Nanoparticle Heterogeneity: Realizing Strengths by Embracing the Differences

ACS Spring 2024 | New Orleans | GSSPC Symposium
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## Schedule Overview

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<td>Zac O'Dell</td>
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<td>Katherine M. Hatzis</td>
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<td>2:00 – 2:25</td>
<td>David J. Masiello</td>
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<td>2:25 – 2:50</td>
<td>Peter N. Njoki</td>
<td>Engaging Undergraduate Students in Heterogeneous Nanomaterials Research at a Primarily Undergraduate Institution</td>
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2:50 – 3:15  Michelle L. Personick  *Engineering Complexity in Metal Nanoparticles: Defects and Interfaces*

**Coffee Break (3:15 pm – 3:30 pm)**

3:30 – 3:55  Bo Zhang  *Ultrafast Transient Electrochemical Behavior of Single Nanoparticles*

3:55 – 4:20  Peng Chen  *Single-Particle Photoelectrodes: Inter-Facet and Intra-Facet Differences*

4:20 – 4:45  Caleb M. Hill  *Pipets as Tools for Imaging Chemical Reactivity at the Single Nanoparticle Level*

4:45 – 5:05  Ayush Agarwal  *Accelerating Nanoparticle Characterization: High Throughput Size Resolved Elemental Analysis using SMPS-ICP-MS*

5:05 – 5:25  J. Ethan Batey  *Ultrahigh-Throughput Single-Particle Hyperspectral Imaging of Plasmonic Nanoparticles*

**Keynote Lecture & Closing Remarks (5:25 pm – 6:00 pm)**

5:25 – 5:50  Christy F. Landes  *Active Control of Energy Transfer in Plasmonic Nanorod-Polyaniline Hybrids*

5:50 – 6:00  Ekta Verma  *Closing Remarks*

**Networking Social (6:30 pm – 8:30 pm)**
Critical need: High-throughput methods for screening single-nanocrystal properties

Sara E. Skrabalak | Indiana University – Bloomington

This presentation will outline the critical need for high-throughput tools and methods that screen single-nanocrystal properties. Colloidally prepared nanocrystals present a variety of different features (e.g., planes, edges, and defects), and the distribution of these features will vary from one nanocrystal to the next even in the best nanocrystal samples. Given the strong structural dependence of many nanocrystal properties, such heterogeneity is a double-edged sword. On the one hand, this heterogeneity provides ample opportunity to discover new structures with useful properties. On the other hand, this heterogeneity makes structure-property correlation challenging as common property measurements (e.g., by absorption spectroscopy, cyclic voltammetry) are based on the ensemble, meaning that each nanocrystal may contribute by a different (unknown) amount. This heterogeneity likely affects the performance of nanocrystals samples for a broad range of applications where important minority features may be hidden. To identify and ultimately leverage this heterogeneity, high-throughput tools and methods that screen the properties of nanocrystals at the single-particle level are needed. Advances developed by the Center for Single-Entity Nanochemistry and Nanocrystal Design will be discussed. These advances include a new high-throughput electrochemistry platform consisting of 96 electrochemical cells dimensioned to match common 96-well plates, which is compatible with electroanalysis and electrosynthesis of metal nanocrystals. These advances also include the application of calcite-assisted localization and kinetics (CLocK) microscopy to metal nanocrystals, with CLocK images encoding spectral data that can be used to extract nanocrystal size and orientation rapidly and accurately, without the need for electron microscopy.
Many applications of metal nanoparticles (NPs) require precisely controlled optical properties that are governed by the NP shape, morphology and composition. Finding reaction conditions for the synthesis of NPs with targeted characteristics is a time-consuming and resource-intensive trial-and-error process, however closed-loop NP synthesis enables the accelerated exploration of large chemical spaces without human intervention. Here, we introduce the Autonomous Fluidic Identification and Optimization Nanochemistry (AFION) self-driving lab that integrates a microfluidic reactor, in-flow spectroscopic NP characterization, and machine learning for the exploration and optimization of the multidimensional chemical space for the photochemical synthesis of metal NPs. By targeting spectroscopic NP properties, the AFION lab successfully identified reaction conditions for the synthesis of different types of NPs with designated shapes, morphologies, and compositions, including gold tetrapods that have not been previously synthesized via seedless photochemical reaction. Data analysis provided insight into the role of reaction conditions for the synthesis of the targeted NP type and the impact of a specific condition on NP quality. This work shows that the AFION lab is an effective exploration platform for on-demand synthesis of metal NPs.
Using Precursor Libraries to Control Nanocrystal Properties

Mark P. Hendricks | Whitman College

Nanocrystal reactions are often controlled by changing the reaction conditions: time, temperature, reagent concentrations and ratios; or through the addition of Lewis acids or bases that influence the reaction. While effective, these changes often affect multiple reaction parameters and therefore make it difficult to isolate which variable of the reaction caused a change to the nanocrystal properties. One method to avoid this complication is to use a library of similarly-structured precursors that undergo the same mechanism, where the structural differences of the precursors influence the nanocrystal reaction in a systematic way. This enables rational control over nanocrystal properties and supports studies that correlate how and why the structural differences of the precursors impact the nanocrystal properties. This talk will highlight the use of precursor libraries to synthesize a variety of nanocrystals, including substituted thiourea and single-source precursors for controlling the formation of metal sulfide nanocrystals.
The ability to precisely control nanoparticle shape, size, and composition is useful in many fields, including catalysis and plasmonics. Seed-mediated strategies have proven effective for preparing a wide variety of structures, but the understanding of how to selectively grow corners, edges, and facets has limited the development of a general strategy to control structure evolution. We have developed a synthetic strategy for directing the site-specific growth of anisotropic seeds to prepare a library of designer nanostructures. This strategy leverages nucleation energy barrier profiles and the chemical potential of the growth solution to control the site-specific growth of nanoparticles into exotic shapes and compositions. This strategy can be used to not only control where growth occurs on anisotropic seeds but also controls the exposed facets of the newly grown regions. The observation that one can control the microscopic shape of single crystals based upon control of particle building block and crystal symmetry has important fundamental and technological implications for this novel class of colloidal matter.
Can Seeds Help? Toward Improved Anisotropic Nanoparticle Homogeneity for Room-Temperature Reactions

Teri W. Odom | Northwestern University

This talk will discuss the growth mechanisms of anisotropic gold nanoparticles at room temperature. We will focus on a simple synthetic system starting from a gold salt and Good’s buffers as reducing and stabilizing agents. Using correlative microscopy and spectroscopy tools, we will analyze the formation of anisotropic gold nanoparticles as a function of reaction time. We will describe differences in anisotropic features as a function of pH and buffer concentration and how large nanoparticle seeds can guide the growth process toward increased homogeneity. Implications for exploiting nanoparticle heterogeneity in applications will be discussed.
Investigating the pH Dependent Surface Structure of Citrate Coated Silver Nanoparticles

Clyde A. Daly Jr. | Haverford College

One of the most common coatings for silver nanoparticles is citrate because of its low cost and relatively benign biological effects. The citrate molecules do not bind very strongly to the nanoparticle surface; instead their surface binding is mainly favorable because the citrate molecules replace bound water molecules which then have more configurational freedom. Experiments have observed that citrate coated nanoparticles change in their surface charge as the pH of the solution changes, which is often attributed to the protonation or deprotonation of citrate molecules on the surface. However, there is little simulation evidence of this hypothesis. In our work, we build computational models of citrate coated silver nanoparticles under the assumption that the mixture of citrate protonation states present in solution are what would be expected from the pKa’s of citrate. We then calculate the surface charge of the resulting nanoparticles and find a correlation to experimental zeta potential measurements. We also generally quantify the surface coating structure of the nanoparticles.
Heterogeneity at the Atomic and Molecular Levels in and on Noble Metal Nanorods

Catherine J. Murphy | University of Illinois Urbana-Champaign

When chemists create colloidal nanomaterials with ligand shells, the natural tendency is to picture the Platonic ideal of a single nanorod with a uniform ligand shell. In the case of rods containing multiple metals, it is generally assumed that the nanoscale composition is predicted by the bulk phase diagram of the metals. In this talk I will show how the experimental reality of ligand density, ligand dynamics, and spatial location of metals within individual metallic nanorods are far more complex than the community may have imagined.
Several methods can identify the chemical species on nanomaterials’ surfaces, yet less is known about the heterogeneity and dynamics of these surface ligands. In 2019, Murphy et. Al. functionalized a series of gold nanospheres (AuNS) with one ligand (MTAB) featuring a thiol group, a 16-carbon alkyl chain, and a bulky trimethyl ammonium headgroup in a D2O environment; using NMR tools such as T2 (spin-spin relaxation) measurements, they determined that ligand motion sharply decreased as NP diameter increased due to the reduction in radius in curvature. This talk will feature a continuation of that work in which ligands analogous to MTAB but featuring shorter or longer alkyl chains are appended onto AuNSs and investigated with NMR spectroscopy to connect the effects of ligand length/van der Waals interactions on ligand conformation and dynamicity on AuNS. This work will create a fuller picture of ligand behavior on AuNS, which will inform many of their potential applications.
Every ligand counts: Quantitative analysis of nanoparticle surface ligands via TD-DART-MS

Kevin Cavey | Middle Tennessee State University

The characterization of surface ligands on colloidal nanoparticles remains an underdeveloped area of research. The methods currently used for surface characterization often lack specificity and sensitivity, making the identification of specific ligands and ligand mixtures complicated, if not impossible. To overcome these limitations, thermal desorption–direct analysis in real-time–mass spectrometry (TD-DART-MS) was used to discriminate between bound and unbound species in colloidal suspensions of cadmium selenide quantum dots (QDs). Beyond the identification of bound species, we show that this method allows for the quantitative analysis of ligands on the surface of QDs. The oleate coated QDs were analyzed and the quantity of oleate was determined by an external calibration curve constructed based on oleic acid standard solutions. The 1H NMR was used as a reference method to validate the quantitative results for oleate ligands. Our study demonstrates TD-DART-MS as a valuable tool to study surface chemistry of QDs.
Optical Control over Thermal Distributions in Topologically Trivial and Non-Trivial Plasmon Lattices

David J. Masiello | University of Washington

Emergent from the discrete spatial periodicity of plasmonic arrays, surface lattice resonances (SLRs) are characterized as dispersive, high-quality polaritonic modes that can be selectively excited at specific points in their photonic band structure by plane-wave light of varying frequency, polarization, and angle of incidence. Room-temperature Bose-Einstein condensation of exciton polaritons, lasing, and nonlinear matter-wave physics have all found origins in SLR systems, but to date, little attention has been paid to their photothermal behavior. In this talk I will discuss our recent work in combining analytical theory and numerical calculations to investigate the photothermal properties of SLRs in periodic 1D and 2D arrays of plasmonic nanoparticles. Specifically, I will describe how to create steady-state SLR thermal gradients spanning from the nanoscale to hundreds of microns that are actively controllable using light despite heat diffusion. I will also examine the surprising ability to localize thermal gradients at the lattice edges in topologically non-trivial SLR dimer lattices, thereby establishing a class of extraordinary thermal responses that are unconventional in ordinary materials.
Engaging undergraduate students in heterogeneous nanomaterials research at a primarily undergraduate institution

Peter N. Njoki | Hampton University

Experimental research is an important complement to classroom-based undergraduate education. It fosters an appreciation for the scientific process by linking what is learned in the classroom with the hands-on laboratory experience. Performing research alongside a faculty mentor helps undergraduate students better understand design of experiments, interpretation of data, exploration of the literature, communicating results, and teamwork. This presentation describes heterogeneous nanomaterials research by undergraduate students at a Primarily Undergraduate Institution. Heterogenous copper-silver nanoparticles were synthesized using microwave irradiation technique which has been shown to shorten reaction time and enhance reaction control. The morphology of the nanoparticles was influenced by the reaction temperature and the sequence of reducing the reaction precursors. When silver ions were reduced on copper nanoparticles seeds at hydrothermal temperature, spherical nanoparticles were formed whereas concurrent reduction of copper ions and silver ions at identical hydrothermal temperature produced shaped nanoparticles. Results from characterization of the optical, size, morphological, and composition of the nanoparticles using ultraviolet-visible (UV-vis) spectroscopy, scanning transmission electron microscopy, and X-ray photoelectron spectroscopy will be discussed. Implications of the results to the design of multifunctional nanoparticles will also be discussed.
Meeting competing demands for performance and sustainability in applications such as catalysis requires the design and synthesis of increasingly complex nanostructured materials with finely tuned and multifunctional surfaces and interfaces. This added complexity, in turn, provides more opportunity for the introduction of structural and compositional heterogeneity during nanoparticle synthesis. As a result, it is necessary to continually develop creative synthetic approaches for minimizing heterogeneity and/or for tuning architectural parameters such as composition, defects, and interfaces in a controlled and deliberate manner to accurately correlate these features with the performance of the material.

This talk will describe approaches developed in our lab for differentially tuning the rates of competing chemical processes to manage complexity during nanomaterials synthesis in order to generate materials with tailored defects and interfaces. In the first approach, visible light illumination is used as an orthogonal parameter to the chemical composition of the reaction solution to modulate the relative rates of reductive and oxidative chemical processes during materials synthesis. This approach enables the formation of hybrid nanostructures composed of a plasmonic metal and a more reactive metal, with both core-shell and core-satellite architectures. It also provides a means for reconfiguring monometallic Ag nanoparticles to tune defect structure while maintaining the same overall surface facet structure and surface-binding molecules, to separate the influence of these parameters in studies of materials performance and reactivity. The second example is an integrated electrochemical approach that enables the synthesis of shaped nanoparticles directly on electrode surfaces. While nanoparticles synthesized in colloidal solution can be processed and cast onto electrodes, doing so adds an additional fabrication step—increasing time and cost—and faces challenges in controlling particle dispersion on the surface. Our approach links metal nanoparticle synthesis with real-time monitoring of chemical changes in the reaction solution using a combination of colloidal particle synthesis, electrochemical particle synthesis, and electrochemical measurements. This provides a pathway for the directed adaptation of the extensive library of existing shaped colloidal nanoparticle syntheses to growth on a surface—something that remains a non-trivial challenge.
Ultrafast Transient Electrochemical Behavior of Single Nanoparticles

Bo Zhang | University of Washington

We have been studying some unique and interesting electrochemical behavior of single metal nanoparticles during their collision on an ultramicroelectrode (UME). In one study, we observed the ultrafast proton reduction and H-adsorption process when single Pt nanoparticles collide on a carbon UME. These reduction and adsorption processes appear as individual sharp current spikes on top of their continuous reduction waves for hydrogen reduction and evolution. In a second study, we saw similar transient current spikes when Pt nanoparticles collide and catalytically oxidize hydrazine (N2H4) molecules on a carbon UME. These spikes are likely generated from the catalytic oxidation of the pre-adsorbed N2H4 molecules on the Pt nanoparticles upon their collision on the carbon surface. If time permits, I will also describe another interesting observation related to transient formation of nanobubbles and their possible effect on determining the adhesion probability of nanoparticles during their collision on an UME.
Single-particle photoelectrodes: inter-facet and intra-facet differences

Peng Chen | Cornell University

This presentation will describe our recent work in using single-particle/single-molecule imaging approaches to study (photo)(electro)catalysis on nano- and micro-scale particles. I will first give some background on single-molecule, super-resolution fluorescence imaging of catalytic reactions on single catalyst particles, with some examples. I will then focus on a recent study of photoelectrocatalytic properties of particulate semiconductor photocatalysts, important for many solar energy conversion technologies. In anisotropically shaped photocatalyst particles, the different constituent facets differ in their electronic and thus photoelectrocatalytic properties, and between different facets they may form inter-facet junctions at their adjoining edges, analogous to lateral two-dimensional (2D) heterojunctions or pseudo-2D junctions made of few-layer 2D materials. Using subfacet-level multimodal functional imaging, we uncover inter-facet junction effects on anisotropically shaped bismuth vanadate (BiVO4) particles and identify the characteristics of near-edge transition zones on the particle surface, which underpin the whole-particle photoelectrochemistry. The imaging tools, the analytical framework and the inter-facet junction concept pave new avenues towards understanding, predicting and engineering (opto)electronic and photoelectrochemical properties of faceted semiconducting materials, with broad implications in energy science and semiconductor technology.
Pipets as Tools for Imaging Chemical Reactivity at the Single Nanoparticle Level

Caleb M. Hill | University of Wyoming

Metallic and semiconducting nanoparticles (NPs) are attractive platforms for a variety of applications in catalysis, energy conversion, and sensing. Unfortunately, the complex, heterogeneous nature of these systems makes it difficult to elucidate how different chemical, electronic, and morphological factors impact their performance. In this presentation, it will be shown how Scanning Electrochemical Cell Microscopy (SECCM), which utilizes electrolyte-filled pipets as high-resolution probes, can be used to characterize the chemical behavior of individual NPs. First, studies will be presented which employ correlated SECCM and electron microscopy measurements to enable key structural factors controlling reactivity to be identified for shape-controlled colloidal NPs and high entropy alloy systems. Experiments will also be presented which leverage SECCM as a powerful, instantly reconfigurable tool for the fabrication of ordered NP arrays. Together, these studies demonstrate the utility of electrochemical microscopy techniques for fundamental studies into the chemical properties of discrete, nanoscale chemical entities.
Accelerating Nanoparticle Characterization: High Throughput Size Resolved Elemental Analysis using SMPS-ICP-MS

Ayush Agarwal | École Polytechnique Fédérale de Lausanne

Over the past decades, the production of engineered nanoparticles has increased from thousands of kilograms to thousands of tons! The simultaneous analysis of physical and chemical properties of nanoparticles is crucial for understanding their properties and behaviour. This work presents the hyphenation of a scanning mobility particle sizer (SMPS) with inductively coupled plasma mass spectrometer (ICP-MS), allowing for simultaneous size and elemental analysis of nanoparticles. The system demonstrated the ability to characterize nanoparticles in highly concentrated suspensions. Validation of results was performed using various techniques, including single-particle ICP-MS, low-resolution transmission electron microscopy (LR-TEM), and scanning transmission electron microscopy energy-dispersive X-ray spectroscopy (STEM-EDX). This establishes the SMPS-ICP-MS system as a promising high throughput, complementary analytical tool for characterizing nanoparticles. This work has broad implications for understanding and controlling nanomaterial properties in different industrial and research contexts.
Ultrahigh-Throughput Single-Particle Hyperspectral Imaging of Plasmonic Nanoparticles

J. Ethan Batey | University of Arkansas

Plasmonic nanoparticles (NPs) have become increasingly useful in recent years for their roles in nanomedicine, cellular biology, energy storage and conversion, photocatalysis, and more. At the single-particle level, NPs have heterogeneous physical and chemical properties which are not resolvable in ensemble measurements. We have developed an ultrahigh-throughput spectroscopy-microscopy system for characterization of NPs at the single-particle level using phasor analysis. This method enables quantification of spectral and spatial information on large numbers of NPs using a single snapshot with high temporal resolution and localization precision (sub-5 nm). Our method is characterized using the localized surface plasmonic resonance (SPR) scattering spectra of gold nanospheres (AuNSs) of many different sizes. Comparing to the conventional optical grating method which suffers low efficiency in characterization due to spectral interference caused by nearby nanoparticles, our phasor approach enables high-throughput analysis of single-particle SPR properties in high particle density. We see up to 10-fold greater efficiency in single-particle characterization using the spectra phasor approach when compared to a conventional optical grating method.
Active Control of Energy Transfer in Plasmonic Nanorod-Polyaniline Hybrids

Christy F. Landes | University of Illinois at Urbana-Champaign

I will discuss recent work in which we showed that polyaniline (PANI), which readily hybridizes onto gold nanorods (AuNRs), undergoes switchable energy transfer when the PANI chemical structure is modulated between its emeraldine base and salt forms. Energy transfer between plasmonic nanoparticles and polymer acceptors is a possible means to overcome fast competing processes such as rethermalization. Energy transfer had not previously been identified, despite the fact that AUNR/PANI hybrids have been widely studied for refractive index switching. Because energy transfer depends strongly on spectral overlap between the donor (AuNR plasmon) and the acceptor (PANI absorption) spectra, small changes in overlap within a heterogeneously broadened colloidal sample are enough to obscure the effect in ensemble spectroscopy. Single-particle scattering studies exploit the innate heterogeneity of AuNR/PANI samples because we were able to examine the spectral overlap 1 hybrid at a time and show that strong energy transfer can occur when overlap is maximized in either the emeraldine base or salt forms. Thus, the small differences in AuNR/PANI structure turn out to be an advantage when we look one hybrid at a time.
Travel Awardees

Names listed here are in alphabetical order.

Isa Ahmed.................................................................Wayne State University

James Batey..............................................................University of Arkansas

Samuel Holloway......................................................Jackson State University

Abigail Ramirez.......................................................Lewis & Clark College

Kenzie Stewart.........................................................Lewis & Clark College

Morgan Stucky...........................................................Western Washington University
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Rebecca Skalla
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Ekta Verma
Indiana University
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Sanjay Sridhar
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Meeting Logistics

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Madison Edwards
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Marketing Liaison

Oluwasegun “Segun” Wahab
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Event Coordinator
About CSENND

NSF Center for Single-Entity Nanochemistry and Nanocrystal Design.

Our vision for the NSF Center for Single-Entity Nanochemistry and Nanocrystal Design is to address one of the biggest challenges in nanocrystal chemistry – the inherent heterogeneity of nanocrystals – by creating the scientific toolkit and chemical knowledge to separate individual nanocrystal responses from bulk property measurements. This research is supported by the NSF Centers for Chemical Innovation Program Grant #221062 from the Division of Chemistry.

Nanocrystals are a driver of innovation because they display properties distinct from their bulk form. For example, bulk gold appears a lustrous yellow, but gold nanocrystals can appear nearly any color depending on their specific size and shape. However, the way in which nanocrystals are made introduces variations from one crystal to the next in the same sample, meaning that each one may have different properties. This heterogeneity provides ample opportunity to discover new nanocrystals with useful properties. On the other hand, this heterogeneity makes the discovery of the nanocrystals with exceptional properties incredibly challenging, similar to finding the needle in a haystack.

Conceptually, this challenge is similar to the screening challenge in the field of drug discovery and design, where there is a large experimental space from which a led drug has to be identified. CSENND is inspired by the conceptual similarities and is creating the tools to accelerate the discovery and design of nanocrystals with exceptional properties and aims to transform the way the chemical community handles nanocrystal heterogeneity. For Phase 1 of CSENND, these efforts will be directed toward nanocrystals for catalysis and chemical sensing.

Learn more about CSENND with the QR code below.