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POWDER X-RAY DIFFRACTION STUDY OF A ONE-DIMENSIONAL CADMIUM(II) ISONICOTINATE COORDINATION POLYMER

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Abstract
A one-dimensional cadmium(II) coordination polymer based on the isonicotinate ligand was investigated by powder X-ray diffraction (PXRD). The compound, formulated as catena-poly[(μ_2-isonicotinato-κ^2O,O′:κN)(κ^2-O,O′-nitrato)(diaqua)cadmium(II)]·(N,N-dimethylamine), belongs to the class of one-dimensional coordination polymers. Powder X-ray diffraction analysis was performed to evaluate the phase purity and structural integrity of the synthesized material. The experimental PXRD pattern was compared with the simulated pattern generated from single-crystal X-ray diffraction data. A good agreement between the positions of the major diffraction peaks in the experimental and simulated patterns confirms the successful synthesis of the target phase. Minor differences in peak intensities can be attributed to preferred crystal orientation, particle size effects, and sample preparation conditions. Furthermore, the absence of additional diffraction peaks indicates that no detectable crystalline impurities are present in the sample. The sharp and well-defined reflections observed in the diffraction pattern also suggest a high degree of crystallinity of the synthesized material. The results demonstrate that the bulk material possesses the same crystal structure as that determined from single-crystal analysis and confirm the phase purity and crystallographic homogeneity of the obtained coordination polymer.

Keywords: Cadmium(II), isonicotinate, coordination polymer, catena-poly, PXRD, crystal structure.

Introduction

Coordination polymers derived from pyridine carboxylate ligands have attracted considerable attention because of their diverse structural architectures and promising applications in catalysis, gas storage, molecular recognition, luminescent materials, and crystal engineering. The structural versatility of these compounds arises from the ability of pyridine carboxylate ligands to coordinate metal ions through multiple donor atoms, resulting in the formation of extended networks with different dimensionalities and topologies. Such materials have become an important class of functional crystalline solids owing to their tunable structural and physicochemical properties. Among pyridine carboxylate ligands, isonicotinate (4-pyridinecarboxylate) is particularly important because it contains both pyridine nitrogen and carboxylate oxygen donor atoms. This dual coordination functionality enables the ligand to act as an effective bridging linker between adjacent metal centers and facilitates the construction of one-dimensional, two-dimensional, and three-dimensional coordination architectures. In addition, the rigid aromatic backbone of the isonicotinate ligand often contributes to the formation of stable crystalline frameworks through coordination bonding and supramolecular interactions. Cadmium(II) ions are widely employed in coordination chemistry due to their flexible coordination environments, variable coordination numbers, and ability to generate structurally diverse coordination polymers. The combination of Cd(II) ions with isonicotinate ligands

frequently leads to the formation of extended polymeric structures with interesting structural and physicochemical properties. Therefore, cadmium-isonicotinate systems continue to attract interest as model compounds for studying coordination polymer assembly and crystal engineering principles.

Results and Discussion

The PXRD pattern of the synthesized cadmium(II) isonicotinate coordination polymer was recorded in the 2θ range of 10-40° and compared with the simulated pattern generated from single-crystal X-ray diffraction data. As shown in Figure 1, the experimental diffraction pattern exhibits excellent agreement with the simulated profile, indicating that the bulk material corresponds closely to the crystallographic phase determined from single-crystal analysis. The most intense reflections observed in the experimental pattern are located in approximately the same angular positions as those in the simulated diffractogram. In particular, the characteristic reflections indexed as (002), (200), (210), (021), (212), (221), and several higher-angle reflections show good correspondence with the calculated pattern. This agreement confirms that the crystal structure obtained from single-crystal X-ray diffraction is representative of the bulk sample and demonstrates the successful formation of the target cadmium(II) isonicotinate coordination polymer. Although some variations in relative peak intensities are observed between the experimental and simulated patterns, such differences are commonly encountered in coordination polymers and polycrystalline materials. These discrepancies may arise from preferred orientation of crystallites, differences in crystallite size distribution, sample packing effects, and instrumental parameters during data collection. The intensity variations do not significantly affect the overall correspondence between the two diffraction patterns and therefore do not indicate structural changes or phase transformation.

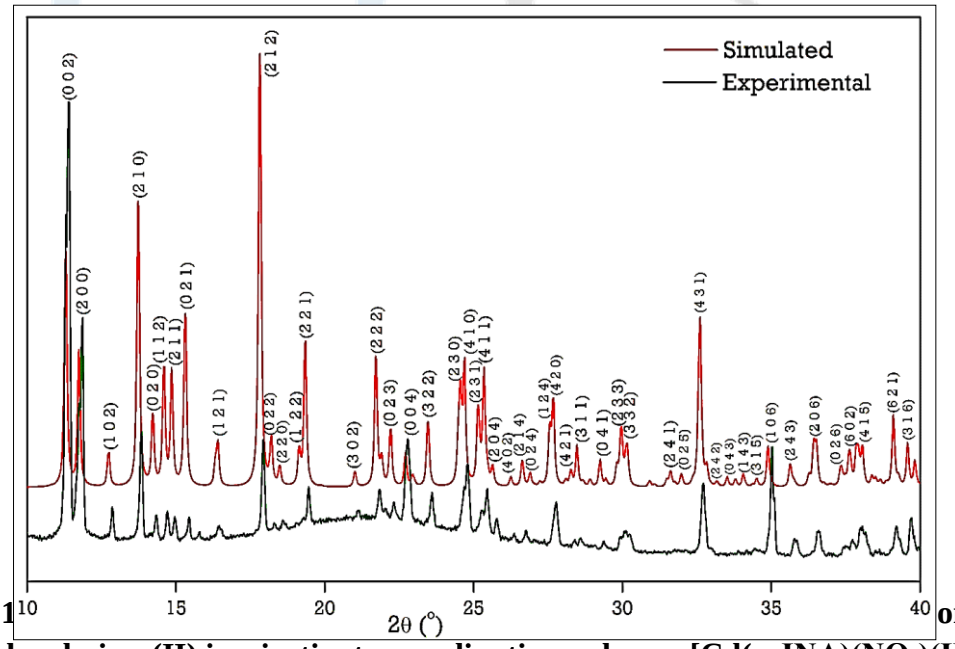

Figure 1 one-dimensional cadmium(II) isonicotinate coordination polymer $[Cd(\mu\text{-}INA)(NO_3)(H_2O)_2]_n$.

Furthermore, the diffraction peaks are relatively sharp and well-defined, suggesting a high degree of crystallinity in the synthesized material. The absence of broad amorphous halos indicates that the sample possesses predominantly crystalline character. Importantly, no significant additional diffraction peaks attributable to secondary crystalline phases or synthetic by-products were

detected throughout the investigated diffraction range. This observation demonstrates the high phase purity of the obtained coordination polymer. The close agreement between the experimental and simulated PXRD patterns provides strong evidence that the bulk material retains the same structural arrangement as the crystallographically characterized single crystal. Therefore, PXRD analysis confirms both the structural integrity and crystallographic homogeneity of the synthesized cadmium(II) isonicotinate coordination polymer. These findings further support the successful synthesis of a pure and highly crystalline one-dimensional coordination polymer suitable for subsequent physicochemical investigations.

Conclusion

Powder X-ray diffraction analysis confirmed the successful synthesis of a one-dimensional cadmium(II) isonicotinate coordination polymer formulated as catena-poly[(μ_2-isonicotinato-κ^2O,O′:κN)(κ^2-O,O′-nitrato)(diaqua)cadmium(II)]·(N,N-dimethylamine). Comparison of the experimental PXRD pattern with the simulated pattern derived from single-crystal X-ray diffraction data revealed excellent agreement in the positions of the major diffraction peaks, demonstrating that the bulk material possesses the same crystal structure as that determined crystallographically.

The observed diffraction peaks were sharp and well-defined, indicating a high degree of crystallinity of the synthesized material. Minor differences in peak intensities between the experimental and simulated patterns are attributed to preferred orientation effects, crystallite size distribution, and sample preparation conditions rather than structural variations. Furthermore, the absence of additional diffraction peaks associated with secondary phases confirms the high phase purity and crystallographic homogeneity of the obtained compound.

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