

Evaluating paleoenvironmental and landscape mobility dynamics: stable isotope  
and strontium isotope analyses of ostrich eggshell at Spitzkloof Rockshelter, South  
Africa

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## Abstract

Recent debates have focused on the roles that environmental change played in cultural shifts associated with the appearance of ‘modern’ symbolic behaviour in *Homo sapiens*. Marginal environments such as the arid to semi-arid region of the Northern Cape in South Africa continue to have sporadic climatic reconstructions, making it difficult to evaluate comparisons between modern symbolic behaviour and environmental fluxes. The appearance of ostrich eggshell (OES) beads at archaeological sites in South Africa by 50 ka has been thought to represent early forms of personal ornamentation reflecting modern human behaviour. Recent ethnographic research has shown that OES beads were traded and exchanged between networks of hunter gatherers as a method of moderating risk across the southern African landscape (Mitchel, 1996). Isotope analyses were performed to assess climatic changes and where hunter gatherers might have been using the landscape for subsistence and risk moderating strategies. Strontium isotope analyses can be used as a tool for evaluating the movement of objects as they reflect a specific geochemical signature sourced from the landscape. Carbon, oxygen, and strontium isotope ratios were measured on six modern and twenty-eight archaeological OES fragments and twelve OES beads from Spitzkloof Rockshelter in the Northern Cape radiocarbon dated to between 52-14 ka cal BP using ostrich eggshell. Variation in  $^{87}\text{Sr}/^{86}\text{Sr}$  ratios was observed between modern and archaeological fragments. Interestingly, the beads show more variance when compared to the fragments, suggesting that they demonstrate a wider range of transport over the fragments. The carbon and oxygen isotope data show a general trend consistent with the published literature for MIS 3 (57-29 ka) and MIS 2 (28-14 ka). The  $\delta^{13}\text{C}$  values during MIS 2 indicate a steady increase in  $\text{C}_3$  habitat preferences, which is likely indicative of the increased landscape area for grazing as a result of the lowered sea levels (Dewar and Stewart, 2016). It is also at this time that we see the introduction of ostrich eggshell beads displaying a strong coastal  $^{87}\text{Sr}/^{86}\text{Sr}$  signal indicating that hunter-gatherers likely exploited these large stretches of the landscape beside the coast at the time when sea levels were very low. Isotopic analyses of ostrich eggshell represent a novel approach to exploring questions of environmental fluctuations in combination with resource exploitation and movement of goods between regions.

## 1. Introduction

The Middle Stone Age (MSA) which dates between ca. 280 and 30 ka (Jacobs et al., 2008) and Later Stone Age (LSA) between ca. 40 and 2 ka (Backwell et al., 2008) in southern Africa have been argued as a time for the shaping of ‘modern’ *Homo sapiens*. MSA archaeological sites along the coast show that this time period was associated with coastal adaptations such as the procurement of marine resources as well as increased forms of symbolic material culture (Marean et al., 2007; Will et al., 2013; Jerardino, 2016). Early forms of symbolic meaning include engraved ochre blocks and shell beads from Blombos Cave (75 ka) (Henshilwood et al., 2002; Henshilwood et al., 2004; d’Errico et al., 2005), the presence of ostrich eggshell in the form of containers, a flask mouth found at Apollo 11 dated to 70 ka (Vogelsang et al., 2010), engraved ostrich eggshell fragments at Diepkloof Rockshelter (60 ka) (Texier et al., 2010; Texier et al., 2013) and manufactured beads by 50 ka (Kandel and Conard, 2005; Bednarik, 2005; Texier et al. 2010). These forms of symbolic meanings appear across the southern African landscape, at different times and different technological contexts (Texier et al., 2010; Hodgson, 2014; Lee-Thorp and Ecker, 2015).

Ostrich eggshells (OES) were likely first used by hunter-gatherers as containers for holding items such as water and when shells would break, the fragments would sometimes be recycled into beads (Kandel and Conard, 2005). Recent ethnographic research has shown that ostrich eggshell beads were traded and exchanged between networks of hunter-gatherers as a method of moderating risk across the southern African landscape (Wadley, 1987; Mitchel, 1996; Hitchcock, 2012). Fluctuations in ecological productivity on the landscape would lead to unpredictable food and water supply thus surviving on such a marginal landscape meant keeping close ties with other hunter-gatherer networks. When key food or water resources were scarce in a group’s home area, these exchange networks or partnerships with other groups, provided shared resources until conditions improved (Mitchell, 1996). San groups in Botswana partake in reciprocal exchanges to maintain social ties but also to reduce the economic risks to their groups (Cashdan, 1985). Thus, the movement of goods between regions has been considered an important component in the MSA/ LSA and researchers have attempted to study this but it has



been difficult to identify. However, there is still debate about whether or not research can or should use this form of analogy for the Middle Stone Age.

Over the last couple of decades, archaeologists have applied geological scientific methods such as isotope geochemistry to investigate historical climate reconstructions, paleodietary studies and population movements (Sealy et al., 1991; Gilbert et al., 1994; Johnson et al., 1997; Sealy, 2006; Slater et al., 2014; Lee Drake et al. 2014). While carbon and oxygen isotopic analyses have been the most frequently used in archaeological sciences, strontium isotopic analyses have gained momentum as a consistent tool for tracing geochemical signatures through the environment. Few geochemical studies have been performed on ostrich eggshell fragments and beads despite the fact that these items have been consistently recovered at archaeological sites across southern Africa (Mellars, 2006). Strontium isotope analyses can be used as a tool for evaluating the movement of objects, animals, and people as they reflect a specific geochemical signature sourced from the landscape (Price et al., 2002; Knudson et al., 2010; Radloff et al., 2010; Copeland et al., 2011; Copeland et al., 2016).

Ostrich eggshell use has mainly focused on retrieving carbon-14 data for dating purposes as well as paleoenvironmental information through light stable isotope analyses. In this study, I apply in-depth analyses on ostrich eggshell through two research avenues: strontium isotope analyses to infer landscape and mobility dynamics, and paleoenvironmental applications using carbon and oxygen to reconstruct past environments during occupation at Spitzkloof Rockshelter.

## 2. Objectives

The purpose of this thesis is to develop an in-depth assessment into the roles that climatic fluctuations played in the behaviour of hunter gatherers, specifically their subsistence and risk moderating strategies. This will be conducted on modern and archaeological ostrich eggshell samples using light stable isotope and strontium isotope analyses. Fitting with the AMEMSA project framework, these methods will provide new insights into the past, including paleoenvironmental reconstructions, and landscape and mobility dynamics for the Namaqualand region of South Africa.

### Objective 1: Paleoenvironmental Applications

The aim of this objective is to assess past climatic changes in the area during the occupation at Spitzkloof Rockshelter using carbon and oxygen light stable isotope analyses and eggshell thicknesses as a proxy for aridity (Rahn et al., 1979; Brand et al., 2012; Ecker et al., 2015).

- What is the distribution of  $\delta^{18}\text{O}$  and  $\delta^{13}\text{C}$  at Spitzkloof Rockshelter through time? Is there a statistical difference between the strata through time?
- The null hypothesis of this objective is that there are no significant differences within the stable isotopes through time (ie. through the strata).
- If the null hypothesis is rejected, that there are statistical differences, what does this mean for climatic changes during the Middle and Later Stone Age? How does this associate with the strontium isotope values in the fragments and beads?
- If the null hypothesis cannot be rejected, no statistical difference, how does it compare to published paleoenvironmental data for the region?

### Objective 2: Landscape and Mobility Dynamics

The aim of this study is to assess whether strontium isotope analyses can be used for tracking past movements of ostrich eggshell and ostrich eggshell beads on the landscape.

- Are there differences in the  $^{87}\text{Sr}/^{86}\text{Sr}$  between the modern and archaeological samples? Are there differences between the beads and the fragments? Is there a coastal versus terrestrial signal in the strontium values?

- The null hypothesis of this objective is that there are no differences between the modern and archaeological fragments and the ostrich eggshell beads.
- If the null hypothesis is rejected, it would mean that the samples have strontium isotopic variability and thus could be exhibiting different localities. I will investigate this further by comparing the results to known  $^{87}\text{Sr}/^{86}\text{Sr}$  values for given regions in the published literature and to the paleoenvironmental data to discern if this movement is the result of a changing environment.
- If the null hypothesis cannot be rejected, no  $^{87}\text{Sr}/^{86}\text{Sr}$  isotopic difference, I will investigate this further by discerning whether the values are still local or non-local to the site by comparing them to known  $^{87}\text{Sr}/^{86}\text{Sr}$  values for given regions in the published literature.

### **3. Background Information**

#### *3.1 Evidence of Signaling Theory in the Middle and Later Stone Age*

The origins or emergence of modern human behaviour has had two generalized pathways in the literature: the first states that there was a late behavioural emergence in Africa roughly 50 kya (Henshilwood et al., 2004), while the second states that it was a gradual increase in behavioural complexity in Africa beginning approximately 250 kya (Lahr and Foley, 1998; McBrearty and Brooks, 2000; Henshilwood et al., 2004). However, there is disagreement on how modern behaviour is defined (Henshilwood & Marean, 2003) and how we demonstrate this through the archaeological record. A few researchers have suggested a list of behavioural traits that could be theorized as evidence for modern behaviour such as blade technology, processing of ochre, worked ostrich eggshell and shellfish fragments (Ambrose, 2001; Bouzougar et al., 2007) however some researchers argue that such a list may not be the best approach for the Middle and Later Stone Age periods (d'Errico and Stringer, 2011). Of particular interest to researchers are the manufacturing of beads from ostrich eggshell and shellfish fragments as way of assessing early stages of communication because it is suggestive of early innovative technology (Texier et al., 2010).

There are limitations to the amount of hypothesizing that can be done about how these people may have communicated. Kuhn (2014) argues that researchers should turn their eyes to biological signaling theory as a means for analyzing signaling strategies of the past. Biological signaling theory proposes that adaptive contexts and evolutionary outcomes can be seen through different signaling strategies (Kuhn, 2014; Sterelny and Hiscock, 2014). Through animal communication, there is a distinction between costly and non-costly signals: costly signals result in consequences for the individual who displays messages such as bright colours inevitably drawing in predators or when the signaler wants to convey a dishonest message. In Paleolithic archaeology, costly signaling has been used as a term to explain showy but inefficient forms of hunting and elaborate funerary rituals (Kuhn, 2014), ultimately adding little or no benefit to the individual. These principles of biological signaling theory could then be applied to the archaeological record, specifically what we see as traits of modern human behaviour in the Middle Stone Age, South Africa. The most common signaling media found during the Middle Stone Age is the introduction of pigments (ochre) and personal ornamentation (Henshilwood et al., 2004; Jacobs et al., 2008; Henshilwood & d’Errico, 2009; Shultz and Maslin, 2013).

One way to get a glimpse into past hunter gatherer societies is to look to recent descendant populations such as the !Kung San people living in the Kalahari Desert in Botswana and using it as an analogy to hypothesize past networks and ways of survival. Past hunter-gatherer societies used ostrich eggshell for decoration of their bodies, social information, and reciprocal exchange networks between tribes and social rank (Kandel & Conard, 2005; Wadley, 2010; Stiner, 2014). Through the archaeological record, researchers have noticed a change in how ochre and ostrich eggshell were manufactured and employed, adding to the complexity of what signals were being sent. For ochre production, a change in the geological mineral materials is seen through time with an increase in colour diversity as well as production methods (Wadley, 2010; Henshilwood et al., 2014). Ochre being one of the earliest forms of communication is argued to be associated with a non-costly signal (Kuhn, 2014). Depending on geographic location, areas could have several different geological outcrops, all with the materials to make ochre. If ochre was used to decorate the body, the person only has a finite space of skin to put the ochre on and thus a finite quantity of signaling to others. Ostrich eggshell bead sizes have changed over time from small beads with large aperture holes for LSA hunter gatherers to large more robust beads in pastoralist societies (Jacobson, 1987; Hodgson, 2014; Stiner, 2014). The

introduction of ostrich eggshell and shellfish beads lends to greater potential of costly signaling. The productions of such intricate pieces with no literal survival purposes have been used for personal decoration and social status. The quantity of beads on an individual can be counted and when worn with numerous beads can show costly signaling (Kuhn, 2014).

### *3.2 Biogeographical Theory for the AMEMSA Project*

The AMEMSA project (Adaptations to Marginal Environments in the Middle Stone Age) aims to look at: how, when, and under what environmental conditions were marginal environments occupied in the Middle Stone Age. While sites in southern Africa have shown evidence for symbolic behaviour, the majority of this research has been geographically focused along the southern coastline, an area that has consistently had rich and predictable resources. The biogeographical model developed by Yellen (1977), hypothesizes that when environments are variable, it is more advantageous for hunter-gatherer groups to remain adaptive or mobile, practicing subsistence strategies that exploit a diverse range of food resources. Spitzkloof Rockshelter, located in northern Namaqualand of South Africa contains occupational evidence during the Later Stone Age and late Middle Stone Age. Following a biogeographic model, project AMEMSA at Spitzkloof Rockshelter investigates whether hunter-gatherers occupying these harsh environments were adapting to changes in the environment and/or changes to the procurement of food resources in order to remain resilient. The objectives outlined for this research will specifically use a biogeographical model to interpret these hunter-gatherer occupations at Spitzkloof from multiple avenues under the framework of the AMEMSA project.

### *3.3 Geologic Setting of Namaqualand and Richtersveld*

Namaqualand covers approximately 45 000 km<sup>2</sup> and is made up of seven regions based primarily on the flora, climate, and structure of the physical environment (Desmet, 2007). It is located between the Atlantic Ocean and Bushmanland 100 km to the east and bounded by the Orange River in the north and Olifants River to the south. The region is complex and diverse in geological substrates (Figure 1). In the north is the Richtersveld bioregion where Spitzkloof Rockshelter is located. The Richtersveld consists of mostly metamorphic rocks including

quartzite, schists and shales that are arranged in a north-south alignment, making the area known for its remote valleys and heavily eroded peaks (de Villiers and Söhnge, 1959). Spitzkloof Rockshelter is located within the Gariep supergroup dated to 750-650 Ma and is approximately 40 km inland from the modern coastline. The area is now mainly made up of coastal sand dunes from younger aeolian and alluvial sediments from increased erosion over the years (Dewar and Stewart, 2012, Reid, 2015).

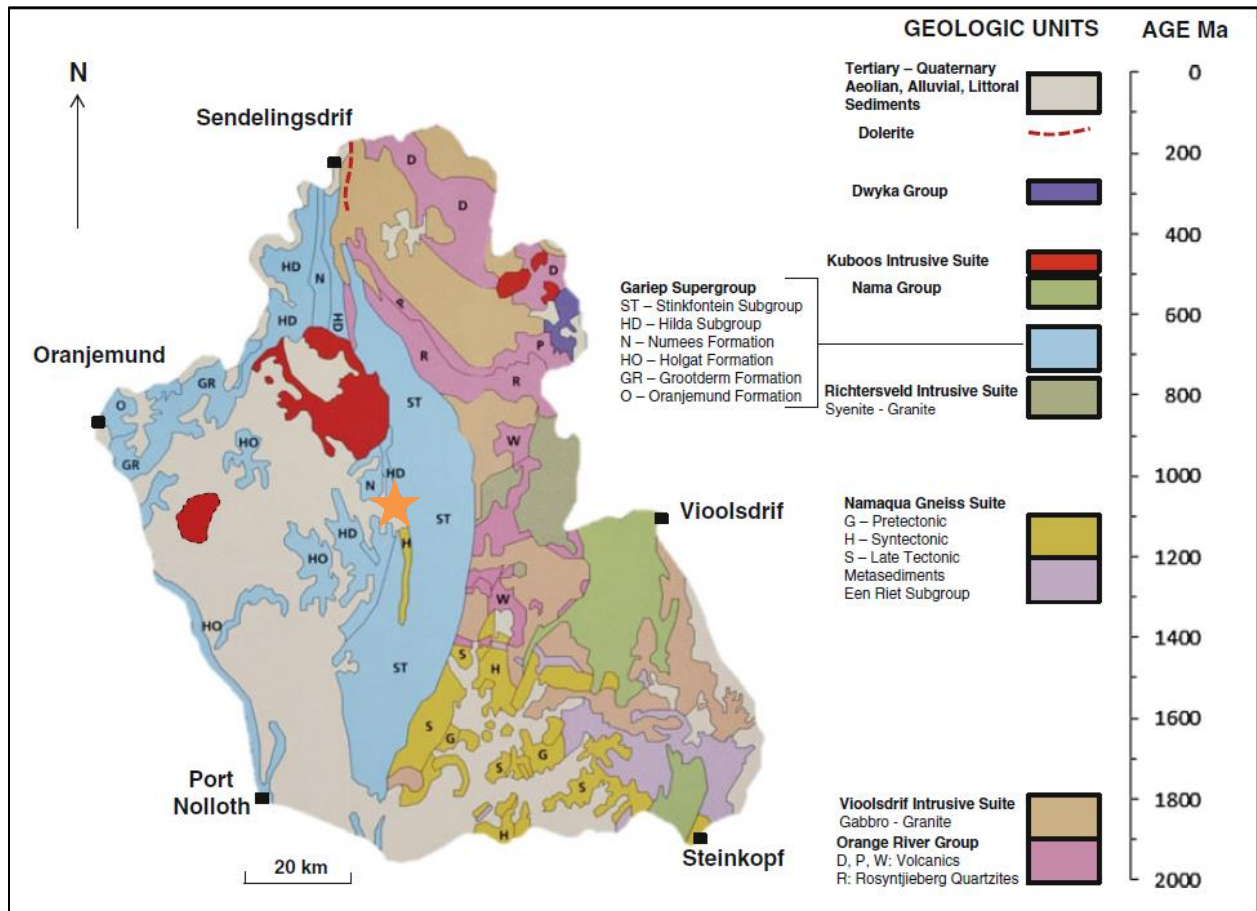


Figure 1: A simplified geological map for northern Namaqualand (Reid, 2015). The orange star represents the location of Spitzkloof.

### 3.4 Floristic Setting

The northern part of Namaqualand is listed under the Succulent Karoo floristic region consisting mostly of  $C_3$  and succulent vegetation and has been recognized as a hotspot for biodiversity and endemism (Cowling et al., 1999; Driver et al., 2003). Currently, Namaqualand

receives about 50 mm in the northwest to 200 mm further south however the area generally receives less than 150 mm during the winter months (Cowling et al., 1999). Namaqualand is located almost entirely within the winter-rainfall zone meaning that the majority of precipitation that does fall happens mostly within a three to four month period from June-September. The presence of the Atlantic Ocean next to Namaqualand helps to moderate the temperatures (max summer temperature  $< 30^{\circ}\text{C}$ ). This moderating effect also has influences in the winter months when low pressure formations off the coast cause hot, dry air to move westward from the interior escarpments (max winter temperatures  $> 35^{\circ}\text{C}$ ) (Desmet, 2007). The constant flow of air between the ocean and land also provide coastal fogs that can extend as far as 80 km inland (MacKellar et al., 2007). We still do not know the degree of which coastal fogs support the region hydrologically but they do provide an alternative source of moisture to the  $\text{C}_3$  and succulent plants (Weldeab et al., 2013).

### *3.5 Paleoenvironments of Namaqualand*

Paleoenvironmental data for the region have come from offshore cores from the Namaqua Mudbelt (Hahn et al., 2015), hyrax middens (Carr et al., 2006; Chase et al., 2010; Carr et al., 2016), and other archaeological sites along the fringes of the Namaqualand region such as Apollo 11 (Vogelsang et al., 2010), an MSA and late LSA occupational site approximately 100km northwest of Spitzkloof, and Erb Tanks, a Middle and Later Stone Age site in central Namibia (McCall et al., 2011). A synthesis by Chase and Meadows (2007) combined these sources to interpret the movements of the boundaries of the winter rainfall zone for the region during glacial and interglacial periods; the study concludes that humid conditions persisted during glacial periods and dryer conditions during interglacial times. Glacial and interglacial periods also promote changes to the position of coastlines due to sea level fluctuations. Changes to the coastline for the region could have significantly influenced the extent to which the winter rainfall atmospheric systems and coastal fogs extended inland, altering the availability of water and nutrients to the flora and fauna (Dewar and Stewart, 2016). Dewar and Stewart (2016) synthesize what is currently known about past environments for the Namaqualand region between MIS 6-2 as well as how the distance to the coastline would have fluctuated. Dated strata

at Spitzkloof Rockshelter align within MIS 2 (29-14 ka) and show two pulses of occupation (23.5-23 ka cal BP and 19-17 ka cal BP) which is also when the distance to the coast was at a near maximum (Table 1) with another pulse of occupation occurring during MIS 3 at approximately 51 ka (Dewar and Stewart, 2016). The region likely experienced cool moist conditions with increased water availability during MIS 2. Dewar and Stewart (2016) propose that the occupation and colonization of Namaqualand's coastal plains by grasslands, specifically during MIS 2, was the result of either increased precipitation with the movement of the coastline westward or reduced evaporation due to lowering temperatures (Stewart and Jones, 2016 pp.10). Dewar and Stewart (2016) argue that an increase of surface land area due to lower sea levels would coincide with increased carrying capacity for larger grazing species such as ungulates. Apollo 11 also exhibits archaeological faunal material of grazing species for this time period (Vogelsang et al., 2010). By the end of MIS 2, around 14 ka, the environment for the region was characterized by increasing aridity which approached present day values; this aridification coincides with a decrease in occupation at both Apollo 11 and Spitzkloof (Dewar and Stewart, 2016).

### *3.6 The Biology and Ecology of Ostriches in South Africa*

The South African ostrich (*Struthio camelus australis*) is the main subspecies found in southern Africa. The preferred habitat of this large flight-less bird includes well vegetated semi-arid open grasslands. They have evolved to be able to tolerate harsh weather conditions such as deserts which exhibit extreme temperature shifts with little water. The bulk of an ostrich's water needs are obtained through the vegetation it eats, particularly by feeding in the morning and on hygroscopic plants which absorb moisture through the air (Louw, 1972; Bertram, 1992). The ostriches eat a variety of species and plants including succulents, shrubs, grasses and small fruits (Bertram, 1992). Ostriches tend to lay on average 8-12 eggs at the end of, or just after, the rainy season and like most animals, depends on the availability of nutrients to have a large and successful hatching and rearing period.

The biometry of an ostrich's eggshell is made up of five layers; three calcified layers which preserve well and two non-calcified layers that do not preserve in the archaeological record (Ecker et al., 2015). Pore canals are also present through these layers which help the



embryo obtain oxygen via diffusion through the pores and into the shell. These layers along with the pore canals protect the embryo inside as well as supply adequate oxygen for development (Board, 1980). Eggshell conductance which includes eggshell thickness and porosity is a measure of the quantity of gas (oxygen and carbon dioxide) that can diffuse through the pores per unit time; both parameters can change in size in order for the embryo to remain in optimal conditions. For example, thicker shells and low porosity indicate a decrease conductance or gas exchange with the atmosphere and vice versa. These parameters could be used as additional proxies for past environmental conditions during the time that the eggs are being laid (Ségalen et al., 2006; Ecker et al., 2015), because there is a relationship between egg conductance and humidity (Board and Scott, 1980; Ecker et al., 2015). Ostriches that breed in very arid environments tend to have a thicker eggshell and a low degree of eggshell porosity, aimed at protecting the embryo from drying out and avoiding water loss. Inversely, ostriches that breed in humid environments will have an increased degree of porosity and thinner shell to increase gas exchange. Research by Stein and Badyaev (2011) demonstrated that these changes can occur rapidly within a single ostrich over each season.

### *3.7 Stable Isotope Analyses of Archaeological OES*

Light stable isotope analyses are practiced extensively in archaeological studies for paleoecological and paleoclimatic reconstructions. Central to the analysis of light stable isotopes is the examination of fractionation, or partitioning, of an element's isotopes through the environment. Light elements such as carbon, oxygen, and hydrogen have specific isotope fractionations as a result of changes in pressure and temperature, meaning they are mass dependent. Strontium has a larger atomic mass in comparison to carbon, oxygen and nitrogen making its kinetic and equilibrium fractionation numbers negligible, meaning it retains the same  $^{87}\text{Sr}/^{86}\text{Sr}$  ratio from its original location (Bentley, 2006). We can use ostrich eggshell for stable isotope analyses because the ostrich eggshell is composed of mineralized calcite (96%) and organic proteins (4%). This dense ratio of calcite to protein makes the eggshell highly resistant to decay, weathering and burning (Johnson et al., 1997).

An important aspect to consider when using ostrich eggshell for stable isotope analyses, specifically strontium, is the occurrence of pseudo replication. Within each stratigraphic unit

there can be hundreds of ostrich eggshell fragments, several of which could have come from the same individual (one whole eggshell). When comparing strontium isotopic values, another stable isotope such as carbon and oxygen should be used to discern the number of individuals present in the sample size. One eggshell can present only one  $^{87}\text{Sr}/^{86}\text{Sr}$ ,  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  value, assuming fragments with the same strontium, carbon and oxygen values came from the same eggshell.

### *3.7.1 Carbon Isotopes*

Carbon isotopes have been used extensively as a tool for inferring paleovegetation. Plants utilizing  $\text{C}_3$ ,  $\text{C}_4$  or CAM pathways fractionate atmospheric carbon differently, resulting in a geochemical tracer, which can be linked to a photosynthetic pathway.  $\text{C}_3$  (Calvin pathway) plants include trees, shrubs, and some grasses,  $\text{C}_4$  (Hatch-Slack) plants include some tropical grasses and shrubs while CAM (Crassulacean acid metabolism) plants, include some succulents which may adopt both  $\text{C}_3$  and  $\text{C}_4$  photosynthetic pathways (Johnson et al., 1998). The average  $\delta^{13}\text{C}$  of  $\text{C}_3$  plants in South Africa is approximately -26.5 ‰ and  $\text{C}_4$  grasses is approximately -12.6‰ (Johnson et al., 1998).

The  $\delta^{13}\text{C}$  values found from the inorganic portion of an eggshell reflect the vegetation consumed by the animal during the breeding season (Von Schirnding et al., 1982; Bertram, 1992). The fractionation between the plant and eggshell through the ostrich's diet is approximately 15 ‰ (Johnson et al., 1997; Johnson et al., 1998; Lee-Thorp and Ecker, 2015). The ostriches breed and lay their eggs after the rainy season which means that the isotopic composition found in the eggshell will be representative of a brief environmental signal (Bertram, 1992; Johnson et al., 1997).

### *3.7.2 Oxygen Isotopes*

Ostriches are non-obligate drinkers and obtain water mainly through the plant-leaf tissues they eat. The  $\delta^{18}\text{O}$  of the plant-leaf water increases with decreasing relative humidity and increasing temperature (Johnson et al., 1998). Thus, a very high  $\delta^{18}\text{O}$  would correspond to low humidity and increased air temperatures. According to Sharp (2007), the fractionation of  $\delta^{18}\text{O}$  between body-water and the calcium carbonate of the ostrich eggshell is roughly 30 ‰.

### 3.8 Strontium Isotope Analysis

Strontium is an alkaline earth metal with the element symbol Sr and an atomic number of 38. Strontium can readily substitute for calcium because it has a +2 valence and thus behaves similarly, making ostrich eggshell an ideal candidate for strontium isotope analyses. Strontium has four naturally occurring stable isotopes,  $^{84}\text{Sr}$  (~ 0.56%),  $^{86}\text{Sr}$  (~9.87%),  $^{88}\text{Sr}$  (~82.53%) which are non-radiogenic and  $^{87}\text{Sr}$  (~7.04%) which is radiogenic, produced from  $\beta$ -decay of  $^{87}\text{Rb}$  (Elderfield, 1986, Bentley, 2006). Rubidium, which is very similar to potassium in terms of its ionic radius, has a beta decay half-life of  $4.88 \times 10^{10}$  years (Price et al., 2002; Knudson et al., 2010). In geological materials such as feldspar, muscovite, and biotite, strontium substitutes for calcium and rubidium substitutes for potassium (Bentley, 2006). The decay of rubidium to strontium ( $^{87}\text{Rb}/^{87}\text{Sr}$ ) has long been studied for its uses on dating geological materials for geochronological studies. The ratio of  $^{87}\text{Rb}/^{87}\text{Sr}$  is used to measure the relative proportions of the decay of rubidium and the  $^{87}\text{Sr}/^{86}\text{Sr}$  strontium (Aberg, 1995). While rubidium has a constant decay rate, it weathers and erodes from geological materials at varying rates, creating variations in  $^{87}\text{Sr}/^{86}\text{Sr}$  ratios seen in the environment. The  $^{87}\text{Sr}$  content found in a rock or water sample is dependent on how much rubidium is in the rock or how much rubidium has weathered from its parent material and into the tributary system.

Geochemical signatures, specifically strontium isotopes can be used to source a material to a geological area. The premise is that the strontium isotopic ratio in ostrich eggshell is derived from plants consumed by ostriches; the ratio in those plants reflects that of the strontium available to plant via the soil and/or geological substrate underneath (Bentley, 2006; Ericson, 1985). Rivers and their tributaries have shown to provide a good basis for predicting the  $^{87}\text{Sr}/^{86}\text{Sr}$  available to plants and thus animals at different locations because they are depositing the same materials onto floodplains (Bentley, 2006). In food webs, strontium isotopes do not fractionate with trophic levels like that of nitrogen (Blum et al., 2000), thus the eggshell carries the same isotopic signature as the organism's diet (Graustein, 1989; Blum et al., 2000; Slater et al., 2014). For understanding strontium isotopic variability on the landscape, many studies have used specific ranges to identify or accurately reflect variation between samples. Differences in a strontium ratio  $^{87}\text{Sr}/^{86}\text{Sr} > 0.0002$  to the fourth decimal place are considered meaningful in identifying these variations (Price et al., 2012; Slater et al., 2014). For this study project, a ratio

$^{87}\text{Sr}/^{86}\text{Sr} > 0.001$  will be used due to the fact that two different methods were used for strontium analyses (the fragments vs. the beads). Studies along the southwestern Cape of South Africa have also used the underlying bedrock geology to help infer strontium variability (Sealy et al., 1991; Balasse and Ambrose, 2002). A study conducted by Sealy et al., (1991) used a cross-sectional approach of strontium variability from the coastline which would demonstrate a value close to the marine average to further inland which consisted of old Cambrian and pre-Cambrian rocks enriched in  $^{87}\text{Sr}$ . This method was further strengthened when carbon isotopes were added to the interpretation to infer terrestrial system variability (coastal versus inland food sources).

### *3.8.1 Defining $^{87}\text{Sr}/^{86}\text{Sr}$ Strontium Ranges*

When working with strontium isotopic variability, it is important to define the local  $^{87}\text{Sr}/^{86}\text{Sr}$  ratio range for local vs. non-local values. This is important for determining population movements of migratory people at archaeological sites (Slater et al., 2014) and for environmental scientists who are investigating restoration techniques for documenting species populations (Kennedy et al., 2000; Kennedy et al., 1997), both examples approach this issue differently. For human migration studies, Slater et al., (2014) first assessed strontium isotopic variability within the samples, and then defined a range with a 95 % confidence interval for local vs. non-local people. For documenting salmon populations, Kennedy et al., (2000) first assessed strontium isotopic variability in a closed watershed catchment area using geological maps before sampling, which helped to maximize areas with strontium variability. For this research, I will first be assessing the  $^{87}\text{Sr}/^{86}\text{Sr}$  variability for the region for each tributary system to maximize the largest variability possible.

Factors to keep in mind when sampling modern non-migratory animals for  $^{87}\text{Sr}/^{86}\text{Sr}$  values include anthropogenic influences such as fertilizers and human food. Bentley et al., (2004) suggests that a more robust way to assess local values would be to find archaeological faunal remains derived from non-migratory animals. To this effect, these animals would be ingesting  $^{87}\text{Sr}/^{86}\text{Sr}$  values for that area without the added variables of anthropogenic influences. Understanding where particular species are found for the region and what their home ranges are will aid in allowing the preliminary assessments for strontium analyses to move forward. For this research, non-migratory animals were difficult to collect for the location thus local geologies

were first considered, including watershed catchment zones, groundwater movement, topography, and geological substrates. Sediments located in tributary catchment areas were used to define the strontium isotopic variability for the region because rivers carry most of the weathered sediments from the interior to the ocean (Bentley, 2006) (see Materials and Methods section).

## **4. Materials and Methods**

### *4.1 Field Sampling*

The samples were first clustered according to the archaeological layer they were associated with, then to the radiocarbon dated AMEMSA layer (Dewar and Stewart, 2012). Some layers had more samples than others; despite the fact that the presence of eggshells was continuous through the distribution, the number of beads and fragments were variable through the sequence. Fragments and beads were inspected for taphonomic processes as these could potentially alter the isotopic composition within the shells. Table 1 summarizes the layers given, their associated contexts, calibrated radiocarbon dates, the distance from the coastline (Dewar and Stewart, 2016) and their assigned groups by the author. The number of samples for both the carbon and oxygen isotope analyses and strontium isotope analyses were constrained given the costs of the procedures. The same fragment (sample) was used for both strontium isotope analyses and carbon and oxygen isotope analyses, with the exception of one modern fragment (OES-08) which was not analyzed for carbon or oxygen.

Table 1: Summary of the assigned groups and their associated strata, contexts, and radiocarbon ages of ostrich eggshell and distance from the coast (km) from Spitzkloof A, South Africa (Dewar and Stewart, 2016).

Group	AMEMSA Layer	Context	Date in $^{14}\text{C}$ BP	Calibrated dates in cal BP	Distance from the coast (km)
0	Modern	0	0	0	41
1	1	1-4	$14,400 \pm 70$	17,390 - 17,090	56
2	2	5, 5a	N/A	N/A	56-58
3	3	6-10	$15,200 \pm 50$	18,300-18,110	58
4	4	11, 12	$16,250 \pm 60$	19,460-19,240	62
5	5, 6, 7	13-17	$19,750 \pm 80$	23,130-23,670	57
6	8	18, 19	$51,150 \pm 850$	N/A	48

*Note:* Experiments have shown that fossil OES can exhibit older values ( $180 \pm 120$  years too old) (Vogel et al., 2001), thus 180 yrs were subtracted before calibration (Dewar and Stewart, 2016).

#### 4.2 Eggshell Thickness Measurements

For each of the 1,074 fragments from all contexts, shell thicknesses were measured using a caliper, accurate to 0.01mm. The author carried out all measurements. The thickness measurements were assembled into the associated Groups. An ANOVA analysis was first proposed to address whether there were differences in eggshell thicknesses between groups. The assumptions for the ANOVA (tests for normality and equal variances) were not met in the data using the Shapiro-Wilk Test for normality and the Levene's test for homogeneity of variances even after attempts to transform the left-skewed measurements. A Kruskal-Wallis *H*-test is a non-parametric method that does not assume normality of the residuals. The *H*-test demonstrated significant differences between groups which led to performing a Dunn's test for pairwise comparisons with and without a Bonferroni correction. The Bonferroni correction adjusts the p-

value when multiple analyses are being carried out on one data set. The analyses were performed in R statistical software (R Core Team, 2016) using the packages *dunn.test* (Dinno, 2016) and (Fox and Weisberg, 2011).

#### *4.3 Carbon & Oxygen Isotope Analysis*

All analyses for stable isotopes were performed in the Department of Archaeology at the University of Cape Town. Samples were cleaned by sanding a corner of each fragment which removed the sediment and debris from the shell. The fragment was then removed and crushed in an agate mortar and pestle to achieve a powder consistency. Approximately 0.1 mg of sample was used for the Gasbench II, connected to a continuous flow isotope mass spectrometer. The carbonate fraction of the sample was reacted with 100% phosphoric acid at 70°C to release the carbon dioxide for the measurement of  $^{13}\text{C}/^{12}\text{C}$  and  $^{18}\text{O}/^{16}\text{O}$ . The samples were calibrated against NBS19, IAEA CO-1, and MERCK and interspersed between the samples. Light stable isotope analyses could not be performed on the beads as this would cause complete destruction of archaeological artifacts, which was not permitted under the research license.

An ANOVA was used to test for statistical differences between the groups for both carbon and oxygen isotopic ratios. Sample sizes in Groups 2 and 6 were too low, thus those groups were removed before statistical analyses.

#### *4.4 Mapping Strontium Isotope Variability*

Identifying the origin of the ostrich eggshell requires known values for strontium isotope ratios from potential source regions. Thus, Strontium isotope ratios were collected from published literature for the Namaqualand region. Samples considered from published works included geological bedrock material, eroded sediments along the tributaries, suspended load sediments from rivers and tributary water where possible. Five tributaries were chosen in the region: Olifants Tributary, Buffels Tributary, Holgat Tributary, and the Orange River subdivided into lower and upper; these values can be found in Appendix B. Since strontium is best expressed as a mixing system of bedrock, atmosphere, tributary water, and eroding sediments,

the values for each region were averaged to gain a complete representation of how the vegetation in that area would express a strontium isotopic value; we assume that vegetation signals would be coeval with those recorded in ostrich eggshells (see above). A seawater strontium value was also included into all regions except the Upper Orange because the Atlantic Ocean and Benguela Current play a large role in bringing precipitation and moisture to the plants mostly during the winter rainfall months and coastal fogs (Figure 2). The Upper Orange samples were not included in the seawater signal as these values as they come from the summer rainfall zone further upstream and are not influenced by the Atlantic but rather the Pacific.

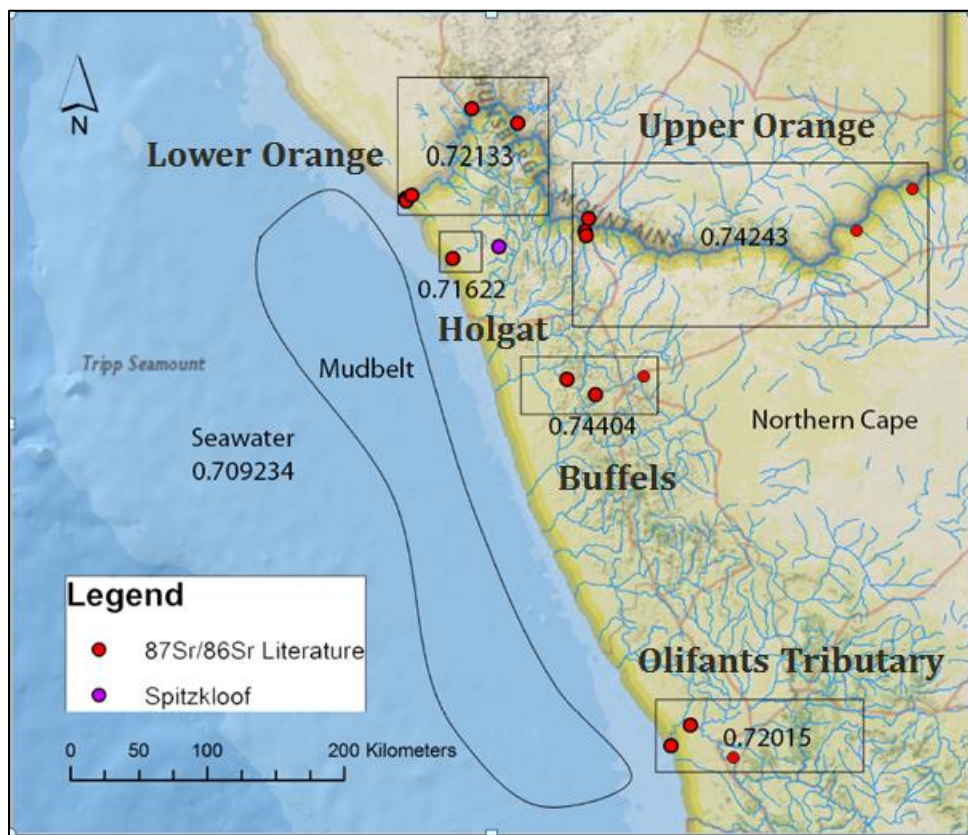


Figure 2: Published strontium isotope values for the five tributary regions and location of Spitzkloof Rockshelter. (Refer to Appendix B-: All localities contain a standard deviation of  $\pm 0.0001$  with the exception of the Olifants Tributary ( $\pm 0.0006$ ). *Note:* The rivers and tributary base layer was retrieved from the Department of Water and Sanitation of the Republic of South Africa ([https://www.dwaf.gov.za/iwqs/gis\\_data/river/rivs500k.aspx](https://www.dwaf.gov.za/iwqs/gis_data/river/rivs500k.aspx)).



#### 4.5 Strontium Isotope Analysis

All analyses for strontium isotopes were performed at the Department of Geological Sciences at the University of Cape Town. Ostrich eggshell fragments were first cleaned using sandpaper to remove dirt and debris from the surface. A sub-sample from each shell fragment of 25-30mg was placed on a Mettler AE240 electronic balance. The samples were prepared in batches of nine with one NIST987 standard sample for a total of ten. The Department of Geological Sciences uses NIST 987 as their strontium carbonate isotopic standard reference. The samples were covered with distilled water and placed in a sonic bath to remove any surficial contaminants. After a five-minute sonic bath, the distilled water was changed and the process repeated for three more sonic bath rinses. 2 mL of nitric acid were incorporated with the sample and put on a hotplate at 140°C overnight to dissolve. Samples were then prepared for column chemistry that removed all other contaminants except the strontium (see Appendix A for full procedure). Four groups (36 samples) were completed for strontium isotope analyses: six modern OES, twenty-eight archaeological fragments using a MC-ICP-MS (Multi-Collector Inductively Coupled Plasma Mass Spectrometer) and twelve OES beads using LA-ICP-MS (Laser Ablation Inductively Coupled Plasma Mass Spectrometer). The external long-term reproducibility according to the NIST 987 standard material is  $^{87}\text{Sr}/^{86}\text{Sr}$   $0.710255 \pm 20$  (2SD, n= 56) (P. le Roux, personal communication, March 2016). The ostrich eggshell beads were prepared separately so as not to destroy the artifacts. LA-ICP-MS method does not carry the same quality of data as the solution analysis, the laser ablation analyses provided  $^{87}\text{Sr}/^{86}\text{Sr}$  solution value 0.7328 2SD= 0.0003. However, due to the fragile nature of the beads, the LA-ICP-MS method proved to be the best option. The surfaces of the beads were first cleaned with the laser by making two small (200 x 700µm) trenches into each bead. Two locations on the bead were used in order to increase confidence in the values obtained due to the lower level in data quality by the laser ablation method.

## 5. Results

### 5.1 Eggshell Thickness Results

Table 2 summarizes the number of eggshell thicknesses measured with their means, ranges, and standard deviations. The Kruskal-Wallis H-test demonstrated significant differences between the groups (chi-squared= 27.753, df=6 p=0.0001). After performing the Dunn's Test for pair-wise comparisons between the groups, there were significant differences found between Group 1 (context 1-4) and Group 4 (context 11, 12) as well as Group 6 and all other groups. After the Bonferroni Correction Test, it was concluded that there were significant differences between groups: 1↔6, 4↔6, and 5↔6 only (all p values < 0.05). Group 6 showed the highest mean thickness (20.2 mm) compared to the other groups as well as a high minimum thickness comparable to Group 2 and Group 0. Figure 3 highlights these distributions in boxplots for Group 0 to Group 6.

Table 2: Measurement of eggshell thicknesses including the number of samples, means, ranges and standard deviations using a caliper (mm).

Group	Age (cal BP)	N	Mean	Max	Min	SD
0	Modern	13	19.7	21	19	0.0061
1	17,390 - 17,090	131	19.4	22	10	0.021
2	N/A	38	19.6	21	18	0.01
3	18,300-18,110	53	19.3	22	12	0.022
4	19,460-19,240	125	19.2	22	12	0.018
5	23,130-23,670	613	19.5	23	11	0.015
6	N/A	101	20.2	23	17	0.01

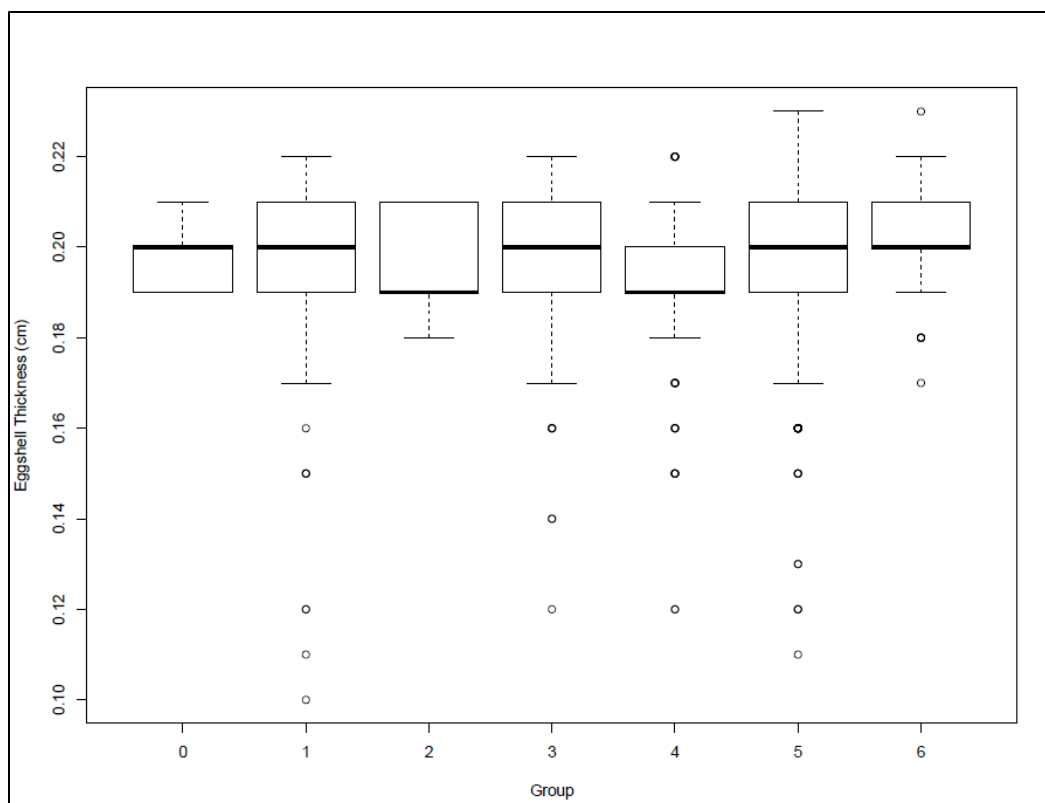


Figure 3: Boxplot showing the eggshell thickness distribution for the modern (Group 0) and archaeological samples (Groups 1-6).

### 5.2 Carbon and Oxygen Light Stable Isotopes

Basic statistics for  $\delta^{18}\text{O}$  and  $\delta^{13}\text{C}$  are given in Table 3. Both carbon and oxygen isotope values demonstrated a normal distribution and a homogeneity of variances between groups. An ANOVA was used to compare  $\delta^{13}\text{C}$  ( $F_{4,25} = 0.801$ ,  $p = 0.536$ ) and  $\delta^{18}\text{O}$  ( $F_{4,25} = 2.614$ ,  $p = 0.0594$ ) between the stratigraphic groups, and demonstrated no statistical difference in either ratio. The distributions of  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  together are shown in Figure 4. Separately, the distributions of  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  values are shown using boxplots in Figure 5 and Figure 6, respectively. The average values for  $\delta^{13}\text{C}$  demonstrate that the ostrich's diet was largely dominated by  $\text{C}_3$  plants with a small contribution of  $\text{C}_4$  or CAM plants. The standard deviation for the  $\delta^{13}\text{C}$  ranged from 0.2 ‰ – 1 ‰. The  $\delta^{18}\text{O}$  values indicate an arid environment throughout the sequence with a  $\delta^{18}\text{O}$  range of 35.4 ‰ - 42.1 ‰. The standard deviation for the  $\delta^{18}\text{O}$  ranged from 1 ‰ - 3.5 ‰. The  $\delta^{18}\text{O}$  values showed to be more variable when compared to the  $\delta^{13}\text{C}$ .

Table 3: Summary of carbon and oxygen light stable isotope analyses for each group, their means, standard deviations and ranges for each Group.

Group	Age (cal BP)	N	$\delta^{18}\text{O}$ (‰)	2SD	Min	Max	$\delta^{13}\text{C}$ (‰)	2SD	Min	Max
0	Modern	5	39.5	2.0	37.5	43.4	-24.2	1.0	-26.0	-23.0
1	17,390 - 17,090	9	37.9	1.0	35.6	39.9	-24.5	0.6	-25.6	-23.5
2	N/A	2	35.4	2.1	33.3	37.5	-23.5	0.2	-23.7	-23.2
3	18,300-18,110	4	37.7	3.5	34.3	42.6	-24.7	1.1	-26.6	-23.8
4	19,460-19,240	3	42.1	1.5	39.9	43.2	-24.3	0.7	-25.1	-23.6
5	23,130-23,670	16	40.4	2.9	34.6	45.7	-23.9	0.7	-25.7	-22.3
6	N/A	1	39.1	N/A			-22.3	N/A		

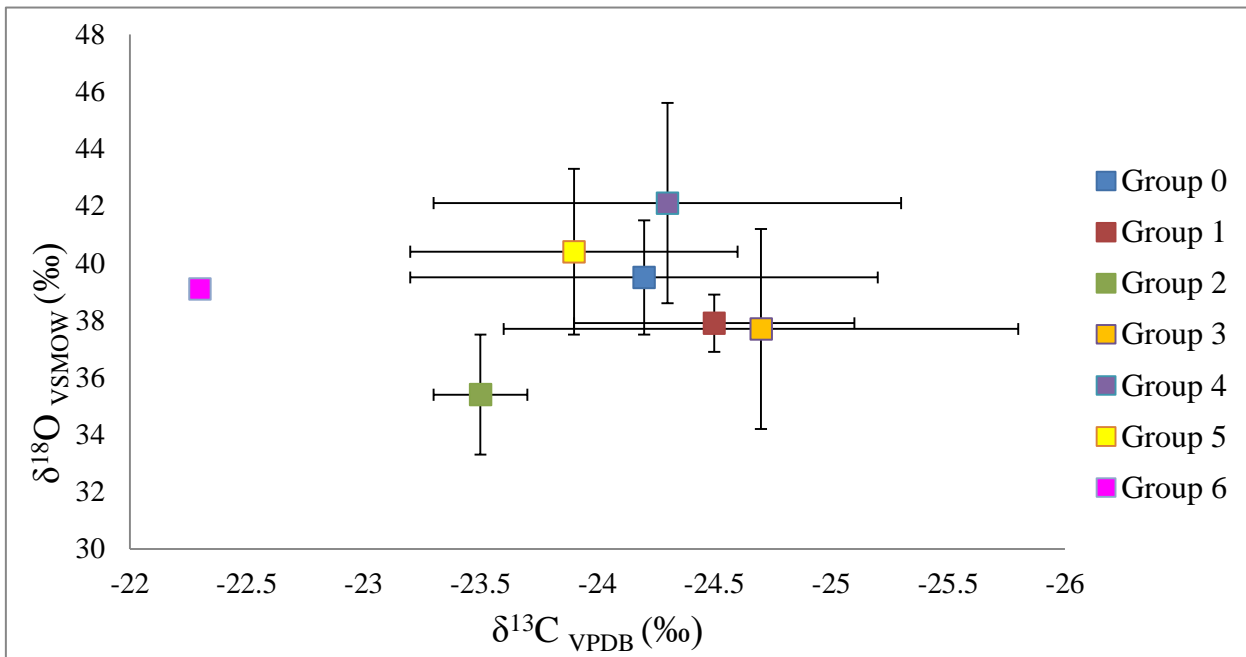


Figure 4: OES fragment isotope results of carbon and oxygen mean values per stratigraphic group. Error bars represent two sigma standard deviations.

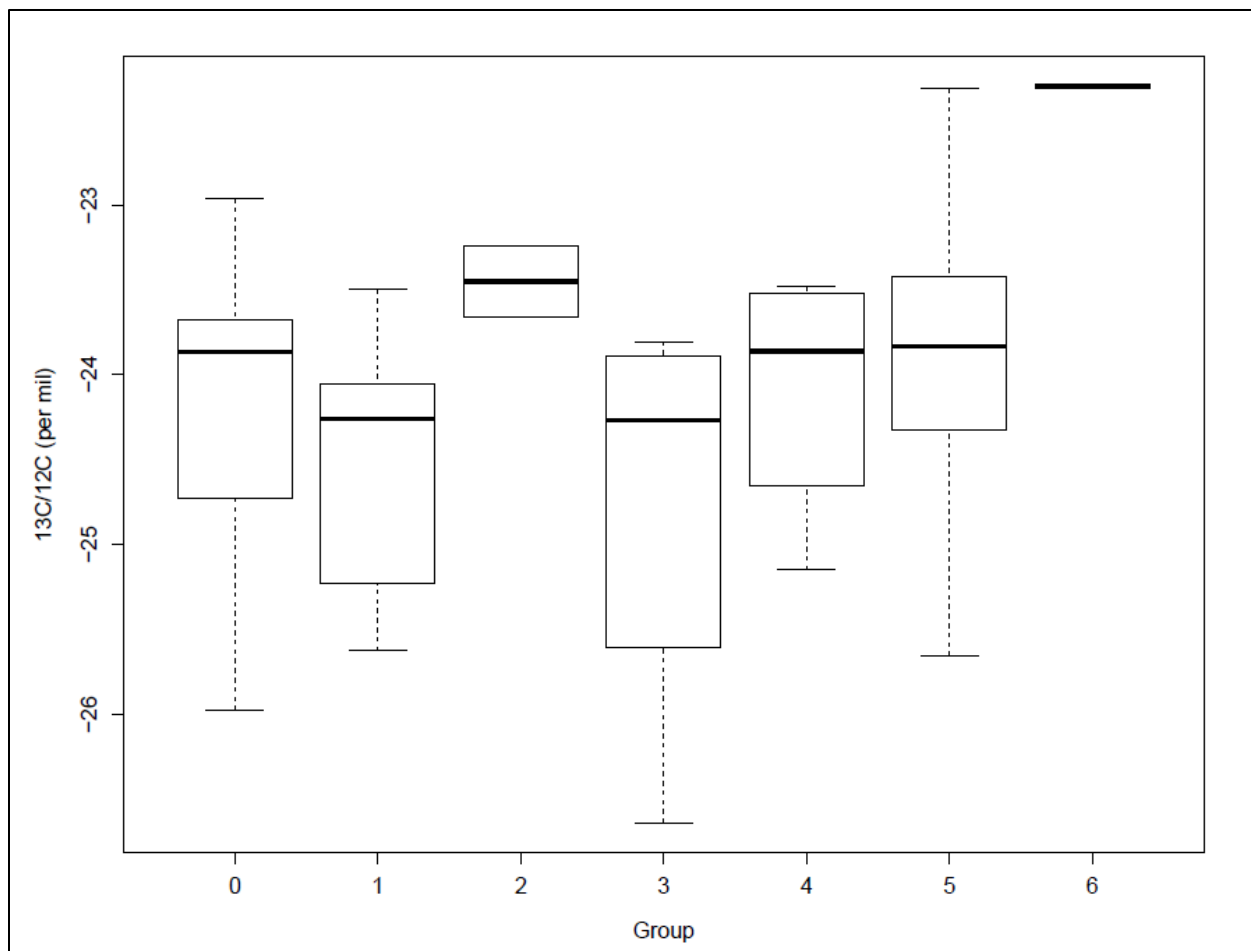


Figure 5:  $\delta^{13}\text{C}$  boxplot showing the distribution of the isotope data for Spitzkloof Rockshelter Groups 0-6. The  $\delta^{13}\text{C}$  values are expressed relative to PDB. Errors bars represent two sigma standard deviation.

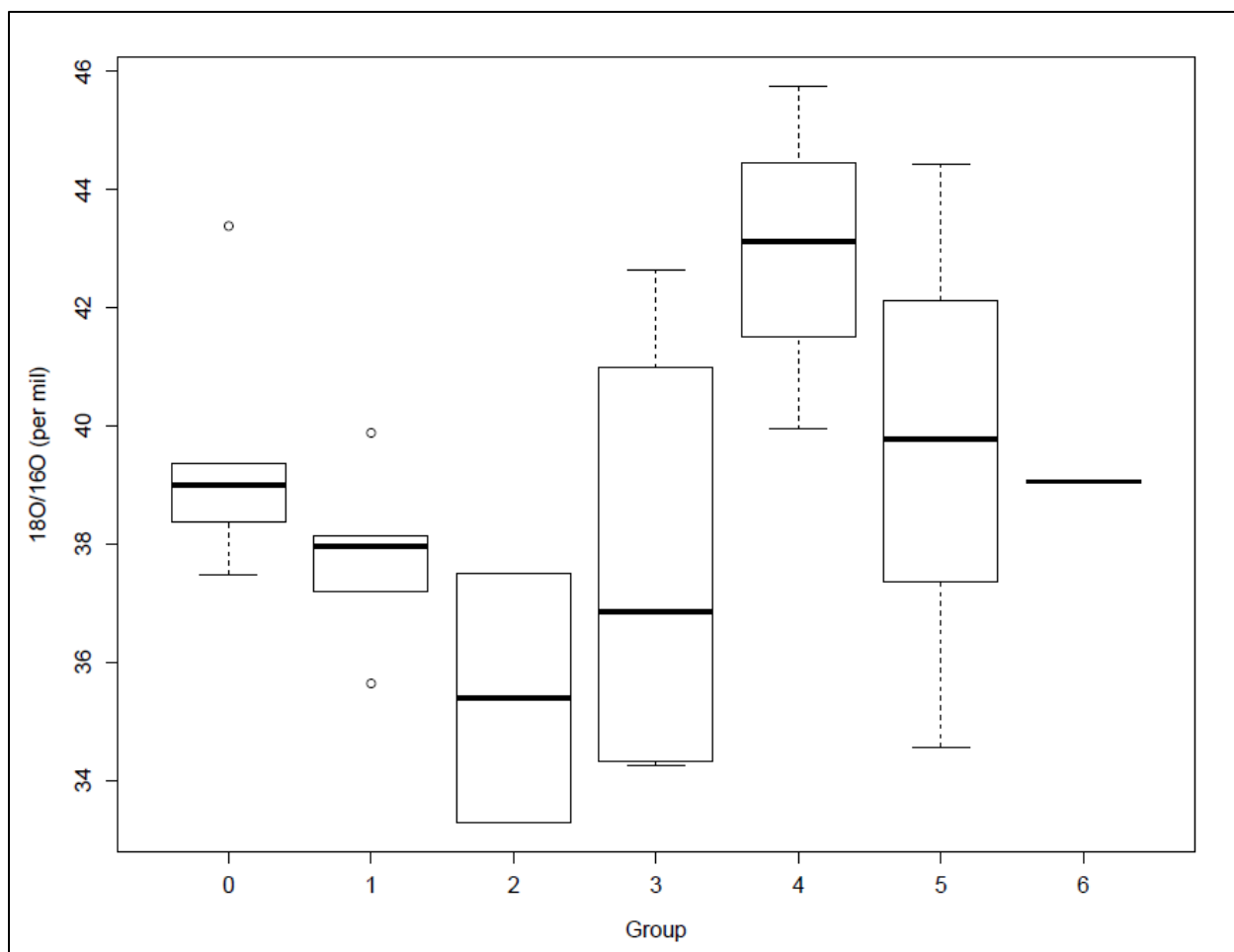


Figure 6:  $\delta^{18}\text{O}$  boxplot showing the distribution of the isotope data for Spitzkloof Rockshelter Groups 0-6. The  $\delta^{18}\text{O}$  values are expressed relative to VSMOW. Error bars represent two sigma standard deviation.

### 5.3 Strontium Isotopes

Results from the strontium isotopes indicate that there is variability between the archaeological fragments and modern samples as well as differences between the fragments and the beads (Figure 7). The isotopic ratios of the modern fragments appear to cluster together between 0.7134-0.7148. The archaeological fragments from Group 1-Group 6 appear to increase in strontium variability through time with Group 5 showing the largest range of strontium isotopic values (0.7109-0.7172), a difference of 0.0063. The beads also show a large range of isotopic variability among all archaeological groups ranging from 0.7096- 0.7199. The beads in fact demonstrate the largest range in strontium values with a difference of 0.0103.

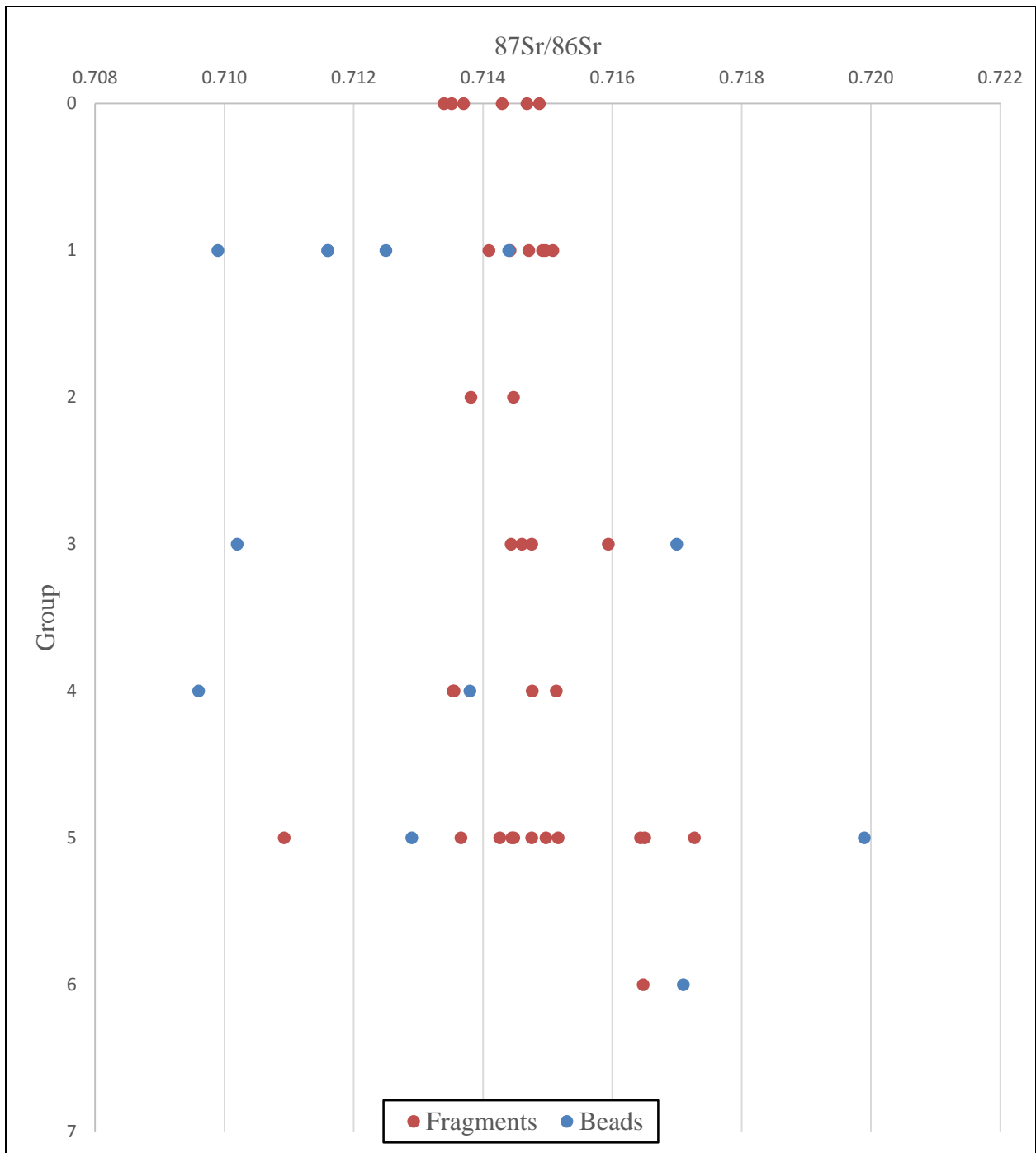


Figure 7:  $^{87}\text{Sr}/^{86}\text{Sr}$  isotope values for OES fragments and beads across the stratigraphic groups according to radiocarbon dates.

## 6. Discussion

### *6.1 Paleoenvironmental interpretation: environmental fluctuations and pulses of occupation at Spitzkloof*

The objectives of the paleoenvironmental analyses presented in this study were to help assess past climatic changes in the area and further extend what we know from the published literature and its relation to the times of occupation at Spitzkloof. More specifically, the analyses of the ostrich eggshell were to help our understanding of this arid to semi-arid region.

#### *MIS 3 (57-29 ka)*

The environment during MIS 3 (57-29 ka) has been interpreted as a time of increased aridity during the early stages (57-51 ka) followed by increasing humidity at approximately 50 ka (Shi et al., 2001). Group 6 (51 ka cal BP) within this study is positioned just at this interface of xeric to humid conditions. The one paleoenvironmental sample from this group ( $\delta^{18}\text{O} = 39 \text{ ‰}$ ,  $\delta^{13}\text{C} = -22 \text{ ‰}$ ) shows indications that the environment was arid. Using a linear mixing model, the  $\delta^{13}\text{C}$  value of  $-22 \text{ ‰}$  demonstrates a proportion of 70%  $\text{C}_3$  browsing diet and 30%  $\text{C}_4$  grazing diet by the ostrich, the highest value for  $\text{C}_3$  browsing seen in the set of samples (Appendix C). Fauna found on the landscape were also indicative of being arid-adapted as well as more inclined to a browsing diet with the availability of woody vegetation on the landscape (Dewar and Stewart, 2016). The highest mean value for eggshell thickness was also found in this layer ( $\mu = 20.2 \text{ mm}$ ) further suggesting arid conditions, confirming other reconstructions for this time period in northern Namaqualand (Shi et al., 2001, Lim et al., 2016).

#### *MIS 2 (29-14 ka)*

The transition from MIS 3 to MIS 2 and the first pulse of occupation at Spitzkloof (Group 5 at 23 ka cal BP) is marked by a decrease in sea levels as well as an increase in grazing species on the landscape (Dewar and Stewart, 2016). While sea levels are dropping and parts of southern Africa are experiencing increased dryness, pollen records in the Namaqualand region suggest an increase in precipitation indicating cool, moist conditions (Shi et al., 2001). According to the available pollen data, this period of increased precipitation continues until 19 ka for the region (Scott et al., 2004; Lim et al., 2016) or the start of the second occupational pulse for Spitzkloof.



The light stable isotopes show both an increase in  $\delta^{18}\text{O}$  ( $\mu= 40.4 \text{ ‰}$ ), and an increase in  $\text{C}_3$  plant vegetation or grazing environment. Ostrich eggshell thickness measurements also become thinner ( $\mu= 19.5\text{mm}$ ) indicating more cool moist conditions.

The period between the first and second occupational pulse is marked by the Last Glacial Maximum (LGM) approximately 21 ka in Namaqualand with the lowest sea level (Dewar and Stewart, 2016) and greatest distance to the coast from Spitzkloof. Group 4 through to Group 1 is represented in this second pulse with relatively similar  $\delta^{18}\text{O}$  and  $\delta^{13}\text{C}$  values. While the ANOVA demonstrated no statistical differences between groups for both carbon and oxygen, the variability in the range of values is present indicating considerable fluctuations in both  $\delta^{18}\text{O}$  and  $\delta^{13}\text{C}$  values. Lim et al., (2016) suggest that the last glacial period was characterized by variable water availability, with fluctuations in temperature and evapotranspiration having a large effect on the hydrological balance. The  $\delta^{18}\text{O}$  values for the region fluctuate considerably within Groups 2, 3, and 4 (17ka- 19ka cal BP) indicating large variability between dry and moist conditions. Scott et al., (2004) recorded pollen data for the LGM and interpreted the environment as being cool and moist however they noted that this could be due to a decrease in overall temperatures which would result in an increase of precipitation and thus moisture. The ostrich eggshell thickness measures (19.5 mm – 19.3 mm) appear to slowly decrease in thickness from 23 ka - 18 ka cal BP until rising back to 19.7 mm for modern xeric conditions similar to the MIS 3 period. The  $\delta^{13}\text{C}$  values indicate a steady increase in  $\text{C}_3$  vegetation or grazing habitat preferences for the ostriches which is likely indicative of the lowered sea levels and thus increased landscape area for grazing habitats and fauna. It is also at this time that we see the introduction of ostrich eggshell beads displaying a strong coastal  $^{87}\text{Sr}/^{86}\text{Sr}$  signal indicating that hunter-gatherers likely exploited these large stretches of the landscape beside the coast at the time when sea levels were very low. Previous studies have shown that sea-levels likely played a role in hunter gather subsistence strategies along the southern coast of South Africa (Fischer et al., 2010; Compton, 2011; Roberts et al., 2016). There are no ostrich eggshell beads from the first occupational pulse at 23 ka cal BP before the LGM which demonstrate a coastal  $^{87}\text{Sr}/^{86}\text{Sr}$  signal however it is at this time that we see a bead indicative of the Olifants Tributary system (approximately 500 km away). This could be indicative of potential trade and/or that hunter-gatherers were using a large range for resources before the LGM. Hunter-gatherers living in the Cape region of South Africa typically do not partake in long distance foraging trips, instead the overall foraging radius is

approximately 8-10 km, or defined as the distance a person can make a return walk in one day (Binford, 1980; Kelly, 1995; Fischer et al., 2010). It is likely that proximity to the coast influenced the subsistence and risk moderating strategies of the hunter gatherers occupying Spitzkloof, providing richer resources in comparison to the interior terrestrial environment.

6.2 Variability in strontium isotopes across landscapes: implications for mobility dynamics

The comparative analysis of strontium isotope ratios presented here demonstrated that there was variability between the modern and archaeological fragments as well as between the fragments and the OES beads at Spitzkloof, however because the sample size was too low, a statistical analyses could be done to accurately assess a variation of 0.001. To assess where the fragments and beads are originating on the landscape requires a comparison of the strontium isotope values with the published literature for landscape strontium isotopic ratios (Figure 8).

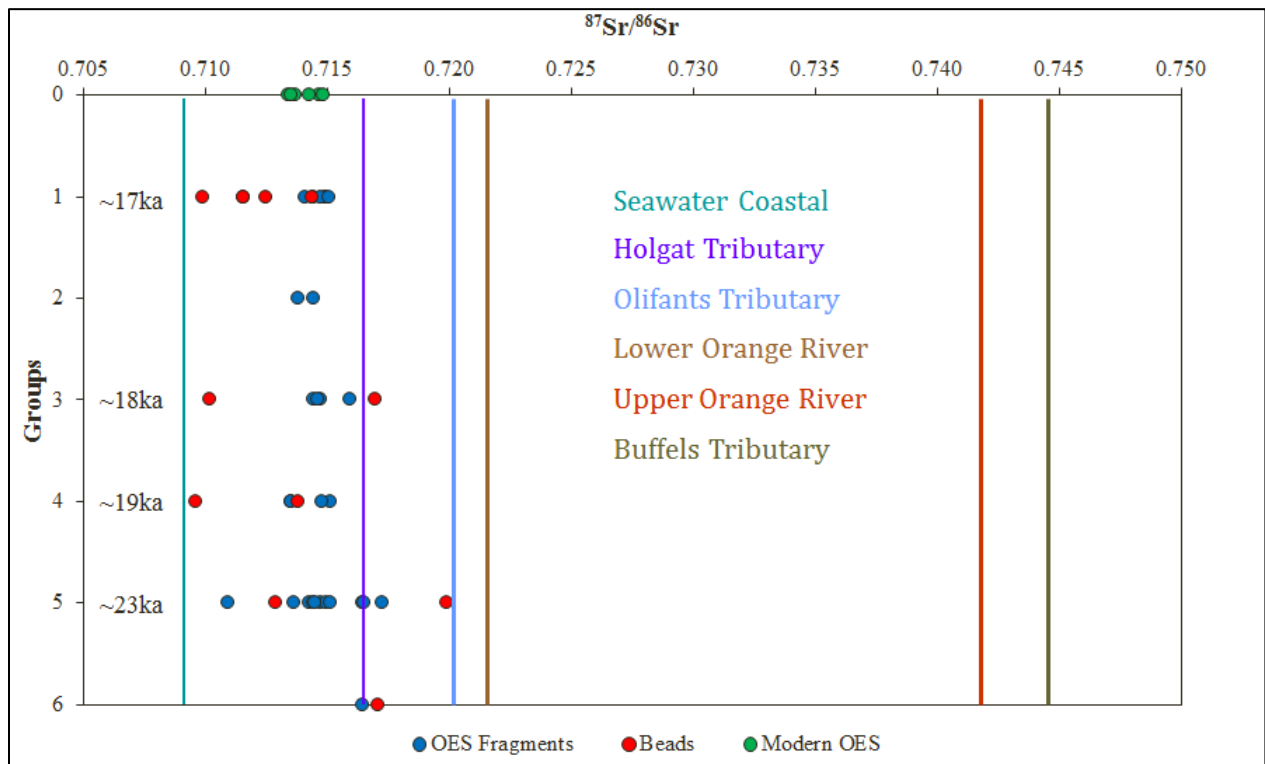


Figure 8: Strontium isotopic ratio results for OES fragments and beads from Spitzkloof archaeological strata with the published literature values for modern strontium isotopic ratios for 6 regions (vertical lines), as measured from geological materials.

The modern fragments collected close to Spitzkloof (sampled locally) differ by approximately 0.001 per mil from the average strontium isotope value for the Holgat Tributary system, which is on the threshold for discerning local versus non-local. Since Spitzkloof is located on a dry riverbed once belonging to the Holgat Tributary system, the data values could be exhibiting an additional strontium source or increase seawater signal, thus slightly lowering the values. The data suggest that the fragments are very likely, local to the site. The archaeological fragments demonstrate strontium isotope values between the Holgat Tributary system and the seawater coastal signal indicating that the fragments may have come from in between these two locales, making it difficult to discern if there is a coastal versus terrestrial signal in the strontium isotopic ratios. Interestingly, no fragments or beads from the study demonstrate a strontium isotopic value close to that of the Upper or Lower Orange River regions, which is approximately 30 km north of Spitzkloof and signifying that this area was likely not exploited during the time of occupation. Instead, the areas most exploited occurred around the Spitzkloof site and to the west where coastal resources are present.

The highest strontium isotope variability was seen in the OES beads at Spitzkloof; the beads showed more variability when compared to the fragments, suggesting that the beads demonstrate a wider range of transport over the fragments, however there could have been an effect on the beads as the result of different processes used to make the measurements (LA-ICP-MS vs. MC-ICP-MS). There are a couple of beads, which indicate a strontium value similar to some of the fragments but there are beads that demonstrate a Sr value much different. Several beads match closely to strontium isotope values found in fragments, which could mean that both the fragments and beads are originating from the same locale. However, since carbon and oxygen isotope analyses could not be performed on the beads, we cannot distinguish whether these beads come from the same eggshells. Aside from the beads which have almost identical strontium values to a few fragments, there are several beads which are statistically different (greater than 0.001) from the modern and archaeological fragments. In Group 5, there is a bead with a much higher strontium value next to the fragments, very similar to a strontium value found in the Olifants Tributary region. The Olifants Tributary system is much further away from Spitzkloof than the Orange River, approximately 500 km away, making this range of transport much greater than any other bead analyzed. At 23 ka cal BP the coastline was 57 km west of Spitzkloof, (Dewar and Stewart, 2016) a similar distance at 18 ka and additionally, the range of strontium

isotope values is similar, with the exception of the one bead comparable to the Olifants. Unfortunately, it is impossible to determine whether the bead reflecting the Olifants Tributary was prepared at this location but we can tentatively say that the ostrich eggshell originated from the region. However there are other potential explanations: the high variability in the data could be skewing the interpretation, and finally the difference between the Olifants and Lower Orange River localities is a difference in  $^{87}\text{Sr}/^{86}\text{Sr}$  of  $0.001 \pm 0.0006$  and if the bead was potentially exhibiting an additional source of lower  $^{87}\text{Sr}/^{86}\text{Sr}$ , then it could have skewed the sample to reflect the Olifants rather than the Lower Orange River locale

Evidence for bead production was seen most heavily at the top of the deposit dated to 17 ka cal BP and the  $^{87}\text{Sr}/^{86}\text{Sr}$  ratios show variability within the group suggesting that a large area of the landscape was being used at the same time. At this depth, there are beads that also have a similar strontium value to that of the seawater or coastal area, which is currently roughly 41 km west of Spitzkloof however, the paleo-shoreline has fluctuated over the time of occupation. At 17 ka cal BP, the coastline was in fact 56 km west due to the lower sea levels (Dewar and Stewart, 2016); this potentially extends the range of exploitation by hunter-gatherers at Spitzkloof. Strontium isotope values for the beads at 18 ka cal BP (58 km from the coast) and 19 ka cal BP (61 km from the coast) also demonstrate a value almost identical to that of a coastal signal which is indicative that hunter gatherers were exploiting resources along the coastline. Because the beads demonstrate a strong  $^{87}\text{Sr}/^{86}\text{Sr}$  coastal signal, we cannot discern where along the coastline the beads or eggshell originated; the coastal strip fully extends both north and south along the Atlantic. However, since the beads do not show values similar to the tributaries (with the exception of one), we can hypothesize that the eggshell likely originated on the coast.

### *6.3 Limitations to the Data and Future Research*

These methods were designed to best suit the needs of the project given the small sample sizes and gaps within both the data and the availability of radiocarbon dates from Spitzkloof. By conducting a literature review of all available strontium isotopic data for the region, I was able to appreciate as well as understand the diverse range of strontium isotopes present on the landscape. We should also consider the potential likelihood that geological rocks of the same age could be present across a large area and exhibit similar strontium values at many outcrops. There is an error of uncertainty with this data and the overall questions and assumptions being asked about

trade and subsistence strategies, it should not be assumed that all of the strontium data is controlled by mobility, however I would argue that strontium isotopes in conjunction with carbon and oxygen offer an exciting and new interdisciplinary avenue for many archaeologists. Further analyses could be done on this data set by increasing the sample sizes for carbon, oxygen and strontium isotopes, this will allow for a strengthened statistical interpretation and discussion of the results. More  $^{87}\text{Sr}/^{86}\text{Sr}$  values of tributary systems in Namaqualand would also strengthen our confidence that the locations presented here are properly representative. If possible, I would also suggest the second method for defining local vs. non-local ranges by designing the research question around archaeological non-migratory mammals (Slater et al., 2014) as a baseline for inferring potential ranges of eggshell transport. In addition, a more thorough investigation into the effects of coastal vs. inland signals (I.e. the overall mixing of strontium from the ocean and interior geological materials) could be a potential avenue for not just archaeologists determining subsistence strategies, but for climate modellers interested in changes to a regional climates or in untangling the cycling of strontium between the atmosphere and biosphere in this unique area.

## 7. Conclusion

Early forms of personal ornamentation (OES beads) are argued to reflect modern human behaviour and the production of such intricate pieces is suggested as a sign of costly signaling. However the application of OES beads also stretched to trade and exchange as a means for moderating risk. Under a biogeographic theory, when environments are variable, it is more advantageous for groups to have exchange networks, and remain mobile and adaptive, thus increasing their chances at survival. Exchange networks have been considered an important component in the Middle and Later Stone Age, but have been difficult to identify when objects appear to have similar morphology and are present across the landscape. Ostrich eggshell is abundant across many MSA and LSA archaeological sites due to its lack of susceptibility to diagenesis, making it an ideal sample to perform strontium, carbon, and oxygen isotope analyses. The objectives of this research were to assess past climatic changes during occupation and compare these to the  $^{87}\text{Sr}/^{86}\text{Sr}$  data and published literature values for given regions to discern if this movement (adaptiveness) was the result of a changing environment. Variations in  $^{87}\text{Sr}/^{86}\text{Sr}$  ratios were present between the modern and archaeological samples while the OES beads demonstrated the highest variability, suggesting that the beads demonstrated a wider range of transport over the fragments. The transition from MIS 3 to MIS 2 and the first pulse of occupation (Group 5 at 23 ka cal BP) was marked by a decrease in sea level as well as an increase in grazing species on the landscape. The  $\delta^{13}\text{C}$  values during MIS 2 indicate a steady increase in  $\text{C}_3$  habitat preferences which is likely indicative of the increased landscape area for grazing as a result of the lowered sea levels. At this time there is also an increase in OES beads showing a strong coastal origin indicating that hunter-gatherers likely exploited these large stretches of the landscape beside the coast at the time when sea levels were very low rather than the interior sections. In conclusion, by using strontium isotope analyses in combination with carbon and oxygen stable isotopes, new insights can be drawn for the roles that climatic fluctuations played in subsistence and risk moderating strategies of hunter gatherers at Spitzkloof.

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## Appendix A: Strontium Isotope Analyses Column Chemistry Procedure for Solution Analysis

*Note:* This procedure is utilized by the Department of Geological Sciences at the University of Cape Town for solution analyses. Additional information about the facilities can be found at <http://www.geology.uct.ac.za/multicollector-icp-ms>

These steps are completed for each group (9 samples per group).

- Add 2mL of nitric acid to each sample and place on a hotplate at 140°C, leave overnight
- Place the samples in a centrifuge to ensure the whole sample is fully dissolved
- Place a waste beaker underneath each column of sample. To remove the nitric acid from the sample, remove the bottom cap and let the acid drip out of the column.
- Add 1mL of 2M HNO<sub>3</sub>- drain into the waste beaker to fully remove any leftover acid (repeat x2)
- Load 1.45mL of sample (0.75mL, 0.7mL) into a clean column
- Wash each sample with 0.5mL of 2M 2BHNO<sub>3</sub> (repeat x6)
- Place a clean labelled 7mL beaker under each column for the strontium collection then remove the tip of each column
- Collect all the strontium by rinsing the columns with 6 x 0.5mL MQ Water
- Dry the samples on a hot plate at 130°C, let cool
- Add 2mL of 0.2HNO<sub>3</sub>
- Ultra-sonicate for 30 minutes

### Cleaning Process

- Place waste beaker underneath the used columns
- Insert 6.2M HCl fully into the columns (repeat x5)
- Rinse the columns with MQ Water
- Add 1mL of 7M 2BHNO<sub>3</sub> (0.1,0.1,0.8)
- Rinse with 3mL of MQ Water (0.1, 0.1, 0.8, 1mL, 1mL)
- Add 3mL of 2M HNO<sub>3</sub> (0.1,0.1,0.8, 1mL, 1mL)
- Wipe the tips of each column and rack with wet paper towel
- Replace the tips and add in 1mL 2M HNO<sub>3</sub>
- Seal the columns until next procedure

Appendix B: Published Strontium Isotope Values for Namaqualand region, Northern Cape, South Africa.

Region	Coordinates	Sample	$^{87}\text{Sr}/^{86}\text{Sr}$	Standard Error	Reference
Lower Orange	-28.58, 16.46	River water	0.71658	0.0001	de Villiers et al., 2000
Lower Orange	-28.6, 16.46	River bed sediment	0.735538	0.0001	Weldeab et al., 2013
Lower Orange	-28.6, 16.46	River suspension sediment	0.732447	0.0001	Weldeab et al., 2013
Lower Orange	-28.56, 16.5	River suspension sediment	0.73658	0.0001	Hahn et al., 2015
Lower Orange	-28.2, 16.7	River water	0.71663	0.0001	de Villiers et al., 2000
Lower Orange	-28.1, 16.9	River water	0.71662	0.0001	de Villiers et al., 2000
Lower Orange	-28.15, 17.2	River water	0.71655	0.0001	de Villiers et al., 2000
Seawater			0.709234	0.0001	Bentley, 2006
<b>Regional <math>^{87}\text{Sr}/^{86}\text{Sr}</math> Average: 0.72133 ± 0.0001</b>					
Holgat	-28.93, 16.77	River bed sediment	0.7232	0.0001	Weldeab et al., 2013
Seawater			0.709234	0.0001	Bentley, 2006
<b>Regional <math>^{87}\text{Sr}/^{86}\text{Sr}</math> Average: 716217 ± 0.0001</b>					
Upper Orange	-28.77, 17.64	River suspension sediment	0.733628	0.0001	Weldeab et al., 2013
Upper Orange	-28.77, 17.64	River bed sediment	0.727351	0.0001	Weldeab et al., 2013
Upper Orange	-28.79, 17.65	River bed sediment	0.76666	0.0001	Weldeab et al., 2013
Upper Orange	-28.7, 17.67	River water	0.71693	0.0001	de Villiers et al., 2000
Upper Orange	-29.69, 17.71	River bed sediment	0.7676	0.0001	de Villiers et al., 2000
<b>Regional <math>^{87}\text{Sr}/^{86}\text{Sr}</math> Average: 0.742434 ± 0.0001</b>					

Buffels	-29.61,17.52	River bed sediment	0.75528	0.0001	Weldeab et al., 2013
Buffels	-29.69,17.71	River bed sediment	0.7676	0.0001	Weldeab et al., 2013
Seawater			0.709234	0.0001	Bentley, 2006
<b>Regional <math>^{87}\text{Sr}/^{86}\text{Sr}</math> Average: 0.744038 <math>\pm</math> 0.0001</b>					
Olifants	-31.68, 18.19	River bed sediment	0.72423	0.0001	Weldeab et al., 2013
Olifants	-31.57, 18.33	River suspension sediment	0.727	0.0006	Hahn et al., 2015
Seawater			0.709234	0.0001	Bentley, 2006
<b>Regional <math>^{87}\text{Sr}/^{86}\text{Sr}</math> Average: 0.720155 <math>\pm</math> 0.0006</b>					

Appendix C: Corrected carbon and oxygen isotope values with standard deviations and approximate C<sub>3</sub>/C<sub>4</sub> proportions for OES fragments.

*Note:* The C<sub>3</sub>/C<sub>4</sub> proportions given are based on the average δ<sup>13</sup>C value of plant foliage in South Africa with C<sub>3</sub> approximately -26.5 ‰ and C<sub>4</sub> approximately -12.6 ‰ (Vogel et al., 1978; Johnson et al., 1998).

Sample	Group	δ <sup>13</sup> C Corrected	δ <sup>13</sup> C SD	C <sub>3</sub> :C <sub>4</sub> Ratio	δ <sup>18</sup> O Corrected	δ <sup>18</sup> O SD
OES-01	0	-25.97	0.22	96:4	38.99	0.07
OES-04	0	-23.67	0.27	80:20	43.38	0.08
OES-06	0	-22.96	0.21	75:25	39.36	0.15
OES-07	0	-23.86	0.26	81:19	38.37	0.13
OES-10	0	-24.72	0.22	87:13	37.48	0.10
OES-124	1	-24.05	0.19	82:18	37.93	0.11
OES-127	1	-24.24	0.44	84:16	39.88	2.08
OES-128	1	-24.27	0.2	84:16	38.14	0.11
OES-139	1	-25.62	0.14	94:6	38.00	0.05
OES-140	1	-25.22	0.21	91:9	35.63	0.12
OES-141	1	-23.49	0.14	78:22	37.18	0.09
OES-163	2	-23.65	0.40	80:20	33.28	0.13
OES-164	2	-23.24	0.23	77:23	37.49	0.13
OES-142	3	-26.64	0.15	99:1	34.39	0.09
OES-143	3	-24.56	0.19	86:14	34.25	0.08
OES-144	3	-23.80	0.20	81:19	39.32	0.11
OES-145	3	-23.96	0.14	82:18	42.64	0.15
OES-146	4	-25.14	0.13	90:10	39.94	0.19
OES-168	4	-23.47	0.17	78:22	45.73	0.11
OES-147	4	-24.16	0.19	83:17	43.07	0.10
OES-148	4	-23.55	0.18	79:21	43.15	0.24
OES-155	5	-23.83	0.16	81:19	37.07	0.09
OES-160	5	-24.52	0.17	86:14	38.84	0.08
OES-149	5	-23.11	0.19	76:24	37.66	0.07
OES-150	5	-25.65	0.20	94:6	40.20	0.23
OES-151	5	-23.43	0.25	78:22	44.43	0.12
OES-157	5	-23.61	0.21	79:21	42.30	0.14
OES-159	5	-23.40	0.17	78:22	42.54	0.09
OES-161	5	-24.36	0.14	85:15	41.94	0.06
OES-169	5	-24.28	0.21	84:16	34.55	0.19
OES-152	5	-22.31	0.17	70:30	39.77	0.07
OES-166	5	-24.03	0.12	82:18	36.24	0.15
OES-162	6	-22.29	0.25	70:30	39.05	0.09

Appendix D: Strontium Isotope Values for the OES fragments and beads.

*Note:* The fragments and bead analyses were performed on different instruments, MC-ICP-MS and LA-ICP-MS, respectively, thus the number of significant digits reflects the accuracy and reproducibility according to each instrument.

Sample	Group	$^{87}\text{Sr}/^{86}\text{Sr}$	2 $\sigma$ standard deviation
Fragment			
OES-01	0	0.713400	0.00014
OES-04	0	0.714681	0.00013
OES-06	0	0.713701	0.00009
OES-07	0	0.713517	0.00012
OES-08	0	0.714874	0.00012
OES-10	0	0.714297	0.00010
OES-124	1	0.714969	0.00010
OES-127	1	0.714422	0.00012
OES-128	1	0.714922	0.00014
OES-139	1	0.714711	0.00010
OES-140	1	0.714094	0.00012
OES-141	1	0.715080	0.00012
OES-163	2	0.714472	0.00013
OES-164	2	0.713815	0.00011
OES-142	3	0.715941	0.00010
OES-143	3	0.714435	0.00010
OES-144	3	0.714753	0.00013
OES-145	3	0.714604	0.00009
OES-146	4	0.715134	0.00012
OES-168	4	0.714763	0.00011
OES-147	4	0.713553	0.00010
OES-148	4	0.713536	0.00011
OES-155	5	0.716437	0.00013
OES-160	5	0.714755	0.00011
OES-149	5	0.716505	0.00012
OES-150	5	0.714976	0.00010
OES-151	5	0.714262	0.00013
OES-157	5	0.714448	0.00012
OES-159	5	0.715163	0.00011
OES-161	5	0.714476	0.00013
OES-169	5	0.710926	0.00009
OES-152	5	0.713661	0.00011
OES-166	5	0.717270	0.00012

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OES-162	6	0.716477	0.00010
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Bead			
	1	0.7125	0.0001
	1	0.7116	0.0001
	1	0.7144	0.0000
	1	0.7099	0.0000
	1	0.7116	0.0001
	3	0.7102	0.0001
	3	0.7170	0.0001
	4	0.7138	0.0001
	4	0.7096	0.0000
	5	0.7199	0.0001
	5	0.7129	0.0001
	6	0.7171	0.0001

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