

## BACKGROUND

EVQ-218 is a high energy nanoparticle manufactured using a patented compound laser ablation process. The rapid, light driven process allows particle formation without typical chemical and biological syntheses. The resulting nanoparticles are stable, due to geometric effect, in HPLC grade water, without added surfactant or capping agents. In depth nanoparticle characterization studies show EVQ-218 is comparable to NIST standards with respect to uniform sphere morphology and the lack of ion emission. Further investigation using STEM-EELS reveals EVQ-218 possesses a bare silver surface, while the "bare" NIST standard has a citrate coating.

## GOALS

Engineered nanomaterial behavior can be a result of group particle properties, individual particle properties, or emitted ion properties. When characterizing a new nanoparticle, it is important to investigate it alongside ideal (NIST) materials, and reaction products (emission-based species i.e. ions, ROS, agglomerates, etc.). The widespread use of nanoparticles, particularly metallic, is leading to a reevaluation of all things nano.

In 2016, the U.S. Army Engineer Research and Development Center (ERDC) issued SOP-F-1 Nanomaterial Dispersion/Dissolution Characterization. This method outlines kinetic dissolution of the nanoparticle of interest, in DI water, Moderately Hard Water (MHW), and mineral rich AAP media. Determination of ionic content relies on ultracentrifugation as most nanoparticles will sediment within 1 hour at 100,000 x g force. Results show EVQ-218 does not follow this rule and prompted additional TEM studies.

## METHODS

EVQ-218 and NIST nanoComposix nanoparticles (10 nm, 20 ppm NanoXact Bare/Citrate, AGCN10: Lot RRR0028) were evaluated side by side as non-ionic/non-emissive comparatives. Studies included DLS particle sizing, TEM particle sizing, kinetic dissolution with ultracentrifugation and ICP-OES, and STEM-EELS surface analysis.

DLS measurements were collected on a Malvern Zetasizer NanoSeries according to NIST standard methods [1]. TEM particle sizing was conducted using a JEOL JEM-2800 STEM, in line with NIST standard methods [2]. Kinetic dissolution [3] was conducted with EVQ-218 and NIST nanoComposix (10 mg/L) NEAT, and in mineral rich synthetic media (mimicking environmental conditions). As both EVQ-218 and NIST nanoComposix are both produced in water (never solid isolate); kinetic sampling was not conducted on NEAT material. ICP-OES data was collected using a Perkin Elmer Optima 8000 with S10 autosampler. Samples were digested with High ORP aqua regia and sonication at 40°C for 40 minutes.

Aliquots were collected at 1 hour, 6 hours, 24 hours, 29 hours, and 48 hours. DLS and ICP-OES data were collected for all 5 timepoints, and 6 hour ultracentrifugation was conducted for 1 hour, 24 hours, and 48 hour time points. Ultracentrifugation was performed using a Beckman Coulter Optima XL-80K Ultracentrifuge with 70Ti rotor. 6.5 mL aliquots were spun at the recommended 100,000 x g force, however EVQ-218 nanoparticles did not sediment. The system was taken to the recommended tube force limit of 150,000 x g force, for up to 18 hours. As a result, the centrifugation was cut down to 6 hours to obtain daily study data.

STEM-EELS was conducted using a JEOL ARM200F. Samples were deposited onto Lacey Formvar/Carbon, 300 mesh, Cu TEM grids (Ted Pella)

## ACKNOWLEDGEMENTS AND REFERENCES

- [1] NIST-NCL Joint Assay Protocol, PCC-1: Measuring the Size of Nanoparticles in Aqueous Media Using Batch-Mode Dynamic Light Scattering.
- [2] NIST-NCL Joint Assay Protocol, PCC-7: Measuring the Size of Nanoparticles Using Transmission Electron Microscopy.
- [3] Miller, L.; and Chappell, M. ERDC/EL SR-16-1 Nanomaterial Dispersion/Dissolution Characterization Scientific Operating Procedure SOP-F-1. (2016)

This work made use of University of Utah USTAR shared facilities supported, in part, by the MRSEC Program of NSF under Award No. DMR-1121252.

## CONTACT INFORMATION

Bretni Kennon, Ph.D. – bretnikennon@evoqnano.com  
William Niedermeyer – williamniedermeyer@evoqnano.com

## RESULTS

At first glance, EVQ-218 and NIST nanoComposix are comparable 10 nm silver nanoparticles. DLS data shows both have narrow size distribution around 10 nm. TEM imaging reveals both have uniform spherical particles, with narrow size distribution and uniform particle density. When comparing surface chemistry, and gravitational effects, the differences between EVQ-218 and NIST become clear.

## GENERAL CHARACTERIZATION

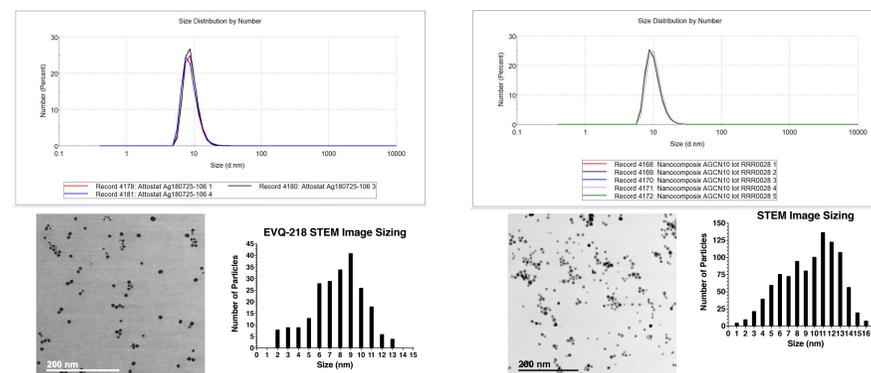


Figure 1: EVQ-218 TEM image based sizing

Figure 2: NIST nanoComposix TEM image based sizing

## KINETIC DISSOLUTION

EVQ-218 is rapidly destabilized in mineral rich water. Upon addition of MHW, EVQ-218 exhibited a full range of color changes within 60 seconds. The yellow solution immediately turned peach, pink, purple, then blue. When exposed to AAP media, EVQ-218 turned a brown grey color. Both MHW and AAP induced particle agglomeration and fallout within 6 hours, with complete fallout within 48 hours. NIST, however it did agglomerate and fallout with AAP. nanoComposix exhibited no color change upon addition to MHW, and turned a similar brown grey color with AAP. NIST material exposed to MHW, did not fall out of suspension over the 48 hour study

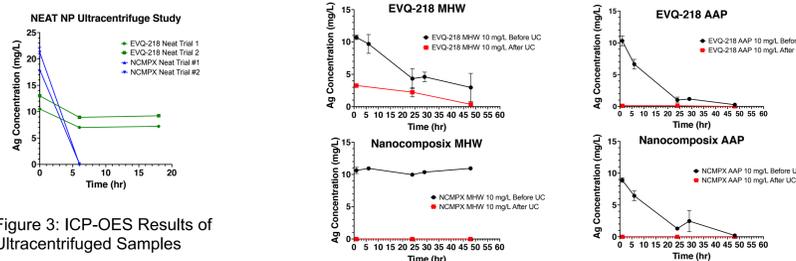


Figure 3: ICP-OES Results of Ultracentrifuged Samples

Figure 4: Kinetic ICP-OES results. Pre and Post Ultracentrifuge.

## ULTRACENTRIFUGATION

Ion emission in metallic nanoparticles often employs ultracentrifugation, particularly when size falls below 40 nm. EVQ-218 maintained 60-70% of original Ag levels after ultracentrifugation far more extreme than literature established levels. NIST nanoComposix readily sedimented under the recommended conditions, as shown in Figure 3. Experimental conditions were taken to the maximum tube ratings, and extended to 18 hours, with no additional sedimentation. To this end, experimental conditions were cut to 6 hours for feasibility.

Post centrifuge samples were imaged according to PCC-7 and revealed a smaller fraction of particles remained, with average size of 1.8 nm. This smaller sized fraction is likely less visible when imaging NEAT material due to contrast blindness from ~10 nm particles.

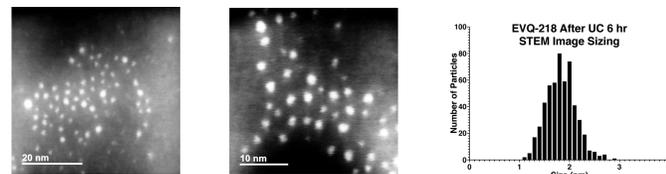


Figure 4: TEM images of EVQ-218 after 6 hours Ultracentrifugation (150,000 x g force).

## IONIC EXPOSURE

EVQ-218 was exposed to AgNO<sub>3</sub> to determine whether analytical methods can differentiate ions from particles. Upon addition to EVQ-218, Ag<sup>+</sup> ions begin to plate out onto the particle surface, immediately deforming the uniform spherical nature. Ions also induce agglomeration as shown in image set figure 5. If EVQ-218 were emissive, uniform particle nature would not be possible. Ions would continuously emit and redeposit, destroying the narrow size distribution. Samples of EVQ-218 produced in 2007 are imaged annually in an ongoing stability study. If EVQ-218 were emissive, you would not maintain the uniform profile 1 year post manufacture, let alone 15 years post manufacture which is seen in figure 6.

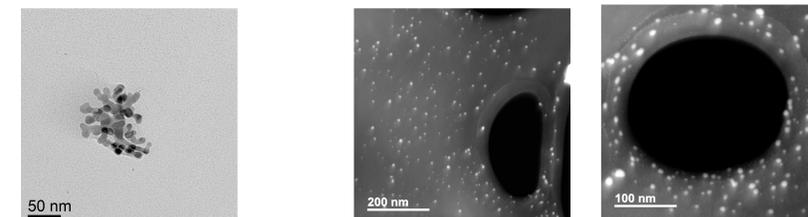


Figure 5: EVQ-218 exposed to AgNO<sub>3</sub> shows altered morphology and metallic density

Figure 6: EVQ-218 produced in 2007, imaged in 2022. Material maintains uniform morphology 15 years post manufacture.

## STEM EELS

EVQ-218, Composite Element Image, Survey Image, High Loss Image, High Loss Spectrum, and Quantification

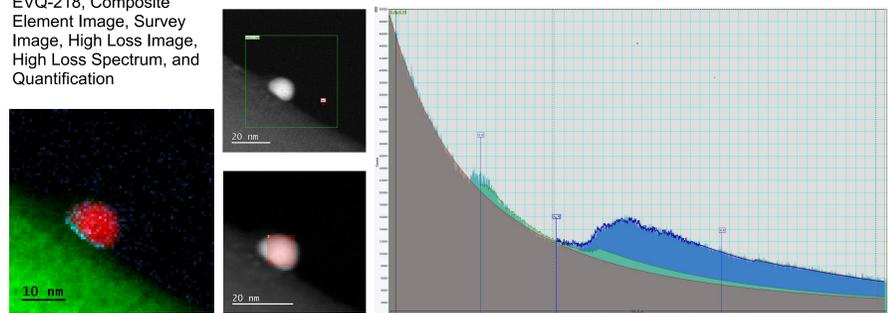


Figure 7: EVQ-218 EELS Mapping

NIST nanoComposix, Composite Element Image, Survey Image, High Loss Image, High Loss Spectrum, and Quantification

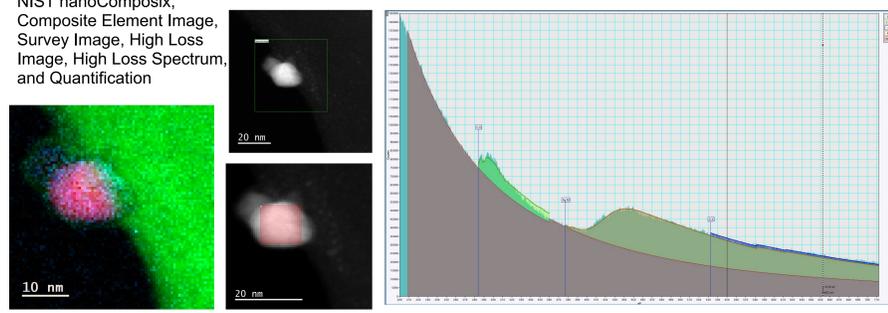


Figure 8: NIST nanoComposix EELS Mapping

## CONCLUSION

EVQ-218 is a new type of silver nanoparticle that is on par with NIST standards. It is produced in a patented high energy process that mitigates the intensive chemical and biosynthetic routes to nanometallics. EVQ-218 does not emit ions and has a unique bare silver surface not seen in NIST standards or comparable silver nanoparticles.