TEM AND SEM SPECTROSCOPY OF as-SYNTHESIZED LEAD SULFIDE QUANTUM DOT (PbS QD)

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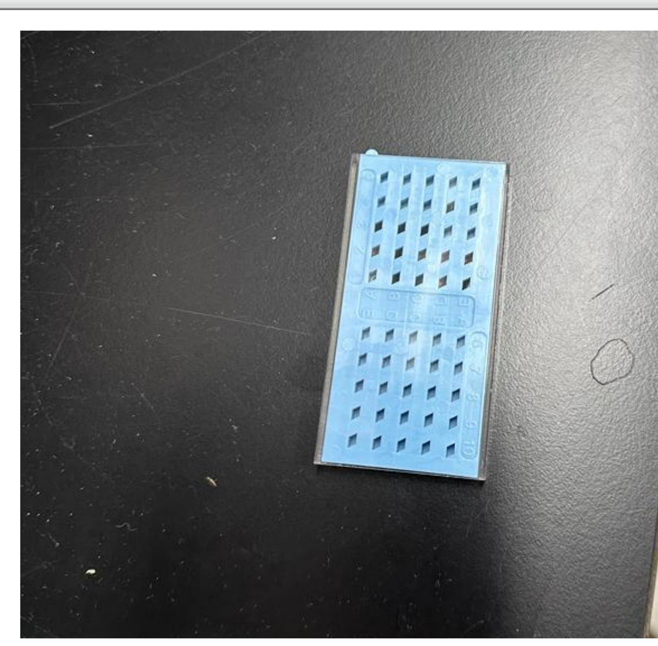
INTRODUCTION

Quantum dots (QD) have emerged as promising synthetic materials for applications in optoelectronic devices, solar cells, bioimaging, etc. due to their size-dependent energy band structure (Jiao et al., 2012). Lead (II) Sulfide PbS quantum dots offer a tunable band gap in the near-infrared region essential biological cells simulations. The Near-infrared (NIR) is between 800-1550nm (Hong et al., 2017)

SAMPLE PREPARATIONS

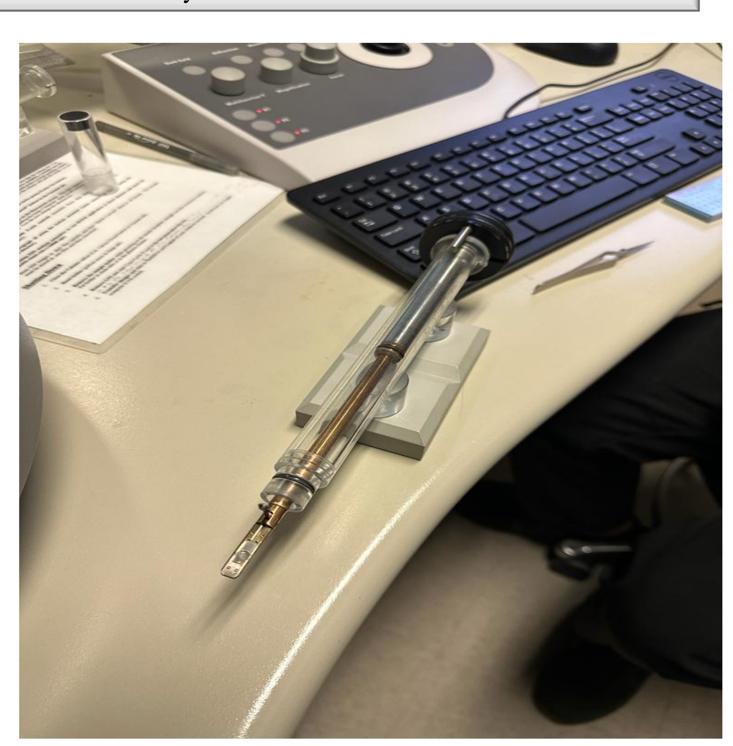
The as-synthesized lead sulfide (PbS) quantum dots (QD) were deposited on a lazy carbon grid and dried.
Liquid nitrogen was used to cool down the TEM detector, this traps charges generated by the detector, so we recorded only signals from the sample. Following the protocol, the sample was gently placed on the sample holder and inserted into the TEM compusstage.
The electromagnetic lenses were adjusted so the beam came in focus on the sample. Images were taken at 20nm, and 5nm magnification.

Lastly, we performed an elemental analysis with both TEM and SEM techniques.
For the SEM techniques, 40 microliters of the as-synthesized PbS QD were deposited on a silicon grid and desiccated for 2hrs. Part of the sample was sputter coated with platinum and the rest uncoated before the SEM imaging. The various elements in the sample are displayed in the spectrum.



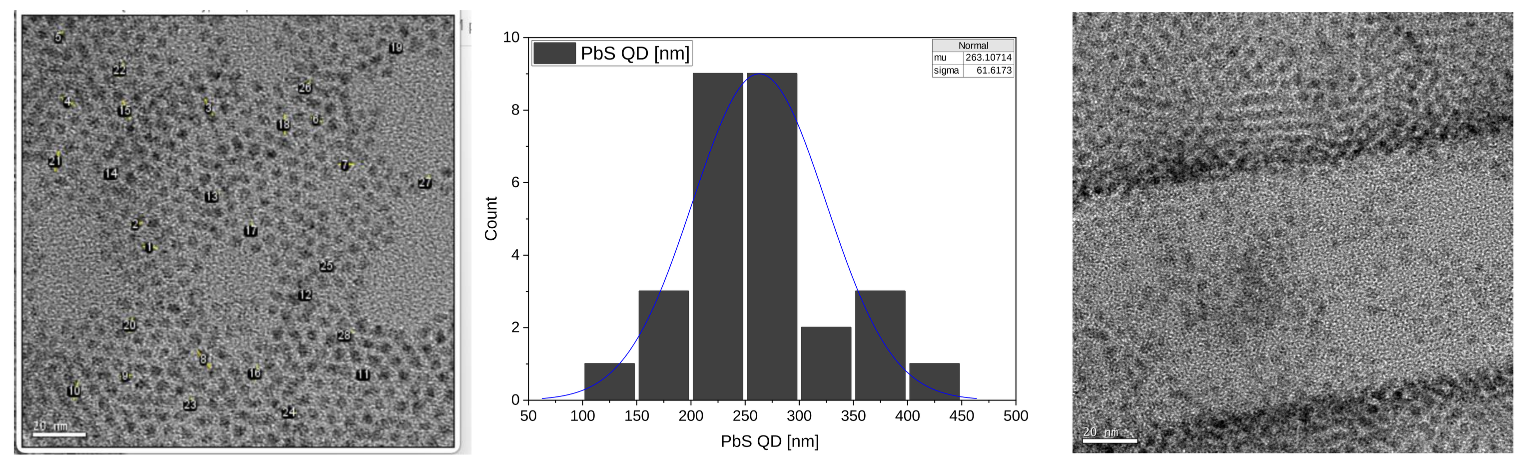
TRANSMISSION ELECTRON MICROSCOPE

High-resolution Transmission electron microscope (HR TEM), Thermo Scientific TEM, and Zeiss-Leo DSM982 SEM of the Institute of Optics (Willmot 206a) at the University of Rochester were used for the analysis.



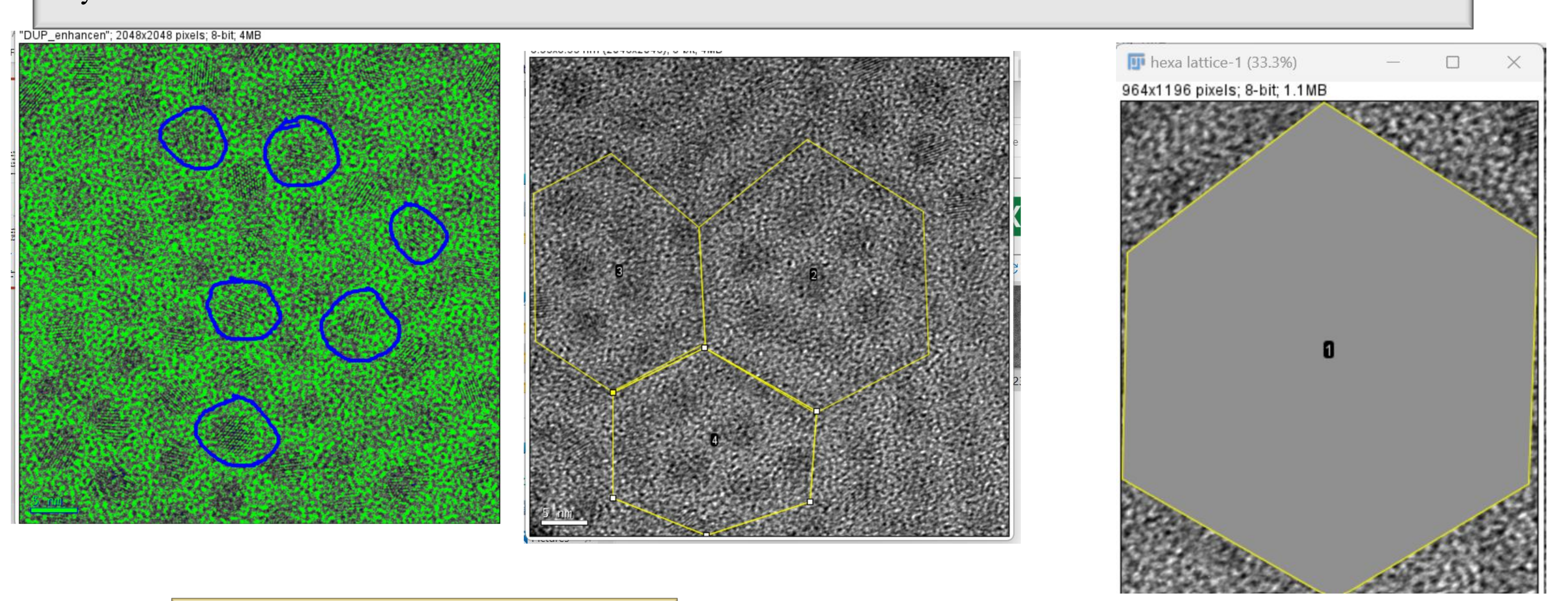
PARTICLE SIZE DISTRIBUTIONS

Under 20nm magnification and an accelerating voltage of 200kV in TEM, we observe a cluster of monodisperse PbS QD on the lazy carbon grid. The image looks pretty good because the sizes of the dots are uniformly distributed, and it shows we did a good job in the synthesis (i.e the right amount of secondary phosphine was used, and more reaction time was allowed to equilibrate).



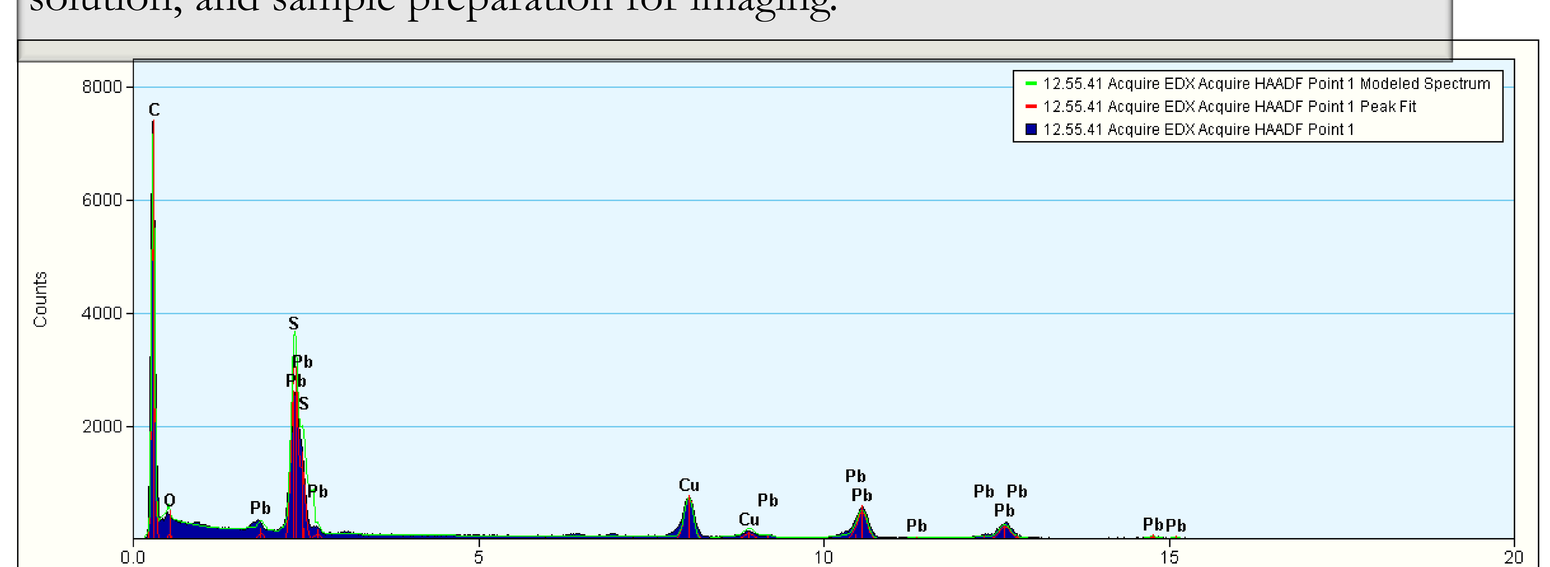
SUPERLATTICE

Under 5nm magnification, we observe superlattice. At this magnification, most of the crystal planes diffract the electron beam giving orderly arranged lattices. More interestingly, we observe a hexagonal crystal structure with approximately 7-8 dots in each unit cell, this depicts the structure of lead sulfide quantum dots. This data gives us an intuition of the material we are working with and its crystal information.



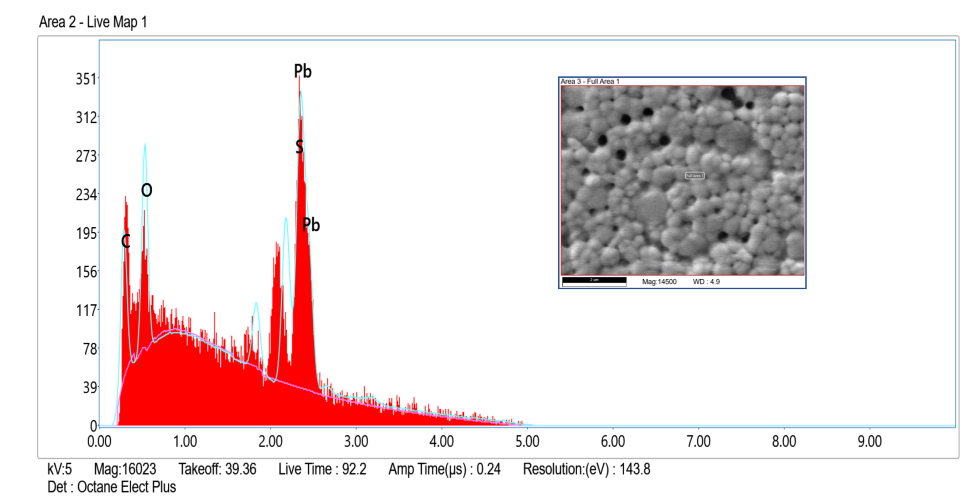
TEM EDS

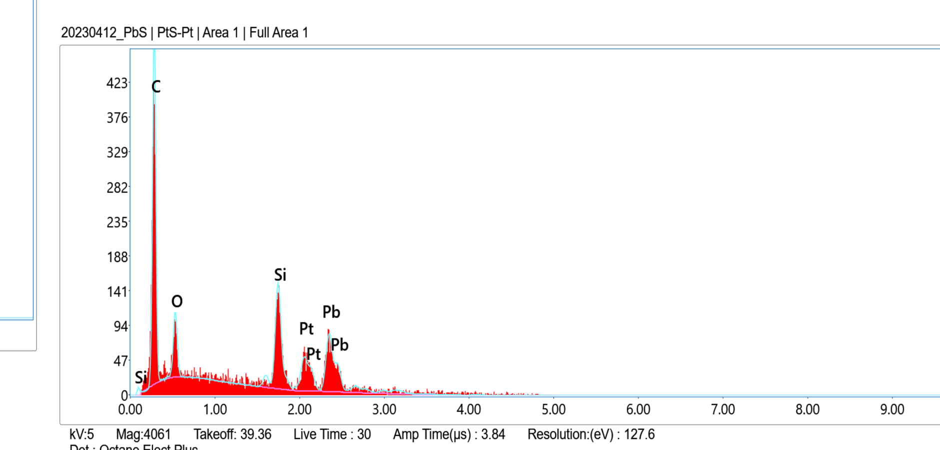
Elemental quantification with the HR TEM gave a clear peak distribution with the various energies. The results agree with the SEM data, but we observed copper (Cu) here because of the back-coated lazy carbon grid with Cu. Due to the higher energy of the electron beam and the thin nature of the sample size, Cu peaks were captured.
These elements are expected because the main components of our PbS QD are lead (Pb), and sulfur (S) with the other associated elements from the solution, and sample preparation for imaging.



SEM EDS

The elemental analysis data with SEM EDS at 2 microns and a working distance of 4.9 mm, shows the K-series energy spectra of each element with its count. The peak of lead overlaps with that of sulfide, due to their energy similarity. The high atomic number of lead (Pb) makes its peak more pronounced than sulfur. Other elements observed are oxygen and carbon. Oxygen came from the lead (II) oxide or oleic acid used, and carbon may be from the oleic acid. Also, we see platinum and silicon in the spectra. Silicon came from the grid used and platinum from the coated part of the sample.





CONCLUSION

With the HR TEM, we are able to observe the shape of our QD and its monodispersity in the solution. The uniformly distributed dots on the carbon grid of nearly equal sizes indicates we used the right amount of phosphine, and reaction time and generally have control of the synthesis and dots size. The SEM and TEM EDX data show that our QD is not contaminated, and the elements observed are expected. Pb and oxygen (O) came from the lead (II) oxide, Sulfur came from the secondary phosphine, and silicon and platinum came from the grid and coating respectively.

RECOMMENDATION

XRD technique could give us more structural information, lattice parameter, and crystallographic orientation of our dots along with planes. In order to analyze a single dot, we could make a more diluted solution.

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