Synthesis, Characterization and Voltammetric Study of Dimethylammonium Lead Halide Perovskites

Jeta Sela[a]
Leon Stojanov,[b] Besarta C. Ramadani,[a] Miha Bukleski,[b] Arianit A. Reka,[a] Valentin Mirceski[b, c] and Slobotka Aleksovska[b*]
[a]*Department of Chemistry, Faculty of Natural Sciences and Mathematics, University of Tetovo, Tetovo, North Macedonia*
[b]*Institute of Chemistry, Faculty of Natural Sciences and Mathematics, Ss. Cyril and Methodius University, Skopje, North Macedonia*
[c]*Research Center for Environment and Materials, Macedonian Academy of Sciences and Arts, Bul. Krste Misirkov 2, 1000 Skopje, Republic of N. Macedonia*
bote@pmf.ukim.mk

Over the past decade, hybrid organic-inorganic perovskites (HOIPs) have attracted significant attention for their optoelectronic properties and potential application in photovoltaics, leading to ongoing exploration and detailed study of both new and existing HOIPs. This research focuses on the synthesis, comprehensive characterization, and cyclic voltammetric study of hybrid lead halide perovskites with the formula $[(CH_3)_2NH_2]PbX_3$, ($DMAPbX_3$), where X represents I^-, Br^-, or Cl^-. A slightly modified synthesis method from literature was employed, using stoichiometric amounts of lead halides (PbX_2) and dimethylammonium halides (DMAX) dissolved in acetonitrile [1] or *N,N*-dimethylformamide [2]. Controlled evaporation yielded $DMAPbI_3$ as a yellow crystalline powder, while $DMAPbBr_3$ and $DMAPbCl_3$ formed colorless hexagonal-like and needle-like crystals, respectively. X-ray powder diffraction (XRPD) confirmed distinct perovskite structures with unique lattice parameters for each halide, while scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDX) revealed well-defined morphologies and homogeneous elemental distribution. IR and Raman spectroscopy revealed characteristic vibrational features and reflected differences in chemical bonding and structural dynamics for each halide variant. Cyclic voltammetry (CV) studies of $DMAPbX_3$ perovskites performed in dichloromethane (DCM) with tetrabutylammonium chloride (TBAC) or tetrabutylammonium perchlorate (TBAPC) as electrolytes, using a paraffin-impregnated graphite electrode (PIGE), demonstrated significant variations in redox behavior due to different halides. The results showed that $DMAPbX_3$ perovskites are electrochemically active, with oxidation (and reduction) currents decreasing in subsequent scans, indicating partial degradation of the perovskite structure.

Keywords: dimethylammonium lead halide perovskites, cyclic voltammetry, XRPD, vibrational spectroscopy, SEM-EDX

References

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