In archaeology worldwide, mapping and comprehending the climatic conditions in which the past populations lived has been regarded as valuable for furthering the understanding of subsistence strategies, site functions and land use patterns. During the past 50 years, stable oxygen isotopes from a large variety of materials found in archaeological sites have been used to assess temperature fluctuations of the ancient climates. Specimens of different marine molluscs have been extensively used in stable isotope analysis, because calcium carbonates ($CaCO_3$) of molluscan shells and opercula are believed to be reliable recorders of their surrounding environment.

In this study, an evaluation of the methodology of stable isotope analysis to determine palaeoenvironmental fluctuations from marine molluscs was conducted. Over the last three decades, advances in procedural techniques and evolved knowledge of marine molluscs and biologically produced minerals have made it essential to re-examine the suppositions and procedures of this method. A number of pitfalls and obstacles within the methodology have been discovered, which can complicate the reliability of earlier palaeoenvironmental reconstructions. This study will determine whether the methodology of stable isotope analysis, as it has been used in the past, must be rejected altogether or whether it is reliable, providing precautions are taken.

For this study, calcium carbonate samples were taken from the operculum of the marine mollusc *Turbo sarmaticus*. Specimens of opercula were collected from four Later Stone Age sites in Garcia State Forest nature reserve on the southern Cape coast, South Africa. The selected opercula were cleaned, examined by microscopy to determine the physical preservation of the surface, and investigated by X-ray Diffractometry (XRD) and Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray System (EDS) for chemical analysis. Lastly, the opercula were examined by mass spectrometry for stable isotope analysis.

The results of this investigation highlight the need for rigorous attention to all stages of the techniques and procedures used. Isotopic studies conducted to determine ancient temperature fluctuations on marine calcium carbonates need careful scrutiny of the material to ensure satisfactory preservation, controls on procedural problems, controls on palaeotemperature equations and an awareness of all the pitfalls and complications of the method. However, if rigorous investigations are performed, the stable isotope analysis can prove valuable for palaeoclimatic reconstructions.