 2013). In addition, three different types of ceramics are found across sites, and the volcanic paste (group 1a), is found only at Tibes. Perhaps, this is evidence for the use of specific pottery for ceremonial activities. This theory is preliminary due to the limited sample size. By continuing research in the vicinity of Tibes in south central Puerto Rico, more in depth understanding of the motivation behind the preferential selection of clay can be investigated.

The second main conclusion is regarding the discernment of function through a materials science approach. Establishing function is both complicated in theoretical and practical analytical terms. Based on the literature, it appears that relating function to use is not straightforward, and requires not only an in depth understanding of ceramic variability in the region, but also ethnographic patterns to guide archaeological research. Although ethnoarchaeology is not expected to provide final answers, it can shed light on unique information regarding social practices, which have remained prevalent in the region. For this reason, function cannot be strictly related to the petrographic pastes. A possible explanation for the correlations discussed in Chapter 6 and 7 is that the volcanic and sedimentary groups (group 1a and 1b), which are “finewares” can be related to ritual activities at Tibes. Especially since the volcanic group 1a was found exclusively at the site. The felsic paste (group 2a and 2b), or "coarsewares," could be related to domestic contexts due to their suitability for cooking and their lack of aesthetic "quality." Nevertheless, it is apparent that a set of additional methods must be employed to assess the physical properties of the ceramics, and to identify for what purposes each paste would be best suited for.

Thirdly, there is evident widespread ceramic variability in the manufacturing technology in south central Puerto Rico. Through mineralogical and chemical data, this research was able to differentiate 4 different raw material sources that are present across sites (except the volcanic group, discussed above). Probably, members of the community surrounding Tibes are sharing resources, and therefore perhaps acting as one larger community entity. As hypothesized by Torres (2012: 345),

> "…local social community, comprised of multiple residential settlements and strongly associated with particular localities, become prominently represented as a coherent sociopolitical entity in the Ostionoid through its stable and proximally related settlements and construction of its ritual integrative facilities."

Therefore, the interaction between sites, reflected in the sharing of raw material sources, can be another line of evidence to confirm that these small residential settlements in south central Puerto Rico are acting as a community and not as separate groups. Another justification for the sharing of clay for pottery production is a continuation in technical practices from the egalitarian Saladoid period by continuing to share clay amongst different communities. Or, that these clays were available at various locations in the landscape, and therefore were not being "shared" at all. In my opinion, it appears that through the reflection of social behaviors in ceramics, we can hypothesize that the sites surrounding Tibes had a common "cultural identity." In addition, there is a scarcity of evidence to support the notion that raw material sources were being controlled by the elite. Although the volcanic paste (group 1a) only appears at Tibes, and therefore could be evidence of the monopolizing of that clay resource for ceremonial activities, the sample size is too small to confirm such an interpretation.

In addition, it might be productive and useful to apply the concept of "spaces of experience," which suggests that the raw material sources being exploited are the product of other factors in people's lives (Gosselain, 2008 as cited in Druc, 2013, p. 492). Raw material sources were collected in areas where other activities were taking place such as agriculture, dwellings, sites of social interaction, or fishing sites. By comparing different time periods, a larger ceramic assemblage, and including a larger sample size of regional clays, questions relating to identity and power can be hypothesized with more certainty.

In the future, it will be paramount to continue studying social processes in south central Pueto Rico. This thesis found a materials science approach in combination with archaeological methods as a fruitful and dynamic way to investigate questions concerning the socio-political processes relating to the rise of social complexity.

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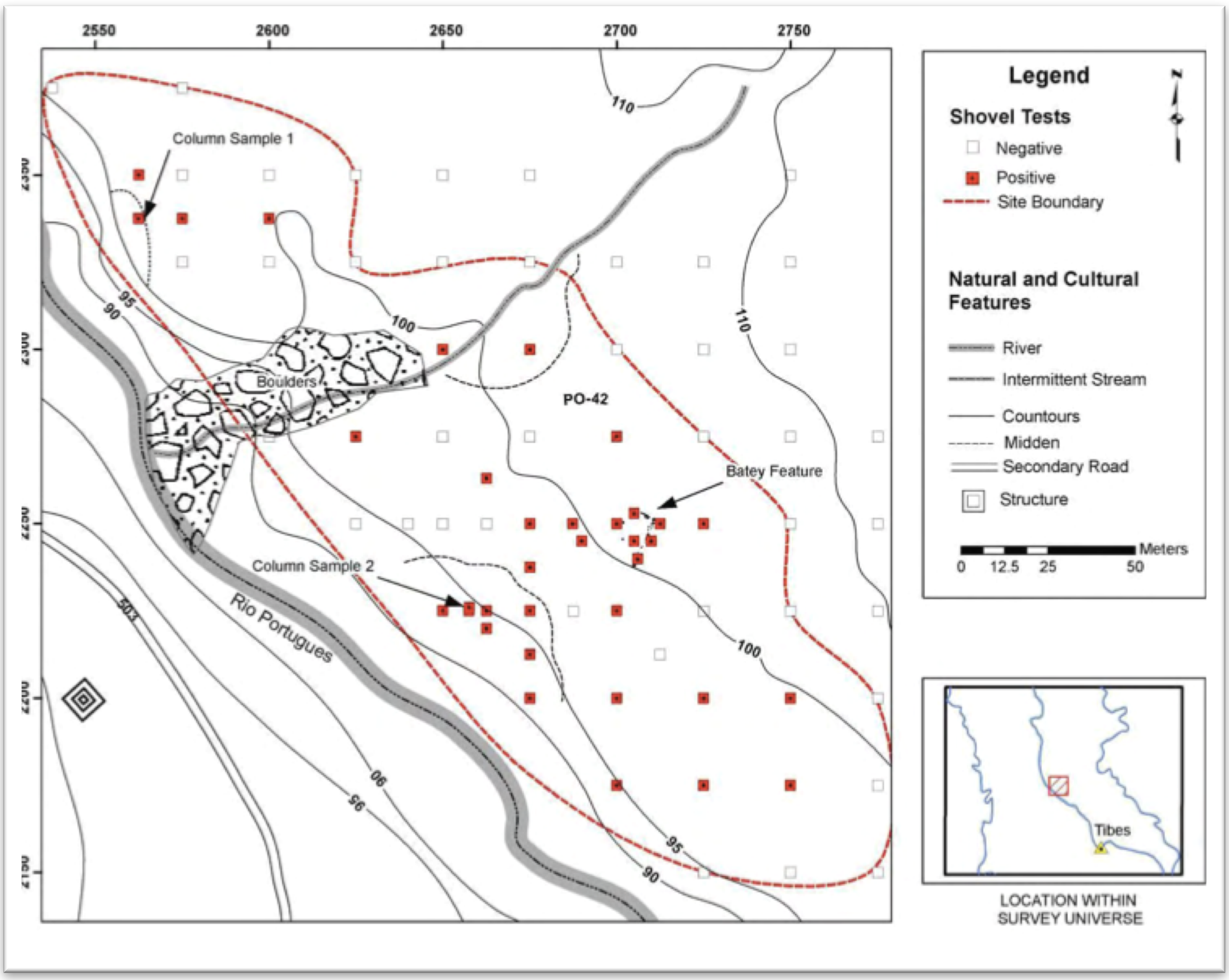
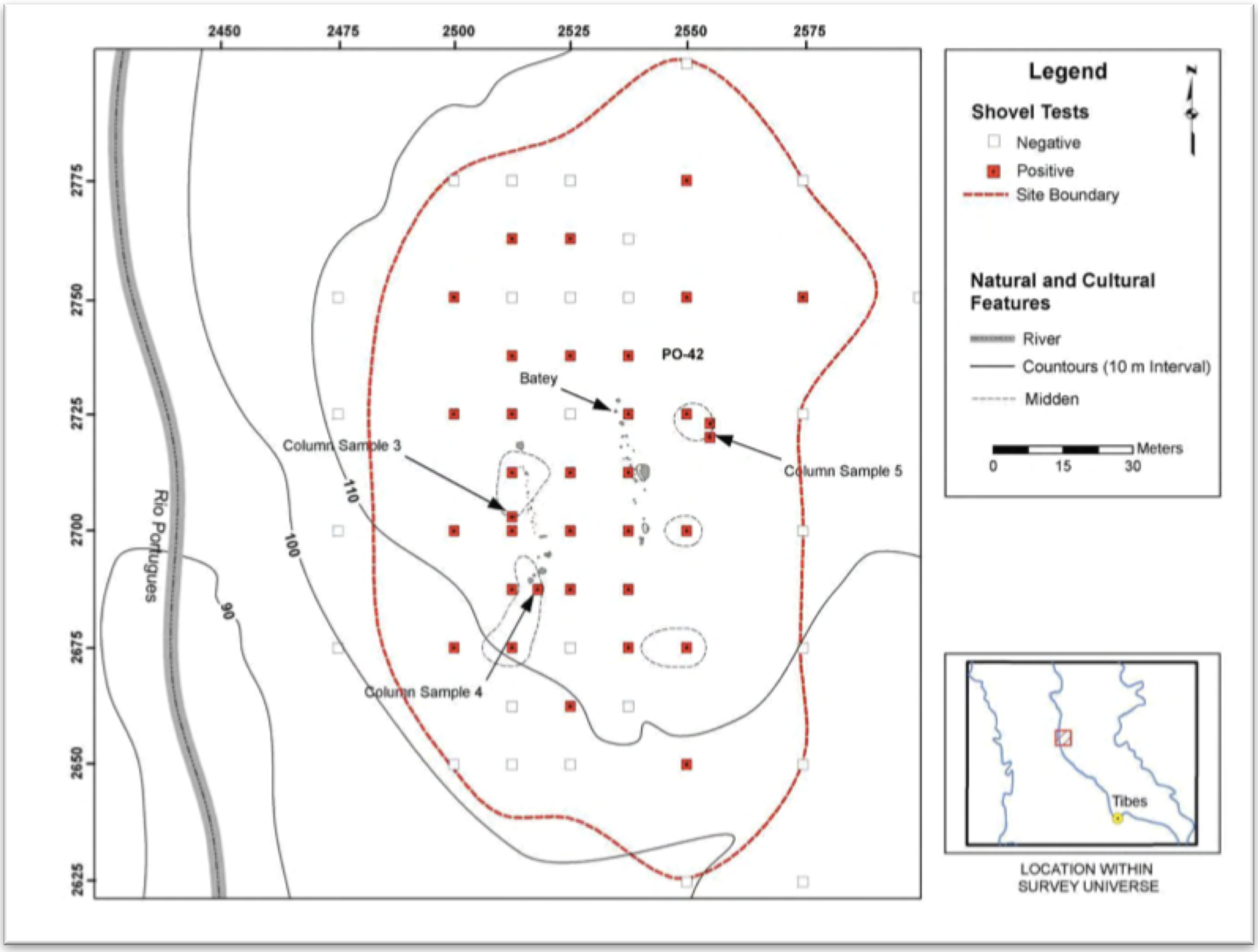
Annex

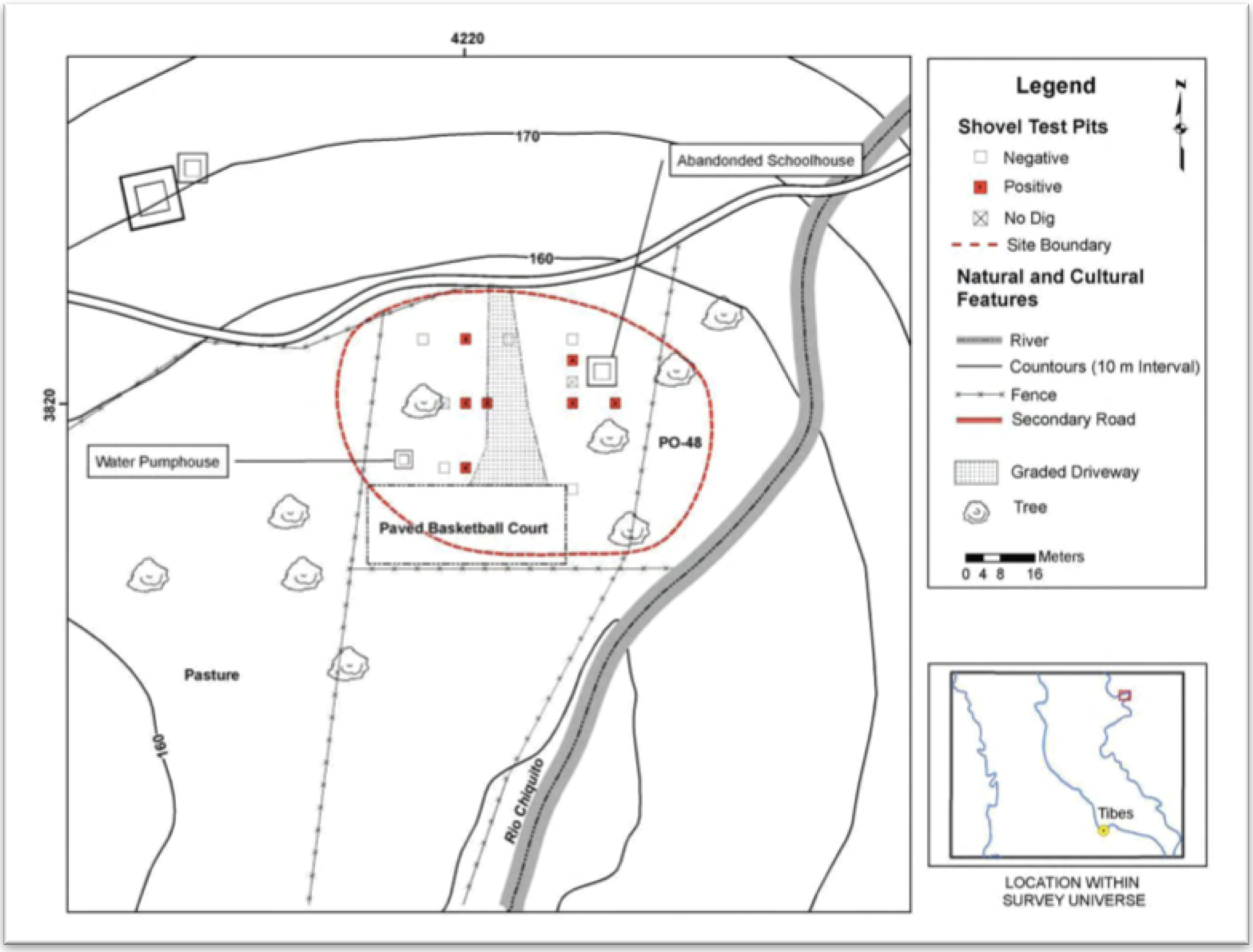
Annex 1 Map of PO-42 showing the shovel test units and the location of the batey feature (Image from Torres, 2012).



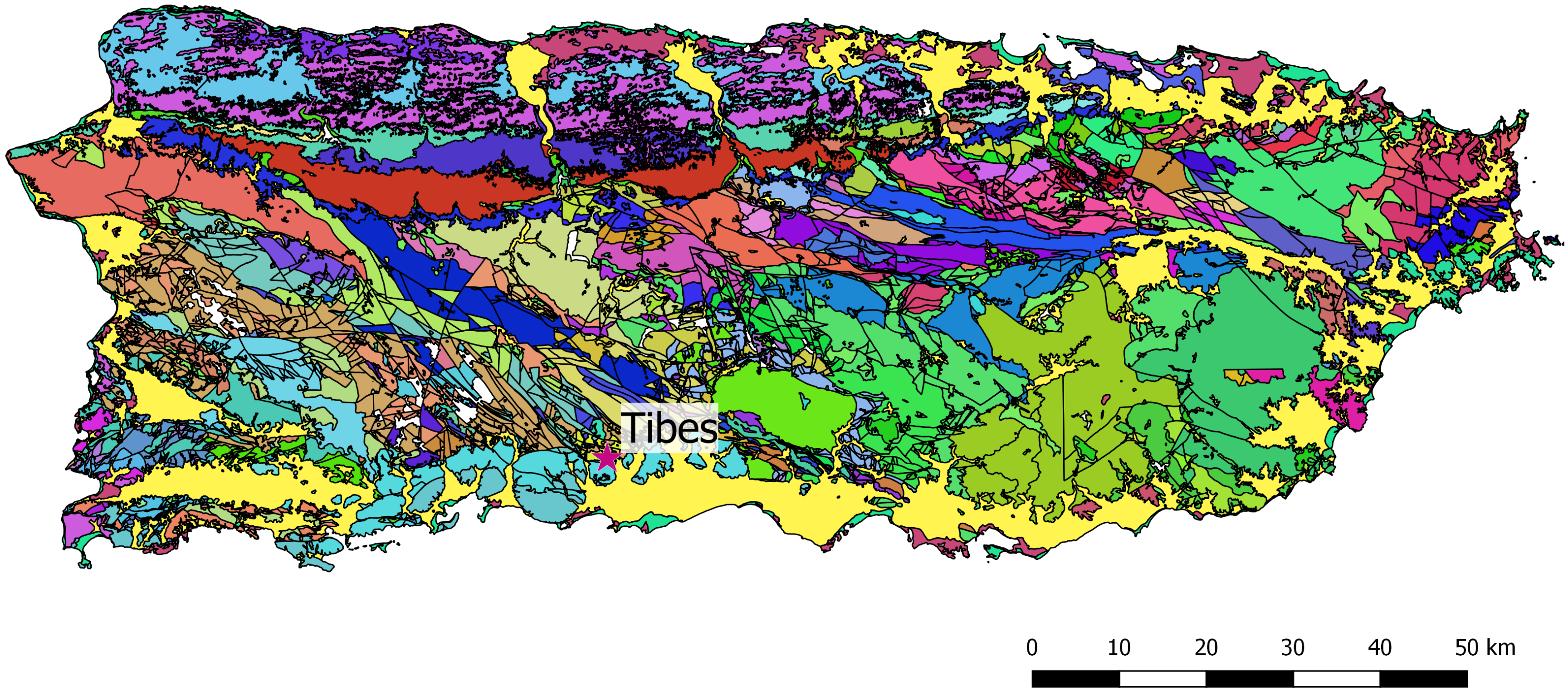
Annex 2 Map of PO-43 showing shovel tests and batey feature (Image from Torres, 2012).



Annex 3 Map of PO-48 site showing shovel tests (Image from Torres, 2012).



Annex 4 Geology Map of Puerto Rico with detailed geological formations adapted from USGS. See next page for the legend.



- af: Artificial fill, Holocene
- Fault, concealed
- Fault, type unknown
- Kabcj: Formations A, B, C, and J, lower ?-upper ? Cretaceous
- Kac: Achiote Conglomerate, Maestrichtian-Santonian
- Kal: Alonso Formation, upper ? Cretaceous
- Kap: Avispa and Perchas Formations undivided, Cretaceous
- Kav: Avispa Formation, upper ? Cretaceous
- Kba: Barrazas Formation, upper Cenomanian
- Kbo: Boqueron Basalt, upper Cretaceous ?
- Kca: Cariblanco Formation, lowest Campanian-upper Santonian
- Kcag: Granodiorite of Caguas Pluton, post middle Albian
- Kcam: Camarones Sandstone, upper ? Cretaceous
- Kcan: Cancel Breccia, upper ? Cretaceous
- Kcar: Carraizo Breccia, lower Turonian-middle Cenomanian
- Kcb: Cambalache Formation, Turonian
- Kce: Celada Formation, post Turonian ? Cretaceous
- Kcg: Cerro Gordo Lava, lower ? Cretaceous
- Kcn: Canovanas Formation, lower Santonian-upper Turonian
- Kco: Concepcion Formation, Maestrichtian-Campanian
- Kcoa: Coamo Formation, lower Maestrichtian-upper Santonian
- Kcot: Cotui Limestone, lower Maestrichtian-upperMOST lower Campanian
- Kd: Daguao Formation, lower Cretaceous ?
- Kdh: Diorite-hornblendite-gabbro, upper ? Cretaceous
- Kdi: Diorite, Campanian ? Albian ?
- Ke: El Rayo Formation, upper to middle Maestrichtian
- Keo: El Ocho Formation, Cenomanian ?
- Kf: Figuera Lava, lower Cretaceous ?
- Kfa: Fajardo Formation, middle ? Albian
- Kfd: Figuera Lava and Daguao Formation Interbedded, Cretaceous
- Kfr: Friales Formation, Campanian
- Kg: Guynabo Formation, lower Santonian-upper Cenomanian
- Kh: Hato Puerco Formation, Turonian-lower Cenomanian
- Ki: Infierno Formation, Turonian
- Kib: Intrusive Daguao Breccia, lower? Cretaceous
- KJa: Amphibolite, Cretaceous ?-pre-Cretaceous ?
- Kja: Jayuya Tuff, Cretaceous ?
- KJas: Amphibolite-Serpentinite, Cretaceous ?-pre-Cretaceous ?
- KJb: Spilitized Basalt, lower Cretaceous ?-pre-upper Kimmeridgian?
- KJc: Cajul Basalt, lower Cretaceous ?-upper Jurassic ?
- KJm: Mariquita Chert, middle Turonian ?-upper Kimmeridgian-lower- Tithonian undivided
- KJs: Serpentinite, Cretaceous ?-pre-Cretaceous ?
- Kl: Lajas Formation, lower ? Campanian-pre Campanian ?
- Klm: La Muda Formation, Maestrichtian-Campanian ?
- Kln: Los Negros Formation, lower ?-upper ? Cretaceous
- Klo: Lomas Formation, upper ? Cretaceous
- Km: Melones Limestone, middle Maestrichtian-upper Campanian
- Kma: Magueyes Formation, middle Albian
- Kmag: Martin Gonzales Lava, upper Cretaceous
- Kmal: Malo Breccia, upper ? Cretaceous
- Kmam: Mameyes Formation, upper ? Cretaceous
- Kman: Manicaboa Formation, upper ? Cretaceous
- Kmar: Maravillas Formation, middle ? Maestrichtian-upper Santonian
- Kmc: Granodiorite of Morovis and Ciales Stocks, post middle Albian
- Kmo: Monacillo Formation, upper Cretaceous
- Kp: Parguera Limestone, lower? Maestrichtian-lower Campanian-upperMOST Santonian
- Kpa: Pajaros Tuff, lower ? Cretaceous
- Kpe: PenoÃ±es Limestone, Maestrichtian
- Kper: Perchas Formation, upper ?-lower ? Cretaceous
- Kpgg: Granodiorite of the plutonic complex of Punta Guayanes, Maestrichtian-Campanian
- Kpgq: Quartz diorite of plutonic complex of Punta Guayanes, Maestrichtian-Campanian
- Kpgqm: Quartz monzonite of plutonic complex of Punta Guayanes, Maestrichtian
- Kpi: Pitahaya Formation, lower Cretaceous ?
- Kpo: Pozas Formation, lower Maestrichtian-upper Santonian
- Kpob: Two pyroxene olivine basalt, lower ? Maestrichtian-Campanian
- Kpsg: Granodiorite of the plutonic complex of Punta Guayanes and granodiorite of San Lorenzo undivided, upper Cretaceous
- Kpsq: Quartz diorite of plutonic complex of Punta Guayanes and of the granodiorite of San Lorenzo, upper Cretaceous
- Kr: Robles Formation, Cenomanian-lower Albian
- Kra: RÃo Abajo Formation, lower Cretaceous ?
- Krp: RÃo de la Plata Sandstone, upper ? Cretaceous
- Ks: Sabana Grande Formation, lower Maestrichtian-Turonian
- Ksl: Granodiorite-Quartz Diorite of San Lorenzo, post Cenomanian ?
- Kslg: Mixed granodiorite-diorite of San Lorenzo complex and diorite hornblendite-hornblende gabbro, Cretaceous
- Kslq: Quartz Diorite Facies of Granodiorite of San Lorenzo, upper ? lower ? Cretaceous
- Kso: Santa Olaya Lava, upper ? Cretaceous
- Kt: Torrecilla Breccia, lower Albian
- Kta: Tabonuco Formation, middle or lower Cenomanian-Albian and ? Aptian
- Kte: Tetuan Formation, Campanian-Santonian
- Kto: Tortugas Andesite, upper ? Cretaceous
- Ku: Granodiorite-quartz diorite of the Utuado batholith, post upper Paleocene- Santonian ?
- Kv: Vista Alegre Formation, upper ? Cretaceous
- Normal fault
- Qa: Alluvium, Quaternary
- Qb: Beach Deposits, Quaternary
- Ql: Landslide Deposits, Holocene-Pleistocene ?
- Qs: Swamp Deposits, Holocene-Pleistocene ?
- QTs: Blanket Sand Deposits, Quaternary ? late Tertiary ?
- Ta: Aguada Limestone, lower Miocene
- Tay: Aymamon Limestone, lower Miocene
- Tc: Cuevas Formation, lower middle Eocene
- Tca: Carreras Siltstone, early ? Paleocene ?
- Tcb: Cibao Formation, lower Miocene-upper Oligocene
- Tcbg: Guajataca Member, Tertiary
- Tcbga: Almirante Sur Sand Lentil, Tertiary
- Tcbm: Montebello Limestone Member, Tertiary
- Tcbmi: Miranda Sand Member, Tertiary
- Tcbq: Quebrada Arenas Member, Tertiary
- Tcbr: RÃo Indio Limestone Member, Tertiary
- Tcm: Camuy Formation, lower Pliocene-upper Miocene
- Tco: Corozal Limestone, lower Eocene ?-Paleocene
- Td: Dacite, Tertiary
- Tfb: Fault Breccia, middle Eocene ?-post middle Eocene ?
- Tg: Guayo Formation, middle Eocene ? -post middle Eocene ?
- Tga: Gabbro, upper ?-middle Eocene
- Tgua: Guanajibo Formation, upper Miocene
- Thp: Hornblende quartz-diorite, Tertiary ?
- Thrust fault
- Tj: Jicara Formation, lower Eocene-upper Paleocene
- Tjd: Juana Diaz Formation, middle Miocene-lower Oligocene
- Tjo: Jobos Formation, Oligocene ?-pre-Oligocene?
- TKa: Anon Formation, Eocene-middle Campanian
- TKahp: Augite-hornblende porphyry, Tertiary ?-upper Cretaceous
- TKal: Lago Garzas-Anon Formations Interbedded, upper Eocene?-upper Campanian
- TKam: Anon and Maricao Formations Interbedded, Paleogene ?-late Cretaceous ?
- TKamo: Anon and Monserrate Formations Interbedded, middle Eocene
- TKap: Augite andesite porphyry, Tertiary ?-upper Cretaceous ?
- TKas: Alkali syenite, Tertiary ?-upper Cretaceous ?
- TKay: Anon and Yauco Formations Interbedded, upper Maestrichtian-middle Campanian
- TKaym: Anon, Yauco, and Maricao Formations Interbedded, lower Paleogene ?-Campanian ?
- TKci: Cibuco Formation, upper Cretaceous ?-Paleogene ?
- TKda: Dacite, Tertiary-upper Cretaceous ?
- TKdi: Diorite, Tertiary ?-upper Cretaceous ?
- TKgm: Granodiorite quartz monzonite, Tertiary-upper Cretaceous ?
- TKgu: Guaracanal Formation, lower Paleogene-upper Cretaceous
- TKh: Hornblende quartz-diorite porphyry, Eocene
- TKha: Hydrothermally altered rock, Tertiary ?-Cretaceous?
- TKhda: Hornblende dacite, Tertiary-late Cretaceous
- TKk: Quartz keratophyre, Tertiary ?-upper Cretaceous ?
- TKl: Lago Garzas Formation, middle Eocene-Campanian
- TKlam: Lago Garzas, Anon, and Maricao Formations Interbedded, Tertiary ? -Maestrichtian ?-Campanian ?
- TKly: Lago Garzas and Yauco Formations Interbedded, Tertiary ? -Maestrichtian ?-Campanian ?
- TKm: Maricao Formation, Tertiary ? -Maestrichtian-Campanian
- TKmly: Maricao, Lago Garzas, and Yauco Formations Interbedded, lower Paleogene ?-Campanian ?
- TKmv: Metavolcanic Rocks, upper Cretaceous ?-Paleogene ?
- TKmy: Maricao and Yauco Formations Interbedded, Tertiary ?-Maestrichtian ?-Campanian ?
- TKn: Naranjito Formation, lower Paleogene ? upper Cretaceous ?
- TKqd: Quartz diorite-granodiorite, Tertiary ?-upper Cretaceous ?
- TKy: Yauco Formation, Tertiary ?-middle ?-lower ? Maestrichtian-Cenomanian
- Tl: Los Puertos Formation, lower Paleocene
- Tla: Lares Limestone, upper ? Oligocene
- Tm: Monserrate Formation, middle Eocene
- Tmu: Mucarbones Sand, lower Miocene ? upper Oligocene
- Tor: Ortiz Formation, Paleogene ? -Neogene ?
- Tpa: Palmarejo Formation, Eocene-Paleocene
- Tpo: Ponce Limestone, upper Miocene-Pliocene
- Tr: RÃo Culebrinas Formation, middle Eocene
- Tra: Raspaldo Formation, lower Eocene-upper Paleocene
- Trd: RÃo Descalabrado Formation, lower middle Eocene
- Trhp: Rhyodacite porphyry, Tertiary
- Trp: RÃo Piedras Siltstone, upper Paleocene
- Ts: San Sebastian Formation, middle ?-upper Oligocene
- Ty: Yunes Formation, middle Eocene-upper Paleocene

Annex 5 Instrumental limits of detection for the two round of ICP-MS analysis. Left, first and second batch; Right, third batch.

First and Second Batch	
Name	DL (ppb)
P	0.580820889
Sc	0.017516165
V	0.011173988
Mn	0.020597171
Co	0.01932988
Ni	0.041484576
Cu	0.015021359
Zn	0.382572683
Ga	0.00623951
Ge	0.010171819
Rb	0.023380956
Sr	0.03227818
Y	0.006553086
Zr	0.012488812
Nb	0.030405413
Sn	0.014194284
Sb	0.045845613
Cs	0.002040722
Ba	0.027468261
La	0.000902577
Ce	0.000956295
Pr	0.000329941
Nd	0.002194527
Sm	0.001469269
Eu	0.001839218
Gd	0.002032055
Tb	0.00055179
Dy	0.002114452
Ho	0.000976646
Er	0.001234222
Tm	0.001365725
Yb	0.000778016
Lu	0.001008172
Pb	0.007920823
Th	0.005664166
U	0.000320304

Third Batch	
Name	DL (ppb)
P	0.477501252
Sc	0.014454433
V	0.013344117
Mn	0.011143872
Co	0.00697697
Ni	0.130631136
Cu	0.029775439
Zn	0.307965226
Ga	0.01053755
Ge	0.033023072
Rb	0.025000248
Sr	0.011819997
Y	0.005045702
Zr	0.013542969
Nb	0.022453146
Sn	0.004970539
Sb	0.032522947
Cs	0.013087064
Ba	0.015286519
La	0.000407598
Ce	0.001095584
Pr	0.000632436
Nd	0.002318963
Sm	0.003655909
Eu	0.001309301
Gd	0.002363585
Tb	0.000940082
Dy	0.002196799
Ho	0.001038069
Er	0.001242924
Tm	0.001271442
Yb	0.001003447
Lu	0.000680445
Pb	0.01469082
Th	0.003355457
U	0.000622487

Annex 6 Score plot for PC1, PC2, PC3, and PC4.

Sample ID	Site	Group	PC1	PC2	PC3	PC4
117_c	Tibes	1a	2.75808	0.64443	-0.63831	-0.28836
117_f	Tibes	1a	2.68975	0.41143	-0.07232	-0.51336
117_h	Tibes	1a	0.91178	-0.12168	-0.96812	0.00753
294_d	Tibes	1a	0.99222	0.95329	0.46624	-0.61406
294_i	Tibes	1a	1.34528	0.84686	0.24446	0.17511
62_d	Tibes	1a	2.22986	0.58068	-0.73976	0.1342
62_e	Tibes	1a	1.23032	0.8957	0.27685	-0.79502
117_a	Tibes	1b	0.99592	-1.40168	-1.00779	0.63228
294_b	Tibes	1b	0.631	-1.68319	-1.10171	0.44854
294_a	Tibes	1b	1.33043	-1.34871	0.11332	0.72774
294_e	Tibes	1b	1.14338	-1.65697	0.10946	1.80976
FS.257_a	PO-48	1b	0.55392	-1.61595	-1.14524	-0.00501
FS.75_a	PO-42	1b	-0.13041	-2.29295	-1.73297	-0.1203
FS.76_h	PO-42	1b	0.00352	-2.15255	-2.20496	-0.81776
117_b	Tibes	2a	-0.29794	1.34493	1.47549	-0.99933
117_d	Tibes	2a	-0.65026	0.07536	0.56996	-0.68555
117_e	Tibes	2a	-0.9917	0.88446	-0.34548	1.71229
117_i	Tibes	2a	-0.57099	0.5826	-0.53554	1.30956
294_c	Tibes	2a	-1.13239	0.34033	-0.81884	-0.30145
294_f	Tibes	2a	-0.71397	-0.6014	0.36706	-0.88746
294_g	Tibes	2a	-0.95652	-0.06712	-0.5931	-1.05122
294_h	Tibes	2a	-1.02996	0.26085	-0.67006	-0.57622
62_a	Tibes	2a	-1.0792	-0.21911	0.0261	-0.77205
62_c	Tibes	2a	-1.06917	-0.41022	0.0641	-0.66471
62_f	Tibes	2a	-0.86448	-0.31841	0.04519	-1.20065
62_g	Tibes	2a	0.17637	2.08993	0.85072	1.26255
62_h	Tibes	2a	0.36665	0.32145	0.43997	-0.20868
294_J	Tibes	2a	-0.7938	0.23255	-1.10862	-0.67083
62_J	Tibes	2a	-0.14957	-0.27875	1.34832	-1.35706
FS.246_a	PO-43	2a	-0.70797	0.04245	-0.4428	0.23742
FS.253_a	PO-48	2a	-0.58608	0.88888	-0.1325	1.00874
FS.76_a	PO-42	2a	-1.06785	0.31865	-0.54179	0.28975
FS.76_c	PO-42	2a	-0.14735	0.44476	-0.1578	0.04644
62_i	Tibes	2a	-1.2079	-0.44799	0.24066	-0.14556
FS.232_a	PO-43	2a	-0.49501	0.17641	-0.12775	-1.36183
FS.245_b	PO-43	2a	0.05482	0.76003	-0.01655	1.0549
FS.246_b	PO-43	2a	0.08745	1.40932	-0.67999	0.58106
FS.255_a	PO-48	2a	0.31314	0.38641	0.75802	-0.87041
FS.263_a	PO-48	2a	-0.76678	1.5532	-0.31632	-0.83561
FS.75_b	PO-42	2a	-0.2214	0.67203	-0.5276	-0.30865
FS.76_b	PO-42	2a	-0.721	1.26848	-0.4049	-0.86005
FS.76_g	PO-42	2a	-1.08079	0.08468	-0.03842	0.09738
62_b	Tibes	2b	-0.63371	-1.32776	2.78529	-0.56202
FS.245_a	PO-43	2b	-0.01427	-0.5833	1.63775	0.86936
117_g	Tibes	2b	0.45312	-0.09745	0.658	3.87167
FS.76_f	PO-42	2b	-0.20383	-1.04994	2.63378	-0.1439
117_J	Tibes	2b	0.01731	-0.79503	1.9585	1.34085

Annex 7 Loading plot for PC1, PC2, PC3, and PC4.

	PC1	PC2	PC3	PC4
MgO	0.859	0.300	-0.185	0.082
Co	0.856	0.195	0.141	0.244
Ni	0.852	0.188	-0.080	0.073
Cr	0.785	0.189	0.040	0.087
Mn	0.782	0.109	0.080	0.525
Cu	0.743	0.156	0.053	0.394
Th	-0.676	-0.010	0.094	0.021
Ga	-0.660	0.275	0.433	-0.191
Al2O3	-0.635	0.382	0.270	-0.139
SiO2	0.578	-0.482	-0.393	-0.287
Zn	0.518	-0.200	-0.127	0.340
U	0.201	-0.782	-0.232	0.126
Pb	0.170	-0.735	-0.363	-0.034
CaO	0.032	0.677	-0.058	-0.137
Sb	-0.403	-0.643	0.056	-0.041
Zr	0.366	0.606	-0.140	0.189
S	-0.341	0.581	-0.048	0.010
TiO2	-0.052	0.516	0.918	-0.130
V	0.056	-0.468	0.835	-0.168
Nb	-0.046	0.196	0.734	0.066
Fe2O3	-0.022	-0.002	0.639	0.264
Na2O	0.354	-0.151	0.612	0.329
Ba	0.417	0.220	0.118	0.843
K2O	-0.527	0.308	-0.313	0.778
Rb	0.357	-0.265	0.266	0.730
Sr	0.433	0.213	0.061	0.611
P	0.221	0.140	-0.038	0.432