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Synthesis, Modification, and Characterization of Hydroxide-Derived Nanomaterials

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Abstract

Discovered in 2020, quaternary ammonium hydroxide, quats, derived nanomaterials, or QDNs, are a family of easily synthesized, low-dimensional, transition metal oxides. Typically, QDNs are formed from a combination of transition metal precursor mixed with a highly concentrated, symmetric quat solution, reacted at standard pressure and almost ambient temperatures. Most of the work featured herein focuses on expanding the options for synthesizing members of this family of materials.

A significant amount of the effort in this thesis is on the two-dimensional, 2D, Mn-based, MnO_2 oxide with the birnessite structure (δ-MnO_2), henceforth referred to as QDBs. This investigation is primarily driven by the desire to increase the safety around QDNs by moving away from tetramethylammonium hydroxide, TMAOH, as the primary reactant. There were found successes synthesizing QDB with other symmetric quat hydroxides, but not with any hydroxide, leading to questions on the requirements necessary to form QDB. These questions led to successes in synthesizing birnessite with inexpensive, widely available, non-toxic, Mn_3O_4, in combination with simpler hydroxides, through the addition of an oxidizing agent. Also studied is QDB's ability to be easily ion-exchange cations as other birnessites are known to do. Through ion exchange QDB can find application in dye adsorption, outperforming other forms of birnessite by more than a factor of five, and in biological situations after exchanging the hazardous TMA cation with a biocompatible one, such as Li.

This work continues by investigating the reaction between TiB_2 and TMAOH that results in 1D lepidocrocite, 1DL. This study systematically examined the reaction mechanism for 1DL and determined the importance of carbon dioxide to the reaction kinetics. It also concludes that in the case of TiB_2, 1DL can be synthesized with much lower concentrations of TMAOH than previously used, creating a material that is functionally, structurally, and optically identical to the standard 1DL, but with a different morphology.