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Siliceous microfossils in the calcareous paleosols from fluvial-eolian deposits of the Angastaco Formation (Oligocene–Miocene), Salta Province, Argentina

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Recibido: 25 de abril 2025 - **Aceptado:** 25 de junio 2025 - **Publicado:** 13 de agosto 2025

Para citar este artículo: Nahuel Espinoza, Alejandro Zucol, Cecilia del Papa, & Ricardo Nestor Melchor (2025). Siliceous microfossils in the calcareous paleosols from fluvial-eolian deposits of the Angastaco Formation (Oligocene–Miocene), Salta Province, Argentina. *Publicación Electrónica de la Asociación Paleontológica Argentina* 25(2): 39–48.

Link a este artículo: http://dx.doi.org/10.5710/PEAPA.25.06.2025.546

ISSN 2469-0228

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ARTÍCULO Año 2025 - 25(2): 39–48

Publicación Electrónica
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SILICEOUS MICROFOSSILS IN THE CALCAREOUS PALEOSOLS FROM FLUVIAL-EOLIAN DEPOSITS OF THE ANGASTACO FORMATION (OLIGOCENE–MIOCENE), SALTA PROVINCE, ARGENTINA

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Abstract. This work reports the first occurrence of siliceous microfossils in the Angastaco Formation (upper Oligocene–Upper Miocene) in the Salta Province, Argentina. Two samples were collected from the Quebrada Escalera section to conduct a prospective analysis for phytoliths in paleosols, aiming to infer paleovegetation and paleoenvironmental conditions. The sedimentary succession comprises ephemeral sheet-like fluvial deposits and fluvial-eolian interaction deposits with calcareous paleosol development on eolian dunes. The microfossil analysis revealed multicellular and unicellular phytoliths, diatom frustules, chrysostomataceae stomatocysts, cutinous epidermal fragments, carbonised microremains, and sponge spicules. The samples showed varied abundances of autochthonous microremains suggesting different hydrological conditions. Sample 2550, with abundant diatoms and stomatocysts, indicates a lentic water environment while sample 2551, rich in sponge spicules, suggests higher energy conditions. The phytolith assemblage, including Poaceae, Cyperaceae, and Arecaceae indicates wet interdune and/or extradune areas.

Key words. Phytoliths. Andean foreland basin. Fluvial-eolian deposits. Micromorphology.

Resumen. MICROFÓSILES SILÍCEOS EN PALEOSUELOS CALCÁREOS DE DEPÓSITOS FLUVIO-EÓLICOS DE LA FORMACIÓN ANGASTACO (OLIGOCENO–MIOCENO), PROVINCIA DE SALTA, ARGENTINA. Este trabajo reporta la primera ocurrencia de microfósiles silíceos en la Formación Angastaco (Oligoceno superior–Mioceno Superior) en la provincia de Salta, Argentina. Se recolectaron dos muestras de la sección Quebrada Escalera para realizar un análisis prospectivo de fitolitos en paleosuelos, con el objetivo de inferir la paleovegetación y las condiciones paleoambientales. La sucesión sedimentaria comprende depósitos fluviales efímeros mantiformes y depósitos de interacción fluvio-eólica con desarrollo de paleosuelos calcáreos sobre dunas eólicas. El análisis de microfósiles reveló fitolitos multicelulares y unicelulares, frústulos de diatomeas, estomatocistes de chrysostomataceas, fragmentos de tejido epidérmico cutinoso, microfósiles carbonosos y espículas de espongiarios. Las muestras mostraron abundancias variadas de microfósiles autóctonos, lo que sugiere diferentes condiciones hidrológicas. La muestra 2550, con abundantes diatomeas y estomatocistes, indica un ambiente acuático léntico mientras que la muestra 2551, con abundantes espículas de espongiarios, sugiere condiciones de mayor energía. La asociación de fitolitos, que incluye Poaceae, Cyperaceae y Arecaceae indica zonas de interduna y/o extraduna húmedas.

Palabras clave. Fitolitos. Cuenca de Antepaís Andina. Depósitos fluvio-eólicos. Micromorfología.

THE OBJECTIVE of this work is to report the first occurrence of siliceous microfossils from the Angastaco Formation (upper Oligocene–Upper Miocene), Salta Province, Argentina. The study of phytoliths in paleosols allows for the determination of paleovegetation and, in turn, the establishment of vegetation patterns according to the paleoenvironment (Dormaar & Lutwick, 1969; Kelly *et al.*, 1998; Raigemborn *et al.*, 2018; Bellosi *et al.*, 2021). Phytoliths have proven to be reliable indicators of the proportion of grasses versus trees in many ecosystems, as they can be preserved in well-oxidized sediments that often lack pollen and macrofossils (Strömberg, 2011). In eolian landscapes, periods of soil formation are often brief, and burial by clastic deposits occurs rapidly. As a result, the ''inheritance'' of phytoliths in these environments may be less representative of the main environmental conditions, as the stratigraphic significance of phytoliths depends on the duration of soil development prior to burial (Boyd, 2005). However, phytoliths represent valuable indicators that must be contrasted with physical/chemical sedimentary features.

Phytolith studies in the Paleogene–Neogene of Argentina have mostly focused on the Patagonia region (*e.g.*, Raigemborn *et al.*, 2018; Bellosi *et al.*, 2021). The record of phytoliths during the Oligocene–Miocene in northwestern Argentina is very scarce. Cotton *et al.* (2014) conducted phytolith studies in the Andalhuala Formation (Upper Miocene–Pliocene), Catamarca Province. The objective of the mentioned study was to determine the origin of the expansion of C4 grasslands. Similarly, Ghosh *et al.* (2020) conducted phytolith studies, in the Jesús María, Guanaco and Piquete formations (Middle Miocene–Lower Pliocene) and Palo Pintado Formation (Upper Miocene–Lower Pliocene) in the Salta Province, with the same purpose.

GEOLOGICAL SETTING

The study area is located within the Eastern Cordillera geologic province (Turner & Mon, 1979) in the Tonco Valley (Fig. 1.1, 2). This valley consists of north-south depressions separated by mountain ranges with a basement core, bordered by reverse faults that predominantly verge to the west (Hongn & Seggiaro, 2001; Carrera & Muñoz, 2008). The basement of the area is composed by the Puncoviscana Formation, characterized by Precambrian-Cambrian low-grade metamorphic rocks, overlain by Mesozoic to Cenozoic, mostly non-marine clastic successions (Hongn & Seggiaro, 2001). Lower Cretaceous to middle Eocene rocks are included in the Salta Group (Fig. 1.2) that represents the filling of an intra-continental rift basin (Salfity & Marquillas, 1994; del Papa, 1999, 2006; Sabino, 2004; Hongn *et al.*, 2011). The middle Eocene to Pleistocene sedimentary succession corresponding to the Payogastilla Group (Díaz & Malizzia, 1983) represents the Andean foreland sedimentation (Starck & Vergani, 1996). The Payogastilla Group is more than 6,000 m thick and consists of coarsening upward alluvial sedimentary succession that includes four lithostratigraphic units (Díaz & Malizzia, 1983). From the base to top these units are: Quebrada de los Colorados (middle Eocene–upper Oligocene), Angastaco (upper Oligocene–Upper Miocene), Palo Pintado (Upper Miocene–Lower Pliocene) and San Felipe (Lower Pliocene–Lower Pleistocene) (Fig. 1.2).

The Angastaco Formation (del Papa *et al.*, 2013a) is approximately 3,500 m thick and is composed of two members. The lower Tin Tin Member is ~450–600 m thick and is composed of gravelly and sandy ephemeral unconfined fluvial channel deposits, gravelly fluvial channels deposits with perennial flow, fluvial-eolian interaction deposits, and eolian deposits composed of eolian dunes and dry and wet interdunes. The upper Las Flechas Member is composed of ~3,780 m of fluvial and alluvial conglomerate and sandstone. The age of the Angastaco Formation spans from the late Oligocene ~28 Ma (based on stratigraphic correlation according to Aramayo *et al.*, 2017) to the Late Miocene (based on two volcanic tuffs dated at 8.8 ± 0.5 Ma U/Pb in zircon and 8.13 ± 0.05 Ma U/Pb in zircon; after Carrapa *et al.*, 2012; Payrola *et al.*, 2020). The three radiometric ages available for the lower aeolian Tin Tin Member are Early Miocene (Aquitanian-Burdigalian): 21.4 ± 0.7 Ma (U/Pb, detrital zircons, Carrapa *et al.*, 2012), 21.0 ± 0.8 Ma (U/Pb, zircons in a tuff, del Papa *et al.*, 2013b), and 17.46 ± 0.08 Ma (U/Pb, zircons in a tuff, Payrola *et al.*, 2020). These ages could potentially correlate with the Oligocene–Miocene Eolian Belt (OMEB) identified in western and northwestern Argentina (Ciccioli *et al.*, 2023).

MATERIAL AND METHODS

This is a preliminary study of two paleosol samples. Siliceous microfossils samples were collected from the lower part of the Tin Tin Member exposed in the Quebrada Escalera section (Fig. 1.3), which was measured using standard sedimentological techniques, a Garmin eTrex® 10 GPS device, and a Brunton Geo 5010 compass. Lithofacies were defined based on lithology, grain size, color (GSA rock Color Chart), sorting, and sedimentary structures. Two samples, each weighing approximately 150 g, were taken for siliceous microfossils study at 55 m and 65 m of the section (Fig. 1.3). These samples were incorporated into the sedimentary collection of the Paleobotany Laboratory at

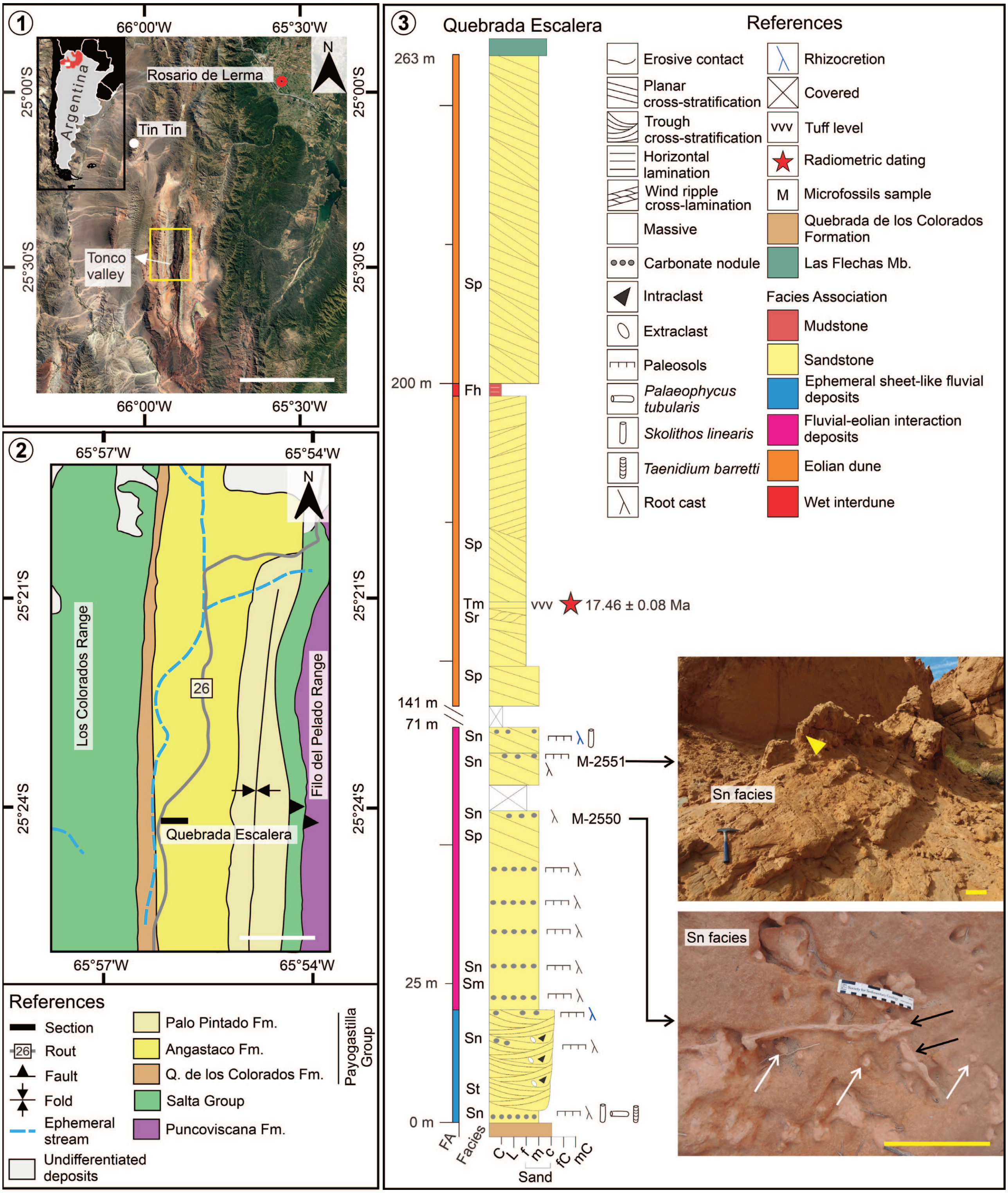

Figure 1. 1, Google Earth™ satellite image showing the study area location the Salta Province, Argentina. The yellow rectangle indicates the location of figure 1.2. Scale bar= 30 km. **2,** Geological map of study area and location of analyzed section (modified from Salfity & Monaldi, 2006). Scale bar= 2km. **3,** Sedimentary log of the Tin Tin Member including a radiometric dating taken from Payrola *et al.* (2020). The photographies show the location of samples for microfossil analysis. Scale bar= 20 cm. Black arrows: horizontal root cast; white arrows: vertical root cast; yellow triangle: bottom of the bed. See facies description in Table 1.

CICYTTP-Diamante, with the following identification numbers: ID 5237 (microscope preparation 2550) and ID 5238 (microscope preparation 2551). In the laboratory, the samples were prepared following the methodology proposed by Zucol *et al.* (2010a).

The methodology for concentrating microremains followed conventional guidelines for biomineral concentration in clastic materials, with particular caution to preserve and concentrate elements formed by resistant organic matter, such as cutin and sporopollenin. The process involved several chemical treatments: soluble salts were removed by repeated washing with distilled water; carbonates were eliminated by treating the samples with 10% diluted hydrochloric acid; disaggregation was achieved using sodium hexametaphosphate; organic matter was removed using hydrogen peroxide; and finally, the pH of the remaining material was neutralized to obtain a "clean sample". These steps were designed to eliminate compounds that could cause agglomeration of micropaleontological elements. Grain size separation was performed on two fractions: a fine fraction (less than 5 μm) by siphoning, and a coarse fraction (greater than 250 μm) by sieving. The fraction between 5 and 250 μm, ranging from fine silt to fine sand, was further used for densitometry separation into two grain size fractions: 5–53 μm and 53–250 μm. Densitometry separation was carried out using an aqueous solution of sodium polytungstate at 2.3 g/cm^3. The concentrates were mounted on a cover slide and affixed with Canada Balsam for fixed microscope preparations and with Cedarwood Oil for liquid microscope preparations. Microscopic observations were conducted using a Nikon E200 petrographic microscope at the Paleobotany Laboratory (CICYTTP-Diamante). Photographs were taken using a Nikon Coolpix 990 digital camera.

The description of phytoliths was based on a phytolith morphotype classification, with names and acronyms detailed in Neumann *et al.* (2019). The classification draws on previous morphological descriptors follows Madella *et al.* (2005) and the guidelines proposed by Twiss *et al.* (1969), Bertoldi de Pomar (1971), Twiss (1992), Kondo *et al.* (1994), and Zucol (1996). These sources were also used to determine the botanical affinities of diagnostic morphotypes. The acronyms used align with the morphotype definitions provided by Zucol *et al.* (2010b) and their group names according to Raigemborn *et al.* (2018).

RESULTS AND DISCUSSION

Depositional environment

The Tin Tin Member features a lower ephemeral sheet-like fluvial deposits, middle fluvial-eolian interaction deposits, and, in the upper part of the section, eolian deposits comparable to an erg composed of eolian dunes and dry and wet interdunes (Fig. 1.3) (see Espinoza, 2024).

Four facies and two facies associations were observed in the lower section of the Tin Tin Member (Table 1). The latter included ephemeral sheet-like fluvial deposits (FA1) and fluvial-eolian interaction deposits (FA2), which are analyzed below.

TABLE 1 – Summary of the sedimentary facies in the lower section of the Tin Tin Member with their description and interpretation.

Facies	Description	Interpretation
Sp	Well-sorted sandstone with large-scale planar cross-stratification	Subaerial migration of dunes with straight to low-sinuosity crests
Sm	Massive sandstone	Possible hyperconcentrated flow events
St	Sandstone with trough cross-stratification	Subaqueous migration of sinuous-crested and linguoid dunes
Sn	Sandstone with carbonate nodules	Soil development with carbonate precipitation
Fh	Mudstone with horizontal lamination	Subaqueous setting of mud
Tm	Massive tuff	Volcanic ash fall

Ephemeral sheet-like fluvial deposits (FA1): This facies association was observed in the lower part of the section, with a thickness of 20 m, composed of St, Sm and Sn facies (Fig. 1.3). The St facies is stacked in a package of approximately 10 m thick, consisting of tabular sand bodies composed of coarse-grained sandstone with a greyish orange color (10YR 7/4) and trough cross-stratification. The sets exhibit a fining-upward tendency, starting with a level of fine-grained gravel and ending with coarse-grained sandstone. The Sm facies consists of poorly-sorted, medium- to coarse-grained sandstone, a greyish orange color (10YR 7/4) and massive appearance. The Sn facies consists of coarse-grained sandstone with silt-clay matrix and colors ranging from moderate orange-pink (10R 7/4) to moderate reddish orange (10R 6/6) and contains carbonate nodules. The nodules are non-coalescent, averaging 0.96 cm in size (range= 0.42-2.58 cm; n= 27) and show vertical size gradation, with the largest ones located at the top of the beds. The parent material of the Sn facies is massive or trough cross-stratified sandstone facies similar to underlying beds. It also presents trace fossils including root casts, rhizocretions, *Skolithos linearis*, *Taenidium barretti*, and *Palaeophycus tubularis*.

This facies association is interpreted as ephemeral sheet-like fluvial deposits with high-sinuosity and linguoid dunes based on poor sorting of sandstones, tabular geometry of the sand bodies, cross-stratification and fining-upward tendency (Bridge, 1993; Miall, 1996; Billi, 2007). The association of carbonate nodules with root casts, and rhizocretions indicate development of calcareous soils. The paleosols document either the ephemeral nature of the fluvial deposits or/and their development in interfluvial areas (Deluca & Eriksson, 1989).

Fluvial-eolian interaction deposits (FA2): This facies association was observed between 20 m and 71 m of the logged section, and it is composed of the Sp, Sm and Sn facies (Fig. 1.3). The Sp facies consists of well-sorted, medium-grained sandstone with a light brown color (5YR 6/4) and rounded grains. It features large-scale planar cross-stratification, with foreset slope ranging from 17° to 40°. The Sm facies consists of poorly-sorted, medium- to coarse-grained sandstone with silt-clay matrix, a greyish orange color (10YR 7/4) and massive appearance. The Sn facies is characterized by medium-grained sandstone with colors ranging from moderate orange-pink (10R 7/4) to moderate reddish orange (10R 6/6) and contains carbonate nodules. The parent material of the Sn facies is massive or planar cross-stratified sandstone facies. The carbonate nodules are non-coalescent, averaging 4.2 cm in size (range= 1-9.8 cm; n= 17) and show vertical size gradation, with the largest nodules at the top of the layers. Thin sections reveal micrite (16.2% by volume) and patches of sparite (15.2% by volume). These levels also contain abundant root casts, rhizocretions, and *Skolithos linearis*. Two samples were collected from this facies (Fig. 1.3) for a preliminary analysis of siliceous microremains.

This facies association is interpreted as fluvial-eolian interaction deposits developed in low relief areas, where fluvial and eolian processes inter-fingered. The precipitation of carbonate nodules is attributed to pedogenic processes, based on their association with rhizoliths (Klappa, 1978, 1980). Paleosols development is compatible with an incipient stage of calcic soils (stage II to III of Machette, 1985) due to the development of sparse carbonate nodules and carbonate content. The Sp facies corresponds to subaerial migration of dunes with straight to low-sinuosity crests. The massive sandstones (Sm facies) suggest hyper-concentrated flows (Scherer & Lavina, 2005) from sporadic flooding after high-intensity rainfall (Mountney & Jagger, 2004). The Sn facies is the result of soil-forming processes on a parent material composed of sandstones of eolian (Sp facies) and fluvial origin (Sm facies). In both cases, indicate low rate of deposition and the stabilization of the surface.

Microfossils

The analysed samples (2550 and 2551) were taken from the Sn facies with an eolian parent material (Sp facies). Both samples contain microremains including small cutinous epidermal tissue fragments (Fig. 2.1), diatom frustules (Fig. 2.2), carbonised microremains (Fig. 2.3), sponge spicules (Fig. 2.4), chrysostomataceae stomatocysts (Fig. 3.1), multicellular (Fig. 2.1) and unicellular phytoliths (Fig. 3.2–9).

Most of the phytoliths are unicellular, with a notable presence of bulliform elements, exhibiting polyhedral and fan-shaped forms (Fig. 3.7–8). Among the macro-

Figure 2. Multicellular phytoliths and non-phytolith microremains of the analyzed samples. **1,** Graminoid and dicot epidermal fragment and arecoid vascular conduction elements. **2,** Diatoms. **3,** Microcharcoal particles. **4,** Fragmented and complete spicules of siliceous sponges. Scale bar in 4= 20 µm is valid for all the panels.

phytoliths, elongated elements with various contours (smooth, spiny and wavy) and shapes (prismatic to irregular) were observed. Worn and/or fragmented phytoliths were rare, typically showing superficial wear. For grass silica short cell phytoliths (GSSCP) bilobate, truncated cones, oblongs, boat-shaped, and small prismatic elements with smooth or wavy contours were found (Fig. 3.2).

Sample 2550 was characterized by a high abundance of well-preserved diatoms to a lesser extent, broken frustules. Chrysostomataceae stomatocysts were also common and

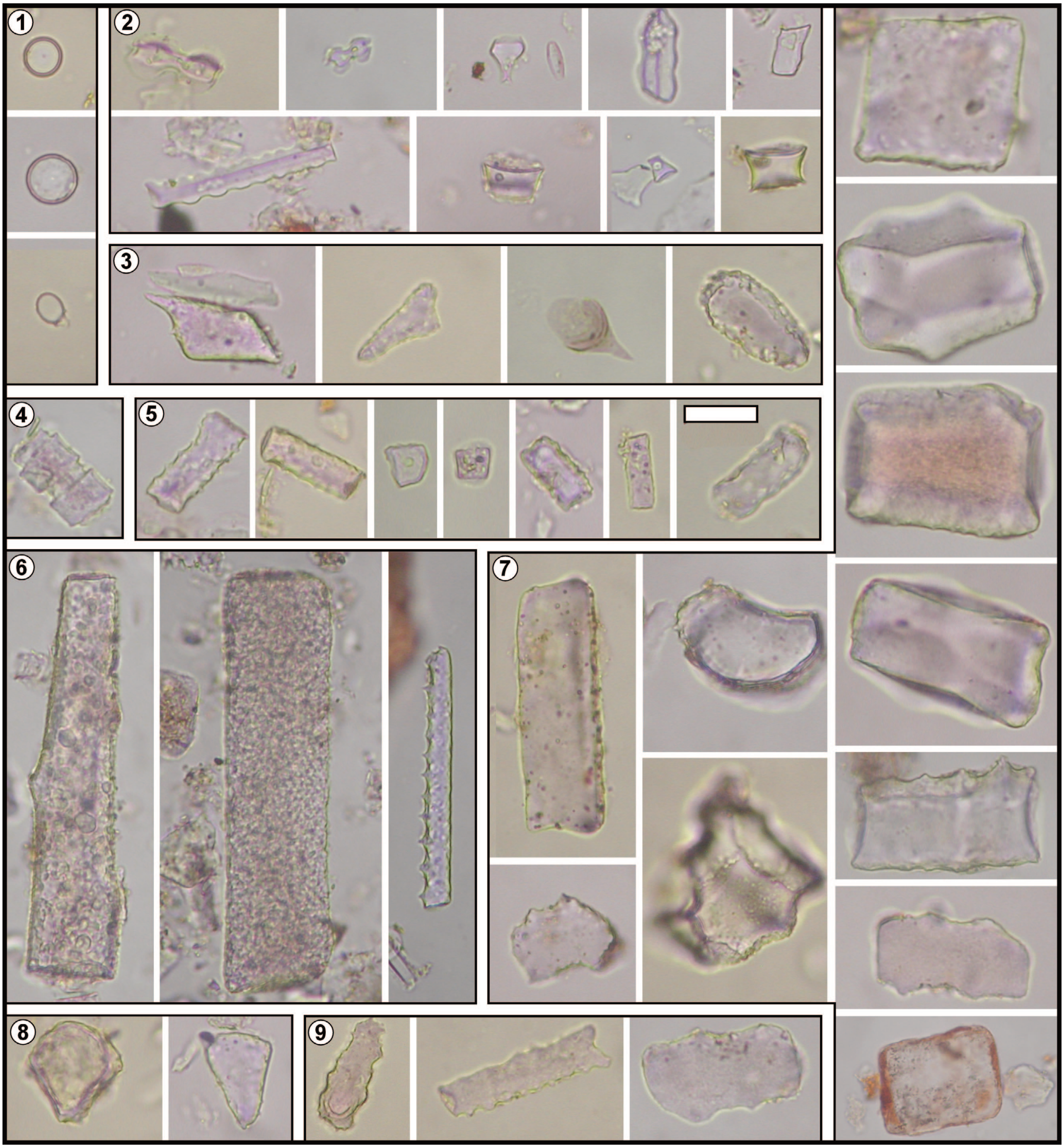

Figure 3. Different phytolith and stomatocysts observed in the Tin Tin Member of the Angastaco Formation. **1,** Chrysostomataceae stomatocyts. **2,** Grass silica short cell phytoliths (GSSCP). **3,** Point-shaped phytoliths. **4,** Tracheary annulate phytolith. **5,** Short prismatic phytoliths. **6,** Different graminoid and cyperoid types of elongated phytoliths. **7,** Cuneiform Bulliform phytoliths. **8,** Fan-shaped elements. **9,** Epidermal elongate graminoids phytoliths. Scale bar in 5= 20 μm is valid for all the panels.

exhibited both smooth and spiny surfaces (Fig. 3.1). These microfossils suggest a lentic water environment with periods of water stress, which is not recorded in the measured section.

In contrast, sample 2551 contained abundant sponge spicules, mostly fragmented smooth macroscleras, although some complete elements were also observed. The high abundance and size of these sponge remains indicate transport processes under higher-energy water conditions. Additionally, cutinous tissue fragments were found exclusively in this sample.

Despite these differences, both samples shared several micropaleontological features. Microcharcoals, generally mimicking cellular forms, were present in both samples (Fig. 2.3), and each contained a substantial amount of microremains, mostly autochthonous and exhibiting minimal wear. The phytolith content appeared similar in both abundance and diversity across the two samples.

CONCLUDING REMARKS

From the facies analysis it can be interpreted that the lower portion of the section is composed of sheet-like deposits from ephemeral streams with development of calcisols. The fluvial deposits corresponding to mid-distal positions of alluvial fans (distal 'braided' sheetflood) (Miall, 1996), based on correlations with other sections in the Cerro Tin Tin area (see Espinoza, 2024). The middle and upper portions of the studied section consist of eolian dunes and ephemeral fluvial deposits, both exhibiting the development of calcisols. The sampled calcisols developed on eolian dunes contain phytoliths. The low percentage of small phytoliths and the lack of a detailed count prevent a precise estimation of the climatic and moisture conditions for the grasses. However, the presence of Poaceae, Cyperaceae and Arecaceae suggests that the microremains originated from humid interdune or extradune areas, which were reworked and deposited in eolian dunes (Sp facies). Humid interdune or extradune facies associated with lentic water environment and periods of water stress were not recorded in the measured section. Thus, information provided by phytoliths, diatoms and sponge spicules, support the interaction of eolian and fluvial processes and give additional information to that offered by the facies analysis. A detailed and systematic study of phytoliths can offer insights into the paleobotanical and paleoclimatic conditions of this sedimentary succession.

ACKNOWLEDGEMENTS

This work is part of the PhD thesis of NE at the Universidad Nacional de Córdoba (Argentina). Funded by projects PICT2019-114 from the Agencia Nacional de Promoción Científica y Tecnológica of Argentina, PIP2021-2023-146 from CONICET to RNM, PIP 666 from CONICET to CdP and IAS Postgraduate Research Grants (2019) to NE. We thank the editor, and two anonymous reviewers for their constructive reviews that improved the manuscript.

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doi: 10.5710/PEAPA.25.06.2025.546

Recibido: 25 de abril 2025

Aceptado: 25 de junio 2025

Publicado: 13 de agosto 2025