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Title: Supramolecular Framework-Driven Electrical Conductivities and Hydrogen Evolution Activities of

Hybrid Nickel(II)-Cerium(IV) Complex Salts Cooperativity

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**Evolution reactions** 

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Abstract:

This work highlights the design, synthesis, structural characterization, electrical conductivities, and hydrogen evolution activities of a new pair of hybrid 3d(Ni)-4f(Ce) block metal complex salts, [Ni(phen)2(NO3)]2[Ce(NO3)6] (1) and [Ni(bpy)3][Ce(NO3)6][Ce(NO3)2(H2O)5]NO3 (2) containing phen and bpy ligands; [phen = 1,10-phenanthroline and bpy = 2,2'-bipyridine]. Crystal structural analysis divulges that complex salt 1 adopts two units of monocationic Ni(II) complex with one dianionic Ce(IV) complex unit while complex salt 2 exists in an association of one unit of dicationic Ni(II) complex with two complex units of Ce along with a counteranionic nitrate. The Ni(II) ions exist in distorted bicapped square pyramidal coordination geometry, while the Ni(II) center in 2 exists in an octahedral geometry. The cerium ion in 1 exists in dodecahedron geometry while the first and second Ce ions hold dodecahedral and tricapped trigonal prism coordination geometries, respectively. Supramolecular interactions reveal that predominant nonclassical forces like O $\cdots$ H,  $N \cdots H$ ,  $\pi \cdots \pi$ ,  $O \cdots \pi$ , and  $O \cdots O$  are interactive to shape highly ordered crystalline frameworks. Complex salt 2 exhibits a unique formation of the supramolecular cage-type framework by the cerium complex units, leading to the inclusion of Ni(II)-complex units into the supramolecular cages. The complex salts (1, 2) were employed to fabricate the Schottky devices to unveil the fate of the hybrid salts in charge transport applications. Carrier mobility  $(\mu)$  for 1 and 2 were determined as  $3.02 \times 10$ -6 and  $8.022 \times 10$ -5 m2 V-1 s-1 with respective transit time( $\tau$ ) of  $2.60 \times 10$ 10–7 and  $9.67 \times 10-9$  s attributing the excellent candidature of complex salt 2 in transport properties. The hybrid salts were also found to be highly active electrocatalysts for proton reduction in 1 M aqueous KOH solution at room temperature. The overpotential values of 1 and 2 were determined to be 730 mV and 687 mV at a current density of 10 mA cm-2 with 0.081 s-1 and 0.225 s-1 as turnover frequencies. The supramolecular interactions-driven crystalline framework sheds light on the electrical conductivities and casting the hydrogen evolution activities for the newly designed hybrid d-f type complex salts.

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