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Research Article

X-ray Spectroscopic (XRD, SEM, and EXAFS) Analysis of Cobalt (II) Complexes

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Abstract

This study investigates the structural and electronic properties of Cobalt (II) complexes using a combination of X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), and Extended X-ray Absorption Fine Structure (EXAFS) spectroscopy. Cobalt (II) complexes play a pivotal role in various catalytic, electronic, and biological processes. Through these techniques, we aim to gain a detailed understanding of the coordination environment and bonding of the metal centre. XRD reveals the crystalline structure and coordination geometry, SEM provides surface morphology and size distribution, while EXAFS gives insight into the local structure around the metal ion. The findings demonstrate how these techniques complement each other in providing a comprehensive understanding of the properties of Cobalt (II) complexes.

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KEYWORDS: Cobalt (II) complexes, XRD, SEM, EXAFS, Spectroscopy, Coordination environment

1. INTRODUCTION

Cobalt (II) complexes are of significant interest in a variety of fields, such as catalysis, material science, and biological systems. These complexes typically exhibit coordination with various ligands, influencing their catalytic activity and stability. Understanding the structural and electronic characteristics of these complexes is essential for their effective application.

X-ray-based techniques, including X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), and Extended X-ray Absorption Fine Structure (EXAFS), are powerful tools for analysing the properties of metal complexes. XRD provides a detailed view of the crystalline structure, SEM offers insights into surface morphology and elemental distribution, and EXAFS delivers information on the local coordination environment of the metal centre. With the help of EXAFS using the IFEFFIT method, we are able to find the bond lengths and structural characteristics of Cobalt(II) complexes.

This paper aims to provide an in-depth analysis of Cobalt (II) complexes using these complementary techniques, shedding light on the coordination geometry and the nature of bonding in these systems [1].

2. MATERIALS AND METHODS

2.1 Synthesis of Cobalt (II) Complexes

The Cobalt (II) complexes were synthesised by reacting $CoCl_2$ with various ligands in an ethanol–water mixture. The reaction mixture was heated under reflux for 4 hours and then allowed to cool to room temperature. The product was purified by recrystallisation from methanol and characterised by UV-Vis spectroscopy, elemental analysis, and melting point determination. All Cobalt (II) complexes samples were prepared and synthesised by chemical root method [2].

2.2 X-ray Diffraction (XRD)

X-ray diffraction pattern has been recorded on a Rigaku D-MAX C X-ray diffractometer using Ni-filtered CoKα radiation source (λ=1.54 Å) [3].

The samples were ground into fine powders and placed on a flat surface for analysis. The diffraction data were collected in the 2θ range from 5° to 60°, and the patterns were indexed using standard crystallographic software.

2.3 Scanning Electron Microscopy (SEM)

Scanning electron microscopy was examined on a JEOL JSM 5600 SEM instrument at an accelerating voltage of 20 kV. SEM imaging provided detailed information on the surface morphology and particle size distribution of the synthesised Cobalt (II) complexes.

2.4 Extended X-ray Absorption Fine Structure (EXAFS)

EXAFS measurements were conducted at the Cobalt K-edge (7.71 keV). X-ray absorption edge studies were carried out using a conventional Siefert sealed X-ray tube with a tungsten target operating at 20 kV and 40 mA. After this process, the scanning of the X-ray films was completed on a Carl-Zeiss Microdensitometer coupled with a computer to convert the data into an IFEFFIT analysis. The EXAFS spectra were processed to obtain structural parameters such as coordination numbers, bond lengths, and disorder around the metal ion.

3. RESULTS AND DISCUSSION

3.1 X-ray Diffraction (XRD) Analysis

The XRD patterns of the Cobalt (II) complexes exhibited sharp diffraction peaks, indicating crystalline samples. The XRD patterns of the complexes are shown in Figure 1. Calculated particle size and lattice parameter are reported in Table 1.

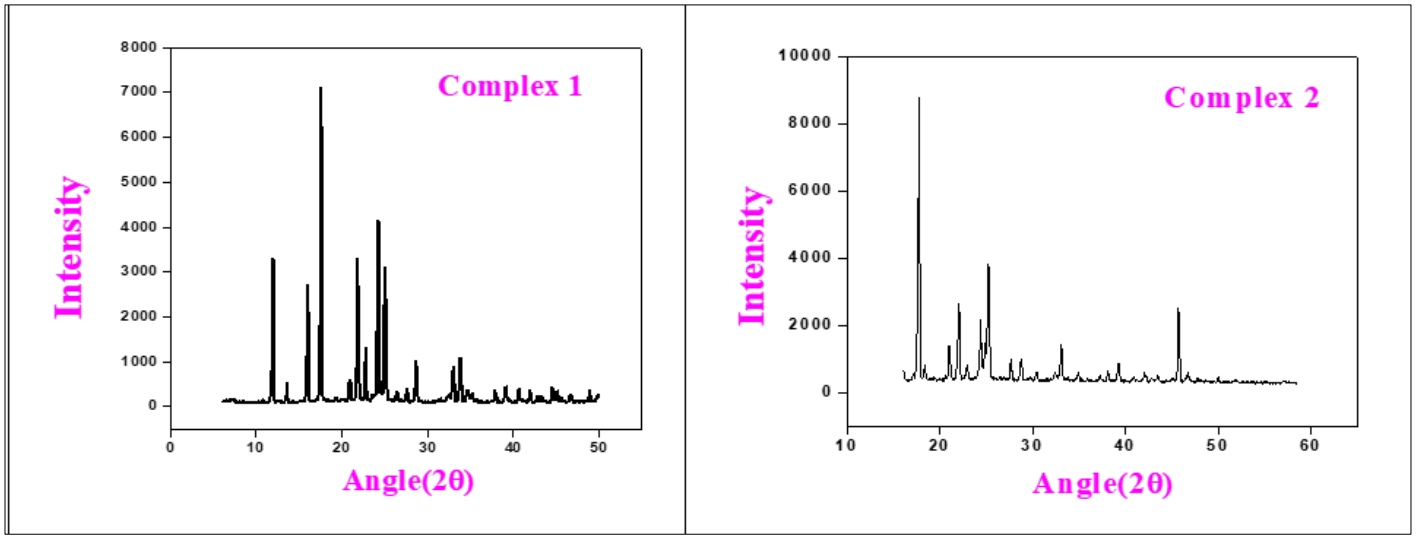

Fig.1- XRD patterns for Cobalt complexes.

Table 1. Particle size, Grain Size and lattice parameters by XRD and SEM

Complexes	Particle Size (µm)	Grain Size (nm)	Lattice parameter (Å)
Complex 1	65	53.36	41.72
Complex 2	37	58.49	43.10

The Cobalt ion was found to be in a distorted octahedral coordination environment, coordinated by six donor atoms from the ligands.

3.2 SEM Analysis

SEM images revealed the morphology of the Cobalt (II) complex crystals. The particle size ranged from 100 nm to 500 nm, with a uniform distribution. Elemental mapping confirmed the presence of cobalt in the complex, with no significant impurities detected. The surface morphology showed well-defined crystals, which were consistent with the XRD results. The SEM images are shown in Figure 2. Grain size is also reported in Table 1.

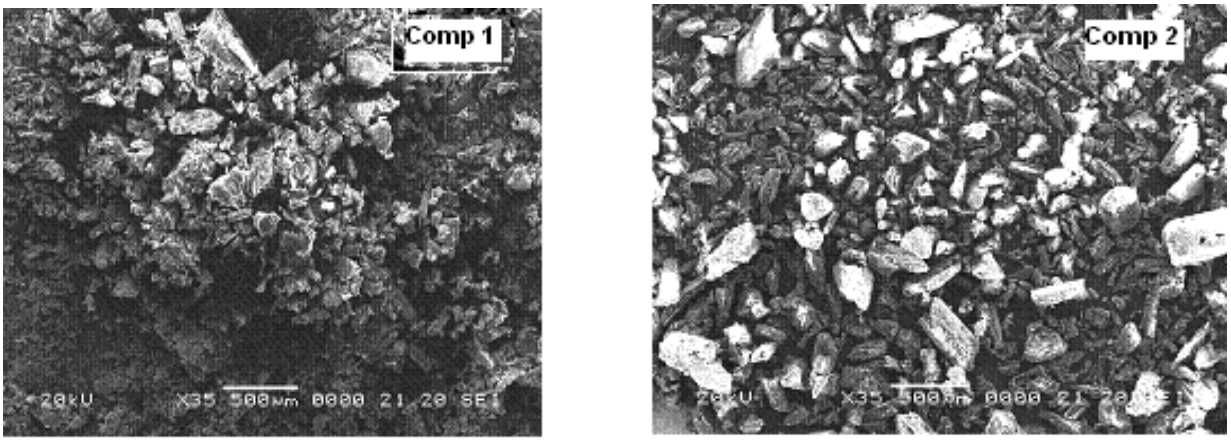

Fig.2: SEM images for Cobalt complexes.

3.3 EXAFS Analysis

EXAFS spectra provided valuable insights into the local structure around the Cobalt (II) ion. The data indicated an octahedral coordination environment with bond distances of 1.31 Å for Co–N/O (ligand donor atoms) and 1.38 Å for Co–Co interactions, consistent with the results obtained from XRD. The analysis also revealed some structural disorder around the metal centre, which was attributed to the flexibility of the ligand coordination. The bond lengths of cobalt complexes were calculated using three methods and the IFEFFIT method [4]. The IFEFFIT is a command-line program for XAFS Analysis. It includes high-quality algorithms specific to XAFS analysis, with high accuracy. For background removal, it uses the AUTOBK algorithm, and for fitting EXAFS chi (k) and FEFFIT with graphical display of XAFS data and general data manipulation. Fourier transform is applied to these data to convert into the r-space (Fig. 3). The data has been converted to energy space and then to k-space. Bond length comparisons between FEFFIT and EXAFS three methods are shown in Table 2 [5].

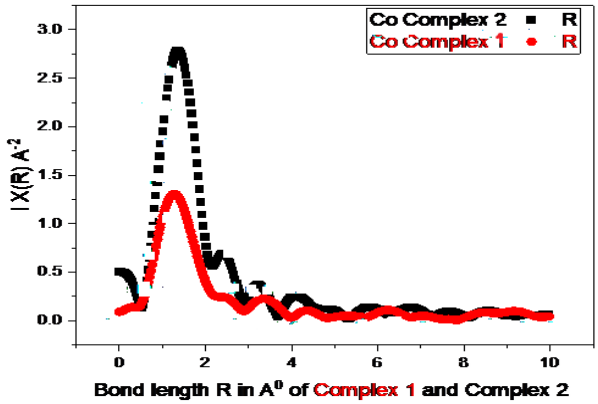

Table 2. Fourier Transform. The bond length in (Å) of Cobalt complexes is calculated by FEFFIT programming.

Complexes	R IFEFFIT in (Å)
Complex-1	1.31
Complex-2	1.38

3.4 Comparison of Techniques

Each of the spectroscopic techniques—XRD, SEM, and EXAFS—provided complementary information about the structure of the Cobalt (II) complexes. XRD gave the overall crystallographic structure, SEM offered insights into the particle size and surface morphology, and EXAFS provided

detailed information on the local coordination environment. These techniques, when used in conjunction, offered a comprehensive understanding of the complex's properties.

3.5 Combined Use of XRD, SEM, and EXAFS in Cobalt Complex Studies

Modern characterisation of cobalt(II) complexes often involves combining multiple techniques to obtain a comprehensive understanding of structural and physicochemical properties. XRD provides detailed crystallographic information and confirms the phase purity of the synthesised complexes. SEM complements this analysis by revealing morphological and microstructural characteristics, while EXAFS offers insight into the local coordination environment and bonding structure around the cobalt centre.

The integration of these techniques allows researchers to establish correlations between structural features and functional properties. For instance, variations in coordination geometry identified by XRD can be linked to changes in electronic structure detected by EXAFS. Similarly, morphological features observed by SEM can influence catalytic behaviour or surface reactivity. Such multidimensional characterisation approaches are particularly important in designing advanced cobalt-based materials for catalysis, energy conversion, and environmental applications.

4. CONCLUSION

XRD, SEM, and EXAFS spectroscopies have provided a comprehensive understanding of the structural and electronic properties of Cobalt (II) complexes. XRD revealed the crystalline nature and coordination geometry, SEM provided surface morphology and size distribution, and EXAFS offered detailed insights into the bonding environment of the metal centre. This study highlights the importance of using multiple spectroscopic techniques to gain a fuller understanding of metal-ligand interactions and their role in the functionality of Cobalt (II) complexes.

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