

Salinity reconstruction of the *chaîne opératoire* of briquetage along the Tyrrhenian coast in Central Italy using diatoms

Majoi N. Nascimento - *University of Amsterdam, Institute for Biodiversity and Ecosystem Dynamics
Department of Ecosystem and Landscape Dynamics (IBED - ELD)*

Introduction

After the discovery of a large complex of early salt production sites in Central Italy, evidence shows that between the Final Bronze to Early Iron Age, there was a transition between a small-scale and labor-intensive industry known as briquetage to a large-scale salt production in salterns (Alessandri 2013, 2019, Sevink et al. 2020). Briquetage consists of preparing and boiling a solution of seawater in ceramic jars to obtain solid salt, whilst salterns are a more efficient production methodology that uses a series of ponds for salt precipitation and is associated with early urbanization. Both methods of salt production go through the same steps (Weller 2015, Tencariu 2015), which are concentration, or salt enrichment to produce brine; crystallization, by artificial (fire) or natural (sun) evaporation process; conditioning, or moulding; and packaging. It is probable that diatoms dwelled these ceramics/salterns. Diatoms are external, box-like, two-part siliceous skeleton of single-celled algae. These unicellular organisms range from approximately 10-100 μm in size and can present different morphological forms, including centric and pennate (Figure 1). Diatoms inhabit all marine and non-marine aquatic and humid environments, including soils and trees, and can occur in a wide range of environmental conditions (Round 1990). Here we suggest that diatoms can be useful in identifying the different salinity levels between the enrichment and crystallization steps at the ceramic jars (briquetage method) or ponds (saltern method); provide examples in which diatoms were successful in responding to questions relevant to archaeological research and describe how the methodology should be applied.

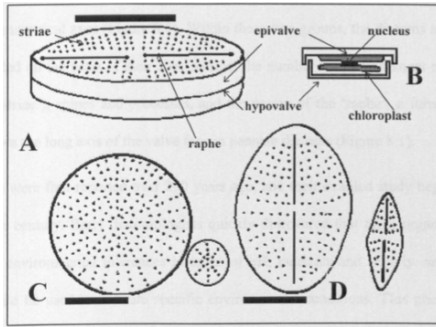


Figure 1. Morphology of the diatom siliceous skeleton. View of a complete box-like, two-part siliceous skeleton of a diatom cell (A), schematic cross-section with internal organelles (B), example of centric diatoms (C) and example of pennate diatoms (D). Scale bars = 100 / μ m (A) and 10 / μ m (C and D). Source: Brasier (1985) and Barber and Haworth (1981).

Diatoms have been used in different natural and artificial systems over time to identify ecosystem's response to change in climate, hydrology or anthropogenic impacts (Birks et al. 1990, Baker 2001, Cennamo et al. 2012, King et al. 2017, Druzhinina et al. 2022). Specifically for archaeological settings, diatoms have been used to recover an array of information on past environmental and human behaviour (Denys 1992, Ashley 2009, Bathurst 2010, Cennamo 2012, Grana and Prieto 2021). Diatoms from sediments and/or soil profiles has been proved particularly successful. One example is on the timing of establishment of an early Roman port near pudding Lane in London was defined based on sedimentary diatoms present in the excavation. The archaeological site was originally constructed by Romans on a riverine area and the abrupt change in diatom populations indicated when marine sediments started to be deposited (Battarbee 1983). Diatoms were used to define differences in salinity on cores from the Caprolace lagoon, the region of interest of this work in Central Italy, helped elucidate the timing of changes in the region's coastal hydraulic system (Alessandri et al. 2019). Diatoms associated with dated macrofossils pointed to the presence of a salt marsh with initially shallow and brackish waters, changing to a more saline environment. Later sedimentary transition points to a deeper lagoon with still saline conditions around 1500 cal BCE. This probably represented a lagoon that was open to the sea, and the subsequent increase in water level. This finding is important because it excludes the existence of a well-developed closed coastal dune system as is present today, and was developed before the 19th century. In this system the species indicating high salinity was *Cyclotella cf. quillensis* L.W.Bailey 1921 (Fig. 1), whilst the ratio between the sum of planktic and benthic species indicated differences in water levels.

Most of the studies using diatom from pottery have been used to associate those pottery to possible clay sources (Gibson 1986, Håkansson and Hulthén 1986, Battarbee 1988, Riederer 2004, Solá and Morales 2007), or to investigate clay preference by people (Alhonen & Matiskainen 1980). To the best of our knowledge, the efficiency of diatoms to elucidate the levels of salinity form content of archaeological pottery has not been yet performed and will be tested.

Identifying different salinity levels using diatoms

Diatom as a tool to identify different levels of salinity has been widely and successfully used in archaeological research (Battarbee 1988, Stone and Yost 2020). Although as a group, diatoms can live on a wide range of salinity levels, the biodiversity, species composition, and relative abundance of each species will be greatly affected by those same levels (Bae et al 2020). For biodiversity levels usually decrease as the gradient in salinity increases (Kirkwood and Henley 2006). Diatoms occur in hypersaline environments with salinity levels above 100 ppt (kilos of salt per kilo of water in parts per thousand), with few species occurring at salinity levels of saturation or 300 ppt (Clavero et al 2000, Silvestre et al. 2001, Kirkwood and Henley 2006, Gomes et al. 2018, Bae et al. 2020, Lopez-Fuerte 2020). To identify the different salinity levels between the enrichment and crystallization steps at the ceramic jars (briquetage method) or ponds (saltern method) we assume that either species composition and relative abundance will change towards the increase of taxa that have preference and/or are tolerant to high levels of salinity, and/or that biodiversity will decrease as salinity increases on jars or salterns used for different stages withing enrichment and crystallization.

Methods

The procedure for preparation of diatoms for microscopic analysis is similar for both sediment and pottery samples (Battarbee 1988, 2001 – Figure 2). For coarse sedimentary/soil material, coarse mineralogic material can be removed by sieving. For clays and silts floatation can be used in some cases. For pottery, the ratio of clay to diatoms can be high, so centrifuging at 2000 rpm for 2 min to keep the clay fraction (< 2 μm) in suspension and then decanting the supernatant is recommended. For this project we suggest that small pieces or powder of pottery to be taken from the section of the jars that used to be the inside and soak it in a 10% solution of Oxylatophosphonic acid (H₂PO₄) at 50 °C for 7-10 days, remove the coarse material and fine clays by differential settling. The fine clays will be decanted after settling for 2 hours (Hakansson and Hulthen 1986). All diatom samples should then be digested according to standard procedures using concentrated hydrogen peroxide (H₂O₂ at 35%, Battarbee et al., 1988, 2001), and then mounted on microscope slides using Naphrax (refractive index 1.7). Quantitative analysis would be performed according to Battarbee et al. (2001) using a Zeiss (Axioskop 2 plus) light microscope equipped with a high-resolution digital camera, under a 1000× magnification.

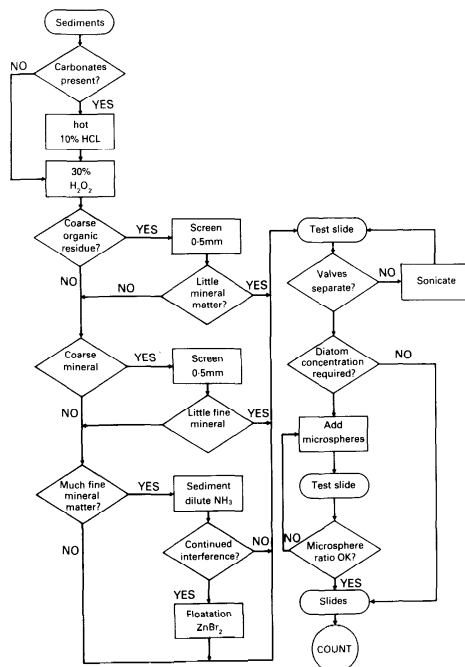


Figure 2: Schematic flowchart for the chemical preparation of diatoms from sedimentary records and pottery. Source Battarbee 1988.

At least 400 valves should be counted per slide for the sediment/soil samples and for pottery, since diatom concentrations are often low, the identification of few individuals per sample would have to be enough, although low valve density is usually the case of studies that contemplate discovering the origin of the clay, and we still do not know if this applies here. Published reference material will be used for the identification and classification of diatoms (Hudstedt 1927-1966, Krammer and Lange-Bertalot 1986; 1988; 1990; 1991, Patrick and Reimer 1966; 1975, Salden 1978, Schmidt 1874-1959, Van Heurck 1880-1885, van der Werf and Huls 1957-1974).

References

Alessandri, Luca, et al. "Salt or fish (or salted fish)? The Bronze Age specialised sites along the Tyrrhenian coast of Central Italy: New insights from Caprolace settlement." *PloS one* 14.11 (2019): e0224435.

Alessandri, L., 2013. *Latium Vetus in the Bronze Age and Early Iron Age / Il Latium Vetus nella età del Bronzo e nella prima età del Ferro*. BAR International Series, 2565, Oxford.

Alhonen, P. E. N. T. T. I., et al. "Application of AAS, diatom analysis and stylistic studies to Finnish Sub-Neolithic pottery." *Bulletin of the Geological Survey of Finland* 52 (1980): 193-206.

Ashley, G. M., Tactikos, J. C., & Owen, R. B. (2009). Hominin use of springs and wetlands: Paleoclimate and archaeological records from Olduvai Gorge (~1.79–1.74 Ma). *Palaeogeography, Palaeoclimatology, Palaeoecology*, 272(1–2), 1–16.

Bae, Hanna, et al. "Shift in benthic diatom community structure and salinity thresholds in a hypersaline environment of solar saltern, Korea." *Algae* 35.4 (2020): 361-373.

Baker, Paul A., et al. "The history of South American tropical precipitation for the past 25,000 years." *Science* 291.5504 (2001): 640-643.

Barber, Horace G., and Elizabeth Y. Haworth. *A guide to the morphology of the diatom frustule: with a key to the British freshwater genera*. Freshwater Biol. Assoc., 1981.

Bathurst, Rhonda R., Davide Zori, and Jesse Byock. "Diatoms as bioindicators of site use: locating turf structures from the Viking Age." *Journal of Archaeological Science* 37.11 (2010): 2920-2928.

Battarbee, R. W. (1988). Use of diatoms in archeology: A review. *Journal of Archaeological Science*, 15, 621–644.

Battarbee, R. W., Jones, V. J., Flower, R. J., Cameron, N. G., Bennion, H., Carvalho, L., & Juggins, S. (2001). Diatoms. In J. P. Smol, H. J. B. Birks, & W. M. Last (Eds.), *Tracking environmental change using*

lake sediments (Terrestrial, algal, and siliceous indicators) (Vol. 3, pp. 155–202). Dordrecht, The Netherlands: Kluwer Academic Publishers.

Birks, H. J. B., et al. "Diatoms and pH reconstruction." *Philosophical transactions of the royal society of London. B, Biological Sciences* 327.1240 (1990): 263-278.

Brasier, M. D. "Evolutionary and geological events across the Precambrian–Cambrian boundary." *Geology Today* 1.5 (1985): 141-146.

Cennamo, Paola, et al. "A survey of the algal flora of anthropogenic caves of Campi Flegrei (Naples, Italy) archeological district." *Journal of Cave & Karst Studies* 74.3 (2012).

Clavero, Ester, et al. "Salinity tolerance of diatoms from thalassic hypersaline environments." *Journal of Phycology* 36.6 (2000): 1021-1034.

Denys, L. (1992). On the significance of marine diatoms in freshwater deposits at archaeological sites. *Diatom Research*, 7(1), 195–197.

Druzhinina, Olga, et al. "Anthropogenic impact on the landscape of the Vishtynets Upland (Kaliningrad region, SE Baltic) in prehistory and Middle Ages: A multi-proxy palaeoenvironmental study." *Quaternary International* (2022).

Gibson, A. M. (1986). Diatom analysis of clays and Late Neolithic pottery from the Milfield Basin, Northumberland. *Proceedings of the Prehistoric Society*, 52, 89–103.

Gomez, Fernando Javier, et al. "Calcium carbonate precipitation in diatom-rich microbial mats: the Laguna Negra hypersaline lake, Catamarca, Argentina." *Journal of Sedimentary Research* 88.6 (2018): 727-742.

Grana, Lorena, and Gabriel Prieto. "Marine diatom remains as bioindicators of the uses of pre-Hispanic fishing gear recovered in ritual contexts at Huanchaco, north coast of Peru." *Journal of Archaeological Science: Reports* 39 (2021): 103167.

Håkansson, H., & Hulthén, B. (1986). On the dissolution of pottery for diatom studies. *Norwegian Archaeological Review*, 19(1), 34–38

King, D. J., Searcy, M. T., Yost, C. L., & Waller, K. (2017). Corn, beer, and marine resources at Casas Grandes, Mexico: An analysis of prehistoric diets using microfossils recovered from dental calculus. *Journal of Archaeological Science: Reports*, 16(October), 365–379.

Kirkwood, Andrea E., and William J. Henley. "Algal community dynamics and halotolerance in a terrestrial, hypersaline environment 1." *Journal of Phycology* 42.3 (2006): 537-547.

Lange-Bertalot, Horst, et al. Freshwater benthic diatoms of Central Europe: over 800 common species used in ecological assessment. Vol. 942. Schmitten-Oberreifenberg: Koeltz Botanical Books, 2017.

López-Fuerte, Francisco Omar, et al. "Halamphora siqueirosii (Bacillariophyta), a new diatom species isolated from a hypersaline evaporation pond in Baja California Peninsula, Mexico." *Phytotaxa* 451.2 (2020): 132-44.

Riederer, J. "Thin section microscopy applied to the study of archaeological ceramics." *Hyperfine Interactions* 154.1 (2004): 143-158.

Round, Frank Eric, Richard M. Crawford, and David G. Mann. *Diatoms: biology and morphology of the genera*. Cambridge university press, 1990.

Sevink, J., Neef, W. De, Alessandri, L., Hall, R. van, Ullrich, B., Attema, P.A.J., 2020. Protohistoric briquetage at Puntone (Tuscany, Italy): principles and processes of an industry, based on the leaching of saline lagoonal sediments. *Geoarchaeology*. doi:10.1002/gea.21820.

Solá, Patricia, and Marcelo R. Morales. "Caracterización petrográfica y biológica de un tiesto cerámico estilo " incaico " hallado en Susques (Jujuy)." *Intersecciones en antropología* 8 (2007): 361-364.

Stone, Jeffery R., and Chad L. Yost. "Diatom microfossils in archaeological settings." *Handbook for the Analysis of Micro-Particles in Archaeological Samples*. Springer, Cham, 2020. 23-64.

Sylvestre, Florence, et al. "Modern benthic diatom distribution in a hypersaline coastal lagoon: the Lagoa de Araruama (RJ), Brazil." *Hydrobiologia* 443.1 (2001): 213-231.

Tencariu, Felix-Adrian, et al. "Briquetage and salt cakes: an experimental approach of a prehistoric technique." *Journal of Archaeological Science* 59 (2015): 118-131.

Weller, O., 2015. First salt making in Europe: an overview from Neolithic times. *Documenta Praehistorica* 42.