Supporting Files

Phytolith collection and processing

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Sampling and collection methods

Samples for phytolith analysis were collected vertically from artificial strata at contiguous 5 cm intervals. When obvious changes in the natural stratigraphy crosscut the arbitrary 5 cm sampling unit, the sample level was adjusted to avoid mixing distinct sediments and/or soils. Samples were collected beginning at the bottom of the profile and moving upward to avoid contamination of samples by sediments falling from above. For each sample, approximately 60 g of sediment was loosened using a metal trowel and collected in a plastic dustpan. The sediment was then transferred into a clean plastic Ziploc bag that was labeled with the date, transect, column, and level. All tools were cleaned with distilled water between samples. Thirty-six of these sediment and soil samples were analyzed.

Extraction Procedure

First, each sample was crushed lightly with a mortar and pestle, and then passed though a 16-mesh geological sieve to extract rocks and large soil particles. The pH level of the sample was then measured and recorded.

Carbonate removal. Each sample was washed in a 10% solution of hydrochloric acid (HCl) and placed in a hot water bath. Additional 10% HCl was added periodically until the sample ceased to react. After samples were centrifuged and the supernatant decanted, 50 ml of strong acid (equal parts HCl and nitric acid (HNO_3)) were added. The sample was stirred and replaced in the hot water bath until all reaction ceased. Distilled water was added to dilute the acid and the sample was allowed to cool. After cooling the sample was centrifuged for 2 minutes at 2,000 RPM and the supernatant decanted (centrifuging and decanting was always carried out for this duration and speed unless otherwise noted). This was followed by two rinses with distilled water.

Organic material removal. Bleach was added to the sample and the sample was then placed in a hot water bath for five minutes. The time limit was closely observed for this step, as prolonged exposure to bleach etches and can potentially destroy opal silica. The sample was then rinsed with distilled water twice. Next, technical grade hydrogen peroxide (H_2O_2, 27%) was added and the samples returned to the hot water bath. Additional H2O2 was periodically added to the samples until all reaction ceased. After diluting the remaining H_2O_2 with distilled water, centrifuging and decanting the supernatant, the samples were then rinsed once more with distilled water.

Mechanical separation of phytoliths from sediment. To disperse the aggregate of particles in the sample, the sample was transferred to a 500 ml plastic centrifuge bottle and a 0.1% solution of sodium EDTA (Na_2H_2EDTA) was added. The sample was then placed

overnight in a mechanized reciprocating shaker at low speed. The sample was then passed through a 60-mesh geological screen using a jet of distilled water. The > 250 micron fraction that remained in the screen (i.e., those particles larger than the largest known phytoliths) was discarded and the < 250 micron fraction retained. The screened sample was subject to centrifuge sedimentation, which removes the clay particles from the sample (see Lentfer and Boyd (1999) for a published description). This procedure involves adding a warm 1% solution of dishwashing detergent in distilled water (the detergent deflocculates the aggregated clay particles), stirring well and then centrifuging the sample for 5 minutes at 3,000 RPM and decanting the sample. This process was repeated for each sample until the supernatant was clear of clay.

Heavy liquid flotation. A solution of zinc iodide (ZnI_2) calibrated to a specific gravity of 2.3 was added to each sample, which was then well-stirred and then centrifuged for 5 minutes at 3,000 RPM. The resulting supernatant contained all material with a specific gravity of < 2.3 (i.e., phytoliths), while the heavier sediment collected at the bottom of the centrifuge tube. This sediment was set aside. The heavy liquid supernatant with phytoliths was poured into a sterile test tube and distilled water added to reduce the specific gravity of the heavy liquid to > 1.5. This step allowed the phytoliths to settle at the bottom of the tube after 10 minutes of centrifuging at 3,000 RPM. The supernatant (now free of phytoliths) was then discarded. These steps were repeated with the remaining sediment until no silica residue, visible as a film on the surface of the heavy liquid, was present in the sample. The resulting concentration of silica was washed in distilled water twice, each time being centrifuged for 10 minutes at 3,000 RPM. It was then dried in an oven for approximately one day, weighed and stored in glass vials. The remaining sediment was washed twice in distilled water and archived at the University of Missouri.

Slide Mounting. A clean glass slide was labeled with the project name and the laboratory assigned identification number. Next a metal spatula was sterilized with rubbing alcohol and used to transfer a small amount of Canada Balsam to the slide. The mounting medium was then sprinkled with 0.001 g of sample extract. A sterile metal probe was used to thoroughly mix the Canada Balsam and extract. A clean glass slide cover was then pressed over the Balsam and the extract mixture until the material was evenly distributed under the cover slip. The finished slide was dried on an electric slide warmer for a day to eliminate air bubbles and to allow the mounting medium to solidify slightly.

The identification of phytoliths was based on the University of Missouri's comparative collection and the typology developed by Dr. Pearsall and her students (publicly available at: http://www.missouri.edu/~phyto) as well as published sources.

Work Cited

Lentfer CJ, Boyd WE. 1999. An Assessment of Techniques for the Deflocculation and Removal of Clays from Sediments Used in Phytolith Analysis. *Journal of Archaeological Science* 26: 31-44

Archaeological survey methods

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Methods

The systematically covered survey area includes 79 ha in seven non-contiguous survey blocks. Two main blocks, one on either bank of the river, constituted the main systematic survey area. We covered these areas using a crew of two or three people spaced at 20 m. We also recorded other sites that were outside the survey blocks but still adjacent to the floodplain. We obtained the locations of these sites by asking our field assistant to direct us to the largest known sites in the area.

To determine whether a find should be considered a site, the crew looked for artifacts and any evidence of surface modification. A site was defined as any location where artifact density exceeded nine items within a 3 m radius of a findspot or had surface features. Site size was determined by the extent of artifact scatter at the above-defined density criterion. Mounded architecture and wall alignments were also recorded, and they normally fell within the artifact distribution. We also inspected the site and surrounding area for terraces, depressions, anomalous vegetation, and other surface modifications. We did not make surface collections, but noted the presence of any diagnostic artifacts, usually ceramics.