

120 years of nano-silver history: implications for policy makers

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Supporting Information

Details on TEM analysis of water filters

Details on TEM analysis of Algaedyn

TEM analysis of water filters

Various silver impregnated carbon water filter products were sourced from on-line vendors. Products from Zodiac Pool Care Inc. were randomly selected for TEM analysis and were considered to be representative of many impregnated water filter type products registered by EPA for many years. In this study Zodiac Nature2 G (EPA# 67712-1) cartridges were examined using TEM. Sample preparation proceeded with manual disassembly of the cartridge units followed by isolation of black colored spherical carbon pellets. A small quantity of pellets (ca. 10) were randomly selected and manually crushed to a uniform powder consistency using a ceramic mortar and pestle.

For preparation of the TEM samples, small portions (ca. 10 mg) of the as-prepared crushed powder were removed using a spatula and dispersed in a glass vial containing 5mL of ethanol. The glass vial was placed in an ultrasonic bath unit (Biorblock Scientific, TP 690A) for 2 min in order to achieve a visually homogeneous and dilute suspension. Using a pipette a single droplet of the sample suspension was transferred to a TEM grid (SPI, Formvar - C coated Cu grids, 200 mesh) and the ethanol allowed to evaporate. Analysis was performed on a TEM (Tecnai, F30ST, FEI) operated at an acceleration voltage of 300 kV. The microscope was operated in the scanning mode and the Ag-NP were localized using a HAADF (High-Angle Annular Dark Field) detector and analyzed with an EDX (Energy Dispersive X-Ray) system (EDAX).

TEM analysis of Algaedyn

The Algaecide Algaedyn® from Pool Products Packaging Corp (EPA registration #68161-1) was used for analysis. Sample preparation for particle sizing involved suspending the aqueous solution in ultra filtered deionized water and dispersing the silver particles using ultrasonic agitation. A carbon substrate supported by a TEM grid was then dipped in the solution and the sample was dried on a hot plate for 5 minutes. The sample was analyzed using a Hitachi S-5500 high resolution field emission SEM (FESEM) with scanning transmission electron microscopy (STEM) capabilities. Particles were measured using an imaging analysis program with automated particle sizing capabilities. A total of 3531 particles were counted and measured from 23 randomly selected areas on the substrate using the bright field transmission electron mode. Bright field STEM images were acquired and measured for particles greater than 15 nm in equivalent circular diameter at a magnification of 50,000x, whereas particles from 2 to 15 nm were measured from images acquired at a magnification of 350,000x. An analysis of this nature requires that the particle counts obtained at each magnification be adjusted to accurately account for the different areas examined at each magnification. The size bins are based on the equivalent circular diameter. The equivalent circular diameter is calculated for each particle based on the projected area.

The EDS analysis revealed that the particles were comprised of silver with lesser amounts of aluminum. The composition of the material was consistent among particles for each size fraction.