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Molluscs (Isotopes): Analyses in Environmental Archaeology

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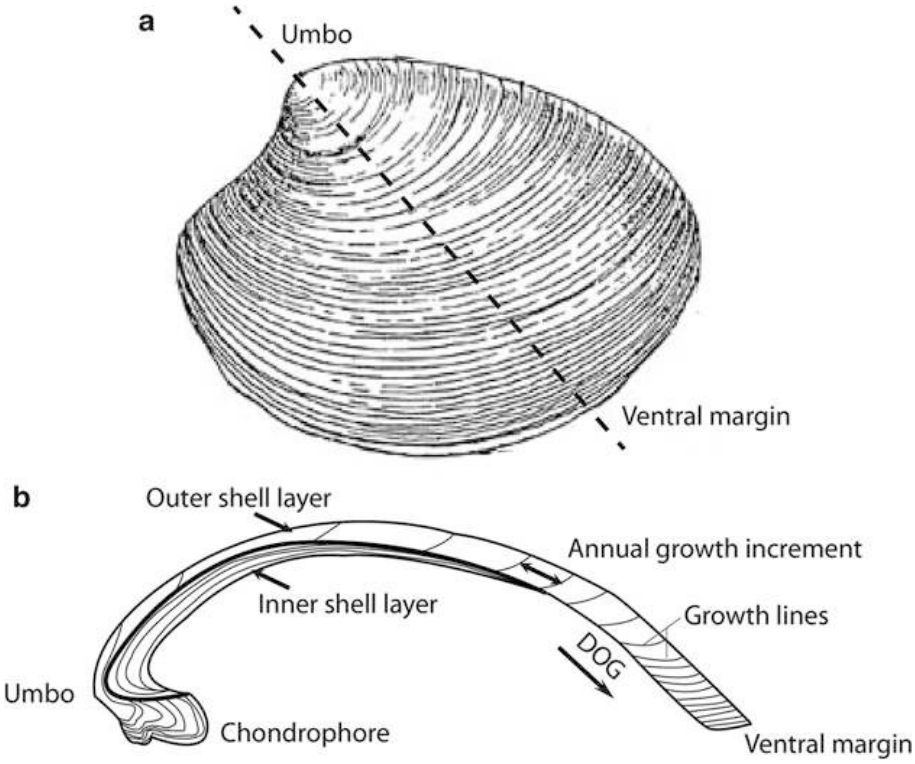
Introduction

Mollusc shells are a common component of many archaeological assemblages. Archaeological mollusc shell accumulations are typically the result of food refuse and may be present within a settlement or as a midden nearby. Mollusc-based artifacts such as shell scrapers and shell beads have also been found in sites from Paleolithic to recent times in many geographic areas. However, some terrestrial mollusc shells may be present in archaeological assemblages even though humans did not actively collect them. Studying the chemistry of



Molluscs (Isotopes): Analyses in Environmental Archaeology, Fig. 1 Idealized morphology of a bivalve shell. The *dashed line* in (**a**) shows the location of the cross section in (**b**). *DOG* direction of growth

shells from archaeological assemblages provides a means to study paleoclimate and paleoenvironmental change that is directly correlated to human activity. Shell chemistry of the final growth increments can also yield information on the seasons in which the shells were harvested. Incorporating climate and seasonality studies within archaeological investigations provides insight on how humans interacted with their environment. Stable oxygen isotope analysis is one of the most common geochemical techniques applied to the analysis of archaeological shells and will be the primary focus of this review.

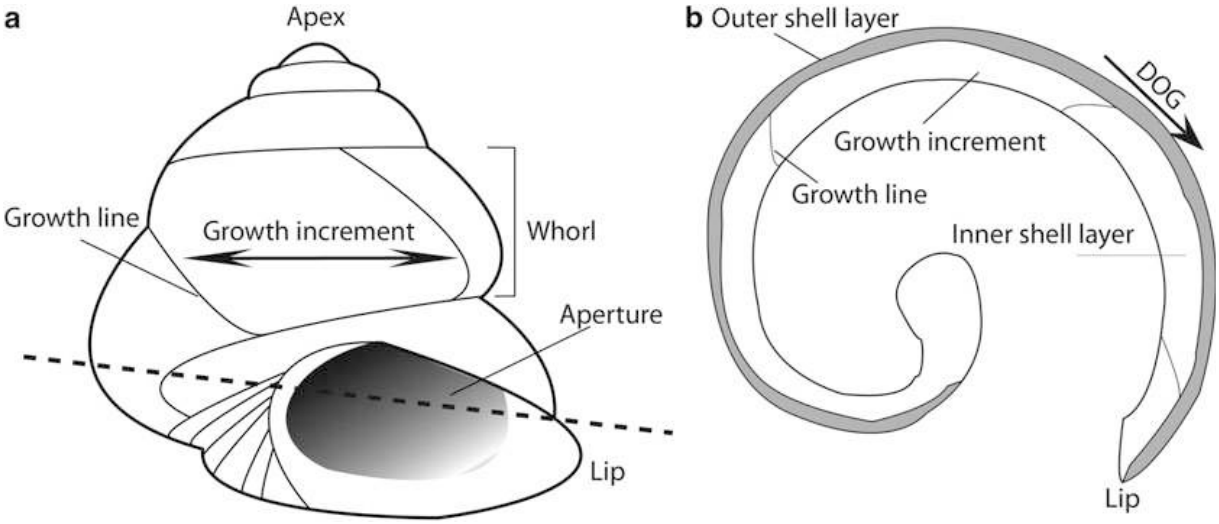
Definition

The Phylum Mollusca

The Mollusca are a phylum of soft-bodied invertebrates whose bodies are often protected by shells composed of calcium carbonate (either calcite or aragonite). Gastropod and bivalve shells are the most commonly preserved mollusc remains in archaeological sites. Both live in marine, freshwater, and estuarine environments, while gastropods also live in terrestrial environments. The usefulness of mollusc shells as high-resolution environmental archives stems from their shell's incremental growth patterns. Bivalve bodies are encased by a shell consisting of two valves, joined by a hinge. The shells grow by depositing successive increments of calcium carbonate from the umbo to the ventral margin (Fig. 1). Gastropods are encased within a shell, which is often coiled. The shell grows by depositing calcium carbonate growth increments from the apex to the shell lip (Fig. 2).

Molluscs generally precipitate their shells in equilibrium with surrounding ambient environmental conditions. Therefore, the physical and chemical properties of mollusc growth increments record changes in the mollusc's ambient environment during growth. Depending on shell growth rates, high-resolution sampling of these shells can yield environmental information from decadal to subdaily timescales. Short-lived species can record seasonality and weather extremes, and long-lived species can record decadal- to centennial-scale climatic oscillations.

Molluscs are widely distributed in varied environments and are abundant from the tropics to the poles. This offers the potential for



Molluscs (Isotopes): Analyses in Environmental Archaeology, Fig. 2 (**a**): Idealized external morphology of a coiled gastropod shell. The *dashed line* in (**a**) shows the location of the cross section in (**b**). *DOG* direction of growth

high-resolution time series analyses from almost every geographic region. Many other quaternary environmental proxies such as ice cores and marine sediment cores are limited in their geographical extent and temporal resolution.

Stable Isotopes

Isotopes are atoms of the same chemical element that have the same number of protons (thus the same atomic number) but different numbers of neutrons (thus different atomic masses). Stable isotopes have nuclei that do not decay to form other isotopes. Chemically, all isotopes of the same element behave identically. However, due to their differences in atomic weight, the lighter isotopes have different kinetic properties, causing them to respond at different rates during biological and geochemical processes. In "light" stable isotopes with low atomic masses, the differences in weight between the isotopes are large enough for physical, chemical, and biological processes to cause fractionation whereby the ratio of heavy to light isotopes in the system changes. Oxygen is the most routinely studied stable isotope in archaeological mollusc shells. It has three stable isotopes ^{16}O, ^{17}O, and ^{18}O. The application of oxygen isotope analysis to mollusc shells uses the ratio of ^{16}O to ^{18}O. As the differences in natural abundances of stable isotopes are usually very small, the ratio of heavy to light isotopes is measured with reference to the isotope ratio in an international standard in delta (δ) notation:

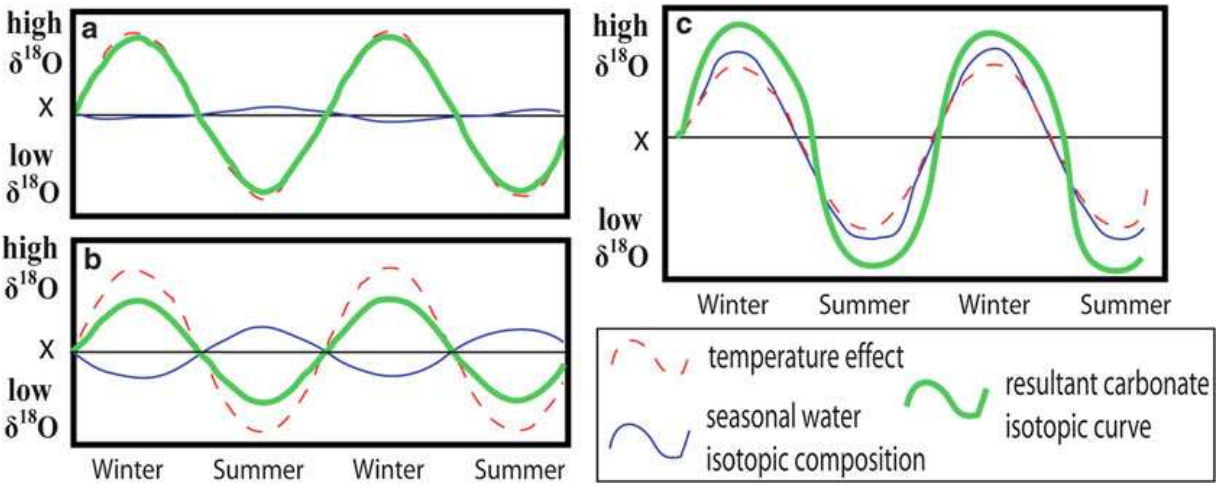
$$\delta X = R_{sample} - R_{standard} / R_{standard} - 1 \times 1000$$

X = the heavy isotope and R = the ratio of heavy to light isotope. Measurements are expressed in parts per mil (‰).

If δX is more positive, the sample has a higher ratio of heavy to light isotopes than the standard.

Oxygen Isotopes in Marine Shells

The isotopic composition of marine shell is controlled by water temperature and the isotopic composition of seawater (Kennett & Voorhies 1996). In modern marine environments in mid-to high latitudes, the role of evaporation and rainfall on the overall isotopic composition of seawater is negligible, making sea surface temperature (SST) the primary fractionation factor (Fig. 3). Warmer temperatures enrich ^{16}O relative to ^{18}O, to generate isotopically negative signatures, while cooler temperatures produce positive signatures. In these temperature-dominated systems, $\delta^{18}O$ amplitude (the difference between maximum and minimum isotopic values) records the range of seasonal water temperature fluctuations experienced by a mollusc during growth. The relationship between shell



Molluscs (Isotopes): Analyses in Environmental Archaeology, Fig. 3 Simple models of seasonal change in shell $\delta^{18}O$ expressed as a deviation from a mean value (X). Temperature is identical in **a**, **b**, and **c**. (**a**): Change in water composition is negligible and temperature is the principal fractionation factor, so the isotope curve closely follows the temperature effect; (**b**): change in water composition is larger. This could be caused by increased summer evaporation or heavier seasonal precipitation; (**c**): higher amplitudes in negative seasonal precipitation combine with temperature to create larger amplitudes in the resultant carbonate isotopic curve (Modified from Dettman & Lohmann 1993)

$\delta^{18}O$, water $\delta^{18}O$, and sea surface temperature has been expressed in several equations, so $\delta^{18}O$ in shells can be used to quantitatively reconstruct sea surface temperature regimes of the past (e.g., Dettman et al. 1999). In tropical regions, interannual temperature changes are minimal, so $\delta^{18}O$ of mollusc shells can be used to reconstruct paleo-$\delta^{18}O$ of the water, which is mainly controlled by the amount and composition of rainfall. In such regions, $\delta^{18}O$ can be used to reconstruct precipitation and monsoon regimes.

Oxygen-Stable Isotopes in Terrestrial Molluscs

Oxygen isotope ratios of terrestrial mollusc shells provide a record of continental climatic conditions. In terrestrial shells, the water ingested by the snails principally controls $\delta^{18}O$. However, due to the variability in the oxygen isotope composition of the environmental water consumed by the snails, there is no worldwide correlation between snail shell $\delta^{18}O$ and a single environmental variable (see Zanchetta et al. 2005 for a review of recent studies): in humid regions at lower latitudes, meteoric water or precipitation tends to dominate the signal, while at higher latitudes in more temperate climates, temperature and humidity are the primary variables. Isotopic signatures of land snails from archaeological contexts should be validated in relation to a thorough assessment of isotopic data from live-collected snails of the same or similar species from a similar geographic range and within the expected foraging range of the archaeological site or under experimentally controlled conditions, to provide a baseline for the interpretation of the paleoenvironmental and archaeological data.

Oxygen Isotopes in Estuarine Shells

The mixing between marine and freshwater complicates the interpretation of stable isotope ratios in estuarine shells. In estuarine environments, oxygen isotopes and shell growth may be differently affected by factors such as turbidity, temperature, and salinity variations. In tropical regions, where temperature remains more constant throughout the year, oxygen isotopes from estuarine shells can be used to detect salinity changes from freshwater inputs including changes in paleorainfall/paleomonsoon and seasonal variations in runoff (e.g., Kennett & Voorhies 1996). Outside of the tropics the situation is more complicated as both temperature

and salinity variation can differently affect the oxygen isotope record (Fig. 3).

Oxygen Isotopes in Freshwater Shells

Freshwater molluscs present a greater challenge for interpretation than marine molluscs, as the oxygen isotope ratios preserved within shell carbonate preserve evidence of various environmental effects. The $\delta^{18}O$ composition of the ambient freshwater can vary significantly with changes in evaporation and precipitation (e.g., Fritz & Poplawski 1974). In lakes with closed catchments and long water residence time, the local precipitation-evaporation balance is likely to be the controlling parameter on water $\delta^{18}O$ composition. By contrast the controlling parameters in lakes with open catchments and low water residence time are likely to be precipitation $\delta^{18}O$ and/or temperature. In river and lake systems, high evaporation results in more positive $\delta^{18}O$. This has the effect of reducing the seasonal amplitude signal (Fig. 3). River systems may be enriched along their flow paths with isotopically heavy or light rainwater, which will either dampen or heighten the amplitude signatures of the shell (Fig. 3). In order to apply $\delta^{18}O$ analysis for paleoclimatic reconstructions in freshwater systems, a thorough study of modern populations of the proxy species is necessary in order to interpret the paleorecord.

Historical Background

Urey (1947) was the first to reveal that the oxygen isotope composition of biogenic carbonate is temperature dependant, showing that variations in the temperature of the water from which the carbonate was precipitated should lead to predictable variations in the $\delta^{18}O$ ratios of the carbonate. Soon after, Epstein et al. (1951) formulated an empirical paleotemperature equation based on isotopic analyses of molluscs with known growth temperatures.

Season of Collection

The initial focus of stable oxygen isotope analysis on archaeological mollusc shells was on determining the season of shell collection. Shackleton (1973) was the first to recognize that the $\delta^{18}O$ value of the last deposited growth increment reflects the environmental conditions at the time of the animal's death. Season of collection analysis is possible when a primary variable affects the stable isotope composition of the shell. For example, in tropical marine settings, water temperature is fairly constant but $\delta^{18}O$ of the water is affected on a seasonal basis by the $\delta^{18}O$ composition and amount of rainfall. In temperate marine settings, water $\delta^{18}O$ is fairly constant so $\delta^{18}O$ in mollusc shells is primarily influenced by sea surface temperature. Season of collection information is useful for archaeologists because in midden shells, death occurred due to collection by ancient human foragers. Seasonal patterns of shellfish collection bear directly upon interpretations of site use and subsistence behavior, providing important data to aid analyses of foraging practices from the archaeological record. Shellfish season of collection data can be combined with other archaeological data to gain a more complete picture of site use patterns.

These methods have been successfully applied in marine, freshwater, and estuarine environments around the world. In earlier studies, there was debate over the temporal resolution that could be obtained from shell-edge $\delta^{18}O$ values. Killingley (1981) contended that monthly resolution was possible, while Bailey et al. (1983) suggested that only seasonal resolution could be attained. More recently, there has been a growing awareness of the importance of modern validation studies on the proxy species in habitats close to the archaeological study site to understand how factors such as seasonal or ontogenetic variability in growth rates may affect the sample resolution in different environments and latitudes (e.g., Mannino et al. 2003).

The number of samples analyzed in archaeological season of collection studies must balance the need to analyze enough shells per archaeological context to detect meaningful foraging patterns with the need to characterize the pattern of growth to accurately determine season of collection (Mannino et al. 2003). Measuring only the outermost growth increment allows

many shells to be characterized using minimal analyses. This strategy works well to identify seasonal extremes (summer and winter); however, it is more difficult to disentangle signatures from shells collected during transitional conditions (autumn and spring). Sequential analyses covering a year or more of shell growth allow more accurate determination of season of shell collection but require many samples from a single shell, so limit the number of individual shells that can be analyzed. Recent studies have suggested a medium between these two extremes whereby the pattern of stable isotope variation of three or four sequential samples inward from the outermost growing edge can accurately illustrate season of death (Mannino et al. 2003).

Paleoclimate

Mollusc shell chemistry provides an archive of climatic and environmental data. Oxygen isotope records from archaeological mollusc shells allow us to study how humans responded to changing climatic regimes in the past. Emiliani et al. (1964) were the first to apply oxygen isotope analyses to archaeological shells to reconstruct paleoclimate. They studied limpets and top shells from Haua Fteah in Libya and Arene Candide in Italy and identified late Quaternary climatic oscillations that correlated with regional Mediterranean climate records.

Most early applications of stable isotope analysis on mollusc shells focused on grinding up whole shells for bulk analyses to obtain average annual climate records (e.g., Mook 1971). Innovations in mass spectrometry and reductions in analysis costs allowed smaller sample sizes to be analyzed, permitting more routine subsampling of shells to obtain series analyses within a single shell.

Recently, there has been a shift toward combining records of shell growth and geochemistry in order to obtain highly detailed time-resolved records of climate and environment. This research comes under the umbrella of sclerochronology, the study of the physical and chemical variations in the accretionary hard parts of organisms. The time span and resolution that can be obtained for climate records from mollusc shell growth increments depends upon on the sampling method used, the growth rates, and the longevity of the shell. The majority of molluscs exploited by humans have life spans typically less than 10 years. Subsamples from short-lived species produce subseasonally resolved snapshots of the amplitude of the seasonal cycle at the time of shell growth.

In the last decade, highly detailed long-term climate records have been obtained from long-lived bivalve species such as *Arctica islandica* which can live for over 500 years. These slower-growing molluscs can provide annual to decadal records of climate change which may allow identification of climate cycles such as the North Atlantic Oscillation (NAO) as well as long-term climatic oscillations such as Dansgaard/Oeschger cycles throughout the late Pleistocene. Cross-dating techniques, where the individual histories of several shells can be aligned chronologically based on overlaps in their periods of shell growth, allow the reconstruction of longer time series of environmental change. For example, cross-dated archaeological *A. islandica* shells from a Norwegian Stone Age midden have been used to reconstruct a 155-year record of climate related to prehistoric occupations (Helama & Hood 2011).

Shell Sourcing

Another novel application of stable oxygen isotope analyses to archaeological mollusc shells is the use of $\delta^{18}O$ to track foraging ranges. In areas with differential freshwater inputs/salinity regimes but similar temperature ranges, the shape of the seasonal $\delta^{18}O$ curve should be similar, but the absolute values should vary in tandem with salinity (e.g., Andrus & Thompson 2012). Examining the changes in salinity from $\delta^{18}O$ allowed the collection range along the coast to be estimated, allowing more detailed reconstructions of foraging strategies beyond season of collection.

Key Issues/Current Debates

Vital Effects

As with any living creature, molluscan life cycles are complex. An attempt to understand their

growth cycles, physiology, and habitat preferences must be made before their shell morphology and geochemistry can be interpreted with confidence (Schöne 2008).

Shell growth may be modulated by internal rhythms or by environmental factors such as temperature extremes, turbidity, storms, or predation. Under favorable ambient environmental conditions, most molluscs precipitate shell material at regular intervals, termed growth increments. Regular periods of slowdown and cessation of growth result in growth lines that interrupt growth increments with periodicities ranging from subdaily to annual. Shell growth may also cease at irregular timescales as the animal aestivates as a result of ambient environmental conditions that exceed the physiological tolerance of the organism such as increased temperature or salinity. In all cases, there are times when ambient environmental conditions are not recorded in shell growth (Schöne 2008).

Shell growth is generally faster during favorable environmental conditions. Therefore, isotopic records taken at regular intervals across a growth band will be biased toward the animal's physiological optimum. Reproduction may slow growth in some species. Mollusc growth rates also vary throughout their life cycles, tending to decrease as the animals age. Variable growth rates will result in time-averaged values for shell carbonate for the periods of slower shell growth. Research on modern populations has shown that the timing and regularity of growth line and growth increment deposition can vary from species to species and sometimes within a single species across different latitudes and environments. These variations need to be understood in order to apply an appropriate sampling methodology to reliably interpret the isotopic records. When a mollusc's growth rate is understood, the time averaging caused by variable growth rates can be corrected for by mathematically weighting each sample (Schöne 2008).

Understanding the habitat preferences of different mollusc species is also crucial to the interpretation of their isotopic signatures. Different species of molluscs that live contemporaneously within a marine, freshwater, or terrestrial context can have different isotopic composition due to differences in water-isotope composition between different microhabitats. Furthermore, in ancient contexts where chronological resolution is more limited, the range in isotope ratios for each species from a single context can be large. Only by analysis of a significant number of species-specific shells from each sampling interval can a true understanding of environmental change be obtained.

Past Water Composition and the Reconstruction of Sea Surface Temperature

Temperature equations (e.g., Dettman et al. 1999) offer the opportunity to reconstruct sea surface temperature from marine shell $\delta^{18}O$ as long as the $\delta^{18}O$ of the seawater where the shell grew is known or can be estimated. Changes in continental ice volume have caused the oxygen isotope ratios in seawater to vary considerably throughout the Quaternary. Mollusc shell $\delta^{18}O$ is a function of both $\delta^{18}O$ of ambient water and temperature. In order to calculate paleotemperatures from shell $\delta^{18}O$, an estimate of paleo $\delta^{18}O$ of seawater must be used. Paleo-seawater $\delta^{18}O$ is most commonly evaluated indirectly through the analysis of $\delta^{18}O$ in fossil carbonates such as planktonic foraminifera or from analysis of resistant organic compounds (alkenones) preserved in sediment cores. Another method is to directly measure $\delta^{18}O$ from pore water extracted from marine sediment cores. Such studies have shown that the last glacial maximum had the highest $\delta^{18}O$ values in the last glacial cycle corresponding with maximum ice sheet extent. This reflects the $\delta^{18}O$ of past bottom waters which can be extrapolated to surface waters. For a more detailed discussion of seawater $\delta^{18}O$ composition, see Rohling (2007).

These methods of paleo-water $\delta^{18}O$ reconstruction yield average values that cannot account for seasonal fluctuations in water composition or for differences in seawater $\delta^{18}O$ that can occur over small geographic ranges. A change in water $\delta^{18}O$ of just 0.5 ‰ (which is the typical seasonal range for many parts of the

Mediterranean) can lead to a calculated temperature difference of 2 °C. Applying this to the paleorecord where seawater $\delta^{18}O$ is less well constrained could lead to uncertainties of several degrees in temperature reconstructions. These factors must be borne in mind when using mollusc shell stable isotopes for paleothermometry studies.

Diagenesis

In archaeological mollusc shells, recrystallization of the shell carbonate due to diagenesis will alter the isotopic ratios in the shell carbonate. Diagenesis can occur because of cementation (the addition of abiotic carbonate after death) or because of dissolution and re-precipitation of carbonate within the shell after death. If these phases are analyzed, they do not represent the chemistry of shell growth but the chemistry of the water at the time of diagenesis. Therefore, care must be taken to avoid sampling regions of shells that are diagenetically altered. This can be done in a variety of ways. High-resolution microscopy can be used to directly observe diagenetic alteration in polished section. Sampling strategies or sampling "maps" can then be devised in order to avoid the recrystallized part of the shell. X-ray diffraction can be routinely applied to detect recrystallization in shells originally composed of aragonite that may be altered to the more stable carbonate form of calcite.

Future Directions

As this review shows, stable oxygen isotope analyses on archaeological mollusc shells can yield valuable high-resolution, quantitative environmental archives from the time of shell growth as well as provide information on prehistoric human subsistence strategies. Future research is likely to focus on four key issues:

1. *Modern validation studies*: It is becoming increasingly evident that inter- and intraspecies differences in growth rates, physiology, and environmental responses can cause variations in the isotopic profiles of mollusc shells. Therefore, before stable isotope analysis is applied to archaeological shells, it is necessary to examine modern populations of the proxy species to understand how isotopic variation in shell carbonate correlates to local environmental conditions. This allows potential isotopic offsets between the environmental signal and the shell carbonate to be identified and, potentially, allows the generation of a more quantitative record of environmental change.
2. *Integration of sclerochronology and cross-dated chronologies*: The integration of detailed studies of shell growth with geochemical sampling enables the generation of high-resolution time-resolved records of environmental change. Cross-dated chronologies can then be constructed to generate continuous, high-resolution records of climate change from archaeological sites.
3. *Combination of stable isotopes with other proxies*: In some mollusc species, element ratios such as Mg/Ca and Sr/Ca serve as proxies for sea surface temperature that are not influenced by the stable isotope composition of ambient water. The combination of independent methods of temperature reconstruction with stable isotope analysis allows the effects of changing water composition from factors other than temperature (such as rainfall) to be disentangled from the isotope record to provide a more robust proxy of temperature. However, the incorporation of strontium and magnesium into mollusc shell carbonate appears to be more variable between and within species, so more work on modern validation of these trace element proxies is necessary before the potential of these integrated records can be realized (e.g., Surge & Walker 2006).
4. *Clumped isotope geochemistry*: This newly emerging field examines the extent to which isotopes such as ^{13}C and ^{18}O bond with or near each other rather than with the surrounding light isotopes. The proportions of ^{13}C–^{18}O bonds in carbonate minerals are sensitive to their growth temperatures, independent of bulk isotopic composition. Therefore, clumped isotopes in ancient carbonate can be

used as a quantitative paleotemperature proxy that requires no assumptions about the $\delta^{18}O$ of waters from which carbonates grew (Eiler 2007). The application of clumped isotope analysis to marine and freshwater shells is rapidly expanding; however, the infancy of the techniques means extensive modern validation studies are needed. Furthermore, large sample sizes are required, currently hindering the application of this technique to seasonality studies. The only study to date that has investigated clumped isotopes of modern terrestrial shells showed the results were complicated by snail ecophysiological adaptations such as shell color, morphology, and behavior (Zaarur et al. 2011). Thus, further work is required to disentangle their influence before this technique can be applied to archaeological terrestrial shells.

Cross-References

- ▶ Isotope Geochemistry in Archaeology
- ▶ Molluscs (Invertebrates): Analyses in Environmental Archaeology
- ▶ Zooarchaeology

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Further Reading

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Molodin, Vyacheslav I.

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Basic Biographical Information

Vyacheslav Ivanovich Molodin (Fig. 1) was born in 1948. He was a Soviet and Russian archaeologist who specialized in archaeology and the prehistory of Siberia. Molodin was born in Orekhovo village of the Brest Region. In 1971, he graduated from the Department of History and Philology at the Novosibirsk State Pedagogical Institute. In 1984, he received his doctorate degree in history.

V.I. Molodin joined the Siberian Branch of the USSR Academy of Sciences in 1971 as postgraduate student of the Institute of History, Philology and Philosophy where he was Head of the Division from 1989 to 1991. In 1992, he became Professor and Deputy Director of the Institute of Archaeology and Ethnography of the Siberian Branch of the Russian Academy of Sciences. He was elected a full member of the Russian Academy of Sciences in 1997. From 1997 to 2001, he was Deputy Chairman of the Siberian Branch of the Russian Academy of Sciences. He was the First Deputy Chairman of the Siberian Branch of the Russian Academy of Science from 2001 to 2007.

Molodin, Vyacheslav I., Fig. 1 Vyacheslav Ivanovich Molodin

V.A. Molodin is a member of the Presidium of the Russian Academy of Sciences, and the Presidium of the Siberian Branch of the Russian Academy of Sciences, as well as the Bureau of the Department of History and Philology of the Russian Academy of Sciences. He is a corresponding member of the German Archaeological Institute.

Major Accomplishments

V.A. Molodin discovered and studied a number of archaeological cultures that existed in