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Sensitive Detection of Dichlorvos Pesticide in Aqueous Phase using a Cobalt (II) Coordination Polymer based on Biphenyl-4-carboxylic acid

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ABSTRACT

A cobalt-based coordination polymer (CP), $[Co(Biphen)_2(Tmp)_2]_n.(H_2O)$, was successfully synthesised and characterised using Fourier-Transform Infrared Spectroscopy (FTIR), ultraviolet visible spectroscopy (UV-Vis), Thermogravimetric analysis (TGA), and Scanning Electron Microscopy (SEM). The MOF appeared as a blue crystalline solid with a melting point of 257 °C and a molecular weight of 889.92 g/mol. It was slightly soluble in Dimethy sulphoxide (DMSO) but insoluble in ethanol, methanol, n-hexane, Dimethylformamide (DMF), and water. FTIR analysis revealed shifts in the C=O band from 1684 cm^{-1} in the free Biphen ligand to 1681 cm^{-1} in the complex, as well as the appearance of carboxylate (COO^-) symmetric and asymmetric stretching bands at 1608 cm^{-1} and 1535 cm^{-1}, respectively, with a $\Delta\nu$ value of 73 cm^{-1}, confirming a chelating bidentate coordination mode. Additional Co-O and Co-N stretching bands appeared at 516 and 621 cm^{-1}, respectively. UV-Vis spectra exhibited ligand-based $\pi\rightarrow\pi^*$/ $n\rightarrow\pi^*$ transitions and d-d transitions characteristic of octahedral Co(II) centers. TGA revealed three decomposition stages with high thermal stability. SEM analysis revealed aggregated, porous micro- to nanosized particles with a cauliflower-like morphology. The compound showed a distinct emission band at 630 nm upon excitation at 318 nm. This was attributed to ligand to metal charge transfer (LMCT) because of cheating the metal to the ligand. The compound also showed high selectivity towards dichlorvos pesticide among other organic compounds tested via luminescence sensing.

Keywords: Coordination polymers, Detection, Fluorescence, Pesticide, Quenching

INTRODUCTION

Pesticides are widely used in modern agriculture to protect crops from pests, diseases, and weeds, thereby enhancing food production and security. However, the excessive and indiscriminate use of pesticides has raised significant concerns due to their persistence in the environment and potential to accumulate in soil, water, and food chains (Rojas *et al*., 2022). Residual pesticides pose serious risks to human health, including endocrine disruption, neurological disorders, and even carcinogenic effects (Singha *et al*., 2017). Conventional methods for pesticide detection, such as gas chromatography, high-performance liquid chromatography (HPLC), and mass spectrometry, provide accurate results but suffer from limitations such as high cost, time consumption, complex sample preparation, and the need for specialised equipment and trained personnel. These constraints make them less suitable for routine monitoring or rapid on-site detection. Therefore, the development of alternative sensing platforms that are cost-effective, highly sensitive and capable of real-time analysis has become an area of intense research interest (Portolés *et al*., 2012; Walorczyk and Drozdzyński, 2012).

Coordination polymers (CPs), a class of crystalline materials formed by the coordination of metal ions with organic ligands, have recently attracted attention as promising candidates for pesticide detection. Their tunable structures, large surface areas, and diverse functionalities enable them to interact selectively with pesticide molecules through mechanisms such as hydrogen bonding, π–π stacking, and metal–ligand interactions (Wang *et al*., 2016; Zhang *et al*., 2018). Moreover, the luminescent, electrochemical, and catalytic properties of many CPs make them highly suitable for designing sensors with excellent sensitivity and detection limits. The use of luminescent coordination polymers, particularly those derived from transition metals and lanthanides, has shown great potential for pesticide detection. These materials often exhibit strong fluorescence, which can be quenched or enhanced in the presence of specific pesticide molecules, thereby enabling a "turn-off" or "turn-on" sensing response. Similarly, conductive and

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electroactive CPs provide opportunities for electrochemical sensing, where pesticide binding alters the electrical properties of the material, leading to measurable signals (Yao *et al*., 2020).

Xu *et al*. (2018) designed a highly luminescent CP based on 1,2,4,5-Tetrakis(4-carboxyphenyl) benzene (H_4TCPB) for detecting parathion-methyl. The CP was able to selectively detect parathion-methyl amongst other pesticides tested by quenching with a significantly low limit of detection. The quenching effect of parathion-methyl is attributed to the electron-rich chemical group of the ligand, which might lead to photo-induced electron transfer. Singha *et al*., 2018 synthesized a 3D cadmium-based metal organic framework $[Cd_3(PDA)_1(tz)_3Cl(H_2O)_4]\cdot 3H_2O$ for the detection of pesticides in aqueous medium and fruit extracts. Wang *et al*. (2023) also prepared a Zn(II) CP for the selective detection of pesticides. The fluorescence quenching was most pronounced for nitenpyram at λ_{ex} = 225 nm and imidacloprid at λ_{ex} = 290 nm. The quenching process may occur via the inner filter effect and fluorescence resonance energy transfer.

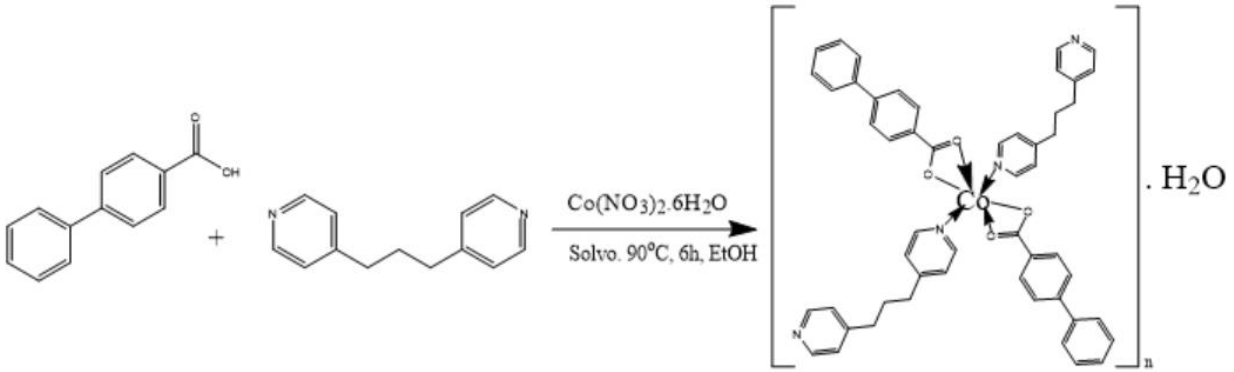
Ongoing research is focused on designing CPs with higher selectivity, lower detection limits, and better stability in real environmental conditions. By integrating CP-based sensors into portable devices, rapid and on-site detection of pesticide residues can be achieved, contributing significantly to food safety, environmental protection, and public health monitoring. This emerging approach highlights the vital role of coordination polymers in addressing global challenges related to pesticide contamination.

MATERIALS AND METHODS

All reagents and chemicals are of analytical grade and were used as received without further purification. Biphenyl-4-carboxylic acid, 4,4-trimethylenedipyridine and pesticide standards were purchased from Sigma Aldrich, Germany. Cobalt nitrate hexahydrate ($Co(NO_3)_2.6H_2O$) was supplied by Alfa Aesar, UK. Solvents: Methanol (99.8%), Ethanol (98.8%), *N, N*-dimethyl formamide (99.0%) and Benzene were obtained from Central Drug House Ltd, New Delhi, India.

Synthesis of $[Co(Biphen)_2(Tmp)_2]_n.(H_2O)$

In a typical solvothermal reaction, 0.198 g (1 mmol) of Biphenyl-4-carboxylic acid (Biphen) and 0.198 g (1 mmol) of 4,4′-trimethylenedipyridine (Tmp) were dissolved separately in 10 mL of ethanol. Subsequently, 0.291 g of $Co(NO_3)_2.6H_2O$ dissolved in 10 mL of distilled water was added to the mixture. The reaction mixtures were stirred at room temperature for 30 minutes and then transferred into a Teflon-lined autoclave, where they were heated at 90 °C for 6 hours. The resulting solid was washed with ethanol and air-dried (Scheme 1)



Scheme 1: Reaction pathway for $[Co(Biphen)_2(Tmp)_2]_n.H_2O$

Instrumentation

The infrared spectra were recorded using a SHIMADZU FTIR-8400S spectrometer using the KBr plate method. UV-Vis absorption spectra were measured in the range 200 to 800 nm using a N4S UV Spectrophotometer with a slit width of 2 nm. The fluorescence emission spectra were recorded on an Agilent Cary Eclipse Photoluminescence Spectrometer set at 2.5 nm slit width for both the excitation and emission. TGA was performed using an SDT-Q600 TA instrument. The sample was heated in air with a heating rate of 10 ºC $min^{-1,}$ and the scan was recorded within the temperature range of 25-900 °C. Scanning Electron Microscopy was conducted on a JOEL-JSM 7600F microscope at 20KV.

Sensing Experiment

The method reported by Singha *et al*. (2018) was adopted for the sensing experiment. The aqueous dispersions of CP were prepared by mixing 2 mg of CP into 4 mL of water, and they were ultrasonicated for 45 min. 1 mM pesticide solution was prepared in acetonitrile solvent, and the dichlorvos solution (analyte) was added to a quartz cuvette containing an aqueous dispersion of CP using a micropipette. A similar procedure was used for the preparation of other organic solvents (ethanol, methanol, DMSO, DMF). The fluorescence data were collected afterwards.

RESULTS AND DISCUSSION

Table 1 shows the physicochemical properties of the synthesised cobalt coordination polymer. The compound is a blue powder with a molecular weight of 867.89 g/mol and a yield of 71 %. The compound is soluble only in dimethyl sulfoxide (DMSO) among other solvents tested. It is thermally stable with a melting point of 257 ºC. The elemental analysis shows the compound is pure with good conformity between experimental and theoretical data.

Table 1: Physicochemical properties of $[Co\ (Biphen)_2(Tmp)_2]_n.H_2O$

Compound	Appearance	Solubility	Mp (ºC)	M.wt (g/mol)	Yield (%)
$Co(Biphen)_2(Tmp)_2]_n.(H_2O)$	Blue powder	Soluble in DMSO	257	867.89	71
Biphen	White	Soluble in DMF, DMSO	228	198.22	-
Tmp	Pale yellow	Soluble in DMF, DMSO	58	198.26	-

Mp = melting point, M.wt = molecular weight

Fourier Transform Infrared spectroscopy

The FTIR spectra of Biphenyl-4-carboxylic acid (Biphen), 4,4′-trimethylenedipyridine (Tmp), and the synthesised $[Co(Biphen)_2(Tmp)_2]_n.H_2O$ coordination polymer is shown in Figure 1. The functional group assignments are summarised in Table 2. The broad absorption band observed around 3428 cm^{-1} in the spectrum of the complex is attributed to O-H stretching vibrations of coordinated water molecules, which is absent in the spectrum of the free Biphen ligand. The C-H stretching vibration in the aromatic rings of Biphen ligand appears at 3012 cm^{-1}, showing slight shifts upon complexation. The IR spectrum of the free Biphen ligand shows a strong C=O stretching vibration band at 1684 cm^{-1}, characteristic of the carboxylic acid group.

Upon complexation with Co(II) ion, the band was shifted to 1681 $cm^{-1,}$ confirming chelation of the carboxylate group to the Co metal. The complex FTIR spectra showed distinctive bands at 1608 cm^{-1}, which represent the coordinated carboxylate $\nu(COO^-)$ symmetric and asymmetric stretching vibrations, respectively. The difference between these bands ($\Delta\nu$) was calculated to be 73 cm^{-1}, which is substantially less than 200 cm^{-1}. The $\Delta\nu$ value indicates that the carboxylate groups do not coordinate to the cobalt centre in a monodentate form, but rather in a chelating bidentate mode (Djoumbissie *et al*., 2021). Additionally, the C–N stretching vibrations in the Tmp ligand, initially observed at 1215 cm^{-1}, shift slightly in the complex spectrum, indicating coordination through the nitrogen atoms of the pyridine rings. In the 516 cm^{-1} and 621 cm^{-1} region, new bands appear in the complex spectrum that are absent in the free ligands. These are assigned to Co-O and Co-N stretching vibrations, confirming metal-ligand coordination (Ant Bursalı, 2024).

UV/Vis Spectroscopy

The UV-Vis spectra of synthesised cobalt-based coordination polymer $[Co(Biphen)_2(Tmp)_2]_n.H_2O$, 4,4′-trimethylenedipyridine (Tmp), and biphenyl-4-carboxylic acid (Biphen) are displayed in Figure 2. The electronic assignments are summarised in Table 3. The spectra of the free ligands display absorption bands in the ultraviolet region. Biphen shows a prominent peak around 255 nm and 334 nm, attributed to $n \rightarrow \pi^*/\pi \rightarrow \pi^*$ transitions within its aromatic system. Similarly, Tmp exhibits a band in the 270 nm and 328 nm region, also due to $n \rightarrow \pi^*/\pi \rightarrow \pi^*$ transitions of the pyridine rings (Abeble Dagnaw, 2021). In the spectrum of the $[Co(Biphen)_2(Tmp)_2]_n.H_2O$ complex, these ligand-based transitions persist but exhibit slight shifts, indicating successful coordination of the ligands to the cobalt (II) center. A moderate band observed around 318 nm is assigned to ligand to metal charge transfer, likely arising from the lone pairs on nitrogen or oxygen atoms now involved in metal coordination. The spectrum of the $[Co(Biphen)_2(Tmp)_2]_n.H_2O$ complex displays a distinct absorption band in the visible region at 669 nm, which are absent in the spectra of the free ligands. These bands are characteristic of $d \rightarrow d$

transitions of the Co(II) ion in an octahedral geometry, confirming the formation of a coordination complex (Pascher *et al*., 2020).

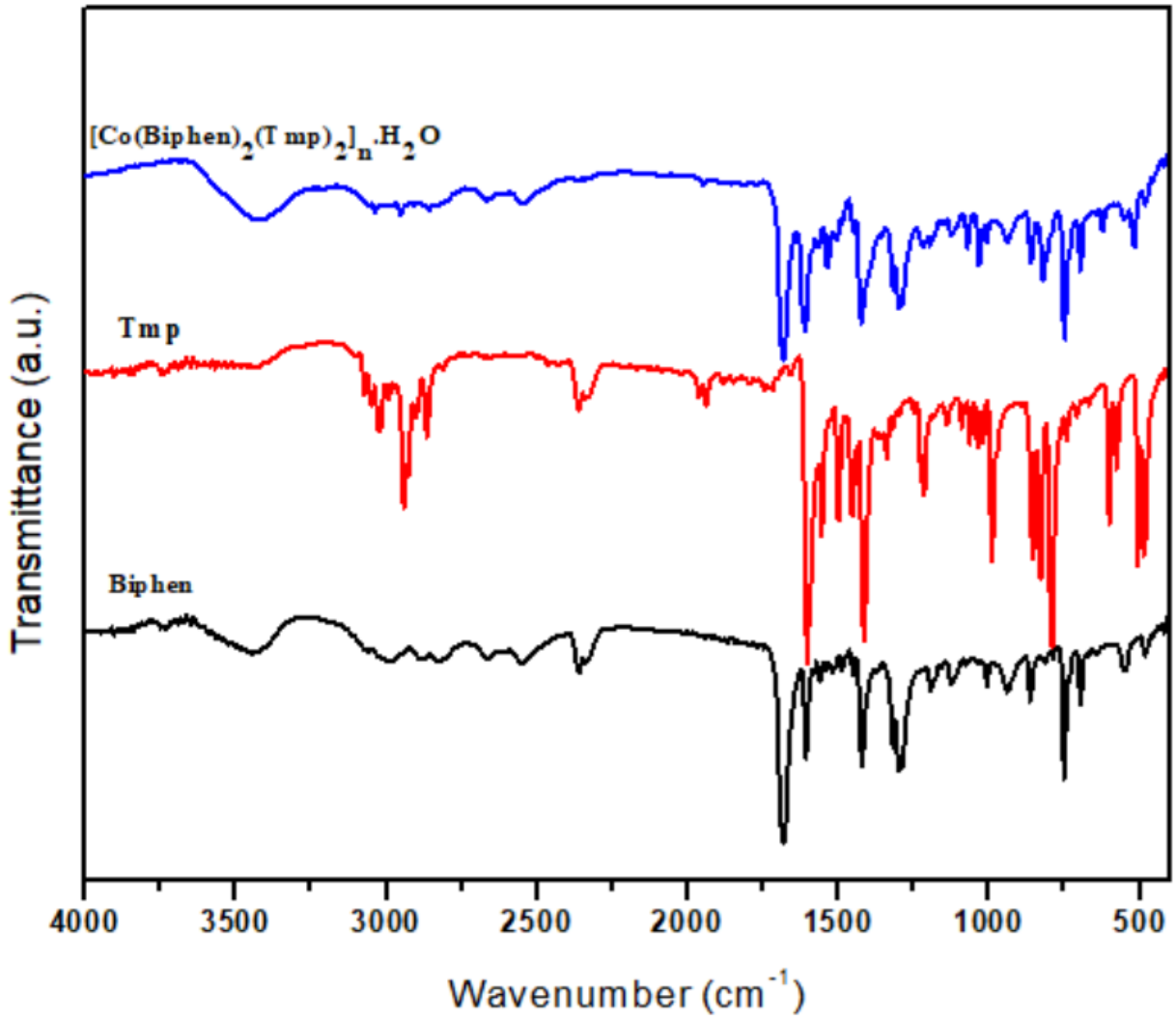


Figure 1: FT-IR spectra of $[Co(Biphen)_2(Tmp)_2]_n.H_2O$ and its ligand

Table 2: FTIR data for $[Co(Biphen)_2(Tmp)_2]_n.H_2O$ and its ligands

Cp/Ligands	***v*(C=N)**	***v*(O-H)**	***v*(C-H)**	***v*(C=C)**	***v*(C-N)**	***v*(C=O)**	***v*(M-O)**	***v*(M-N)**
$[Co(Biphen)_2(Tmp)_2]_n.H_2O$	1678	3428	2955	1030	1118	1608	621	516
Biphen	-	-	3012	1006	-	1684	-	-
Tmp	1604	-	3055	1064	1215	-	-	-

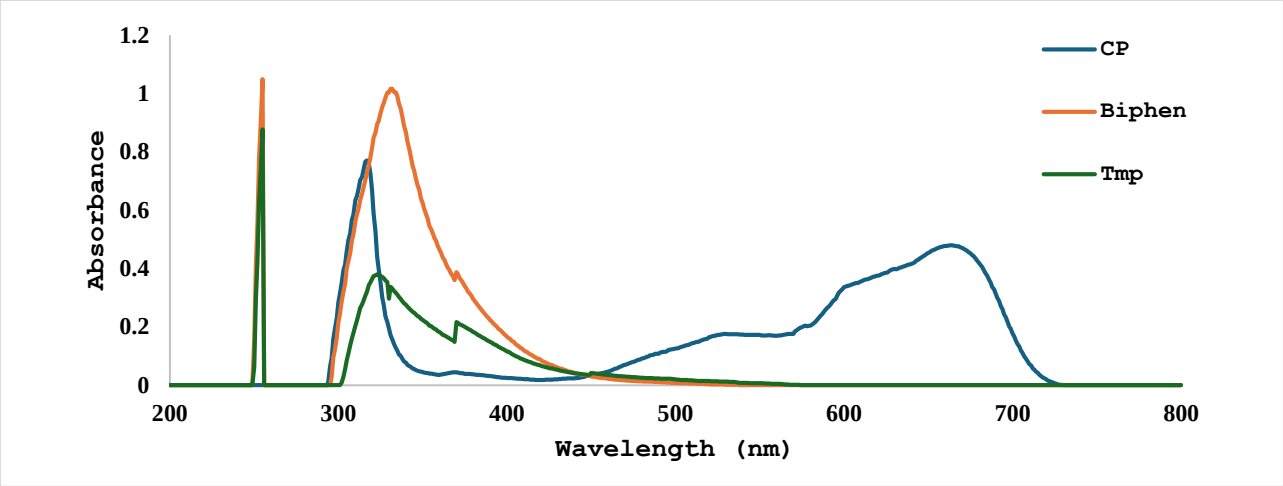


Figure 2: UV/Vis spectra of $[Co(Biphen)_2(Tmp)_2]_n.H_2O$ and its ligands

Table 3: Electronic spectra data of CP and its ligands

Compound/ligand	λmax, nm (E, cm^{-1})	Assignment
Biphen	255 (39,216), 334 (29,940)	π → π*/n → π*
Tmp	270 (37,037), 328 (30,487)	π → π*/n → π*
CP	318 (31,447), 669 (14,948)	LMCT, d → d

Photoluminescent Spectroscopy

Luminescence measurement was carried out on the CP as-synthesised in the solid state and at room temperature. The photoluminescence (PL) spectra of $[Co(Biphen)_2(Tmp)_2]_n.H_2O$ and its free ligands are shown in Figure 3. The Biphen ligand exhibits emission at 640 nm upon excitation at 318 nm, which is assigned to π → π* of an extended aromatic or conjugated ligand that is stabilised in the excited state. On the other hand, the Tmp ligand is blue-shifted with emission wavelength centered at 595 nm attributed to intraligand π → π* singlet transitions (Aazam *et al.*, 2012). $[Co(Biphen)_2(Tmp)_2]_n.H_2O$ showed emission at 630 nm (Excitation = 318 nm) that is slightly blue shift (10 nm) with reduced intensity. This may be assigned to ligand to metal charge transfer (LMCT) because of chelation to the metal (Meng *et al.*, 2019).

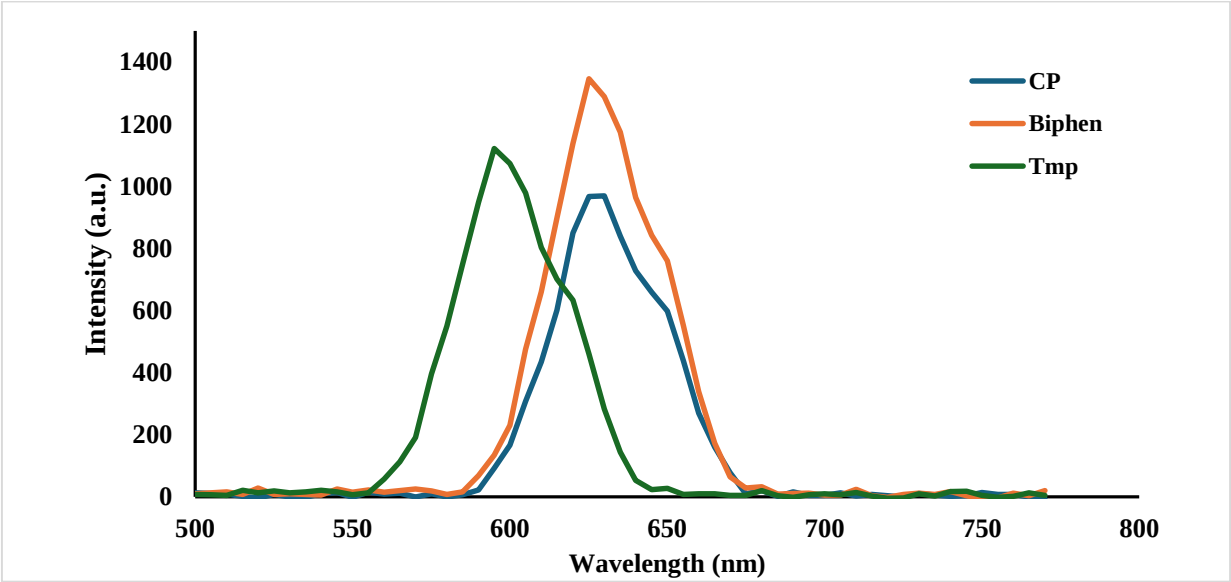


Figure 3: Photoluminescence spectra of $[Co(Biphen)_2(Tmp)_2]_n(H_2O)_2$ and its ligands

Thermogravimetric Analysis

The thermogravimetric analysis (TGA) curve of the synthesised cobalt-based coordination polymer, $[Co(Biphen)_2(Tmp)_2]_n.H_2O$ is displayed in Figure 4. The thermal decomposition profile reveals three stages of weight-loss corresponding to distinct structural changes in the CP. The initial weight loss of approximately 4 % between 25 °C and 298.39 °C is attributed to the removal of two coordinated water molecules. The second decomposition step, occurring from 298.39 °C to 394.29 °C, shows a significant weight reduction from 87.15 %, indicating the thermal degradation of the Tmp ligand. This accounts for a weight loss of 27.5 %, which corresponds well to the expected decomposition range for such an organic moiety. Between 394.29 °C and 453.97 °C, a further weight loss to 52.44 % is observed, likely due to the partial breakdown of the Biphenyl-4,4-dicarboxylate ligand. This minor loss (7 %) may reflect fragmentation of the aromatic backbone or loss of carboxyl groups. The final major decomposition phase occurs between 453.97 °C and 566.6 °C, resulting in a substantial weight loss of 33.4 %, leaving a stable residue of CoO at 19.03 %.

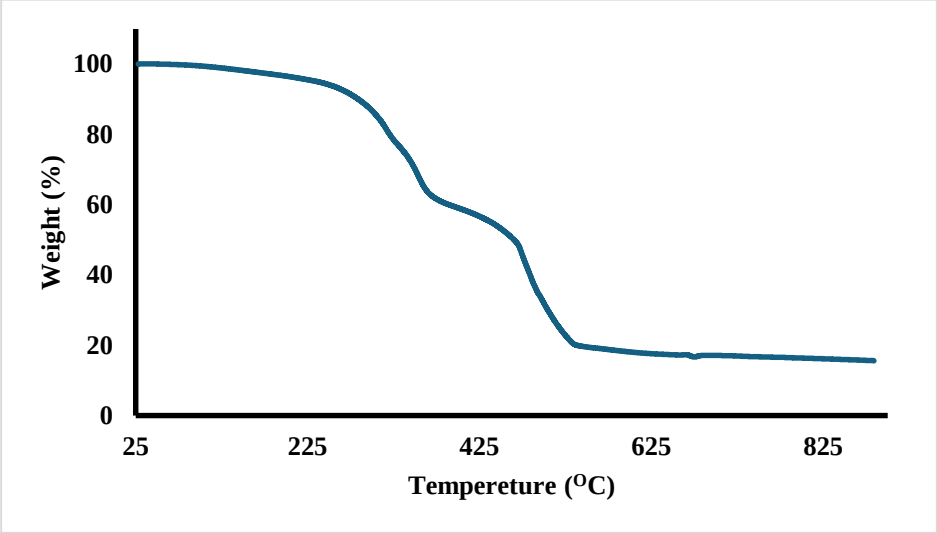


Figure 4: TGA curve of $[Co(Biphen)_2(Tmp)_2]_n(H_2O)_2$

Scanning Electron Microscopy

The surface morphology of the synthesized cobalt-based metal-organic framework $[Co(Biphen)_2(Tmp)_2]_n.H_2O$ was examined using scanning electron microscopy (SEM), and the image is presented in Figure 5. The SEM micrograph reveals a dense and irregularly aggregated structure composed of micro- to nanosized particles. These particles exhibit a rough surface texture and appear to be interconnected, forming clusters with a cauliflower-like morphology. This agglomerated architecture is typical of MOFs synthesized via solvothermal methods, where rapid nucleation and subsequent crystal growth often lead to irregular particle distribution. The image also indicates the presence of voids and interparticle spaces, suggesting the formation of a porous network within the framework (Zhang *et al.*, 2018).

Fluorescence Quenching Analysis

The CP, $[Co(Biphen)_2(Tmp)_2]_n.H_2O$ displayed a clear response sensitivity towards the organic solvents with significant quenching of luminescent intensity upon exposure to dichlorvos with the appearance of a weak band at 725 nm, which may be due to solvent effect. The luminescent quenching is in the order of Dichlorvos > DMSO > DMF > Ethanol > Methanol (Figure 6). The quenching percentages are 6.7 %, 53.0 %, 61.9 %, 66.9 % and 96.7 % for methanol, ethanol, DMF, DMSO and dichlorvos, respectively. The fluorescence intensity of the CP decreased gradually upon incremental addition of dichlorvos (Figure 7). This further confirms the sensitivity of the CP to dichlorvos. The fluorescence data were fitted to stern volmer equation ($\frac{Io}{I} = KSv[Q] + 1$) to give a linear curve with regression value, $R^2 > 0.9$. The stern volmer constant (KS*v*) was found to be 2.8 x 10^3 M^{-1}which indicates the (Figure 8).

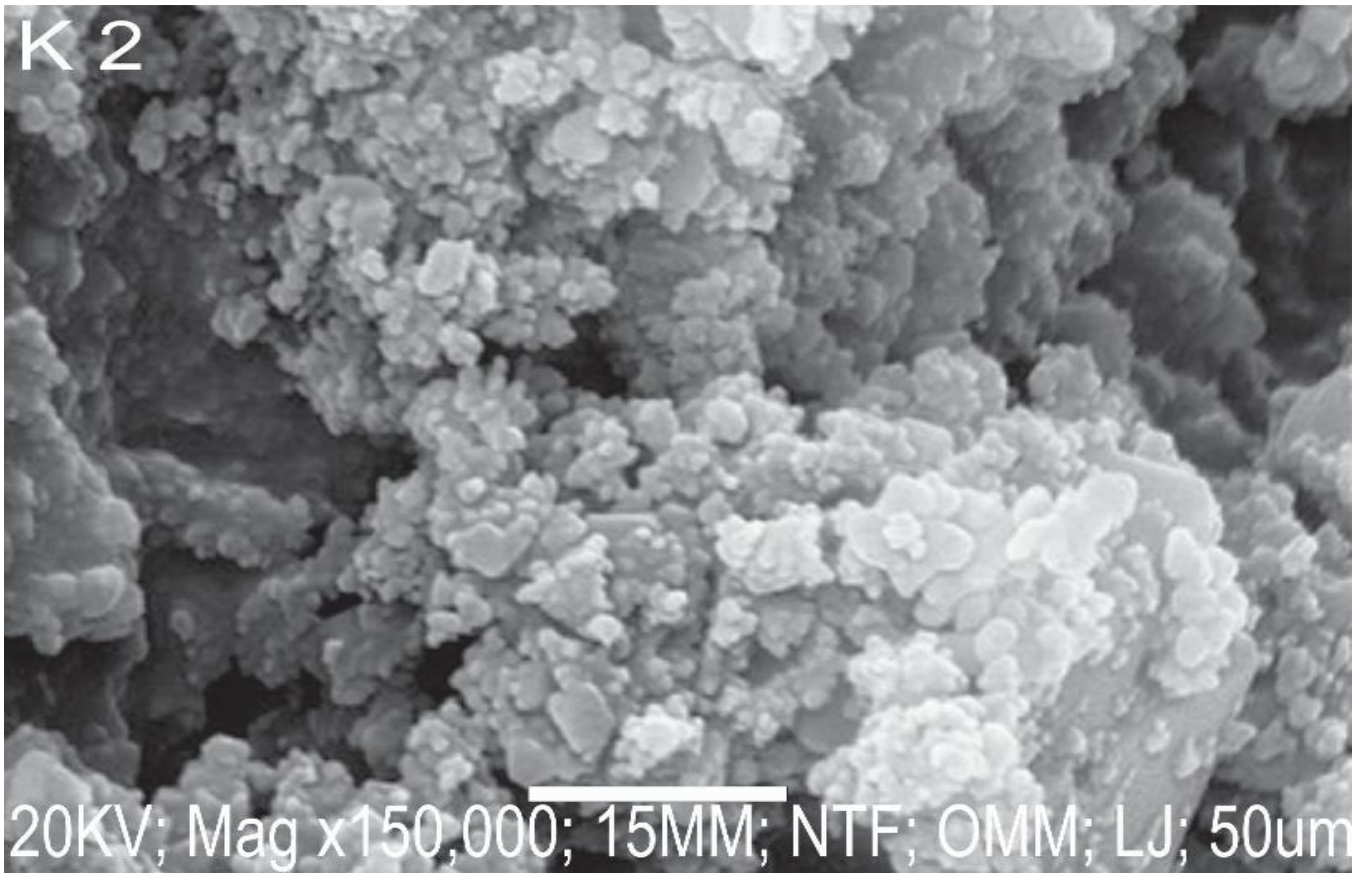


Figure 5: SEM image of $[Co(Biphen)_2(Tmp)_2]_n.H_2O$

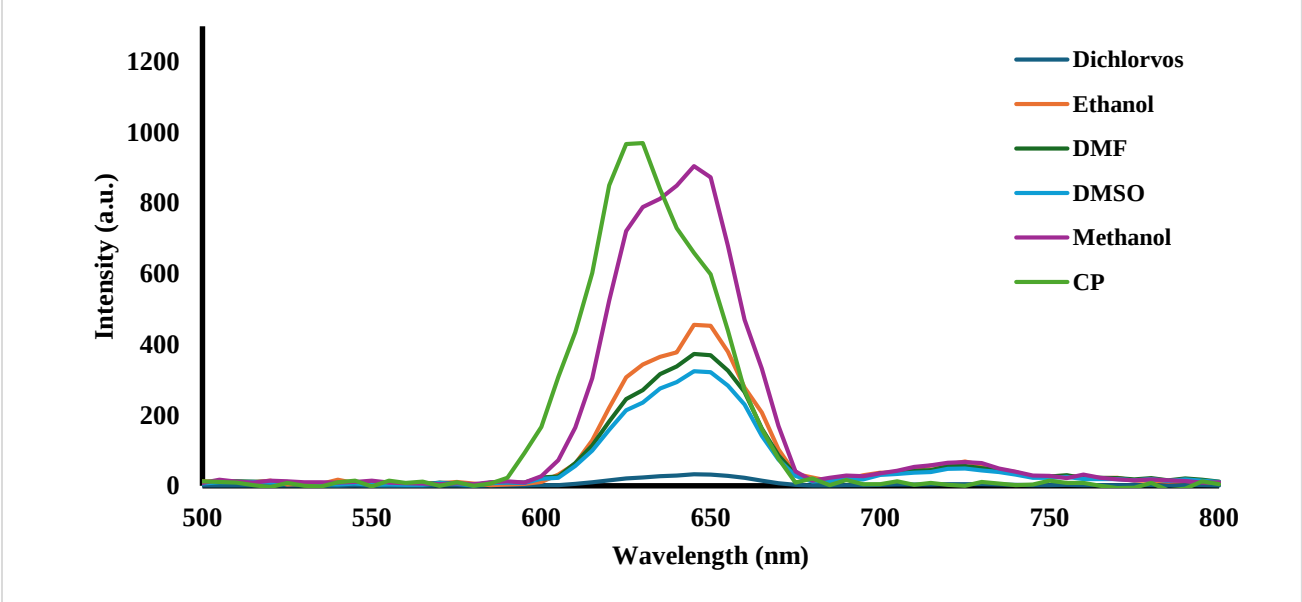


Figure 6: Luminescence intensities of CP in different organics

The mechanism of interaction may be due to photoinduced electron transfer (PET) where P=O of dichlorvos coordinates to open metal sites of the CP, or via hydrogen bond interactions in the pores of the CP. Upon excitation, an electron in the ligand-centred S_1 state can transfer to the low-lying LUMO of dichlorvos, providing an ultrafast non-radiative PET path (Zhang *et al*., 2020; Narayanan *et al*., 2023).

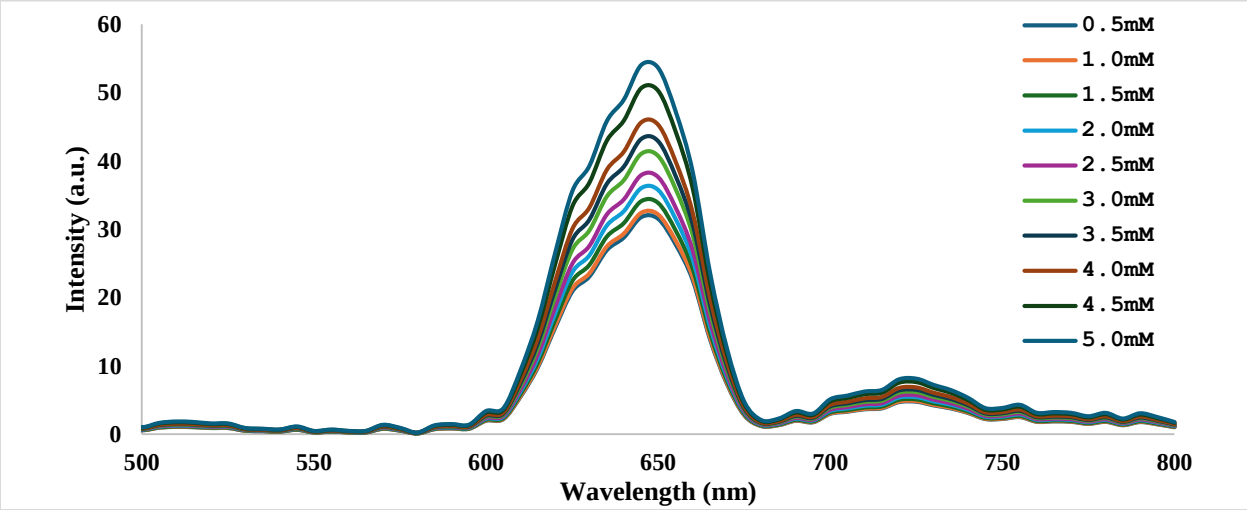


Figure 7: Emission spectra of CP dispersed in DMF with incremental addition of dichlorvos

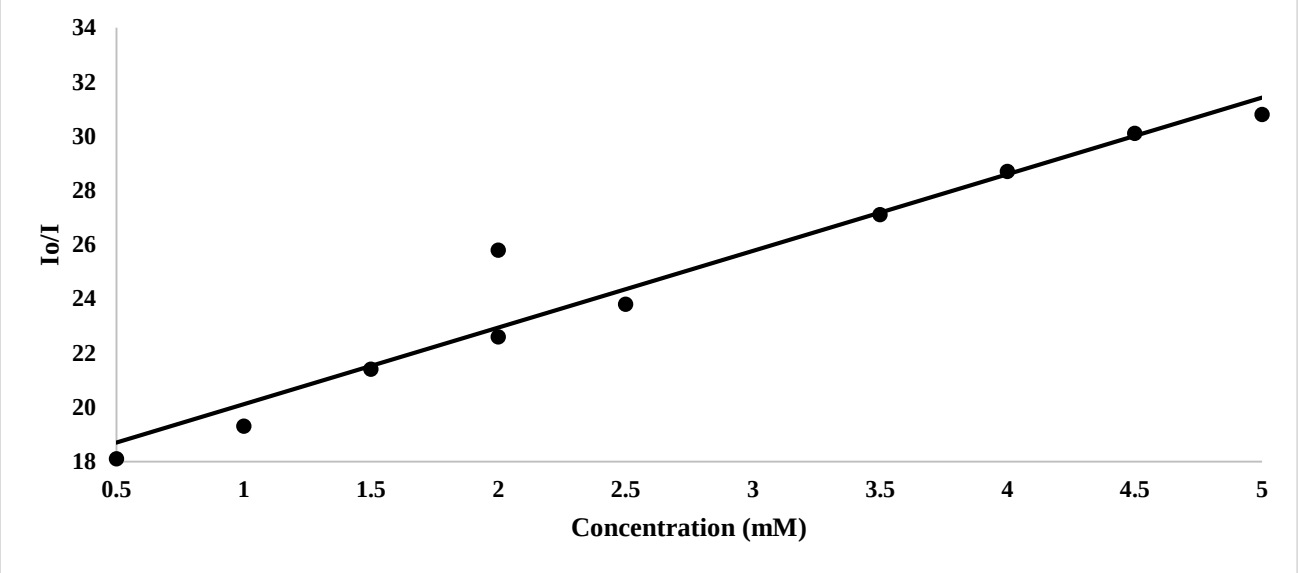


Figure 8: Stern-Volmer plot of CP@dichlorvos

CONCLUSION

A luminescent coordination polymer (CP) was successfully produced and fully characterised via standard physicochemical and spectroscopic techniques, demonstrating structural integrity and stability. Photoluminescence investigations showed that the CP emits light strongly and is highly sensitive to guest analytes. Dichlorvos exhibited a significant quenching effect among the studied solvents and pesticides, with quenching efficiencies above 95%, showing its strong electron-accepting properties. Mechanistic studies show that the primary form of fluorescence attenuation is photoinduced electron transfer (PET), which is aided by the interaction of the electron-rich excited states of the CP with the electron-deficient dichlorvos molecules. Additional contributions from static adsorption on the CP surface, as well as potential dynamic quenching pathways, may increase the sensing response.

These findings show that the synthesised CP is not only structurally robust but also a useful fluorescent sensor for harmful organophosphate pesticides. Its high quenching efficiency, selectivity, and sensitivity highlight its potential for use in chemical sensing, food safety, and environmental monitoring, providing a

viable approach for the development of advanced CP-based sensors.

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