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Research Article

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Posted Date: March 11th, 2026

DOI: <https://doi.org/10.21203/rs.3.rs-8946492/v1>

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Additional Declarations: No competing interests reported.

Synthesis and Structural Elucidation of Co(II) and Mn(II) Coordination Polymers Derived from Isonicotinic Acid

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Abstract

The solvothermal reaction of isonicotinic acid (INA) with Co(II) and Mn(II) afforded isostructural coordination polymers of formula $[M_2(INA)_4]$ ($M = Co, Mn$). The compounds were characterised by FT-IR spectroscopy, elemental (CHN) analysis, solubility studies, magnetic measurements, powder X-ray diffraction and single-crystal X-ray diffraction unit cell determination for the cobalt(II) complex. Infrared spectroscopic data confirmed deprotonation of the isonicotinic acid ligand and coordination through both carboxylate oxygen and pyridyl nitrogen donor atoms. The free ligand, showed absorption bands at 1695 cm^{-1} and 1289 cm^{-1} assigned to $C=O$ and $C-O$ stretching vibrations. Upon coordination, the $C=N$ band shifted from 1580 cm^{-1} in the free ligand to 1560 cm^{-1} in the Co(II) complex and to 1559 cm^{-1} in the Mn(II) analogue, with new absorptions near 500 cm^{-1} and 400 cm^{-1} corresponding to $M-N$ and $M-O$ vibrations. The coordination polymers exhibit low solubility in common solvents, consistent with their extended polymeric structures. Magnetic susceptibility measurements revealed effective magnetic moments of 5.81 BM for the manganese(II) polymer and 4.53 BM for the cobalt(II) polymer, indicative of high-spin octahedral metal centres. PXRD analysis showed the disappearance of characteristic ligand reflections and the appearance of new peaks associated with the coordination polymers, confirming the formation of new crystalline phases. Unit cell determination for the cobalt(II) compound indicated a monoclinic system with space group $P2_1$, ($a = 9.818\text{ \AA}$, $b = 12.804\text{ \AA}$, $c = 10.801\text{ \AA}$) consistent with a reported structure (CCDC 886304).

Keywords Coordination polymers, Isonicotinic acid, Solvothermal, DMF, FT-IR

1. Introduction

Coordination polymers represent an important and rapidly developing class of inorganic–organic hybrid materials [1-3]. Their continued popularity arises from the remarkable diversity of structures they can adopt and from the wide range of properties they may exhibit, making them attractive for applications such as gas storage and separation, catalysis, magnetism, and chemical sensing [4-7]. Structurally, coordination polymers are constructed from metal ions linked by organic ligands into extended one-, two-, or three-dimensional networks, with their final architectures being largely determined by the coordination behaviour of the metal centres and the binding characteristics of the ligands [8].

Organic ligands containing both nitrogen and oxygen donor atoms have been particularly successful in the design of coordination polymers. Among these, heteroaromatic carboxylic acids occupy a prominent position due to their rigidity and versatility [9]. Isonicotinic acid

(pyridine-4-carboxylic acid) is especially notable, as it combines a pyridyl nitrogen atom with a carboxylate group within a single molecular framework [10]. This dual N,O-donor nature allows isonicotinic acid to coordinate to metal centres in multiple ways, often acting as a bridging ligand that promotes the formation of extended polymeric networks through simultaneous metal–nitrogen and metal–oxygen interactions [11].

Cobalt(II) and manganese(II) ions are frequently employed in coordination polymer synthesis because of their flexible coordination preferences and well-defined electronic properties. Cobalt(II) ions are known to adopt a variety of coordination geometries and often give rise to complexes with interesting magnetic and spectroscopic behaviour [12-13]. Manganese(II), on the other hand, typically exists in a high-spin d^5 configuration and readily forms stable coordination compounds and extended frameworks. The use of these metal ions in combination with multifunctional ligands such as isonicotinic acid therefore provides an effective approach for generating coordination polymers with distinct structural features [14].

Although a considerable number of isonicotinate-based coordination polymers have been reported in the literature, direct comparisons between polymers derived from different first-row transition metals under similar synthetic conditions are still relatively limited. In particular, systematic studies involving cobalt(II) and manganese(II) remain useful for clarifying how the nature of the metal ion influences coordination modes, framework dimensionality, and overall structural organisation.

In this study, we report the synthesis and structural elucidation of cobalt(II) and manganese(II) coordination polymers derived from isonicotinic acid. The compounds were prepared under controlled experimental conditions and characterised using spectroscopic and analytical techniques to establish their coordination environments and polymeric nature. The discussion focuses on the coordination behaviour of isonicotinic acid and the role of the metal centres in directing the formation of the extended frameworks. This work adds to the growing body of knowledge on isonicotinate-based coordination polymers and provides further insight into their rational design.

2. Experimental Section

2.1 Materials and Reagents

All chemicals used in this research work were analytical grade and were used as received without further purification. Isonicotinic acid was used as the organic linker, while cobalt(II) chloride hydrate and manganese(II) chloride hydrate served as the metal precursors. N,N-Dimethylformamide (DMF) was employed as the reaction solvent for the solvothermal synthesis.

2.2 Physical measurements

Samples were weighed using an electric meter balance model AB 54. IR spectral analysis was recorded using a Perkin-Elmer FT-IR spectrophotometer in the range 4000 – 400cm⁻¹. Magnetic susceptibility measurements were carried out on a Sherwood Scientific Auto Magnetic susceptibility balance. PXRD measurements were carried out on a PAN analytical Empyrean X'Pert Pro X-ray diffractometer. Copper was used as the X-ray source with a wavelength of 1.5405 Å. Elemental microanalysis (C, H, and N) was determined using a Perkin-Elmer CHNS/O 2400 series II elemental analyser. Single crystal X-ray diffraction was performed on a Bruker Kappa APEX2 diffractometer with a MoK α (λ = 0.71073 Å) I μ S microfocus source at 293 K. the single crystal sample was kept in a small volume of the mother liquor prior to data collection.

2.3 Synthesis of the coordination polymers

Coordination polymers were synthesised by dissolving isonicotinic acid (INA) (3 mmol, 0.369 g) and metal salts (1.5 mmol; MnCl₂·4H₂O, 0.1979 g; CoCl₂·6H₂O, 0.2379 g) in 15 mL of N,N-dimethylformamide (DMF). The mixture was stirred for 10 min and transferred into a 25 mL Teflon-lined stainless-steel autoclave. The reaction mixture was heated from room temperature to 170 °C in 1 h ($\approx$ 2.4 °C min⁻¹) and maintained at this temperature for 72 h under autogenous pressure, followed by slow cooling to room temperature ($\approx$ 0.35 °C min⁻¹).

The resulting coloured crystals were collected by filtration, washed with fresh DMF, and subjected to solvent exchange in methanol for three days with six solvent replacements. The samples were finally activated under dynamic vacuum at 150 °C for 72 h.



Scheme 1: Synthesis of [M₂(INA)₄]_n

3 Results and discussion

3.1 Physical properties

The solvothermal reaction of isonicotinic acid with Co^{2+} and Mn^{2+} cobalt(II) afforded crystalline coordination polymers with distinct physical characteristics. The manganese(II) compound, formulated as $[\text{Mn}_2(\text{INA})_4]$, was obtained as colourless crystalline solids, whereas the cobalt(II) analogue, $[\text{Co}_2(\text{INA})_4]$, crystallised as sinopia-coloured crystals. The difference in colour is consistent with the electronic configurations of the metal centres, with the colourless appearance of the Mn(II) compound reflecting the spin-forbidden d–d transitions of high-spin d^5 Mn^{2+} ions, while the Co(II) compound exhibits visible absorption arising from allowed d–d transitions typical of high-spin Co^{2+} systems.

The isolated yields of the coordination polymers were moderate, amounting to 45.8% and 52.3% for the Mn(II) and Co(II) polymers respectively. These yields are comparable to those commonly reported for solvothermal syntheses of coordination polymers and are indicative of efficient framework formation under the applied reaction conditions.

Table 1 Physical properties of the coordination polymers

Compound	Appearance	% Yield	Decomposition temperature (°C)
$[\text{Mn}_2(\text{INA})_4]$	Colouless	45.8	345
$[\text{Co}_2(\text{INA})_4]$	Sinopia	52.3	323

Mn(II) coordination polymer exhibited a decomposition temperature of 345 °C, while the Co(II) analogue decomposed at 323 °C. The relatively high decomposition temperatures observed for both compounds suggest the formation of robust polymeric frameworks stabilised by extended metal–ligand connectivity. The slightly enhanced thermal stability of the Mn(II) polymer may be attributed to differences in metal–ligand bonding strength and framework packing when compared with the Co(II) system.

3.2 Solubility Behaviour

The solubility behaviour of the synthesised coordination polymers was examined in nine common organic and inorganic solvents, namely acetone, N,N-dimethylformamide (DMF), dimethyl sulfoxide (DMSO), methanol, ethanol, water, benzene, chloroform, and dichloromethane. Both coordination polymers were found to be insoluble in all the tested

solvents, with the exception of slight solubility observed in DMSO. The pronounced insolubility of the compounds in a wide range of polar and non-polar solvents is consistent with their polymeric nature and extended metal–ligand frameworks. Coordination polymers typically exhibit low solubility due to strong metal–ligand bonding and the absence of discrete molecular units. The slight solubility observed in DMSO may be attributed to partial disruption of the coordination network by this strongly coordinating solvent. These solubility characteristics further support the formation of extended coordination polymeric structures rather than mononuclear or oligomeric complexes, in agreement with the spectroscopic, magnetic, and crystallographic data discussed above.

Table 2 Solubility test of the coordination polymers

Compound	Acetone	DMF	DMSO	Methanol	Ethanol	Water	Benzene	Chloroform	Dichloro methane
[Mn ₂ (INA) ₄]	IS	IS	SS	IS	IS	IS	IS	IS	IS
[Co ₂ (INA) ₄]	IS	IS	SS	IS	IS	IS	IS	IS	IS

3.3 FT-IR spectral analysis

Comparison of the FT-IR spectrum of the free isonicotinic acid (INA) with that of the coordination polymers provides clear evidence for metal–ligand coordination through the carboxylate group and the pyridyl nitrogen atom.

In the spectrum of free isonicotinic acid, a broad absorption band observed at around 2409 cm⁻¹ is attributed to the O–H stretching vibration of the carboxylic acid group, indicative of strong intermolecular hydrogen bonding in the solid state. This band disappears completely in the spectrum of the cobalt(II) coordination polymer, confirming deprotonation of the carboxylic acid group during coordination. The presence of aromatic C–H stretching vibrations is evident in both spectra, appearing at 3053.88 cm⁻¹ for the free ligand and shifting slightly to 3065 - 3069 cm⁻¹ in the coordination polymers, reflecting changes in the electronic environment upon coordination.

A strong absorption band at 1695.73 cm⁻¹ in the free ligand is assigned to the $\nu(\text{C}=\text{O})$ stretching vibration of the carboxylic acid group. Upon coordination, this band is replaced by two

characteristic carboxylate stretching bands in the cobalt(II) polymer, observed at 1604 cm^{-1} and 1456 cm^{-1} in the Co(II) compound and 1603 cm^{-1} and 1453 cm^{-1} in the Mn(II) compound, corresponding to the asymmetric $\nu_{\text{as}}(\text{COO}^-)$ and symmetric $\nu_{\text{s}}(\text{COO}^-)$ stretching vibrations, respectively. The separation between these bands ($\Delta\nu \approx 150\text{ cm}^{-1}$) is consistent with a bridging or chelating coordination mode of the carboxylate group, which is commonly observed in coordination polymers derived from isonicotinate ligands.

The band associated with the pyridine ring vibrations of the free ligand, observed around 1409.00 cm^{-1} , shows a noticeable shift in the complex, further supporting the involvement of the pyridyl nitrogen atom in coordination to the metal centre. Similar shifts were also observed in $[\text{Co}(\text{INA})_2] \cdot 2\text{H}_2\text{O}$ [15], $[\text{Zn}(\text{IN})_2]$, and $[[\text{Zn}(\text{IN})_2(\text{NO}_3)(\text{H}_2\text{O})]$ [16].

Additional bands appearing in the low-frequency region of the complex spectrum, notably at 679.76 cm^{-1} , 546.64 cm^{-1} , and 458.12 cm^{-1} , may be assigned to metal–oxygen and metal–nitrogen stretching vibrations, providing further confirmation of coordination. Overall, the observed changes in the FT-IR spectrum upon complex formation clearly demonstrate deprotonation of isonicotinic acid and coordination through both the carboxylate oxygen atoms and the pyridyl nitrogen atom. These features support the role of isonicotinic acid as a multifunctional bridging ligand, promoting the formation of extended coordination polymeric frameworks. Similar coordination behaviour is expected for the manganese(II) analogue, based on its comparable synthetic conditions and analytical characteristics.

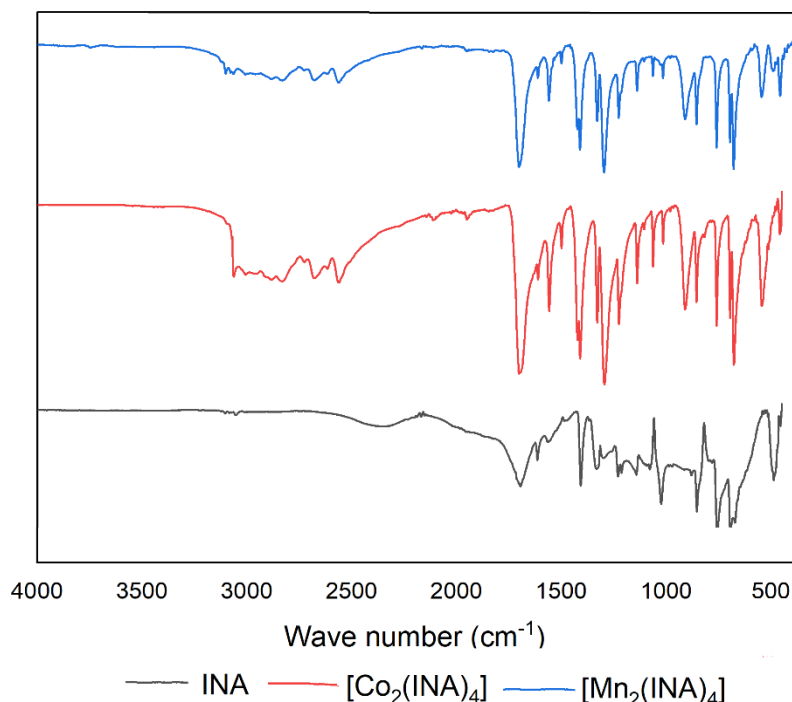


Fig. 1 FT-IR spectra of the free ligand and its coordination polymers

3.4 Magnetic properties

The magnetic susceptibilities of the coordination polymers were measured at room temperature and are expressed as effective magnetic moments (μ_{eff}). The manganese(II) coordination polymer exhibits an effective magnetic moment of 5.81 BM, while the cobalt(II) analogue shows a value of 4.53 BM.

The observed magnetic moment for Mn(II) polymer was 5.81 B.M. this value is in good agreement with the expected spin-only value for a high-spin Mn^{2+} ion (d^5 configuration), for which the theoretical magnetic moment is approximately 5.92 BM. The slight reduction in the experimental value may be attributed to weak antiferromagnetic interactions between neighbouring Mn^{2+} centres within the polymeric framework or to crystal field effects arising from the coordination environment. These results confirm that manganese remains in the +2 oxidation state and adopts a high-spin configuration in the coordination polymer. The effective magnetic moment of 5.67 BM was also documented for $[\text{Mn}(\text{C}_6\text{H}_4\text{O}_2\text{N})_2(\text{H}_2\text{O})_4]$ [17].

In the case of the Co(II) coordination polymer, the measured magnetic moment was 4.53 BM. This is characteristic of high-spin Co^{2+} ions (d^7 configuration) in an octahedral or distorted

octahedral environment. The value exceeds the spin-only magnetic moment (3.87 BM), reflecting a significant orbital contribution to the magnetic behaviour, which is commonly observed for cobalt(II) complexes. The enhanced magnetic moment is therefore consistent with the presence of high-spin Co^{2+} centres within the polymeric structure.

Table 3: Magnetic Susceptibility Measurements

Compound	Mass Susceptibility $\chi_g (\text{erg} \cdot \text{G}^{-2} \cdot \text{g}^{-1})$	Molar Suscetibility $\chi_m (\text{erg} \cdot \text{G}^{-2} \cdot \text{mol}^{-1})$	Magnetic moment μ_{eff}	Spin-only μ (BM)
$[\text{Mn}_2(\text{INA})_4]$	2.094×10^{-5}	1.406×10^{-2}	5.81	5.92
$[\text{Co}_2(\text{INA})_4]$	1.264×10^{-5}	8.59×10^{-3}	4.53	3.87

Overall, the magnetic susceptibility data support the proposed oxidation states and coordination environments of the metal centres in both coordination polymers. The results further corroborate the formation of extended polymeric frameworks containing high-spin Mn^{2+} and Co^{2+} ions, in agreement with the structural and spectroscopic evidence discussed in other sections.

3.5 Elemental analysis

Elemental analysis was carried out to confirm the composition and stoichiometry of the synthesised coordination polymers. For both coordination polymers, the calculated values of the percentages of carbon, hydrogen and nitrogen are in excellent agreement with those observed using a micro-analytical instrument and supports the proposed empirical formulae of the coordination polymers and confirms the successful incorporation of the isonicotinate ligand within the polymeric frameworks.

Table 4: Elemental Analysis of the Coordination Compounds

Compound	% Carbon Calc. (Found)	% Hydrogen Calc. (Found)	% Nitrogen Calc. (Found)
$[\text{Mn}_2(\text{INA})_4]$	38.83 (38.46)	4.34 (4.05)	7.55 (8.03)
$[\text{Co}_2(\text{INA})_4]$	38.42 (38.74)	4.30 (3.92)	7.47 (7.14)

3.5 Single-Crystal X-ray Structural Elucidation of the Cobalt(II) Coordination Polymer

The single-crystal of cobalt(II) coordination polymer crystallises in the monoclinic crystal system with space group $P2_1$. Unit cell determination gave lattice parameters of $a = 9.818 \text{ \AA}$, $b = 12.804 \text{ \AA}$, $c = 10.801 \text{ \AA}$, with $\alpha = 90.00^\circ$, $\beta = 100.97^\circ$, and $\gamma = 90.00^\circ$, consistent with a monoclinic unit cell. The obtained crystallographic parameters show good agreement with a previously reported structure deposited in the Cambridge Crystallographic Data Centre (CCDC 886304), confirming the identity and structural type of the cobalt(II) coordination polymer. On this basis, the structural features of the compound were analysed by reference to the reported crystallographic model.

The structure consists of two crystallographically independent cobalt(II) centres, each adopting a distorted octahedral coordination geometry. Each cobalt ion is coordinated by four oxygen atoms from carboxylate groups of isonicotinate ligands in the equatorial plane, while two nitrogen atoms from pyridine rings occupy the axial positions. This coordination arrangement demonstrates the dual N,O-donor nature of the isonicotinate ligand.

The carboxylate groups act as bridging ligands between neighbouring cobalt centres, leading to the formation of infinite one-dimensional coordination chains. Further interconnection through additional carboxylate bridges results in the generation of an extended three-dimensional polymeric framework. The structure highlights the role of isonicotinic acid in directing the assembly of robust coordination polymers through simultaneous metal–oxygen and metal–nitrogen interactions.

Table 5 Crystallographic data of [Co₂(INA)₄]

Compound	[Co ₂ (INA) ₄ .DMF]
Empirical formula	[Co(C ₂₇ H ₂₃ N ₅ O ₉)]
Molecular weight	671.38
Crystal system	Monoclinic
Space group	P(2) ₁
$a/\text{\AA}$	9.818
$b/\text{\AA}$	12.804
$c/\text{\AA}$	10.801
$\alpha/^\circ$	90.00
$\beta/^\circ$	100.63
$\gamma/^\circ$	90

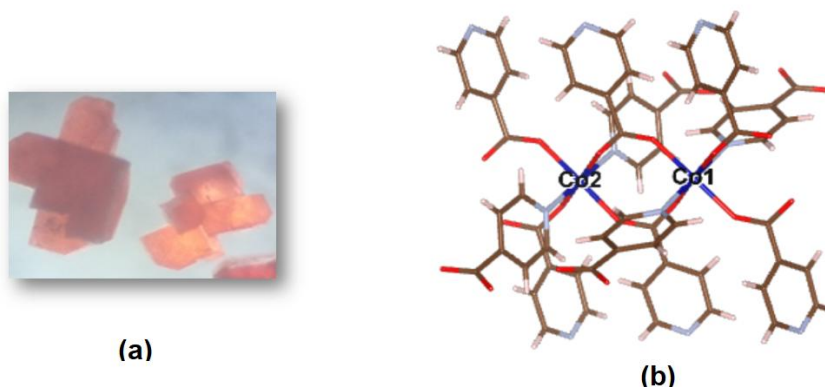


Fig. 2 (a) Microscopic picture of $[\text{Co}_2(\text{INA})_4]$; (b) Connectivity and coordination environment of Co in $[\text{Co}_2(\text{INA})_4\cdot\text{DMF}]$. (Co = blue, O = red, N = grey, C = brown, H = pink. (b) packing diagram

3.4 Powder X-ray Diffraction (PXRD) Analysis

The powder X-ray diffraction (PXRD) patterns of isonicotinic acid (INA) and the synthesised coordination polymers were examined to assess changes in crystallinity and phase formation upon coordination. The PXRD pattern of the free ligand exhibits an intense reflection at $2\theta = 28.24^\circ$, together with a moderate peak at 14.56° , which are characteristic of crystalline isonicotinic acid.

Notably, these reflections are completely absent in the PXRD patterns of both the cobalt(II) and manganese(II) coordination polymers, indicating that the crystal structure of the free ligand is no longer present after coordination. The disappearance of these characteristic ligand peaks confirms successful deprotonation and incorporation of isonicotinic acid into the coordination polymer frameworks.

In contrast, the PXRD patterns of the coordination polymers display new reflections that are not observed in the free ligand. Prominent peaks appear at $2\theta = 10.76^\circ$ and 21.65° , which are characteristic of the newly formed crystalline coordination polymers. The emergence of these reflections provides clear evidence for the formation of new crystalline phases distinct from the parent ligand.

The experimentally observed PXRD pattern shows good agreement with the simulated pattern derived from reported single-crystal data (CCDC 886304), confirming that the bulk material obtained from the solvothermal synthesis is structurally consistent with the known crystal structure. Minor variations in relative peak intensities may be attributed to preferred orientation effects and differences in crystallite size, which are commonly encountered in polymeric coordination compounds.

Overall, the PXRD results demonstrate the successful transformation of isonicotinic acid into crystalline cobalt(II) and manganese(II) coordination polymers and confirm the formation of phase-pure materials with structures distinct from that of the free ligand.

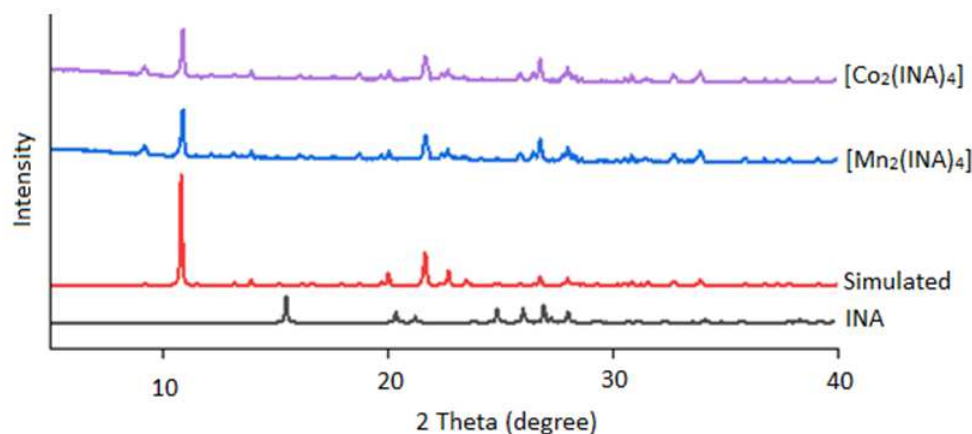


Fig 3 Pxd patterns of Isonicotinic acid, Single crystal of $[\text{Co}_2(\text{INA})_4]$ (simulated) and bulk sample of $[\text{Mn}_2(\text{INA})_4]$

4. Conclusion

In this study, cobalt(II) and manganese(II) coordination polymers derived from isonicotinic acid were successfully synthesised via a solvothermal method and comprehensively characterised using spectroscopic, magnetic, analytical, and diffraction techniques. The physical properties, solubility behaviour, and thermal stability of the compounds are consistent with the formation of robust polymeric frameworks.

FT-IR spectroscopic analysis confirmed deprotonation of the isonicotinic acid ligand and coordination through both carboxylate oxygen and pyridyl nitrogen atoms, highlighting the ligand's dual N,O-donor capability. Magnetic susceptibility measurements revealed high-spin configurations for both metal centres, in agreement with their expected electronic structures and coordination environments.

X-ray diffraction unit cell determination for the cobalt(II) compound, together with comparison to a reported structure (CCDC 886304), established a monoclinic polymeric framework in which cobalt centres adopt distorted octahedral geometries bridged by isonicotinate ligands into extended networks. Elemental analysis further supported the proposed stoichiometries of the coordination polymers. Powder X-ray diffraction studies demonstrated the formation of new crystalline phases distinct from that of the free ligand, while comparison with reported single-crystal data confirmed the phase purity and structural consistency of the cobalt(II) coordination polymer. Single-crystal

Overall, this work demonstrates the effectiveness of isonicotinic acid as a versatile bridging ligand in the construction of coordination polymers and provides insight into the influence of metal ion identity on the structural and physicochemical properties of such materials. The results contribute to the growing understanding of isonicotinate-based coordination polymers and their rational design.

Funding

None declared.

Data availability

The datasets generated and/or analysed during the present study are available from the corresponding author upon request.

Declarations

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Competing interests

The authors declare no competing interests

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