Exploring the Non-Classical Crystallization Pathways of Cobalt Hydroxide Carbonate
Isabelle Lee, Class of 2025

Working alongside Professor Ortoll-Bloch and my labmate Gabriel Bloom-O'Sullivan for this summer research project, our goal as inorganic chemists was to gain a clearer understanding of the crystallization processes of cobalt oxide and its precursors, such as cobalt hydroxide carbonate. Developing a more robust understanding of the crystallization of cobalt hydroxide carbonate and cobalt oxide and further investigating the relationship between the synthesis and crystal structure and properties would have great implications for a broad range of technological applications.[1] Cobalt oxide is used in a wide variety of scientific and industrial processes, such as for clean energy conversion and storage,[2] pigments and dyes,[3] and modification of the rate of certain electrochemical and degradation reactions.[4] The traditional approach to synthesizing cobalt oxide is energy-intensive and costly, as relatively high temperatures are required to synthesize the cobalt oxide product. However, new research shows that cobalt hydroxide carbonate, a precursor to cobalt oxide, can be synthesized at room temperature.[5] This new synthesis method would be highly energy-efficient and cost-effective, but there are remaining gaps in our understanding of how cobalt hydroxide carbonate crystals form when grown via this method. Thus, broadly, our research focused on understanding how cobalt hydroxide carbonate crystals form at room temperature and how this precursor transforms to cobalt oxide via heating. Through our research, we hoped to obtain a better understanding of the new synthesis method and how adjusting the synthesis conditions may affect the crystal properties of the cobalt oxide product, such as size, shape, and assembly, influencing the performance of the resulting cobalt oxide product within various scientific and industrial applications. Our findings will help optimize the synthesis to predictably and reproducibly achieve cobalt hydroxide crystals with the desired properties, creating materials with improved performances.

The first few weeks of our research were dedicated to setting up the physical lab space and the instrumentation for the lab, including the spin coater and X-ray diffractometer (XRD). The XRD is a powerful instrument that identifies and characterizes the structure of crystalline substances, and it was one of the main scientific instruments used for the inorganic project. In addition to the XRD, the scanning electron microscope (SEM) was used to characterize the surface morphology of crystalline substances, producing highly detailed images of surface crystal morphology. Reagents were also ordered during the beginning of the research based on close readings of scientific literature published on non-classical crystallization of metal oxides and metal carbonates, as we intended to replicate or create hybridized experimental procedures that involved the use of the same reagents in literature. For additives, Fast Green FCF, Brilliant Blue R, and amaranth were ordered and added to crystal growth solutions to determine how adding dyes and pigments could impact crystal growth and structure. Cobalt hydroxide carbonate growth replications of Chemistry Honors student Zubin Kenkare, '23, were also created.

Faculty Mentor: Professor Amnon Ortoll-Bloch
Funded by the: Robert Freedman '87, P'17 and Anne Cirillo P'17 Student Fellowship

References

1. Khalil, A. T.; Ovais, M.; Ullah, I.; Ali, M.; Shinwari, Z. K.; Maaza, M. Physical Properties, Biological Applications and Biocompatibility Studies on Biosynthesized Single Phase Cobalt Oxide (Co_3O_4) Nanoparticles via Sageretia Thea (Osbeck.). *Arabian Journal of Chemistry* **2020**, 13 (1), 606–619. https://doi.org/10.1016/j.arabjc.2017.07.004.
2. Su, D.; Dou, S.; Wang, G. Single Crystalline Co_3O_4 Nanocrystals Exposed with Different Crystal Planes for Li-O_2 Batteries. *Sci Rep* **2014**, 4 (1), 5767. https://doi.org/10.1038/srep05767
3. Xia, Y.; Xi, N.; Huang, J.; Wang, N.; Lei, Y.; Fu, Q.; Wang, W. Smalt: An under-Recognized Pigment Commonly Used in Historical Period China. *Journal of Archaeological Science* **2019**, 101, 89–98. https://doi.org/10.1016/j.jas.2018.11.008.
4. Xie, X.; Li, Y.; Liu, Z.-Q.; Haruta, M.; Shen, W. Low-Temperature Oxidation of CO Catalysed by Co_3O_4 Nanorods. *Nature* **2009**, 458 (7239), 746–749. https://doi.org/10.1038/nature07877.
5. Schenk, A. S.; Goll, M.; Reith, L.; Roussel, M.; Blaschkowski, B.; Rosenfeldt, S.; Yin, X.; Schmahl, W. W.; Ludwigs, S. Hierarchically Structured Spherulitic Cobalt Hydroxide Carbonate as a Precursor to Ordered Nanostructures of Electrocatalytically Active Co_3O_4. *Crystal Growth & Design* **2020**, 20 (10), 6407–6420. https://doi.org/10.1021/acs.cgd.0c00576.