Southeastern Nanotechnology Infrastructure Corridor (SENIC)

Research and Education Highlights

Year 8 (October 2022 – September 2023)

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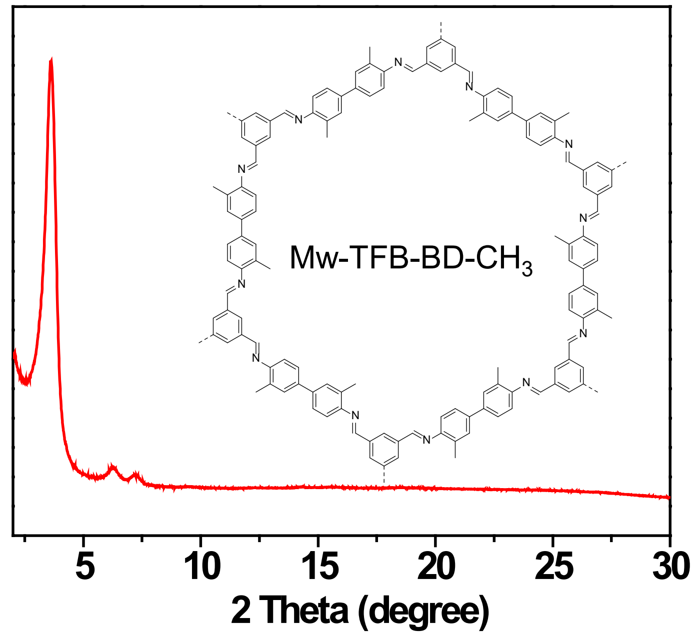
NSF

Imine-Linked Covalent Organic Frameworks for Exceptional Iodine Capture

This work delineates a facile microwave-assisted synthesis of 2D imine-linked covalent organic frameworks (COFs), Mw-TFB-BD-X, (X = $-CH_3$ and $-OCH_3$) under air within merely 1 hour. The resultant COFs possessed higher crystallinity, better yields, and more uniform morphology than those of their solvothermal counterparts. Remarkably, Mw-TFB-BD-CH_3 exhibited exceptional iodine adsorption capacities of 7.83 g g^{-1}, placing it among the best-performing COF adsorbents for static iodine capture to date. Moreover, Mw-TFB-BD-CH_3 can be reused 5 times with no apparent loss in the adsorption capacity. This work establishes a benchmark for developing advanced iodine adsorbents that combine fast kinetics, high capacity, excellent reusability, and facile synthesis.



Powder X-ray diffraction (PXRD) pattern of COF, Mw-TFB-BD-CH_3, showing highly crystallinity.

Ziad Alsudairy, Normanda Brown, and Xinle Li, Department of Chemistry, Clark Atlanta University. PXRD analysis was performed at Materials Characterization Facility at Georgia Institute of Technology.

This work was supported by NSF Award # 2100360.

National Research Priority: DoD Critical Technology Area–Advanced Materials

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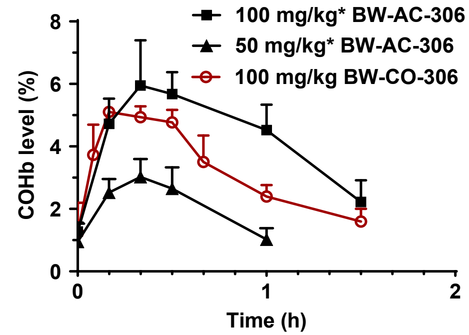
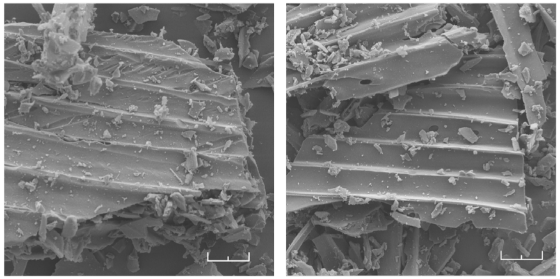
Activated Charcoal Dispersion of Carbon Monoxide Prodrugs for Oral Delivery of CO in a Pill

A novel orally bioavailable solid formulation to deliver a gaseous signaling molecule, carbon monoxide (CO), was developed by adsorbing oxalyl saccharin, a newly developed organic CO prodrug, in the activated charcoal (AC). The resulting solid dispersion formulation addresses key developability issues of this CO prodrug, including the paradoxical problem of low water solubility of the prodrug and the requirement of hydrolysis to release CO, the need for an organic cosolvent, and systemic exposure to the CO prodrug and release byproduct. This formulation allows encapsulation in normal and enteric-coated gel capsules, which enables controllable CO delivery to the upper or lower GI system. Through in-vivo pharmacokinetic studies in mice, the AC formulation showed better CO delivery efficiency of delivering CO through oral administration compared to the prodrug dosed with an organic cosolvent. We envision the wide applicability of this formulation in facilitating the future development of CO-based therapeutics



COHb level in blood following p.o. administration of **BW-AC-306**



SEM image of (A) AC, and (B) **BW-AC-306**. (Scale bar: 20 μm)

Xiaoxiao Yang, Wen Lu, Ladie Kimberly De La Cruz, and Binghe Wang, Dept. of Chemistry, Georgia State Univ., and Minjia Wang and Chalet Tan, Univ. of Mississippi. DSC/TGA were performed at Georgia Tech's IEN.

Partially supported by a SENIC Catalyst grant. Research funded by National Institutes of Health (R01DK119202). International Journal of Pharmaceutics, 2022, 618, 121650.

National Research Priority: NAE Grand Challenge–Engineer Better Medicines

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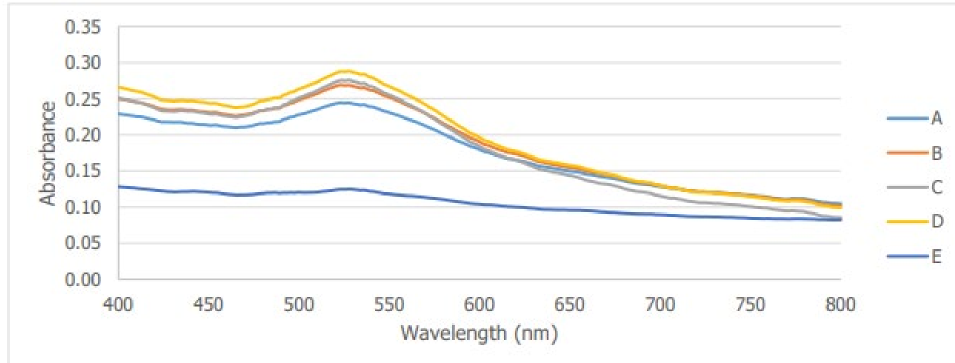
Sensitive Detection of Aflatoxin B1 in Peanuts Using Gold Nanoparticles

This aim of this research was to develop a colorimetric immunoassay for the detection of aflatoxin B1 (AFB1), a dangerous fungal toxin that contaminates a variety of crops. The two major components of the assay are monoclonal anti-AFB1 antibody immobilized on experimentally synthesized gold nanoparticles, and magnetic iron beads covalently bonded to AFB1 antigen. UV-Vis detection of toxin was able to rapidly and sensitively detect AFB1 in peanuts at concentrations of 0.2 ppb. The commercial minimum AFB1 allowable for the US is reported to be 20 ppb, which means the assay can detect 1000x times lower than the allowable limit of consumption]



Average of UV-Vis absorption spectra of supernatant after magnetic separation with different concentrations of AFB (from sample A to E: 800, 500, 200, 100, 50, 20 ng/L).

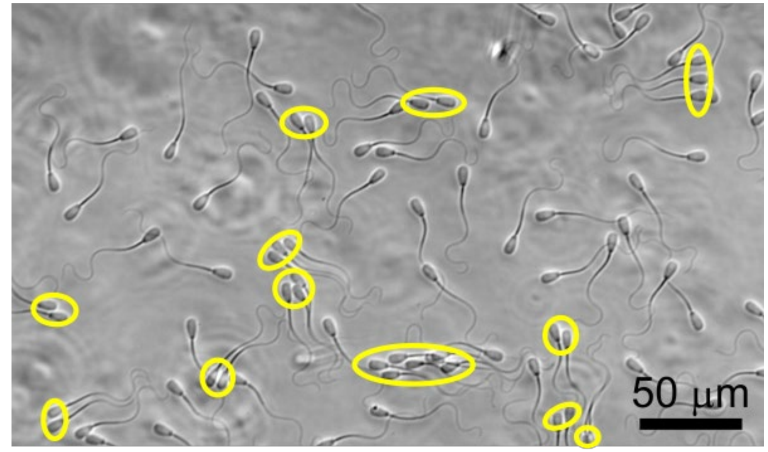
Amber D. Davenport, and Hari P. Singh, Nanotechnology Laboratory, Agricultural Research Station, Department of Agricultural Sciences, College of Agriculture, Family Sciences & Technology (CAFST), Fort Valley State University. Georgia Tech's Institute for Electronics and Nanotechnology facility was utilized for sample analysis.

This work was supported by USDA-ARS grant 335149 "Reducing Aflatoxin Contamination in United States Peanuts" and a SENIC Catalyst grant.

National Research Priority: NSF–Growing Convergence Research

Biological Benefits of Collective Swimming of Sperm in a Viscoelastic Fluid

This work uses a microfluidic device to study motility related biological benefits for sperm to swim collectively when in a viscoelastic fluid. We found that under no flow, collective sperm trajectories are more linear, or sperm swim in a more directed manner. As the flow rate increases, sperm in groups are better aligned against the flow than sperm swimming individually. Finally, under a strong flow that is capable of carrying sperm downstream, we found that sperm swimming close to each other are less likely to move in the flow direction, suggesting the collective swimming protect them from removal by a strong flow.



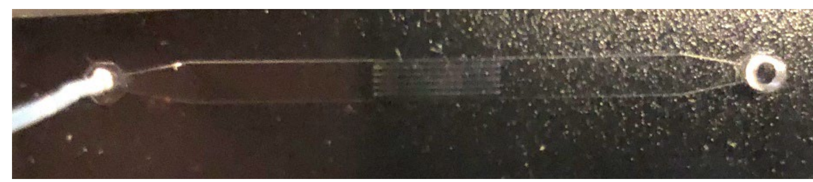


Image of sperm swim collectively in a microfluidic device (shown below) filled with viscoelastic fluid.

Shiva Phuyal and Chih Kuan Tung, Department of Physics, North Carolina A&T State University. Work performed at Joint School of Nanoscience and Nanotechnology.

This work was supported by NIH Award R15HD095411. Front. Cell Dev. Biol. 10:961623 (2022).

National Research Priority: NSF–Understanding the Rules of Life

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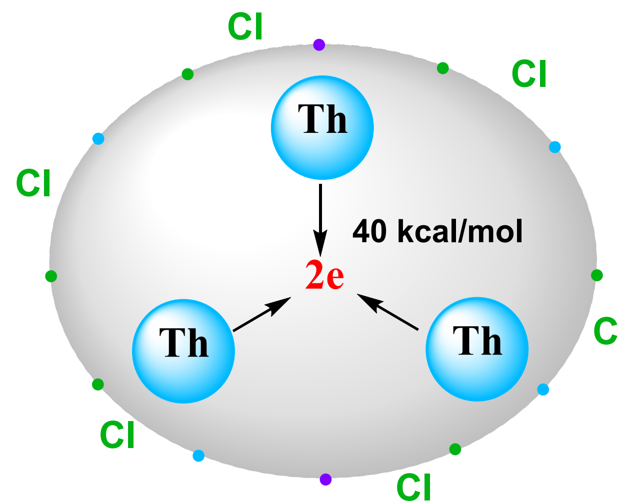
Bonding Nature in the Crystalline Tri-Thorium Cluster: Core-Shell Syngenetic σ-Aromaticity

The aromaticity in transition metal clusters has been fascinating chemists. Recently, Boronski et al. reported a crystalline tri-thorium cluster at the mild experimental condition. In this work, we explore the bonding nature of the tri-thorium cluster with modern ab initio valence bond (VB) theory in order to understand its stability. We propose a new type of core-shell syngenetic bonding model which involves a three-center two electron bond in the Th_3 core and a multicentered $(ThCl_2)_3$ charge-shift bond with 12 electrons scattering along the outer shell. Contributions from both bonds to the stability of the crystalline tri-thorium cluster were differentiated and quantified and compared with the well-regarded σ-aromatic H_3^+ and non-aromatic Li_3^+. We conclude that there is considerable σ-aromaticity in the tri-thorium cluster.

Core-Shell Syngenetic Bonding



Green: Cl; Blue: Th; Purple: K or Free electron

A core-shell syngenetic bonding model is proposed to describe the chemical bond in a stable crystalline tri-thorium cluster.

Xuhui Lin, Southwest Jiaotong University, China; Yirong Mo, Department of Nanoscience, University of North Carolina at Greensboro. Work performed in part at JSNN.

Angewandte Chemie International Edition, 61(37), e202209658 (2022).

National Research Priority: DoD Critical Technology Area–Advanced Materials

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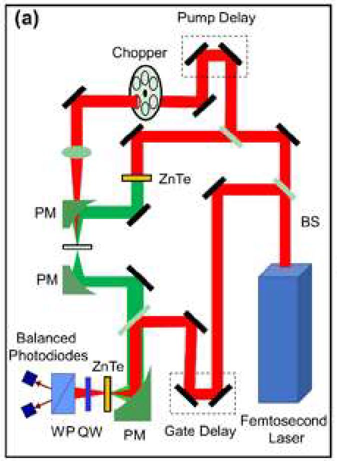
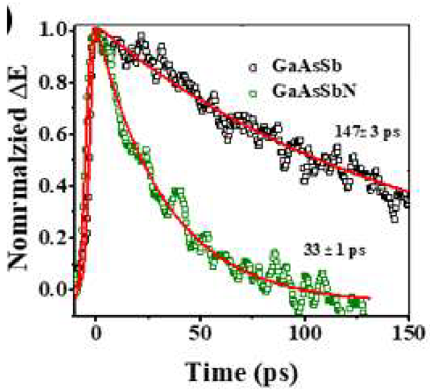
Carrier Dynamics and Transport in Te-doped GaAsSb and GaAsSbN NWs by Correlating Ultrafast Terahertz Spectroscopy

It is established that a small amount of nitrogen (N) incorporation in III-V semiconductor NWs can effectively redshift their wavelength of operation and tailor their electronic properties for specific applications. In this work, ultrafast optical pump-terahertz probe spectroscopy has been used to study non-equilibrium carrier dynamics and transport in Tedoped GaAsSb and dilute nitride GaAsSbN NWs, with the goal of correlating these results with electrical characterization of their equilibrium photo-response under bias and low-frequency noise characteristics. Nitrogen incorporation in GaAsSb NWs led to a significant increase in the carrier scattering rate, resulting in a severe reduction in carrier mobility. Finally, we observed a very fast rise time of ~ 2 ps for both NW materials, directly impacting their potential use as highspeed photodetectors



Schematic of the OPTP experiment



Transient photoinduced change in the THz electric (E) field for GaAsSb and GaAsSbN NWs probed by OPTP spectroscopy

Shanthi Iyer and Rohit Prasankumar, NC A&T State University and Los Alamos National Laboratory. Work was partly performed at JSNN.

Supported by U.S. Army Grant Number W911NF-19-1-0002, Air Force Office of Scientific Research; partly funded by the National Science Foundation (NSF) (Grant: EECS-18322117). Nanotechnology, 33, 425702 (8 pp) (2022).

National Research Priority: NSF–Quantum Leap

National Nanotechnology Coordinated Infrastructure

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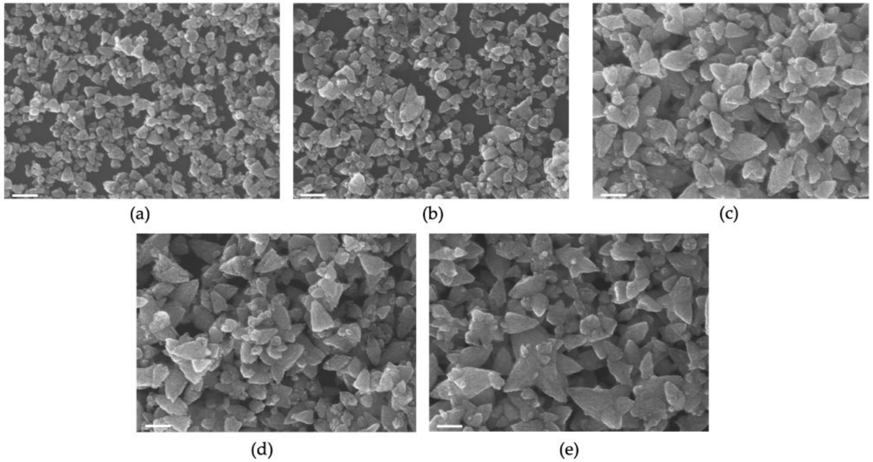
Mg-Doped ZnO Nanoparticles with Tunable Band Gaps for Surface-Enhanced Raman Scattering (SERS)-Based Sensing

This work aims at improving the signal enhancement effect of ZnO when employed as substrate in surface-enhanced Raman scattering (SERS) based applications. We doped ZnO with different concentrations of Mg by co-precipitation to synthesize Mg-doped ZnO nanoparticles with bandgap observed to be modified with the dopant amount. An enhanced Raman signal was observed on molecules using these substrates in colloidal form, which is appropriate for cellular applications and in point-of-care environments. In addition, stability tests and dose-dependent doping for toxicity responses in live cells were carried out to determine the suitability of the substrates in a biological environment. Substrates showed high stability in the first hour of dissolution in a cell media, and it was observed that there was no significant cytotoxic effect resulting from doping Mg with ZnO. Overall, this study provides evidence for the tunability of ZnO substrates and may serve as a platform for applications in molecular biosensing.



FE-SEM images (scale bar: 200 nm) of (**a**) pure ZnO nanoparticles; (**b**) 2% Mg-doped nanoparticles; (**c**) 5% Mg-doped nanoparticles; (**d**) 7% Mg-doped nanoparticles; (**e**) 10% Mg-doped nanoparticles.

Adesoye, Samuel, Saqer Al Abdullah, Kyle Nowlin, and Kristen Dellinger, Nanoengineering, NC A&T State University.
Work performed at Joint School of Nanoscience and Nanotechnology

This research was funded by KL2 Scholar Award from the National Center for Advancing Translational Sciences, NIH, Grant KL2TR002490. Nanomaterials 12, no. 20: 3564.

National Research Priority: DoD Critical Technology Area–Advanced Materials

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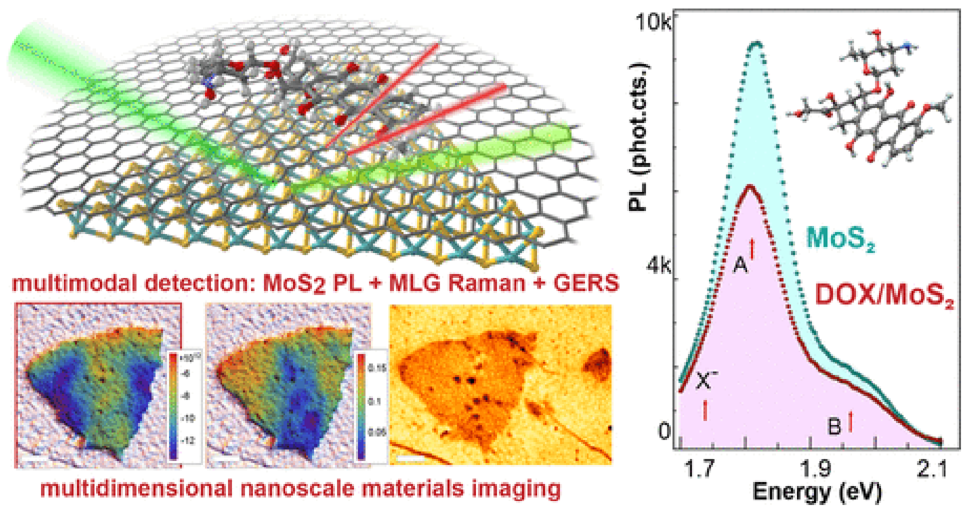
Multidimensional Imaging for Multimodal Label-free Biosensing

Researchers at Penn State University, University of North Carolina-Greensboro, and North Carolina A&T State University developed a multidimensional optical imaging technique to map subdiffractional distributions for doping and strain and understand the role of those for modulation of the electronic properties of the material. This group reported on the optical label-free detection of doxorubicin via three independent optical detection channels (photoluminescence shift, Raman shift, and graphene enhanced Raman scattering). Multidimensional nanoscale imaging allows one to reveal the physical origin for local responses and best strategy for the mitigation of materials variability to enable multiplexed biosensing



Tetyana Ignatova and Slava Rotkin, Penn State and UNC Greensboro. Part of work performed at Joint School of Nanoscience and Nanotechnology

Work partially supported by NSF CHE-2032582) and CHE-2032601. ACS Nano, 16, 2598–2607, 2022

National Research Priority: NSF–Growing Convergence Research

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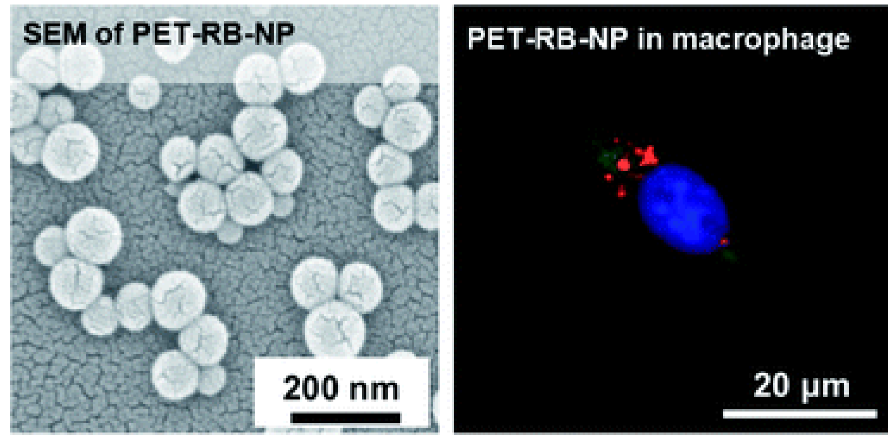
Polyethylene terephthalate (PET) nanoparticles for studies in mammalian cells

Environmental presence of fragmented plastics, derived from high-commodity polymers, is an emerging concern with unknown consequences for human health. As a crucial high-commodity polymer and contributor of plastic waste, PET has infiltrated drinking water, food, and beverages in the form of small-scale debris (i.e., microplastics). Fluorescent nanoparticles (NPs) comprising polyethylene terephthalate (PET) with a hydrodynamic diameter of 158 ± 2 nm were synthesized in a bottom-up approach. Concentration-dependent uptake and cytotoxicity of PET NPs in macrophages are shown. The fabrication of well-characterized NPs, derived from high-commodity polymers, will support future studies to assess effects on biological systems.



FE-SEM images of for PET-RB NPs; fluorescence microscopy of RAW 264.7 cells exposed to PET-RB nanoparticles

Leah Johnson and Ninell Mortensen, RTI International. Part of work performed at Joint School of Nanoscience and Nanotechnology

Nanoscale Advances, 3(2), 2021, 339-346

National Research Priority: NSF–Understanding the Rules of Life

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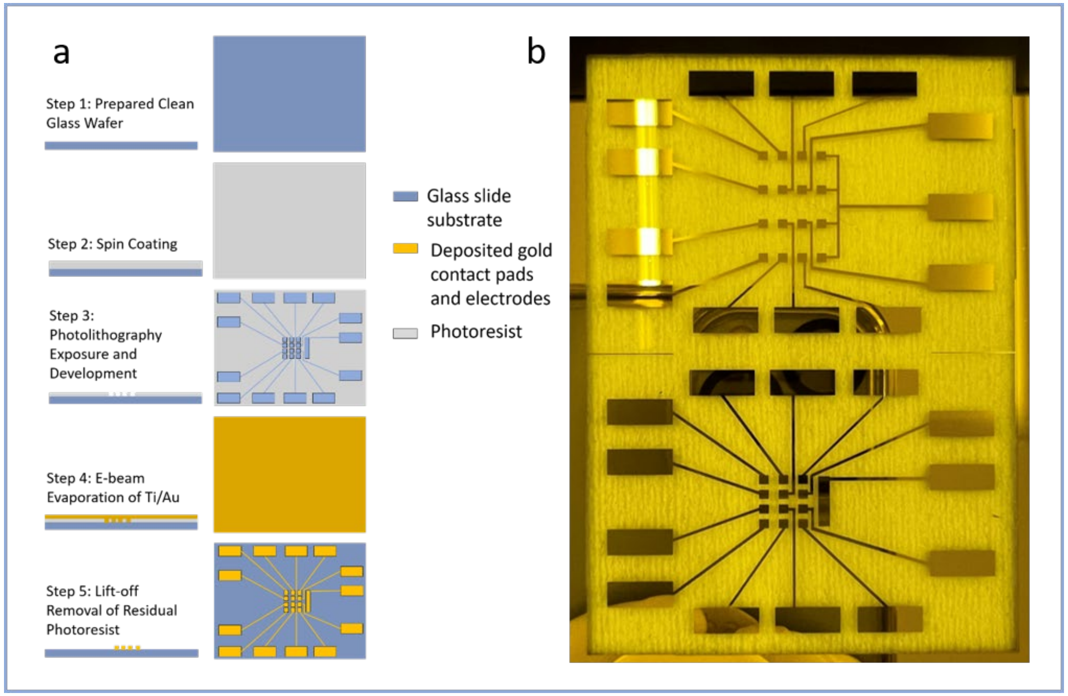
Development of Lab-on-a-Chip Platform for the Study of Extracellular Electron Transfer



(a) Developed fabrication process and (b) digital image of electrode chips with Ti/Au deposition.

This project aimed to develop a lab on a chip (LOAC) platform monitoring the microbial attachment and extracellular electron transfer processes of microorganisms in situ and in real time. The step-by-step process flow of the electrode chip design was completed to ensure consistent fabrication using photolithography and lift off methods shown in Figure 1a. Multiple electrode and flow configurations were also developed with examples shown in Figure 1b. Future work involves finalizing the device with enclosure to enable flow and perform preliminary testing for extracellular electron transfer processes between microorganisms and the deposited solid electrodes on the chip surface.

Mourin Jarin, Ting Wang, Xing Xie; School of Civil and Environmental Engineering, Georgia Institute of Technology
Work performed at Georgia Tech's Institute for Electronics and Nanotechnology.

This work was partially supported by an IEN Core Facility Seed Grant.

National Research Priority: NSF–Understanding the Rules of Life

National Nanotechnology Coordinated Infrastructure

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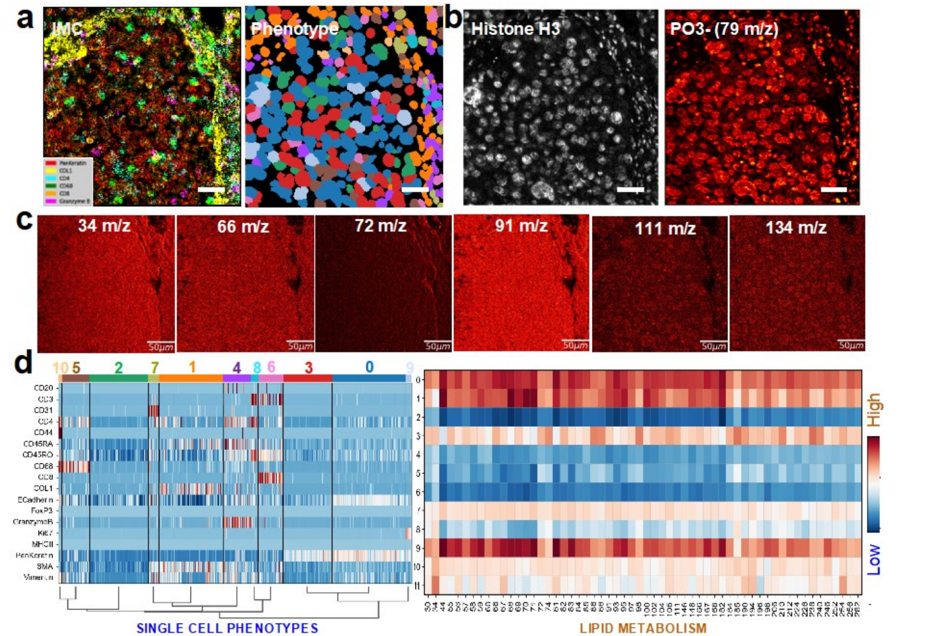
Correlative Spatial Metabolomics And Protein In Situ Imaging

This work aims to decipher spatially resolved metabolic profile at the single cell level by incorporating untargeted spatial metabolomics using (TOF-SIMS) and targeted multiplexed protein imaging (IMC). Human lung adenocarcinoma tissue were labeled by a 20-plex isotope-barcoded antibody library one-time to enhance the contrast of specific immune and cancer cell types then imaged using TOF-SIMS (IONTOF 5 GmbH, Münster, Germany) with a doubly charged 50-kV clustered (Bi3++) bismuth liquid metal ion gun to generate secondary ions from the sample surface, followed by identification of m/z of secondary ions by a TOF analyze. For depth profiling, a Cesium Ion gun (Cs+ ions, 2-kV energy, and microampere current) is used to iteratively sputter away very thin layers of tissue and image across 30-40 depth slices. IMC was used for multiplexed protein imaging for unsupervised single-cell phenotyping. Spatial protein and metabolites imaging modalities were registered using single-cell nuclei features with a cross-correlation metric



(a) IMC multiplex protein imaging (left) with corresponding unsupervised single-cell phenotypes (right) **(b)** Single cell nuclei registration by cross-correlation using Histone H3 protein marker (left) and 3D-SMF PO3- 79 m/z (right) **(c)** Spatial metabolomic profiling examples at different m/z channels **(d)** Comparison of unsupervised clustering of protein phenotype (left) with lipids expression level (right) at single-cell resolution

Thomas Hu, School of Electrical and Computer Engineering, Georgia Institute of Technology. Ahmet F. Coskun, Georgia Institute of Technology and Emory University. Work performed at Georgia Tech's Institute for Electronics and Nanotechnology.

This work was partially supported by an IEN Core Facility Seed Grant.

National Research Priority: NSF–Understanding the Rules of Life

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