



**HAL**  
open science

# Could the grasses have played a role in the earliest salt exploitation? Phytoliths analysis of prehistoric salt spring from Hălăbutoaia - Țolici (Romania)

Mihaela Danu, Claire Delhon, Olivier Weller

## ► To cite this version:

Mihaela Danu, Claire Delhon, Olivier Weller. Could the grasses have played a role in the earliest salt exploitation? Phytoliths analysis of prehistoric salt spring from Hălăbutoaia - Țolici (Romania). *Archaeological and Anthropological Sciences*, 2020, 12 (11), 10.1007/s12520-020-01228-6. hal-02984560

**HAL Id: hal-02984560**

**<https://hal.science/hal-02984560>**

Submitted on 31 Oct 2020

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



# Could the grasses have played a role in the earliest salt exploitation? Phytoliths analysis of prehistoric salt spring from *Hălăbutoaia* - Țolici (Romania)

Mihaela Danu<sup>1</sup> · Claire Delhon<sup>2</sup> · Olivier Weller<sup>3</sup>

Received: 3 July 2020 / Accepted: 18 October 2020  
© The Author(s) 2020

## Abstract

The salt spring exploitation from *Hălăbutoaia* - Țolici (Neamț County, Romania) dates back to the Early Neolithic and lasted throughout Chalcolithic. The deposit stratigraphy is estimated at 8 m and covers 2500 years of history (c. 6000–3500 BCE). In order to document the possible use of plants, particularly of the grasses, in the salt production process, we realized a detailed study of phytoliths preserved in several archaeological levels of *Hălăbutoaia* site. The most identified morphotypes come from grass family. Analysis revealed an important representation of inflorescence bracts phytoliths (especially ELONGATE DENDRITIC) showing the anthropogenic origin of the assemblages. BILOBATE are also well represented. These forms attest the presence of wild panicoid grasses (e.g. wild millet) and/or cultivated millets in the area. In some very punctual assemblages, forms produced by dicots are well represented. Considering the low phytoliths production by dicots plants, it involves a special accumulation of these types of plants that could be related to woody plants used as fuel for salt production. It is very possible that grasses had a very important role in pyrotechnology used to produce salt, either as the main fuel or as a mean for controlling the temperature or even as a firelighter.

**Keywords** Salt spring · Neolithic · Cucuteni · Criș culture · Phytoliths · Romania

## Introduction

The sub-Carpathian area from Eastern Romania is characterized by a high density of saltwater springs: over 200 are

**Supplementary Information** The online version contains supplementary material available at <https://doi.org/10.1007/s12520-020-01228-6>.

✉ Mihaela Danu  
danum2007@yahoo.com

Claire Delhon  
claire.delhon@cepam.cnrs.fr

Olivier Weller  
olivier.weller@cnrs.fr

<sup>1</sup> Department of Biology, Faculty of Biology, Alexandru Ioan Cuza University of Iași, Romania, Carol I Blvd., No. 20A, 700506 Iași, Romania

<sup>2</sup> CNRS, CEPAM, UMR 7264, Université Côte d'Azur, Nice, France

<sup>3</sup> CNRS, Trajectoires, UMR 8215, Université Paris 1 Panthéon-Sorbonne, Paris, France

registered (Alexianu et al. 2007; Weller and Brigand 2017). The exploitation of some of these dates back from the Early Neolithic, during the Criș culture (Weller and Dumitroaia 2005), and represents one of the earliest evidences of salt production in Europe. Moreover, contrarily to other prehistoric salt springs in Europe (such as those from Germany or France), those from Eastern Romania have been constantly exploited through time, and the natural brine is still used by locals nowadays (Alexianu et al. 1992, 2007). The *Hălăbutoaia* - Țolici (Petricani, Neamț County) saltwater spring, discovered in 2005 (Weller et al. 2007) and highly concentrated in sodium chloride, presents direct and very accurate evidence of salt exploitation during the Neolithic and Chalcolithic periods (Brigand and Weller 2013, 2018; Dumitroaia 1994; Weller and Brigand 2017; Weller and Dumitroaia 2005; Weller et al. 2007, 2015). Its excavation revealed that it was probably a seasonal salt exploitation point (Dumitroaia et al. 2008; Monah 2008), extensively used throughout the Neo-Eneolithic (Weller et al. 2015). Seasonal or not, it is certain that natural brine and salt production was an important aspect of prehistoric activities in this area. Two techniques seem to have been practiced: pouring natural brine

onto combustion structures during the Early Neolithic and evaporation in specific ceramic containers from the Chalcolithic onwards (Sordoillet et al. 2018).

The whole salt extraction process involves the exploitation not only of the brine itself but also of several other natural resources, of which a major one is fuel. In order to elucidate some traits of the prehistoric salt production process, several analyses were run on two Early Neolithic salt working sites: *Poiana Slatinei* in Lunca (Neamţ County) by archaeobotany and micromorphology (Dufraisse et al. 2010; Weller et al. 2008) and *Hălăbutoaia* near Țolici by palynology (Danu et al. 2010), micromorphology and SEM-EDS analysis (Sordoillet et al. 2018).

In order to better document the use of plants in the salt production process, in particular for the heating of the saltwater and the management of vegetal resources, we carried out a detailed study of phytoliths from the levels linked with salt extraction at *Hălăbutoaia* site. These microremains were very well-preserved in several archaeological levels assigned from Criş to Cucuteni Cultures (c. 6000–3500 BCE). The underlying issues concerned the influence of the environment on the salt extraction process and more specifically the impact of the local vegetation on the salt extraction techniques. As they are a link of the operational chain, the availability of the plants used as fuel may have influenced the whole process, and it may be one of the reasons why this activity could have been seasonal. Moreover, the recourse to phytolith analysis allows tracing non-wood plants, which are often underestimated when the question of fuel is considered.

Phytoliths, amorphous silica (SiO<sub>2</sub>) bodies which are formed both within and between the living plant cells (Currie and Perry 2007; Madella and Lancelotti 2012; Piperno 1988), may provide important data for palaeoenvironmental reconstruction (Borba-Roschel et al. 2006; Carter and Lian 2000; Rovner 1971), and they can be successfully used in palaeoecology (Fredlund and Tieszen 1997; Li et al. 2017; Lu et al. 2007; Murungi et al. 2017; Piperno 1989) and archaeology (Albert et al. 1999; Delhon et al. 2008; Messenger et al. 2011; Ollendorf 1987; Piperno 1988). Due to the physico-chemical properties of opal, these bio-indicators are preserved very well in many sedimentary contexts, being undoubtedly the most sustainable plant fossils known so far (Piperno 2001). Although their taxonomic value is generally inferior to other botanical indicators, the phytoliths analysis presents the huge advantage that these proxies allow the determination of grass subfamilies (which is impossible with fossil pollen). Their use in archaeological context becomes more important as the signal given by these indicators is local: phytoliths are released into sediments or soil and are considered to be mainly the subject of an in situ deposition, as aerial dispersion is most of the times nearly absent, contrarily to what happens with pollen (Lebreton et al. 2017). Phytoliths dispersal can be influenced by strong

winds, but their in situ deposition, which is aided by gravity, remains dominant (An et al. 2015; Garnier et al. 2012; Piperno 2006).

We also tried to correlate the information obtained from phytoliths analysis with palynological research results (Danu et al. 2010), as well as other archaeobotanical data (Dufraisse 2011), in order to interpret it taking into account the ancient environmental conditions from *Hălăbutoaia* - Țolici. Phytoliths found at the site are derived from the palaeoenvironment, although they reflect only specific part of it because the phytolith assemblages results from anthropic activities.

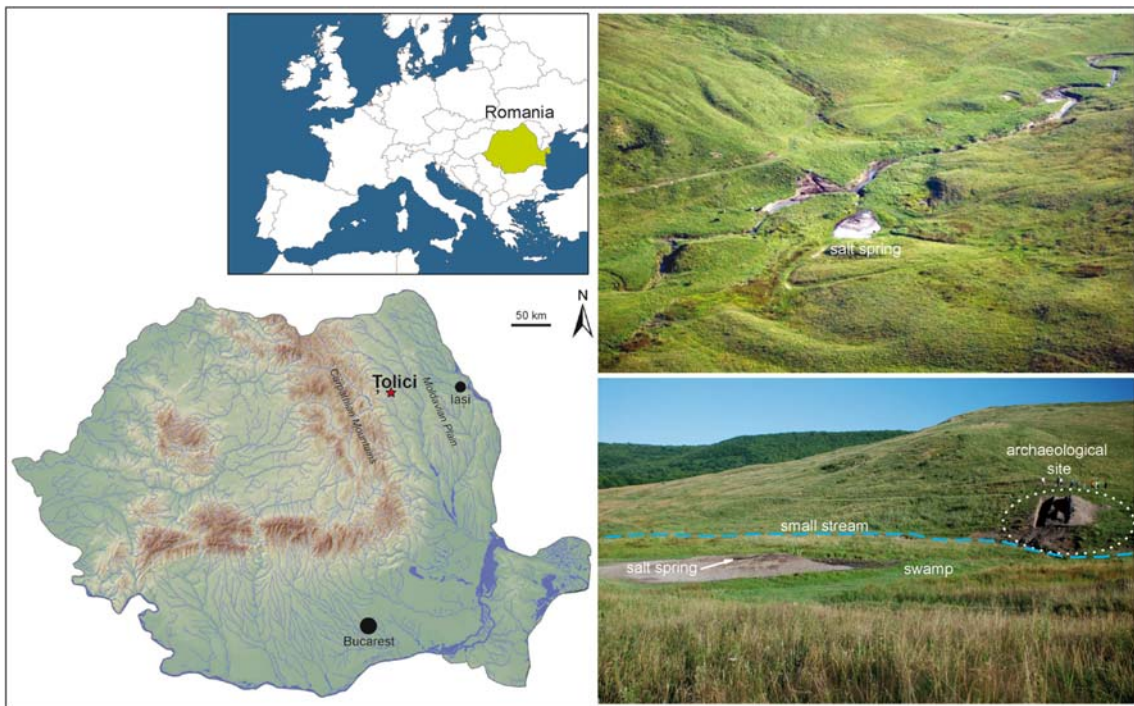
## The *Hălăbutoaia* - Țolici site and its setting

Situated to the East of the Eastern Carpathians, in the contact area between Moldova Valley and the Neamţ Subcarpathian Depression, the Țârpești - Țolici - Petricani micro-region is rich in saltwater springs, one of them being *Hălăbutoaia* - Țolici. Except for a few archaeological surveys, the exploitation of salt in this micro-region received little attention and was therefore poorly understood. Regarding archaeological science, only a few ancient palaeoecological studies have been carried out at the Neolithic settlement of *Râpa lui Bodai* in Țârpești, located 6 km north of the salt spring (Marinescu-Bîlcu et al. 1981). Discovered during our archaeological survey in 2005 (Weller et al. 2007) and then excavated in 2007–2010 (Weller et al. 2015), the *Hălăbutoaia* - Țolici salt spring is situated in a relatively narrow and deep valley, in the south-west of Țolici village, Petricani, Neamţ County (Fig. 1).

The main archaeological deposit is at the base of the east slope of *Hălăbutoaia* hill, 40 m away from a saltwater well. The anthropic deposit has the form of an ovoid mound with diameters of approximately 30 × 20 m; the general slope and the south side of the deposit are affected by an advanced erosion process revealing large quantities of fragmentary ceramics. Through a large trench in the middle part (SI, 15.5 × 1.5 m), this salt working site occurs as thick archaeological sedimentary accumulations, which are several metres high and comprise numerous lenticular layers of clay, ash and charcoal (Fig. 2).

The Neo-Eneolithic deposits represent a 8-m accumulation of combustion features and ceramics, with 1.30 m below the actual stream *Pârâul Slatina*. The whole sequence indicates the exploitation of the salt spring with typical combustion features and specific ceramic moulds usually known as *briquetage* (Weller 2015). At this salt workshop, only an artificial evaporation process would have been used in order to concentrate, until crystallization was reached, the natural highly concentrated saltwater spring using fire (ignigenous salt).

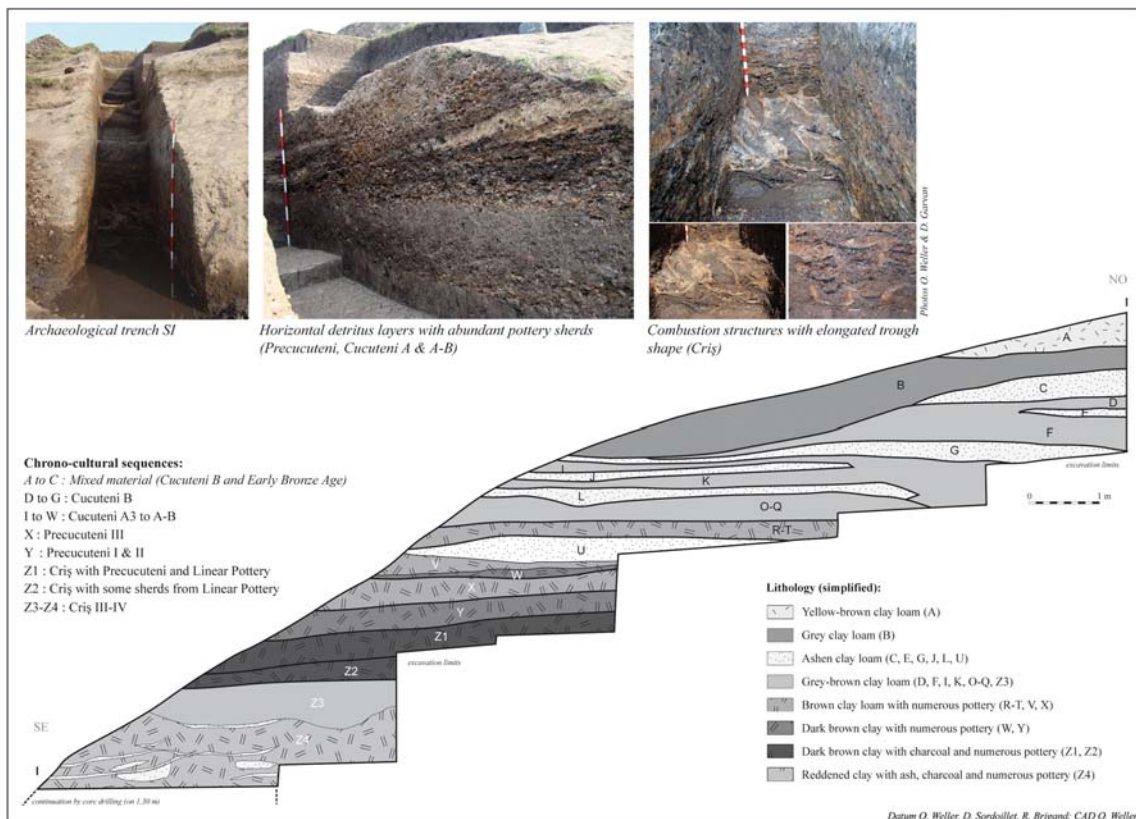
The trench allowed the study of the 7-m-thick visible stratified accumulation. The main litho-stratigraphic characteristics



**Fig. 1** Location of the archaeological site *Hălăbutoaia* at Țolici (Petricani, Neamț). Aerial photography with the salt spring in the middle (above); extension of the archaeological deposit indicated by white dotted ellipse (bottom). Map: R. Brigand & O. Weller; Photos O. Weller

of the 29 recorded strata (from A to Z4) reveal abundant combustion structures, as shown by the superposition of ashy-

charcoal residues on reddened clay (Sordoillet et al. 2018). Radiocarbon dating (Table 1) and abundant typical ceramic



**Fig. 2** Archaeological deposit and section at *Hălăbutoaia* - Țolici (SI)



**Table 1** Radiocarbon dates from *Hălăbutoaia* - Țolici

Stratigraphic unit (section SI and core depth (cm))	Laboratory ID	Material	<sup>14</sup> C age (BP)	Calibrated age (BC) 95.4% probability
SI (C-D)	Poz-30826	Charcoal	4660 ± 35	3620–3600; 3530–3360
SI (O)	Poz-30828	Charcoal	5320 ± 40	4320–4290; 4270–4040
SI (U)	Poz-30829	Charcoal	5300 ± 40	4260–3990
SI (V)	Poz-18749	Charcoal	5520 ± 40	4460–4320; 4290–4270
SI (W)	Poz-30830	Charcoal	5480 ± 40	4450–4410; 4400–4240
SI (W-FA4)	Poz-18753	Charcoal	5650 ± 40	4560–4360
SI (Z3)	Poz-41438	Charcoal	6280 ± 40	5360–5207; 5162–5138; 5129–5120; 5094–5080
SI (Z4)	Poz-18751	Charcoal	6630 ± 50	5630–5480
SI (Z4)	Poz-18752	Charcoal	6660 ± 40	5660–5510
35 (Z4)	Poz-18754	Charcoal	6750 ± 40	5730–5610; 5590–5570
70 (Z4)	Poz-24385	Charcoal	6760 ± 40	5730–5610
152 (Z4)	Poz-24424	Charcoal	6840 ± 50	5840–5630

material place the beginning of exploitation at around 5700 BCE and the end toward 3500 BCE. The well-preserved combustion structures with an elongated trough shape characterize the Early Neolithic exploitation (Criș culture), whereas the horizontal detritus layers with abundant pottery sherds characterize the Chalcolithic ones (Precucuteni and Cucuteni cultures). This remarkable deposit seems to illustrate almost all cultures and chronological stages known in Moldova, starting with Criș and until the end of the Cucuteni culture with Early Bronze Age elements, which makes this site exceptional (Dumitroaia et al. 2008; Weller et al. 2015).

## Material and method

Twenty-six samples from several stratigraphic units (US) were collected for phytolith analysis (Fig. 3; Supplementary material 1, available online). For each US (or combustion structure, all described in Sordoillet et al. 2018 - appendix B), the samples were homogeneous and grouped (see Fig. 3), the maximum distance between the samples being 12 m. Seventeen samples were directly obtained from micromorphological blocks, which insured a twofold interpretation and allowed to consider diverse stratigraphic contexts (mostly ash features with hearths, but also secondary deposits with maintenance and waste). The chronological distribution is as follows: 8 for the Early Neolithic deposits (Criș cultural period), 1 for the Final Neolithic/Early Chalcolithic (Precucuteni) and 17 for the Chalcolithic (Cucuteni, with 11 for Cucuteni A and A-B, and 6 for Cucuteni B).

Phytoliths extraction was carried out in UMR 7264 CEPAM Université Côte d'Azur - CNRS, starting from a sample of sediment of between 1 and 3 g. For chemical preparation, we followed the standard protocol of the laboratory:

- Clay deflocculation with distilled water under magnetic stirring, 200 μm tumbling for coarse particles removal, centrifugation 2000 t. min<sup>-1</sup> for clay elimination
- Decarbonation with concentrated hydrochloric acid (33%) by heat and using the ultrasonic bath
- Organic matter oxidation under hot and ultrasonic action: KOH (10%), nitric acid (30%) and hydrogen peroxide (30%)
- Phytoliths densimetric separation with sodium polytungstate, density = 2.35

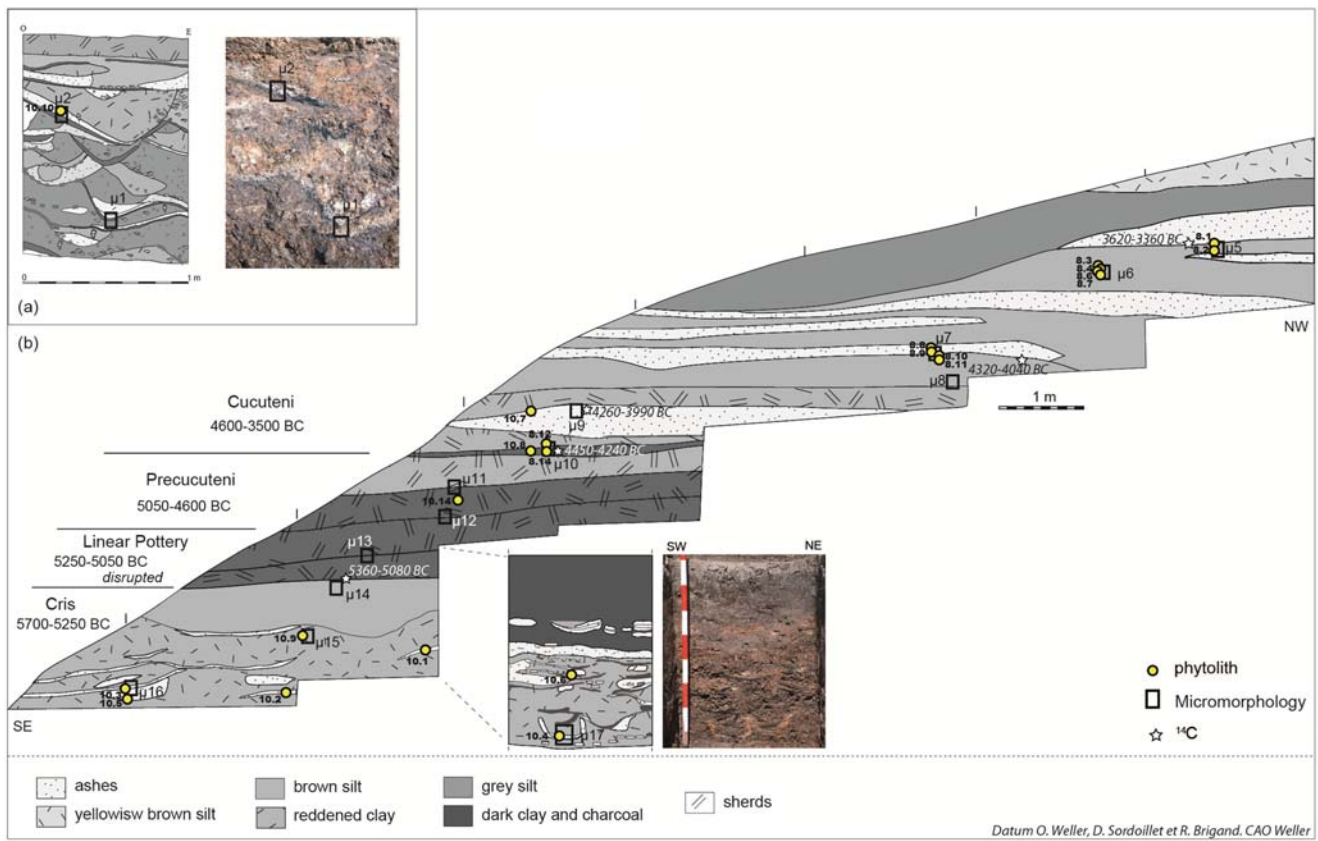
Then, the rinsed and dry extract was poured into ethanol, after which few drops were fixed on a microscopic slide using immersion oil. Observation was made at optical transmission microscope, with magnification from × 400 to × 1000. For each sample, at least 200 phytoliths with an identified morphology were counted. Phytoliths were named following the International Code for Phytolith Nomenclature 2.0 (ICPT 2019).

## Results and discussion

The samples from Țolici were found to be rich in phytoliths, except for few ones: Tol08.11, where phytoliths were less concentrated; Tol08.1 and Tol10.7 in which low concentration of phytoliths did not allow analysis to be done (although two extraction tests were performed for the first one); and Tol10.12 in which no phytoliths were observed.

### Identified morphotypes

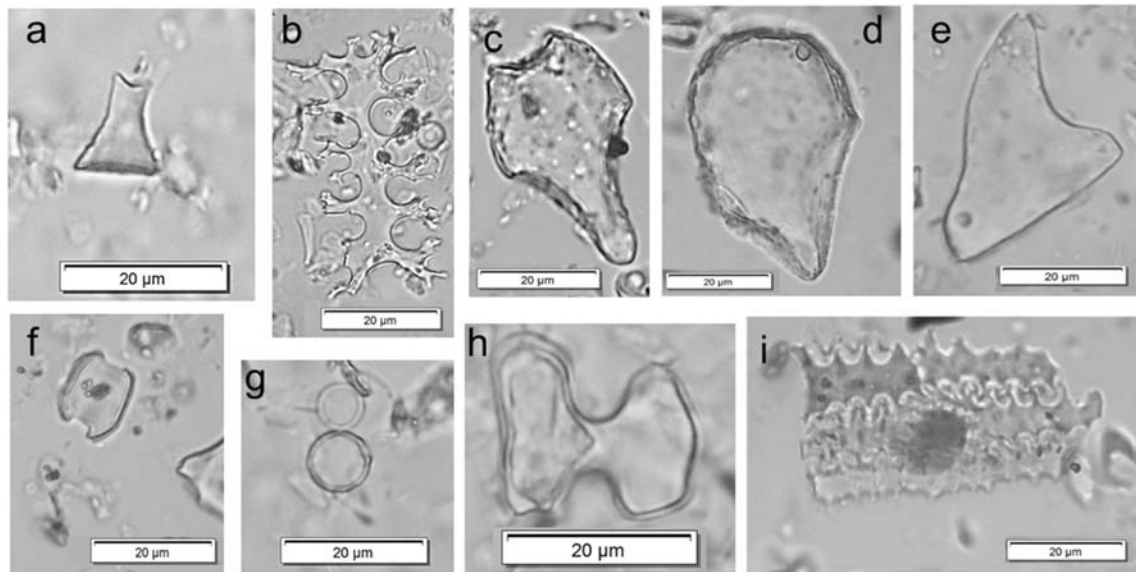
In this study, 13 morphotypes have been distinguished: RONDEL, BILOBATE, CRENATE, SADDLE, PAPILLATE, ELONGATE DENDRITIC, ELONGATE ENTIRE, ELONGATE SINUATE, ACUTE BULBOSUS,



**Fig. 3** Stratigraphic section, radiocarbon dating and sampling. (a) First samples from the elongated hollow at the base of the archaeological mound at Tölici, when it was discovered; (b) Sampling from the different chronological levels of the main stratigraphic section (SI, profil SW)

SPHEROID, BULLIFORM FLABELLATE and BLOCKY, including *Phragmites*-type, tabular polygonal Cyperaceae-type and

TRACHEARY (Fig. 4, Table 2). Articulated phytoliths, also known as silica skeleton, were observed.



**Fig. 4** Examples of phytolith morphotypes identified in *Hălăbutoaia* - Tölici samples: **a** RONDEL, **b** ELONGATE DENDRITIC, **c** BULLIFORM FLABELLATE, **d** BULLIFORM FLABELLATE (*Phragmites*-type), **e** ACUTE

BULBOSUS, **f** SADDLE, **g** GLOBULAR, PSILATE, **h** BILOBATE and **i** silica skeleton made of several ELONGATE DENDRITIC

**Table 2** Phytolith data from *Hätübutoaia* - Tolici

Samples	Site code	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol
Sample	Sample	08.1	08.2	08.3	08.4	08.6	08.7	08.8	08.9	08.10	08.11	08.12	08.12	08.12	08.12	08.12	08.12
Field data	Ref	$\mu 5.s1$	$\mu 5.s2$	$\mu 6.s1$	$\mu 6.s2$	$\mu 6.s3$	$\mu 6.s4$	$\mu 7.s1$	$\mu 7.s2$	$\mu 7.s3$	$\mu 7.s4$	U-C6	$\mu 10.s1$	A7-W			
Phytolith analysis	US	C	D	F'	F'	F	F	L	L	N	N	U	V	W			
	BILOBATE	2	45	19	56	30	45	22	18	30	26	1	24	14			
	RONDEL	13	63	80	55	92	72	79	51	86	49	13	78	140			
	CRENATE		12	11	15	15	24	8	12	9	12	1	6	6			
	SADDLE																
	PAPILLATE			1		4	1			4	2						
	ELONGATE ENTIRE	7	76	69	38	95	55	66	79	59	83	4	70	37			
	ELONGATE DENDRITIC		14	9	19	16	15	5		6	5	1	18	14			
	ELONGATE SINUATE			5	2	4	6	5	1	2	1	1	12	12			
	ACUTE BULBOSUS		4	16	14	8	14	11	43	14	14	3	3	13			
	BULLIFORM FLABELLATE			18	18	6	4	12	16	4	10	8	8	3			
	& BLOCKY																
	CYPERACEAE-TYPE			2			1	14	5	2	9	9		228			
	SPHEROID						2										
	TRACHEARY																
	Total	22	214	212	217	270	239	222	225	216	211	33	207	467			
	BILOBATE/RONDEL + CRENATE	0.15	0.60	0.21	0.80	0.28	0.47	0.25	0.29	0.32	0.43	0.07	0.29	0.10			
	ELONGATE DENDRITIC/E. ENTIRE + E. SINUATE	0.00	0.07	0.05	0.09	0.08	0.07	0.02	0.00	0.04	0.04	0.05	0.10	0.07			

Samples	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol	Tol
Sample	08.14	10.11	10.13	10.14	10.9	10.6	10.3	10.5	10.1	10.10	10.4	10.7	10.2	10.2	10.2	10.2	10.2
Field data	$\mu 10.s3/4$	$\mu 4$ haut	$\mu 4$ bas	Y'	Z3 <sup>W</sup> -C8	A11-C13	$\mu 16.4$ -C11	C12	Z4 <sup>+</sup> -C9	$\mu 2.3$	$\mu 17.2$ -C14	C10	Z4base				
Phytolith analysis	W	FA4 (W)	FA4 (W)	Y	Z3	Z4sup	Z4	Z4base	Z4	horiz profil	Z4	Z4	Z4base				
	34	8	6	15	22	2	12	8	18	12	6	13	13				
	65	33	65	143	185	144	165	33	127	152	146	143	143				
	19	2	2	5	6	2	2	5	5	3	4	3	3				
	66	24	64	43	49	83	42	87	43	57	83	1	1				
	9	7	6	66	6	11	17	12	10	6	11	49	49				
	3	6	24	15	29	35	42	24	21	36	31	9	9				
	11	9	50	22	41	45	24	67	60	30	72	40	40				
	3	9	2	2	3	3	2	20	3	11	4	7	7				
	210	96	222	402	349	335	320	262	292	313	357	291	291				
	0.40	0.24	0.09	0.10	0.12	0.01	0.07	0.21	0.14	0.08	0.04	0.09	0.09				
	0.04	0.13	0.04	0.33	0.02	0.05	0.07	0.08	0.04	0.02	0.04	0.04	0.04				

Most of identified morphotypes are assigned to grasses. A characteristic feature of Cyperaceae family has been identified only once, but it should be emphasized that this morphotype is considered fragile and it could be affected by low preservation (Borba-Roschel et al. 2006). Also, rarely and only in certain levels, SPHEROID, which are considered as characteristic of the dicotyledonous plants (Albert et al. 1999; Alexandre et al. 1997; Bozarth 1992; Delhon et al. 2003; Runge 1999), were registered. These forms do not allow a more accurate identification. Despite being often attributed to dicots, TRACHEARY phytoliths have an uncertain origin (primary, secondary, xylem or phloem?), and it seems that they could be produced by a wide range of plants (ICPT 2019).

Among the grass silica short-cell phytoliths, several morphological features can be distinguished. The main type of silica short cell varies between the grass subfamilies (Rovner 1971, Twiss et al. 1969). The most abundant morphotype is RONDEL. It is widespread in several subfamilies, but in temperate areas it characterizes the Pooideae, which produces it in large amount. The plants from that subfamily exhibit a C3 metabolism and mainly develop in temperate environments. Most of the old-world cereals belong to that subfamily. Even if they were less abundant, BILOBATE short cells were constantly present in variable amounts. They are common in the plants from the Panicoideae subfamily. These species are adapted to a warmer climate and often present a C4 metabolism. They spread over the intertropical areas, with some exceptions, in particular certain wild or cultivated millets which spread over temperate areas. Several C4 spontaneous panicoids (e.g. *Bothriochloa ischaemum*, *Cenchrus incertus*, *Chrysopogon gryllus*, *Digitaria ischaemum*, *Echinochloa colona*, *E. crus-galli*, *Eriochloa villosa*, *Panicum dichotomiflorum*, *P. capillare*, *Setaria pumila*, *Sorghum halepense*) are present on the territory of Romania, most of them being weeds (Ciocârlan 2000; Sârbu et al. 2001; Sârbu and Oprea 2011; Table 3).

Some domesticated panicoids are also currently grown in Romania: *Panicum miliaceum*, *Setaria italica*, *Sorghum bicolor*, *S. cernuum*, *S. dochna* and *S. sudanese* (Table 3). Although the Panicoideae subfamily produces abundant BILOBATE phytoliths, these forms can also occur in few festucoid grasses and some chloridoid grasses (Metcalf 1960).

*Phragmites* sp. (a C3 plant of the grass subfamily Arundinoideae) may be responsible for the sporadic occurrence of SADDLE short cells phytoliths (Ollendorf et al. 1988), which may alternatively come from few species of the Chloridoideae subfamily, which is almost entirely distributed over tropical areas. In the inflorescences bracts (glumes, lemma and palea), these short-cell phytoliths are replaced by PAPILLATE (Delhon 2008).

Long cells gather epidermal cells (ELONGATE ENTIRE, E. DENDRITIC, E. SINUATE), hairs and other epidermal

excrescences (ACUTE BULBOSUS). Bulliform cells, located in the leaves, along the nervures, provide BULLIFORM FLABELLATE and BLOCKY morphotypes. Among BULLIFORM FLABELLATE, the *Phragmites*-type was distinguished (see Gao et al. 2017; Li et al. 2014; Liu et al. 2013; Zhang et al. 2010).

The epidermal cells present morphological variations in vegetative parts (leaves and stems) and at inflorescences level (glumes, lemma and palea). Dendritic forms (ELONGATE DENDRITIC) are produced in the inflorescence bracts, while sinuous forms (ELONGATE SINUATE) are produced rather at the leaves level. Smooth shapes (ELONGATE ENTIRE) are more widespread, and they are the only ones represented in stems.

In some cases, silica skeletons could be observed. These multi-cells phytoliths correspond to silicified epidermal cells in connection (Rosen 1992). In all cases, they originated from grassy epidermis which are either from vegetative parts (ELONGATE ENTIRE morphotypes and short-cells) or from inflorescences bracts (ELONGATE DENDRITIC morphotypes and PAPILLATE). These forms are rarely observed in samples from natural (non-archaeological) contexts because of preservation (bioturbations) or statistical (low concentration) hazard. Their presence is often taken as an evidence of in situ accumulation and decomposition of plants or material of plant origin, in a context of low disturbance (in particular in pastoral sites, in the form of dung or bedding for the livestock: Cabanes et al. 2009; Portillo et al. 2014).

### Phytolith assemblages

Generally, except for the sample Tol10.8, phytoliths derived from grasses dominate all the spectra (Fig. 5, Table 2). High representation of this family is explained by its very high phytolith production. Even so, only an anthropogenic accumulation of grasses in the analysed levels can explain the relative and absolute abundances of grassland phytoliths. In addition, we note a punctual presence of silica skeletons and a good representation of inflorescence bracts phytoliths (especially ELONGATE DENDRITIC forms). Furthermore, the presence of well-preserved elongate dendritic, at exceptionally high ratios, seems to indicate a very good preservation of phytoliths (cf. Cabanes et al. 2009, 2011). In natural contexts, the proportion of DENDRITIC phytoliths remains usually low (Novello and Barboni 2015), while it sometimes reaches higher values in archaeological context (Berlin et al. 2003; Portillo and Albert 2011). DENDRITIC phytolith abundance is often interpreted as an in situ presence of grass spikelets (Dal Corso et al. 2018; Danu et al. 2019; Delhon et al. 2008). All these observations contribute to suggest that most of the studied samples are derived from an important accumulation of various parts of grasses, which could be linked with human activities.



**Table 3** List of C4 Panicoideae species in Romania

Panicoideae species	Indigenous*	Cultivated*	Adventive*	C4**
<i>Bothriochloa ischaemum</i> (L.) Keng	•			(a)
<i>Cenchrus incertus</i> M. A. Curtis			•	(b)
<i>Chrysopogon gryllus</i> (L.) Trin	•			(b)
<i>Digitaria ischaemum</i> Schreb. ex Mühl.	•			(c)
<i>Digitaria sanguinalis</i> (L.) Scop.	•			(b)
<i>Echinochloa colona</i> (L.) Link			•	(b)
<i>Echinochloa crus-galli</i> (L.) Beauv.	•			(b)
<i>Echinochloa frumentacea</i> (Roxb.) Link			•	(d)
<i>Echinochloa oryzicola</i> (Vasing.) Vasing.			•	(e)
<i>Echinochloa oryzoides</i> (Ard.) Frisch			•	(f)
<i>Echinochloa phyllopogon</i> (Stapf) Koss.	•			(g)
<i>Eriochloa villosa</i> (Thunb.) Kunth			•	(h)
<i>Panicum capillare</i> L.			•	(b)
<i>Panicum dichotomiflorum</i> Michx.			•	(b)
<i>Panicum miliaceum</i> L.		•		(b)
<i>Paspalum paspalodes</i> (Michx.) Scribner			•	(i)
<i>Panicum schinzii</i> Hack.			•	(j)
<i>Setaria faberi</i> J. Herrm			•	(k)
<i>Setaria italica</i> (L.) Beauv.		•		(b)
<i>Setaria pumila</i> (Poir) Roem. et Schult.	•			(l)
<i>Setaria verticillata</i> (L.) Beauv.	•			(c)
<i>Setaria viridis</i> (L.) Beauv.	•			(c)
<i>Sorghum bicolor</i> (L.) Moench		•		(c)
<i>Sorghum cernuum</i> (Ard.) Host.		•		(m)
<i>Sorghum dochna</i> (Forssk.) Snowden		•		(n)
<i>Sorghum halepense</i> (L.) Pers.			•	(c)
<i>Sorghum sudanese</i> (Piper) Stapf		•		(c)
<i>Tripsidium ravennae</i> (L.) Scholz			•	(o)

\*cf.: Ciocârlan 2000; Sârbu et al. 2001; Sirbu and Oprea 2011

\*\*References for C4 photosynthetic pathway: (a) Waller and Lewis 1979; (b) Downton 1975; (c) Rhagavendra and Das 1978; (d) Voznesenskaya et al. 2006; (e) Ueno and Takeda 1992; (f) Mitchell and Sheehy 2000; (g) Gibson et al. 1999; (h) Follak et al. 2020; (i) James et al. 2013; (j) Brown and Brown 1975; (k) Bazzaz 1996; (l) Tuba et al. 2003; (m) Mall 2019; (n) Erdei et al. 2009; (o) Shimomae et al. 2015)

Several samples (Tol08.8, 08.9, 08.11, 10.11, 10.13, 10.3, 10.5 and 10.2) are characterized by a low content (each circa. 2%) of phytoliths possibly derived from dicots. Only two other samples contain very high amounts of SPHEROID from dicots (Tol10.8: 22.6% and Tol10.14: 48.8%). The wood from broadleaved trees and conifers, and therefore its ash, contains only few phytoliths (Delhon 2010), which seem to be mainly produced by the green parts (leaves, young branches). Based on low phytoliths production from dicots compared to that of grasses, relatively modest percentages of these morphotypes can be interpreted as meaningful. Low phytoliths production from dicotyledonous plants involves a specific accumulation of these plants in the levels of Tol10.8 and Tol10.14, which could be related to some aspects concerning the use of woody plants as fuel for salt production. Among short cells, RONDEL and CRENATE forms mainly produced by Pooideae are

dominant, but we mention a very good representation of BILOBATE (up to 25.8%), especially at the top of diagram (starting from the Tol10.11 sample). It may suggest the presence of wild panicoids but also of cultivated millets. Concerning the spontaneous species from Panicoideae, we assume that the adventive ones (see Table 3) are less likely and therefore only a few spontaneous species could be involved in the phytolith record of Hălăbutoaia: *Chrysopogon gryllus*, *Dichanthium ischaemum*, *Digitaria sanguinalis*, *Echinochloa crus-galli*, *Setaria pumila*, *S. verticillata* and *S. viridis*. Regarding the domesticated millets, the study of plant macroremains confirms the presence of *Panicum* sp. and *P. miliaceum* in Neolithic sites on the Romanian territory (Cârciumaru et al. 2004; Comşa 1996) but also in Cucuteni-Trypillian sites from Moldavia (Janushevich 1976), Slovakia (where *Setaria viridis* was also mentioned) or Ukraine (Hunt

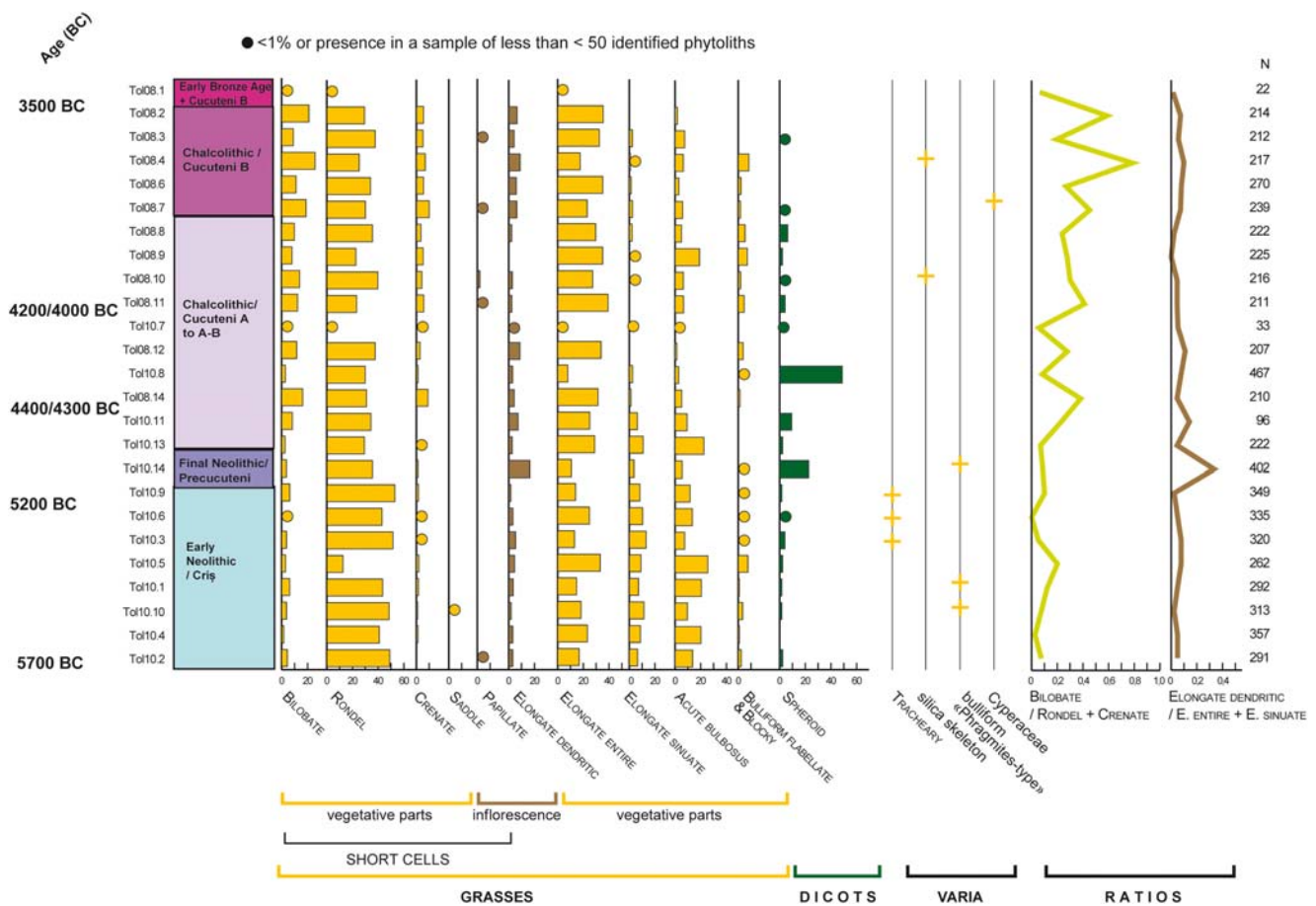


Fig. 5 Phytolith diagram from Hălăbutoaia - Țolici

et al. 2008). Morphotypes typical of panicoid grasses are also attested in other Romanian Cucutenian site from Neamț County, Răucești, but in lower amounts (11% BILOBATE in cultural layer, cf. Danu et al. 2016). At Hălăbutoaia - Țolici, BILOBATE showed varied forms. Beside the so-called “dumb-bell” form (Madella et al. 2005), very elongated morphotypes were observed with bulging extremities connected through a long and thin axis (these forms are often fragmented, broken in the form of half-dumbbells). Also, much more contracted forms, almost square-shaped with a less evidenced central constriction, were noticed. Many intermediate forms also occurred. The identification of millet phytolith has been questionable over time. Some studies showed that there is no valid method for separating *Setaria italica* and *Panicum miliaceum* based on inflorescence phytolith analysis (Harvey and Fuller 2005; Parry and Hodson 1982), but new characters of phytolith identification for husks and leaves of these two species have been developed and evaluated as a reliable way of distinguishing foxtail millet from common millet (Lu et al. 2009; Out and Madella 2016). The possibility of distinguishing panicoid grasses in a fossil phytolith assemblage has been shown in the records of grass short-cell phytoliths (BILOBATE, POLYLOBATE and CROSS) and silica

skeletons from sediments of a Bronze Age settlement situated in northern Italy (Dal Corso et al. 2017). Numerous cut silica skeletons, from grass culms or culms and from grass inflorescences, are derived from panicoid, but also pooid were present in phytolith record, indicating crop processing activities near the site of Fondo Paviani (Italy).

Therefore, several species are probably involved in the abundance of BILOBATE at Hălăbutoaia site, but the absence of local references samples from plants prevented further identification. More research should be carried out in the future on local wild and cultivated Panicoideae.

### Phytolith ratios

It must be noted that the combustion process leaves no easily visible and unequivocal tracks on phytoliths (Parr 2006; Evett and Cuthrell 2017), despite some changes in their refractive index whose observation remains technically difficult (Elbaum et al. 2003). According to Cabanes and Shahack-Gross (2015), pitting and melting become common after the combustion. We did not notice such traces, but considering the archaeological context (large amount of ash and charcoal, cf. Dufraisse 2011; Sordoillet et al. 2018), it is very likely that

these microscopic remains come mainly from plant used as fuel or at least were related to fire purposes. To go further in the interpretation of the phytoliths spectra, we used two ratios developed in order to give a better illustration of the relative variations between selected morphotypes. The ratio of BILOBATE vs. RONDEL and CRENATE (BIL/RON + CRE) is a proxy for the contribution of panicoid grasses vs. pooid grasses in the phytolith record, and the ratio of ELONGATE DENDRITIC vs. ELONGATE ENTIRE and SINUATE (ELO\_DEN/ELO\_ENT + ELO\_SIN) quantifies the proportion of phytoliths from inflorescences bracts vs. phytoliths from vegetative parts.

The BIL/RON + CRE ratio is a simplification of Ic index (“climatic Index”, in which SADDLES also contribute to the divisor) used in tropical regions as a climatic indicator (Twiss 1987, 1992). It draws the proportion of C3 (temperate) grasses against C4 (thermophilic) grasses. It is used here in an unusual way, since variations of the abundance of the forms that characterize the Panicoideae appear to be more related to the selection of certain spontaneous grasses (e.g. wild millets) or cultivated panicoid taxa (cultivated millet) rather than to variations in their abundance in the natural grasslands around the site.

The ELO\_DEN/ELO\_ENT+ELO\_SIN ratio varies either according to season of harvest (presence or absence of spikelets) or to the plant parts selected. The use of straws, of the whole plant, or of residues from spikelets processing can thus be traced. According to Regev et al. (2015), a ratio of leaf-stem to inflorescence which is lower than 1 indicates a tendency toward having more inflorescence (chaff), and a ratio lower than 0.5 indicates predominance of inflorescence phytoliths (most probably an intentional selection of chaff). A ratio higher than 1 indicates a tendency toward having more leaves and stems (e.g. straw, hay), and a ratio above 1.5 indicates predominance of leaves/stems (e.g. straw, hay). Phytolith assemblages from *Hălăbutoaia* - Țolici site show that the ratio ELO\_DEN/ELO\_ENT + ELO\_SIN is lower than 0.5; thus, we assume that may indicate the intentional use of chaff as fuel. Moreover, this ratio may also indicate if wild grasses or cereals are preferentially used. High amounts of grass inflorescence bracts phytoliths are often considered more likely related to cultivated grasses than to spontaneous ones, due to the facts that grains produce bigger spikelets and are normally harvested during seasons when inflorescences are fully developed.

The BIL/RON + CRE ratio shows a curve made of two steps. The first one corresponds to low values (nearly always < 0.2) and stretches from the bottom of the sequence to Tol10.11 sample. The second one corresponds to the increase of the index (nearly always > 0.2 and reaching 0.6 in Tol08.4) in the upper part of the sequence. Thus, the use of panicoid grasses remains as fuel seems to increase in the second part of the sequence. Considering the recurrence of high proportion

of grass inflorescences bracts phytoliths, the recourse to cultivated grasses (or their by-products) seems highly possible. These two observations argue for an agricultural origin of the Panicoideae grasses involved in the record and thus to an increase of the use of their by-products as fuel in the second part of the sequence. Despite they may enhance the main traits perceived through the phytolith diagram, these indices remain complex and their variations potentially linked to several different anthropic or natural phenomena. It is not so simple to decipher all the information they contain; in that purpose, we finally correlated them with data obtained from other domains.

### Correlations with other achaeobotanical data

Palynological analysis of a sediment core taken in a swamp nearby the salt spring and the archaeological site (Danu et al. 2010) made it possible to highlight, during the Cucutenian period, a relatively open vegetation, marked by presence of anthropogenic activities: agriculture, animal farming and salt exploitation. This pollen record (cf. Danu et al. 2010) is dominated by non-arboreal pollen, such as Poaceae (up to 40%) and from herbaceous plants from grassland pasture (Cichorioideae, Anthemideae, Apiaceae, etc.). Within this open environment appear numerous anthropogenic indicators: pollen grains of *Cerealia*, pollen of weeds (*Polygonum aviculare*) and plants of trampled and grazed areas (Plantaginaceae, *Rumex*). Among the non-pollen palynomorphs, coprophilous fungi (Sordariaceae, HdV 55) have been identified suggesting a pastoral presence nearby the site. In this open plant landscape, anthracological data (Dufraisse 2011) highlights opportunistic exploitation, apparently without any selection of particular woody species, of formations such as riverine forests and mixed oak forests as wood fuel supplies. Symmetrically, phytoliths analysis could complete the range of the opportunistic harvest of fuel by also considering the available non-wood materials. The use of Poales herbs as fuel (and maybe for temperature control) is also presumed in other fire-related contexts (Esteban et al. 2018). The high representation of grass phytoliths at *Hălăbutoaia* and their, at least partly, agricultural origin reveals that herbaceous plants have been used as fuel or at least had a potentially important role in operational salt exploitation chain.

Moreover, it appears that this material is not subject to strong selection as various species from various subfamilies are involved and as it could, at a certain extent, be derived from agricultural by-products.

Although the analysis of non-pollen palynomorphs indicated the animal presence in the past landscape of *Hălăbutoaia* - Țolici site (Danu et al. 2010), the presumption of using animal dung as fuel cannot be taken into account because dung has not been observed either macroscopically during the field

excavation or microscopically in the micromorphological analyses (Sordoillet et al. 2018). Furthermore, compared to plant resources (wood and/or grasses), dung is a bad fuel that, in addition, introduces into the ash more phosphates and nitrogen, which is probably not required if it gets mixed with salt.

This complementarity between agro-pastoral systems and salt extraction activities is supported by results of palynological analysis (Danu et al. 2010) which highlight the presence of agro-pastoral markers (anthropogenic pollen indicators and non-pollen palynomorphs like coprophilous fungal spores which strongly suggest the pastoral/breeding activity), as well as the presence of forest exploitation indicators (deforestation, erosion) in close connection with salt exploitation.

## Conclusion

The phytoliths analysis from *Hălăbutoaia* - Țolici saltwater spring made it possible to highlight assemblages with anthropogenic origin in which grasses have a major role, excepting few cases in which dicots are well represented. Phytoliths mostly derived from the subfamily Panicoideae are well represented. Their absolute abundance tends to increase along the sequence, to detriment of those massively produced by plants from the Pooideae subfamily. Given the high sedimentation rate, this increase cannot be due to pollution caused by current vegetation. The abundance of BILOBATE phytoliths and glumes phytoliths emphasizes the possible involvement of cultivated Panicoideae, but more evidences are needed to draw a definitive conclusion. Plant phytoliths were introduced into the site via anthropogenic input, but microscopic observation of phytoliths does not allow the determination of the purpose in which plants were brought. Despite the fact that it is not possible to determine whether phytoliths are burnt or not (burned phytoliths have the same aspect as those that are not burned) their abundance in an archaeological context marked by fire-related activities makes it very likely that their presence may be related to fire purpose. It is thus very possible that grasses have played a role in the salt-making pyrotechnology, whether for ignition, combustion or temperature control.

**Acknowledgements** This research was partly founded by the Commission for Foreign Excavations (French Ministry of Foreign Affairs) and the National Centre of Scientific Research (CNRS, UMR 8215 Trajectoires). Special thanks to the History & Archaeology Museum of Piatra Neamț for their continued cooperation. This work was done also within the project “Development of innovation capacity and increasing the impact of excellence research at UAIC”. This project is funded by the Ministry of Research and Innovation within Program 1 – Development of the national RD system, Subprogram 1.2 – Institutional Performance – RDI excellence funding projects, Contract no.34PFE/19.10.2018.

**Funding** Open Access is funded by the Ministry of Research and Innovation within Program 1 – Development of the national RD system,

Subprogram 1.2 – Institutional Performance – RDI excellence funding projects, Contract no.34PFE/19.10.2018.

**Open Access** This article is licensed under a Creative Commons Attribution 4.0 International License, which permits use, sharing, adaptation, distribution and reproduction in any medium or format, as long as you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons licence, and indicate if changes were made. The images or other third party material in this article are included in the article's Creative Commons licence, unless indicated otherwise in a credit line to the material. If material is not included in the article's Creative Commons licence and your intended use is not permitted by statutory regulation or exceeds the permitted use, you will need to obtain permission directly from the copyright holder. To view a copy of this licence, visit <http://creativecommons.org/licenses/by/4.0/>.

## References

- Albert RM, Lavi O, Estroff L, Weiner S, Tsatskin A, Ronen A, Lev-Yadun S (1999) Mode of occupation of Tabun cave, Mt Carmel, Israel during the Mousterian period: a study of the sediments and phytoliths. *J Archaeol Sci* 26(10):1249–1260. <https://doi.org/10.1006/jasc.1999.0355>
- Alexandre A, Meunier JD, Lézine AM, Vincens A, Schwartz D (1997) Phytoliths: indicators of grassland dynamics during the late Holocene in intertropical Africa. *Palaeogeogr Palaeoclimatol Palaeoecol* 136:213–229. [https://doi.org/10.1016/S0031-0182\(97\)00089-8](https://doi.org/10.1016/S0031-0182(97)00089-8)
- Alexianu M, Dumitroaia G, Monah D (1992) Exploatarea surselor de apă sărată din Moldova: o abordare etnoarheologică. *Thraco-Dacica* 13(1–2):159–167
- Alexianu M, Weller O, Brigand R (2007) Izvoarele de apă sărată din Moldova Subcarpatică. *Cercetări etnoarheologice*. Casa Editorială Demiurg Plus, Iași
- An X, Lu H, Chu G (2015) Surface soil phytoliths as vegetation and altitude indicators: a study from the southern Himalaya. *Sci Rep* 5: 15523. <https://doi.org/10.1038/srep15523>
- Bazzaz FA (1996) Linking physiological, population, and community ecology. Cambridge University Press, New York
- Berlin A, Ball TB, Thompson R, Kittleson D, Herbert SC (2003) Ptolemaic Agriculture, “Syrian Wheat”, and Triticum aestivum. *J Archaeol Sci* 30(1):115–121. <https://doi.org/10.1006/jasc.2002.0812>
- Borba-Roschel M, Alexandre A, Drummond Chicarino Varajão AF et al (2006) Phytoliths as indicators of pedogenesis and paleoenvironmental changes in the Brazilian cerrado. *J Geochem Explor* 88(1–3):172–176. <https://doi.org/10.1016/j.gexplo.2005.08.032>
- Bozarth S (1992) Classification of opal phytoliths formed in selected Dicotyledons native to the Great Plains. In: Rapp G, Mulholland S (eds) Phytoliths systematics, emerging issues. Plenum Press, New York, pp 193–214
- Brigand R, Weller O (2013) Neolithic and chalcolithic settlement patterns in Central Moldavia (Romania). *Documenta Praehistorica* 40:195–207. <https://doi.org/10.4312/dp.40.15>
- Brigand R, Weller O (2018) Neo-Eneolithic settlement pattern and salt exploitation in Romanian Moldavia. *J Archaeol Sci: Reports* 17:68–78. <https://doi.org/10.1016/j.jasrep.2017.10.032>
- Brown RH, Brown WV (1975) Photosynthetic characteristics of *Panicum milioides*, a species with reduced photorespiration. *Crop Sci* 15: 681–685
- Cabanes D, Shahack-Gross R (2015) Understanding fossil phytolith preservation: the role of partial dissolution in paleoecology and



- archaeology. *PLoS One* 10(5):e0125532. <https://doi.org/10.1371/journal.pone.0125532>
- Cabanes D, Burjachs F, Expósito I, Rodríguez A, Allué E, Euba I, Vergès JM (2009) Formation processes through archaeobotanical remains: the case of the Bronze Age levels in El Mirador cave, Sierra de Atapuerca, Spain. *Quat Int* 193:160–173. <https://doi.org/10.1016/j.quaint.2007.08.002>
- Cabanes D, Weiner S, Shahack-Gross R (2011) Stability of phytoliths in the archaeological record: a dissolution study of modern and fossil phytoliths. *J Archaeol Sci* 38:2480–2490
- Cărciumaru M, Pleșa M, Mărgărit M (2004) Omul și plantele. Manual de analiză carpologică. Editura Cetatea de Scaun, Târgoviște
- Carter JA, Lian OB (2000) Palaeoenvironmental reconstruction from the last interglacial using phytolith analysis, southeastern North Island, New Zealand. *J Quat Sci* 15(7):733–743. [https://doi.org/10.1002/1099-1417\(200010\)15:7<733::AID-JQS532>3.0.CO;2-J](https://doi.org/10.1002/1099-1417(200010)15:7<733::AID-JQS532>3.0.CO;2-J)
- Ciocărlan V (2000) Flora ilustrată a României, Pteridophyta et Spermatophyta. Editura Ceres, București
- Comșa E (1996) Viața oamenilor din spațiul Carpat-Danubiana-Pontic în mileniiile 7–4 î hr (the life of people in the Carpatho-Danubian-Pontic region in the 7th–4th millennia b.c., in Romanian). Editura didactică și pedagogică, București
- Currie HA, Perry CC (2007) Silica in plants: biological, biochemical and chemical studies. *Ann Bot* 100:1383–1389
- Dal Corso M, Nicosia C, Balista C, Cupitò M, Dalla Longa E, Leonardi G, Kirleis W (2017) Bronze age crop processing evidence in the phytolith assemblages from the ditch and fen around Fondo Paviani, northern Italy. *Veget Hist Archaeobot* 26:5–24
- Dal Corso M, Out WA, Ohlrau R, Hofmann R, Dreibrödt S, Videiko M, Müller J, Kirleis W (2018) Where are the cereals? Contribution of phytolith analysis to the study of subsistence economy at the Trypillia site Maidanetske (ca. 3900–3650 BCE), Central Ukraine. *J Arid Environ* 157:137–148. <https://doi.org/10.1016/j.jaridenv.2018.06.009>
- Danu M, Gauthier E, Weller O (2010) Human impact and vegetation history on salt spring exploitation (Hălăbutoaia-Țolici, Petricani, Neamț, Romania). *International Journal of Conservation Sciences* 1(3):167–173
- Danu M, Diaconu V, Bejenaru L (2016) Chalcolithic agropastoralism traces in the site of Răucești (Neamț County, Romania): phytoliths and animal remains. *International Journal of Conservation Sciences* 7(4):1071–1080
- Danu M, Messenger E, Carozza JM, Carozza L, Bouby L, Philibert S, Anderson P, Burens A, Micu C (2019) Phytolith evidence of cereal processing in the Danube Delta during the chalcolithic period. *Quat Int* 504:128–138. <https://doi.org/10.1016/j.quaint.2018.03.033>
- Delhon C (2008) Potentiel de l'analyse des phytolithes contenus dans les pâtes céramiques et les matériaux de construction. Dégraissants organiques: identifications, nomenclatures et référentiels, S.n. Cahiers des thèmes transversaux ArScAn 7, 2005–2006: 86–93
- Delhon C (2010) Phytolithes et taphonomie, apport de l'expérimentation à la quantification des phytolithes dans les cendres de bois. *Palethnologie* 2:95–107
- Delhon C, Alexandre A, Berger JF, Thiébaud S, Brochier JL, Meunier JD (2003) Phytolith assemblages as a promising tool for reconstructing Mediterranean Holocene vegetation. *Quat Res* 59:48–60. [https://doi.org/10.1016/S0033-5894\(02\)00013-3](https://doi.org/10.1016/S0033-5894(02)00013-3)
- Delhon C, Martin L, Argant J, Thiébaud S (2008) Shepherds and plants in the Alps: multi-proxy archaeobotanical analysis of neolithic dung from “La Grande Rivoire” (Isère, France). *J Archaeol Sci* 35(11): 2937–2952. <https://doi.org/10.1016/j.jas.2008.06.007>
- Downton WJS (1975) The occurrence of C4 photosynthesis among plants. *Photosynthetica* 9:96–105
- Dufraisse A (2011) Analyse anthracologique du site de Hălăbutoaia à Țolici: bilan des études 2007–2011. In Weller O, Exploitations pré et protohistoriques des sources salées de Moldavie : le cas de Țolici (départ. Neamț, Roumanie) 2011 - 4e année : Bilan des études du site de Țolici, prospections et analyses spatiales en Moldavie sous-carpatique (2008–2011). Rapport MAE: 35–48. Unpublished
- Dufraisse A, Sordoillet D, Weller O (2010) The alteration of Neolithic wood charcoal from the salt spring of Poiana Slatinei in Lunca (Neamț, Romania): a natural evolution or consequence of exploitation techniques?. In: Théry-Parisot I, Chabal L, Costamagno S (eds) Taphonomy of burned organic residues and combustion features in archaeological contexts. *Palethnologie* 2, 117–127. <http://blogs.univ-tlse2.fr/palethnologie/en/2010-10-dufraisse-et-alii/>
- Dumitroaia G (1994) Depunerile Neo-Eneolitice de la Lunca și Oglinzi, județul Neamț. *Memoria Antiquitatis* 19:7–82
- Dumitroaia Gh, Munteanu R, Weller O et al (2008) Un nou punct de exploatare a sării în preistorie: Țolici-Hălăbutoaia, jud. Neamț. In: Monah D, Dumitroaia Gh, Garvăn D (eds), Sarea. De la prezent la trecut. Editura C. Mătasă, coll. Bibliotheca Memoriae Antiquitatis 20, Piatra Neamț, pp 203–224
- Elbaum R, Weiner S, Albert RM, Elbaum M (2003) Detection of burning of plant materials in the archaeological record by changes in the refractive indices of siliceous Phytoliths. *J Archaeol Sci* 30:217–226
- Erdei E, Pepo P, Boros N, Toth S, Szabo B (2009) Morphological and biochemical indicators of drought tolerance in sweet sorghum (*Sorghum dochna* L.). *Cereal Res Commun* 37:157–160
- Esteban I, Marean CW, Fisher EC, Karkanas P, Cabanes D, Albert RM (2018) Phytoliths as an indicator of early modern humans plant gathering strategies, fire fuel and site occupation intensity during the Middle Stone Age at pinnacle point 5–6 (south coast, South Africa). *PLoS One* 13(6):e0198558. <https://doi.org/10.1371/journal.pone.0198558>
- Evettt RR, Cuthrell RQ (2017) Testing phytolith analysis approaches to estimate the prehistoric anthropogenic burning regime on the Central California coast. *Quat Int* 434:78–90
- Follak S, Schwarz M, Essl F (2020) First record of *Eriochloa villosa* (Thunb.) Kunth in Austria and notes on its distribution and agricultural impact in Central Europe. *BioInvasions Records* 9(1):8–16. <https://doi.org/10.3391/bir.2020.9.1.02>
- Fredlund G, Tieszen L (1997) Calibrating grass phytolith in climatic terms: application to late Pleistocene assemblages from Kansas and Nebraska. *Palaeogeogr Palaeoclimatol Palaeoecol* 136(1–4): 199–211
- Gao G, Jie D, Wang Y, Liu L, Liu H, Li D, Li N, Shi J, Leng C (2017) Phytolith reference study for identifying vegetation changes in the forest grassland region of Northeast China. *Boreas* 47(2):2–17. <https://doi.org/10.1111/bor.12280>
- Garnier A, Neumann K, Eichhorn B, Lespez L (2012) Phytolith taphonomy in the middle- to late-Holocene fluvial sediments of Ounjougou (Mali, West Africa). *The Holocene* 23:416–431. <https://doi.org/10.1177/0959683612463102>
- Gibson KD, Foin TC, Hill JE (1999) The relative importance of root and shoot competition between water-seeded rice and *Echinochloa phyllopogon*. *Weed Res* 39:181–190. <https://doi.org/10.1046/j.1365-3180.1999.00135.x>
- Harvey EL, Fuller DQ (2005) Investigating crop processing using phytolith analysis: the example of rice and millets. *J Archaeol Sci* 32: 739–752
- Hunt HV, Vander Linden M, Liu X et al (2008) Millets across Eurasia: chronology and context of early records of the genera *Panicum* and *Setaria* from archaeological sites in the Old World. *Veg Hist Archaeobotany* 17(sup. 1):S5–S18. <https://doi.org/10.1007/s00334-008-0187-1>
- ICPT: Neumann K, Albert RM, Ball T et al. (2019) International code for phytolith nomenclature (ICPN) 2.0. *Ann Bot* 124(2):189–199. <https://doi.org/10.1093/aob/mcz064>
- James TK, Rahman A, Dowsett C, Trolove M (2013) Fenoxaprop for control of yellow bristle grass in pasture and its efficacy on other C4

- grasses. *New Zealand Plant Protection* 66:118–123. <https://doi.org/10.30843/nzpp.2013.66.5718>
- Janushevich ZV (1976) Cultivated plants in south-western USSR according to paleobotanical investigation. Editura Știința, Chișinău
- Lebreton V, Thery-Parisot I, Bouby L et al (2017) Archéobotanique et taphonomie. In: Brugal JP (dir.) *Taphonomie S*, Editions des archives contemporaines, collection Sciences archéologiques. Paris, pp 291–328
- Li N, Jie D, Yong G et al (2014) Response of phytoliths in *Phragmites communis* to elevated CO<sub>2</sub> concentration in Songnen grassland, China. *Quat Int* 321:97–104. <https://doi.org/10.1016/j.quaint.2013.10.007>
- Li D, Jie D, Wang Y, Liu L, Liu H, Gao G, Gao Z, Li N, Shi J (2017) Holocene climate reconstruction based on herbaceous phytolith indices from an AMS C-dated peat profile in the Changbai Mountains, Northeast China. *Quat Int* 447:144–157. <https://doi.org/10.1016/j.quaint.2017.01.015>
- Liu LD, Jie DM, Liu HY et al (2013) Change characters of *Phragmites australis* phytolith in Northeast China. *Chinese J Plant Ecol* 37(9): 861–871. <https://doi.org/10.3724/SP.J.1258.2013.00090>
- Lu HY, Wu NQ, Liu KB et al (2007) Phytoliths as quantitative indicators for the reconstruction of past environmental conditions in China II: palaeoenvironmental reconstruction in the loess plateau. *Quaternary Science Review* 26(5–6):759–772. <https://doi.org/10.1016/j.quascirev.2006.10.006>
- Lu H, Zhang J, Wu N, Liu K, Xu D, Li Q (2009) Phytoliths analysis for the discrimination of foxtail millet (*Setaria italica*) and common millet (*Panicum miliaceum*). *PLoS One* 4(2):e4448. <https://doi.org/10.1371/journal.pone.0004448>
- Madella M, Lancelotti C (2012) Taphonomy and phytoliths: a user manual. *Quat Int* 275:76–83. <https://doi.org/10.1016/j.quaint.2011.09.008>
- Madella M, Alexandre A, Ball T (2005) International code for phytolith nomenclature 1.0. *Ann Bot* 96(2):253–260. <https://doi.org/10.1093/aob/mci172>
- Mall TP (2019) Ethno-veterinary potential of underutilised millets from Bahraich: an overview. *Journal of Medical Research and Health Sciences* 2(10):743–783. <https://doi.org/10.15520/jmrhs.v2i10.126>
- Marinescu-Bîlcu S, Cârciuraru M, Muraru A (1981) Contributions to the ecology of pre- and proto-historic habitations at Tîrpești. *Dacia* 25: 7–31
- Messenger E, Lebreton V, Marquer L, Russo-Ermolli E, Orain R, Renault-Miskovsky J, Lordkipanidze D, Despriée J, Peretto C, Arzarello M (2011) Palaeoenvironments of early hominins in temperate and Mediterranean Eurasia: new palaeobotanical data from Palaeolithic key-sites and synchronous natural sequences. *Quat Sci Rev* 30: 1439–1447. <https://doi.org/10.1016/j.quascirev.2010.09.008>
- Metcalfe CR (1960) *Anatomy of monocotyledons. I. Gramineae*. Clarendon Press, Oxford
- Mitchell PL, Sheehy JE (2000) Genetic modification and agriculture. In: Sheehy JE, Mitchell PL, Hardy B (eds) *Redesigning rice photosynthesis to increase yield*. Proceedings of the Workshop on The Quest to Reduce Hunger: Redesigning Rice Photosynthesis, 30 Nov.–3 Dec. 1999, Los Bafios, Philippines. Makati City (Philippines): International Rice Research Institute and (The Netherlands), Elsevier Science BV, Amsterdam, pp 257–268
- Monah D (2008) Arheologia preistorică a sării în România. Scurt istoric. In: Monah D, Dumitroaia Gh, Garvăn D (eds) *Sarea, de la prezent la trecut, Bibliotheca Memoriae Antiquitatis, XX*, Editura „Constantin Matasă”, Piatra-Neamț, pp 13–39
- Murungi ML, McGlynn G, Lejju JB (2017) Alpine grassland palaeoecology of the Virunga volcanoes, East Africa: a new phytolith record from Mt. Muhavura. *Quaternary International* 434(B):102–116. <https://doi.org/10.1016/j.quaint.2016.01.015>
- Novello A, Barboni D (2015) Grass inflorescence phytoliths of useful species and wild cereals from sub-Saharan Africa. *J Archaeol Sci* 59:10–22. <https://doi.org/10.1016/j.jas.2015.03.031>
- Ollendorf AL, Mulholland SC, Rapp G (1988) Phytolith Analysis as a Means of Plant Identification: *Arundo donax* and *Phragmites communis*. *Ann Bot* 61(2):209–214. <https://www.jstor.org/stable/42770279>
- Ollendorf AL (1988) Archaeological implications of a phytolith study at Tel Miqne (Ekron), Israel. *Ann Bot* 61:209–214. <https://doi.org/10.1179/jfa.1987.14.4.453>
- Out WA, Madella M (2016) Morphometric distinction between bilobate phytoliths from *Panicum miliaceum* and *Setaria italica* leaves. *Archaeol Anthropol Sci* 8:505–521
- Parr JF (2006) Effect of fire on phytolith coloration. *Geoarchaeology* 21: 171–185
- Parry DW, Hodson MJ (1982) Silica distribution in the caryopsis and inflorescences bracts of foxtail millet (*Setaria italica* (L.) Beauv.) and its possible significance in carcinogenesis. *Ann Bot* 49:531–540
- Piperno DR (1988) Phytolith analysis: an archaeological and geological perspective. Academic, San Diego
- Piperno DR (1989) The occurrence of phytoliths in the reproductive structures of selected tropical angiosperms and their significance in tropical paleoecology, paleoethnobotany, and systematics. *Rev Palaeobot Palynol* 61:147–173. [https://doi.org/10.1016/0034-6667\(89\)90067-5](https://doi.org/10.1016/0034-6667(89)90067-5)
- Piperno DR (2001) Phytoliths. In: Smol JP, JJB B, Last WM et al (eds) *Tracking environmental change using lake sediments, volume 3: terrestrial, algal, and siliceous indicators*. Kluwer Academic Publishers, Dordrecht, pp 235–251
- Piperno DR (2006) *Phytoliths: a comprehensive guide for archaeologists and paleoecologists*. Alta Mira Press, Lanham. <https://doi.org/10.1017/S0016756807003159>
- Portillo M, Albert RM (2011) Husbandry practices and livestock dung at the Numidian site of Althiburos (El Médéina, kef governorate, northern Tunisia): the Phytolith and Spherulite evidence. *J Archaeol Sci* 38(12):3224–3233. <https://doi.org/10.1016/j.jas.2011.06.027>
- Portillo M, Kadowaki S, Nishiaki Y, Albert MA (2014) Early Neolithic household behavior at tell Seker al-Aheimar (upper Khabur, Syria): a comparison to ethnoarchaeological study of phytoliths and dung spherulites. *J Archaeol Sci* 42:107–118. <https://doi.org/10.1016/j.jas.2013.10.038>
- Regev L, Cabanes D, Homsher R, Kleiman A, Weiner S, Finkelstein I, Shahack-Gross R (2015) Geoarchaeological investigation in a domestic Iron Age quarter, Tel Megiddo, Israel. *Bull Am Sch Orient Res* 374:135–157. <https://doi.org/10.5615/bullamerschoorie.374.0135>
- Rhagavendra AS, Das VSR (1978) The occurrence of C<sub>4</sub> photosynthesis: a supplementary list of C<sub>4</sub> plants reported during late 1974–mid-1977. *Photosynthetica* 12:200–208
- Rosen AM (1992) Preliminary identification of silica skeletons from near eastern archaeological sites: an anatomical approach. In: Rapp G, Mulholland SC (eds) *Phytolith systematics. Advances in archaeological and museum science, vol 1*. Springer, Boston, pp 129–147. [https://doi.org/10.1007/978-1-4899-1155-1\\_7](https://doi.org/10.1007/978-1-4899-1155-1_7)
- Rovner I (1971) Potential of opal phytoliths for use in paleoecological reconstruction. *Quat Res* 1(3):343–359. [https://doi.org/10.1016/0033-5894\(71\)90070-6](https://doi.org/10.1016/0033-5894(71)90070-6)
- Runge F (1999) The opal phytolith inventory of soils in Central Africa quantities, shapes, classification and spectra. *Rev Palaeobot Palynol* 107:23–53. [https://doi.org/10.1016/S0034-6667\(99\)00018-4](https://doi.org/10.1016/S0034-6667(99)00018-4)
- Sârbu I, Ștefan N, Ivănescu L, Mânzu C (2001) *Flora ilustrată a plantelor vasculare din estul României, II*. Editura Universității Alexandru Ioan Cuza Iași
- Shimomae K, Makabe S, Boriboonkaset T, Chin DP, Igawa T, Khan RS, Mii M, Nakamura I (2015) Enhanced efficiency of agrobacterium-

- mediated transformation by sulfamethazine treatment in ravenna grass, *Erianthus ravennae* (L.) Beauv. *Global Advanced. Res J Agric Sci* 4(11):761–768
- Sîrbu C, Oprea A (2011) Plante adventive în flora României. Editura Ion Ionescu de la Brad, Iași
- Sordoillet D, Weller O, Rouge N, Buatier M, Sizun JP (2018) Earliest salt working in the world: from excavation to microscopy at the prehistoric sites of Țolici and Lunca (Romania). *J Archaeol Sci* 89:46–55. <https://doi.org/10.1016/j.jas.2017.11.003>
- Tuba Z, Raschi A, Lanini GM, Nagy Z, Helyes L, Vodnik D, Sanita di Toppi L (2003) Plant response to elevated carbon dioxide. In: Sanita di Toppi L, Pawlik-Skowronska B (eds) *Abiotic stresses in plants*. Publisher Springer, Dordrecht, pp 157–204. <https://doi.org/10.1007/978-94-017-0255-3>
- Twiss PC (1987) Grass-opal phytoliths as climatic indicators of the Great Plains Pleistocene. In: Johnson WC (ed) *Quaternary environments of Kansas*. Guidebook series 5. Kansas Geological Survey, Lawrence, pp 179–188
- Twiss PC (1992) Predicted world distribution of C<sub>3</sub> and C<sub>4</sub> grass phytoliths. In: Rapp G, Mulholland S (eds) *Phytolith systematics emerging issues*. Plenum Press, New York, pp 113–128
- Twiss PC, Suess E, Smith RM (1969) Morphological classification of grass phytoliths. *Soil Sci Soc Am J* 33(1):109–115. <https://doi.org/10.2136/sssaj1969.03615995003300010030x>
- Ueno O, Takeda T (1992) Photosynthesis pathways, ecological characteristics, and the geographical distribution of the Cyperaceae in Japan. *Oecologia* 89:195–203. <https://doi.org/10.1007/BF00317218>
- Voznesenskaya EV, Franceschi VR, Chuong SDX, Edwards GE (2006) Functional characterization of phosphoenolpyruvate carboxylase-type C<sub>4</sub> leaf anatomy: immuno, cytochemical and ultrastructural analyses. *Ann Bot* 98:77–91. <https://doi.org/10.1093/aob/mcl096>
- Waller SS, Lewis JK (1979) Occurrence of C<sub>3</sub> and C<sub>4</sub> photosynthetic pathways in North American grasses. *J Range Manag* 32(1):12–28
- Weller O (2015) Neolithic and chalcolithic settlement patterns in Central Moldavia (Romania). *Documenta Praehistorica* 42:185–196. <https://doi.org/10.4312/dp.42.12>
- Weller O, Brigand R (2017) Recherches systématiques autour des sources salées de Moldavie. Bilan 2011–2013 et synthèse de 10 ans de prospections. *Memoria Antiquitatis* (2016) 31–32:163–270
- Weller O, Dumitroaia Gh (2005) The earliest salt production in the world: an early Neolithic exploitation in Poiana Slatinei-Lunca, Romania. *Antiquity* 79 (306). <http://www.antiquity.ac.uk/projgall/weller/>
- Weller O, Brigand R, Alexianu M (2007) Cercetări sistematice asupra izvoarelor de apă sărată din Moldova. Bilanțul exploatareilor din anii 2004–2007 efectuate în special în județul Neamț. *Memoria Antiquitatis* 24:121–190
- Weller O, Dumitroaia G, Sordoillet D et al (2008) Première exploitation de sel en Europe. Techniques et gestion de l'exploitation de la source salée de Poiana Slatinei à Lunca (Neamț, Roumanie). In: Weller O, Dufraisse A, Pétrequin P (eds) *Sel, eau et forêt. D'hier à aujourd'hui*. Actes de colloque international (Arc-et-Senans, 2006). Cahiers de la MSH Nicolas Ledoux 12 (Homme et Environnement 1). Presses Universitaires Franche-Comté, Besançon, pp 205–230
- Weller O, Brigand R, Dumitroaia G et al (2015) A pinch of salt in the prehistoric eastern Carpathians mountains (Ro.). In: Alexianu M, Curcă RG, Cotiuță V (eds) *Salt effect. From the ethnoarchaeology to the anthropology of salt*. BAR international series 2760. Archaeopress, Oxford, pp 125–133
- Zhang J, Lu H, Wu N et al (2010) Phytolith evidence for rice cultivation and spread in mid-late Neolithic archaeological sites in central North China. *Boreas* 39:592–602. <https://doi.org/10.1111/j.1502-3885.2010.00145.x>

**Publisher's note** Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.