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TRANSMISSION ELECTRON MICROSCOPY OF METALLIC NANOPARTICLES EXTRACTED FROM GEOMATERIALS; PERSISTENCE AND GENERATION DURING CHEMICAL DIGESTION

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Geoscientists frequently use a range of reactive solutions to separate different material phases from a specimen. Reactive solutions typically include acids of increasing aggressiveness along with bases, oxidants, and non-aqueous solutions each to target particular components. In this study, we isolated and characterized nanoparticles found in Bull Creek, OK using multi-step chemical and physical extractions. Selective extractions by means of hydrochloric acid and acidic dichromate were used to separate nanoparticles from soil samples where nanodiamonds have been previously identified [1,2]. Identifying nanodiamonds from geomaterials presents many challenges, including possible misidentification via Transmission Electron Microscopy (TEM) due to similarities with copper nanoparticles. For example, background signal contamination when using X-ray analysis (EDXA) with copper TEM grids [1], and d-spacings that differ by less than 1% make electron diffraction (SAED) an unreliable tool to unambiguously characterize nanodiamonds [3]. Using TEM and wet chemical analysis, we examined the potential for the destruction, production, and perseverance of metallic nanoparticles during a soil digestion intended to isolate diamond nanoparticles. We found that copper nanoparticles persisted throughout hydrochloric acid and acidic dichromate digestion. Furthermore, a range of common digestion solutions created metal-bearing nanoparticles and/or other precipitates when drop-cast onto TEM grids with the excess solution immediately wicked away by filter paper. We found that gold grids produced an abundance of nanoparticles when exposed to an acidic dichromate digestion matrix; these gold nanoparticles were highly beam-sensitive within the TEM. In comparison, molybdenum grids were destroyed by the matrix; nickel grids appeared unchanged without nanoparticle formation. While metallic nanoparticles may persist through soil digestion steps, a range of aqueous-based solutions have the potential to generate artifact nanoparticles on grids (particularly Au and Cu) within short reaction times. This demonstrates complex interactions between metallic TEM grids and aqueous solutions commonly used for geomaterial digestion and suggests that extreme care must be taken to perform control experiments between solution matrices and grid materials when analyzing nanoparticulate materials.

[1] Bement et al. (2014) *Proc Natl Acad Sci USA* 111, 1726-1731.

[2] Kennett et al. (2009) *Science* 323, 94.

[3] Daulton et al. (2017) *Journal of Quaternary Science* 32, 7-34.