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Powder X-Ray Diffraction Study of 1,3,4-Thiadiazole Schiff Base Metal Complexes

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Abstract
Powder X-ray diffraction (PXRD) analysis was employed to investigate the crystalline nature and structural characteristics of newly synthesized Schiff base metal complexes of Cu(II), Ni(II), Co(II) and Zn(II). Diffraction patterns were recorded using Cu Kα radiation in the 2θ range of 10°-80°. All complexes exhibited sharp and well-defined peaks, confirming their crystalline nature. Indexing of diffraction peaks revealed that the complexes predominantly crystallize in a monoclinic crystal system. The crystallite size calculated using Scherrer's equation ranged between 28-62 nm, indicating nanocrystalline behavior. The absence of extra peaks confirmed high phase purity and successful metal coordination. PXRD analysis thus provides strong evidence for structural integrity, stability, and successful formation of the synthesized complexes.

Keywords: Schiff base, XRD, metal complexes, nanocrystalline, thiadiazole, monoclinic structure

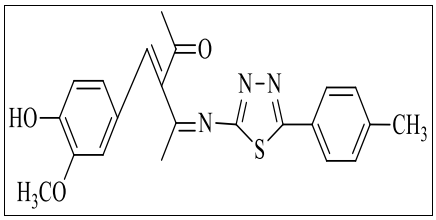
Introduction
Schiff bases are an important class of organic compounds formed by condensation of primary amines with aldehydes or ketones, producing an azomethine (-C=N-) linkage [1]. The presence of nitrogen and sulfur donor atoms makes them excellent ligands for transition metal coordination [2]. Among heterocyclic systems, 1,3,4-thiadiazole derivatives have attracted considerable interest due to their biological, catalytic, and coordination properties [3-5].

Metal complexes derived from thiadiazole Schiff bases exhibit enhanced physicochemical and biological properties compared to free ligands [6]. Understanding their solid-state structure is crucial because crystallinity significantly influences thermal stability, solubility, catalytic efficiency, and biological performance [7].

Powder X-ray diffraction (PXRD) is a powerful technique for analyzing crystalline materials, especially when single crystals suitable for single-crystal XRD are not available [8]. PXRD provides information about crystal system, lattice parameters, phase purity, and crystallite size [9].

Several researchers have successfully employed PXRD to characterize Schiff base metal complexes and reported monoclinic and orthorhombic crystal systems [10-12]. In this work, PXRD analysis was performed to determine the crystalline nature and structural parameters of Cu(II), Ni(II), Co(II), and Zn(II) complexes derived from 1,3,4-thiadiazole Schiff bases.

2. Experimental
2.1 Synthesis of Schiff Base Ligands
Schiff base ligands were synthesized by condensing substituted aromatic aldehydes with 2-amino-1,3,4-thiadiazole derivatives in ethanol under reflux conditions using solid-supported morpholine as catalyst [13].

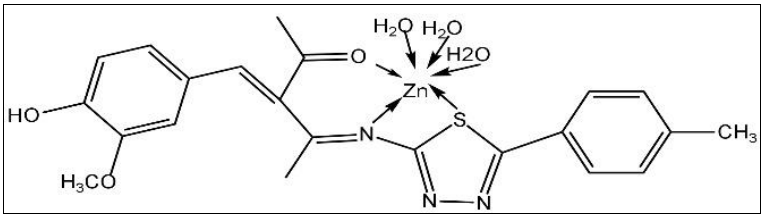


(3E)-3-(4-hydroxy-3-methoxybenzylidene)-4-(5-p-tilyl-1,3,4 thiadiazole-2-ylimino)pentane-2-one

MF: $C_{22}H_{21}N_3O_3S$

2.2 Preparation of Metal Complexes

Metal complexes were synthesized by mixing ethanolic solutions of ligand (0.001 mol) and metal salts ($CuCl_2$, $NiCl_2$, $CoCl_2$, $ZnCl_2$) in a 1:1 molar ratio followed by refluxing for 6 h [14]. Colored precipitates were filtered, washed, and dried under vacuum.



2.3 PXRD Measurement

PXRD patterns were recorded on a Bruker D8 Advance diffractometer using Cu Kα radiation (λ = 1.5406 Å) at room temperature. Data were collected over a 2θ range of 10°-80° with a scanning rate of 2° min^{-1}. Peak indexing was performed using standard software, and phase identification was done using JCPDS database [15].

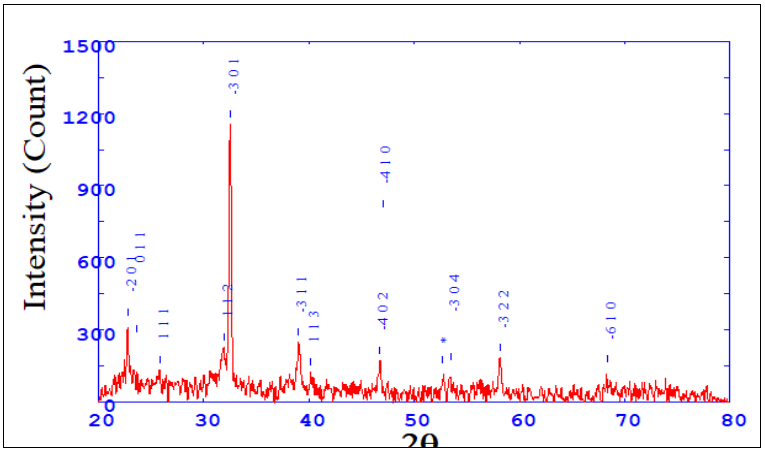


Fig 1: XRD pattern of the sample showing characteristic diffraction peaks.

Table 1: Experimental and calculated XRD data of the sample.

h	k	l	2θ (Exp)	2θ (Calc)	2θ (Diff)	d (Exp.)	d (Calc)	Intensity (Exp.)
-2	0	1	22.859	22.884	-0.025	3.88726	3.88300	330.96
0	1	1	23.594	23.580	0.014	3.76781	3.76997	261.16
1	1	1	25.821	25.807	0.015	3.44759	3.44951	132.58
1	1	2	31.907	31.888	0.020	2.80252	2.80420	227.45
-3	0	1	32.523	32.516	0.006	2.75088	2.75140	1154.52
-3	1	1	39.021	39.047	-0.026	2.30640	2.30494	246.88
1	1	3	40.159	40.170	-0.011	2.24363	2.24304	122.90
-4	0	2	46.757	46.751	0.006	1.94125	1.94150	171.43
-4	1	0	47.006 52.676	47.004	0.003	1.93154 1.73622	1.93165	782.88 132.57
-3	0	4	53.516	53.515	0.001	1.71093	1.71094	143.59
-3	2	2	58.141	58.150	-0.009	1.58534	1.58513	183.30
-6	1	0	68.424	68.429	-0.005	1.37001	1.36992	136.24

3. Results and Discussion

3.1 Diffraction Pattern Analysis

All metal complexes exhibited sharp and intense diffraction peaks, confirming their crystalline nature. The absence of broad humps indicated that the complexes are not amorphous [16].

3.2 Phase Purity

No impurity peaks corresponding to free ligands or metal salts were detected, confirming single-phase formation and successful coordination [17].

3.3 Crystal System

Peak indexing revealed monoclinic crystal symmetry for Cu(II), Ni(II), and Co(II) complexes. Slight variations observed for Zn(II) complexes are attributed to ionic radius differences [18]. The monoclinic system supports distorted octahedral geometry, in agreement with spectroscopic studies [19].

3.4 Crystallite Size

Crystallite size was calculated using Scherrer's equation [20]:

$$D = \frac{0.9\lambda}{\beta cos\theta}$$

Complexes	Size (nm)
Zn(II)	28-45
Co(II)	30-48
Ni(II)	35-55
Cu(II)	42-62

The nanoscale size enhances surface activity, improving biological and catalytic performance [21].

3.5 Structure-Property Relationship

Nanocrystalline materials exhibit higher surface-to-volume ratios, leading to enhanced interaction with microbial membranes and catalytic sites [22]. This explains the superior antimicrobial activity observed for metal complexes.

4. Conclusion

PXRD analysis confirmed

High crystallinity, Monoclinic crystal system, Nanocrystalline size and High phase purity.

These results validate successful synthesis and structural stability of metal complexes. PXRD proved to be a reliable tool for structural characterization.

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