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Surface Species in Graphene Liquid Cells for Transmission Electron Microscopy

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Availability of water for domestic, agricultural, and industrial use rests upon a foundation of scientific and engineering knowledge. While society currently investigates new technologies to increase supplies of clean water, water purification could become more efficient if it was further investigated at the molecular level. The initial stages of water purification focus on having the contaminant particles cluster within water together before being removed mechanically. The resulting water is then sent through membranes to be filtered. If particulates are not completely removed, the water sent through these membranes cannot be filtered properly and, therefore, is not safe for human consumption. Current treatments utilize trivalent salts to form clusters of charged particulate contaminants that are subsequently removed.

In this work, we will use gold (Au) nanoparticles suspended in liquids to model these interactions. The aim of this study is to quantify the aggregation of particles that takes place in the early stages of water purification by utilizing in-situ scanning transmission electron microscopy (STEM).[1,2] Since liquids cannot be placed directly into the high vacuum environment of a STEM, we utilize graphene-based liquid cells (GLCs) in order to characterize the nanoparticles within them (Figure 1). These GLCs contain small quantities of gold nanoparticles in solution sandwiched between two single layer graphene sheets (Figure 1). Although it is only a single atomic layer thick, the graphene prevents water from escaping into the vacuum chamber of the STEM. In addition, the low electron scattering of the graphene allows for the imaging of nanoparticles in the encapsulated water by STEM without much interference.

The preparation of GLCs requires graphene deposited on Cu substrates via chemical vapor deposition (CVD) to be freed from the Cu by wet chemical etching. The etching process often leaves undesirable surface species on the etched graphene. Depending upon the chemicals used for the etching process, the properties of the water-graphene interface could be altered by this contamination, which might influence the behavior of nanoparticles. While a small amount of this contamination is to be expected, we use energy dispersive x-ray analysis (EDX) and Raman spectroscopy to reveal the chemical species that have been absorbed onto the graphene film.

Raman spectra shown in Figure 2 of the liquid on graphene films demonstrate that the most prominent contaminants are silica (SiO_2), hydrocarbons (CH_2), and C_2H_5NO. The implementation of fabrication steps to reduce the level of contamination will be discussed and the effect of improved liquid purity will be demonstrated. Additionally, we will analyze the role that electrostatic interactions play in the aggregation and movement of gold nanoparticles within GLCs using STEM imaging. The goal is to develop an optimized approach for creating clean GLCs to better reveal the interactions of charged nanoparticles and their role in water purification. [4]

References:

[1] C. Wang et al., *Adv. Mat.*, **26(21)** (2014)
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[3] J. Jokisaari et al., Microscopy and Microanalysis, **23**, 878-879 (2017)

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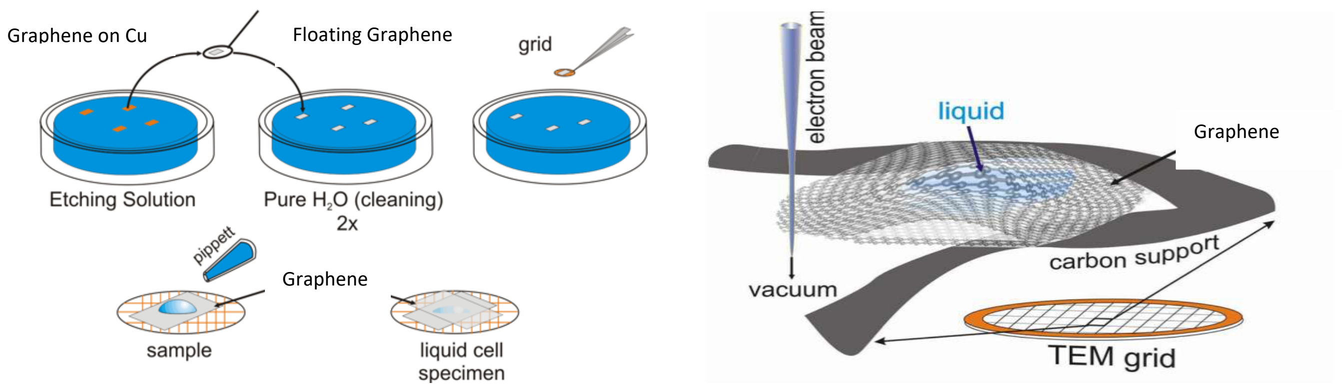
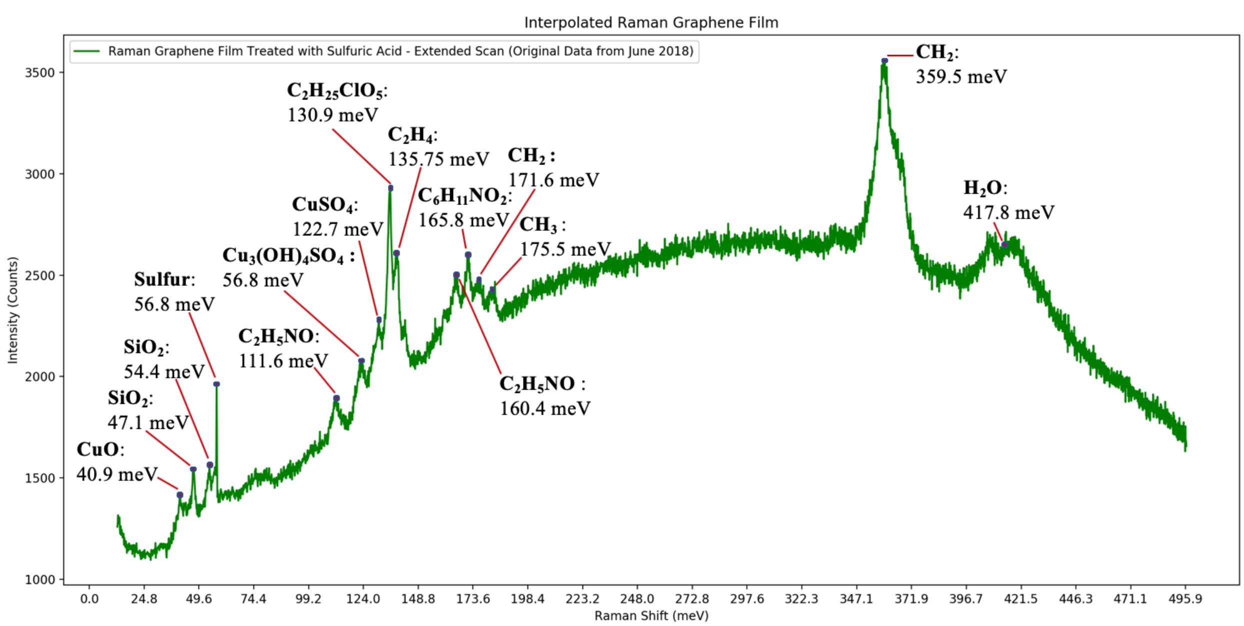


Figure 1: Process to create graphene liquid cells (left); Graphene liquid cell showing the liquid between two thin films (right) [3]



Figures 2: Smoothed Raman scans of a wet graphene film treated with sulfuric acid.

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