

Supplementary Material for “An interplay of dryland and wetland: millet and rice cultivation at the Peiligang site (8000 – 7600 BP) in the middle Yellow River Valley, China”

Soil phytolith processing protocol

Jiajing Wang

Removing carbonates [fume hood]

1. Break up sediment clumps with pestle and mortar.
2. Weigh 1 g of sediment into a 15 ml centrifuge tube.
3. Add 10% HCL acid to the tubes until the samples stop fizzing. Do not seal the tubes to allow the release of gas (optional: place the tubes in a hot water bath at ~50 °C).
4. Add distilled water to 15 ml, vortex gently, then centrifuge for 5 min at 1500 rpm, and discard the supernatant. Repeat this step twice (repeat one more than if the supernatant is still yellowish)

Deflocculation

1. Add 5% Calgon solution (sodium hexametaphosphate) to 15 ml, place in a shaker overnight or at least 8 h.
2. Vortex, then centrifuge for 5 min at 1500 rpm and discard the supernatant.

Removing clays

1. Fill the tubes with warm water and vortex. Centrifuge 3 mins at 2000 rpm. Decant. Repeat the warm water rinses until the supernatant is clear—clays are removed.

Removing organic matter [fume hood]

1. Add 30% H₂O₂, place the tubes in a hot water bath at ~50 °C until the reaction is over (normally a few hours). Do not seal the tube to allow the release of gas.
2. Add distilled water to 15 ml, vortex gently, then centrifuge for 5 min at 1500 rpm and discard the supernatant. Repeat this step twice.

Removing heavy matter

1. Add sodium polytungstate (SPT) solution of specific gravity 2.34 to 4 ml, vortex and centrifuge for 10 at 1000 rpm.
2. Pipette supernatant into a new set of 15 centrifuge tubes.
3. To rinse the SPT, add distilled water to 15 ml, vortex, then centrifuge for 5 min at 1500 rpm.
4. Keep pellet at bottom and discard supernatant for SPT recycling.
5. Repeat steps 3 and 4 twice.
6. The sample is ready to be mounted or stored.