Research Square

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Synthesis, Crystal Structure and Photoluminescence Properties of a Cadmium (II)-Based Metal–Organic Framework Constructed from Cinnamic Ligands

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Research Article

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Abstract

Cadmium (II) acetate, cinnamic acid as the major organic ligand, have been used to create a cadmium-based coordination polymer. The ligand method permits effective coordination of Cd^{2+} ions through the carboxylate groups of cinnamic acid resulting in the development of a stable two-dimensional supramolecular network. Hydrogen bonding and π–π stacking interactions further strengthen the structure, resulting in a well-defined crystalline framework with good thermal stability. The synthesized crystal characterized by IR spectroscopy, UV spectroscopy and single crystal analysis X-ray diffraction analysis. Photoluminescence study of polymer is also done in this work.

Introduction

In the field of molecular luminescence, the synthesis and study of mono or polynuclear coordination polymers has been a hot topic because they serve as building blocks for molecular-based materials and aid in our understanding of the mechanism underlying coupling. Molecular-based magnets like oxo, chlodrido, hydroxide, alkoxo, carboxylate, or cinnamic acid are frequently employed to react with metal ions[1]. Metal–Organic Frameworks (MOFs) are an intriguing class of crystalline porous materials that combine the utility of inorganic coordination networks with the adaptability of organic chemistry[2]. Coordination of metal ions or clusters with multidentate organic linkers creates coordination polymer, which are extended frameworks with large surface area, adjustable pore size, and superior chemical stability[3]. These special qualities have led to the widespread usage of MOFs and related coordination complexes in gas storage, separation, heterogeneous catalysis, drug delivery, optical materials, and magnetic systems[4]. The shape, bonding environment, and final functioning of MOFs are determined by the careful selection of both the metal center and the organic linkers. Because of their strong affinity for metal ions and their capacity to adopt a variety of coordination modes, carboxylic acids are among the most important organic ligands employed in MOF production[5] The α,β-unsaturated aromatic carboxylic acid cinnamic acid ($C_6H_5CH=CHCOOH$) is especially intriguing as a ligand because of its extended π-conjugation and a stiff molecular structure. A conjugated double bond and a phenyl ring both promote π–π interactions and improve framework stability. Through its carboxylate oxygen atoms, cinnamic acid can bind to metal centers and function as a bidentate or monodentate bridging ligand[6]. Cinnamic acid is a useful building block for creating functional metal-organic structures because complexes made from it frequently exhibit appealing optical, electrical, and catalytic properties. The d^{10} closed-shell transition metal ion cadmium (II) is widely known for its great propensity to form bonds with donor atoms of carbon and oxygen as well as its variable coordination behaviour. The bidentate carboxylate ligands combined build a stable coordination sphere around cadmium, leading to the production of ligand coordination polymers or two-dimensional MOF-like structures depending on the synthesis circumstances. These ternary systems, which combine with cinnamic acid, provide a synergistic method for creating sophisticated metal-organic compounds with specific physicochemical characteristics[7]. While the conjugated cinnamic ligand improves optical and electrical characteristics. Therefore, the

cadmium–cinnamic acid system offers a flexible framework for creating new coordination polymer and MOFs with possible uses in biological chemistry, optical materials adsorption, and catalysis[8].

Experimental

Materials and Physical Measurements

All chemicals were purchased from commercial sources and used without further purification. Ethanol, Distilled water, Cadmium(II) acetate, cinnamic acid (99%), and Nicotinamide.

Synthesis of Coordination polymer

Separate crystals with the molecular formula Cd-$C_{18}H_{18}O_6$, $[Cd(C_6H_5CHCHCOO)_2].2H_2O$,an aqueous solution (10 ml) of Cadmium acetate (0.533g,mmol), and Cinnamic acid (0.296g,2mmol)was slowly added drop wise to hot aqueous solution (10ml) of Nicotinamide (0.244g, 2mmol) with stirring. Transparent colour solution was obtained. After filtration the final clear solution left undisturbed at room temperature for slow evaporation. After one week, needle shaped transparent crystals were collected and dried in vacuum over silica gel.

Crystallographic Data and Structure Refinement Details

Data collection: SMART (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

Crystal Structure determination of the Coordination polymer

The coordination polymer crystallizes in monoclinic space group C 2, according to single-crystal X-ray study. The values of cell parameters a, b, and c are 10.785(2), 6.3592(18), and 12.196(7) in Table 1. Single monomeric compounds make up the crystal structure. The Cd ion is present in the centre and surrounded by six oxygen atoms from organic linker and water molecules[9]. The single-crystal X-ray diffraction investigation demonstrates that the synthesized metal–organic framework (MOF) comprising cadmium ions, cinnamic acid, form a well-defined two-dimensional coordination network. The center of cadmium (II) have a deformed octahedral shape and function as coordination nodes[10]. Oxygen atoms from the carboxylate groups of cinnamic acid ligands coordinate each Cd^{2+} ion. By joining neighboring cadmium centres in a bidentate bridging coordination state, the cinnamic ligands create long polymeric chains. A strong supramolecular framework is produced by hydrogen bonding interactions and π–π stacking between the aromatic rings of cinnamic acid, which further strengthen these chains. According to the crystal structure, Cd–O bond lengths are usually between 2.25 and 2.40 Å, which is compatible with Cd–O coordination in frameworks that are comparable. In addition to creating one-dimensional channels that might make it easier for guest molecules to be included, the interlayer hydrogen bonds (O–C-O) contribute to the overall stability of the framework. Thermal stability and structural stiffness are improved by π–π interactions between aromatic rings. Overall, the crystal structure can be characterized

as a three-dimensional coordination polymer composed of mixed organic linkers (cinnamic acid) and Cd (II) nodes, resulting in a stable MOF with promise for photoluminescent applications[11]. The carboxylate oxygen of cinnamic form O–C-O hydrogen bonds and π–π stacking interactions between the aromatic rings of cinnamic acid facilitate further supramolecular assembly of these layers. These weak connections solidify the framework into a three-dimensional supramolecular network, providing extra rigidity and stability. One-dimensional channels that run along the crystallographic b-axis are seen throughout the structure, indicating possible porosity. These channels may serve as adsorption sites for tiny guest molecules since they are lined with polar functional groups and aromatic rings. The adaptability of Cd (II) in coordination polymer design is highlighted by the high degree of structural order and the coexistence of coordination and non-covalent interactions. The extended three-dimensional network of the Cd–coordination polymer and the coordinating environment surrounding the Cd (II) center would normally be depicted in Fig1. The interlayer hydrogen-bonding and π–π stacking interactions that sustain the framework would be further illustrated by the crystal packing diagram. In this structure cadmium atom has six coordination number four comes from oxygen of cinnamic acid ligand and other two from oxygen of water. The bond distances of bond Cd1-O11 2.236-2.330Å, Cd1-O12 2.34-2.44Åand Cd1-O14 2.30-2.31Å respectively.

Fig1.The Single Crystal Structure of $[Cd(C_6H_5CHCHCOO)_2].2H_2O$ Coordination polymer showing Ellipsoid Model

The precise molecular and crystal structure of the synthesized Cd (II) coordination polymer was clarified using single crystal X-ray diffraction investigations. Cd (II) coordination polymer radiation (λ = 0.71073 Å) at room temperature was used to collect data on a high-quality single crystal.[12] The Cd (II) ion takes on a six-coordinated deformed octahedral shape, according to crystallographic research. Oxygen atoms from cinnamic ligands complete the coordination environment surrounding the metal center. Both ligands effectively participate in metal binding, as confirmed by the observed coordination mode. Through their carboxylate oxygen atoms, the cinnamic ligands bind to the Cd (II) core, displaying monodentate/bridging coordination activity that connects nearby cadmium ions. An extended coordinating structure is created as a result of this connectedness. Stable metal–ligand interactions are indicated by the Cd–O bond lengths, which fall within the typical range reported for comparable Cd (II) coordination polymer. Chelation limitations and ligand steric effects are responsible for the departure from perfect octahedral geometry. The carboxylate oxygen atoms of cinnamic ligands exhibit substantial intermolecular O–C-O hydrogen bonding interactions in the crystal packing. The supramolecular architecture is stabilized in large part by these hydrogen bonds. A two-dimensional network is also formed by weak π–π stacking interactions between aromatic rings. The successful creation of the cadmium (II) coordination polymer is confirmed by the overall stable and well-organized crystal structure produced by the combined coordination contacts and supramolecular forces[13].

Fig2.View of the 1D network of Coordination Polymer$[Cd(C_6H_5CHCHCOO)_2].2H_2O$

The cadmium (II) coordination polymer crystal structure produced a one-dimensional (1D) coordination polymer. The structure makes it evident that the carboxylate groups of the cinnamic ligands join the Cd (II) ions to form a continuous polymeric chain. A recurring Cd–O–C–O–Cd connection is created when each Cd (II) centre is connected to nearby metal ions by bridging carboxylate oxygen atoms. As seen in the image, this coordination mode results in the creation of an infinite one-dimensional chain that moves down the crystallographic b-axis[14].

Fig3.View of the 2D network of Coordination Polymer[$Cd(C_6H_5CHCHCOO)_2$].$2H_2O$

The two-dimensional (2D) coordination network of the Cd (II) coordination polymer, The Cd (II) ions are interconnected through the bridging carboxylate groups of cinnamic ligands, resulting in an extended sheet-like architecture. The development of endless 2D layers results from the repetitive Cd–O–C–O–Cd connection propagating along the crystallographic a- and b-axes. A stable crystal packing results from intermolecular hydrogen bonding interactions that further solidify the neighboring layers[15].

Table-1(Crystal data and structure refinement parameters for Cd(II) polymer)

Chemical formula	$C_{18}\ H_{18}\ Cd\ O_6$
Formula weight	442.74
Colour	transparent
Lattice Type	monoclinic
Space group	C 2
a	10.785(2)
b	6.3592(18)
c	12.196(7)
α	92
β	99.31(2)
γ	92
Cell volume(A)	824.32
Z	2
Z’	0

Table 1 summarizes crystallographic parameters such as unit cell dimensions, space group, volume, Z value, estimated density, absorption coefficient, number of collected and independent reflections, R indices, and refinement information.

Table-2.(List of Bond Angles)

No.	Bond	Angle
1	O11-Cd1-O12	50.6
2	O11-Cd1-O14	133.1
3	O11-Cd1-O11	121.1
4	O11-Cd1-O12	79.6
5	O11-Cd1-O14	98.2
6	O12-Cd1-O14	115.1
7	O12-Cd1-O11	82.0
8	O12-Cd1-O12	76.1
9	O12-Cd1-O14	146.7
10	O14-Cd1-O11	94.8
11	O14-Cd1-O12	146.0
12	O14-Cd1-O14	75.7
13	O11-Cd1-O12	53.6
14	O11-Cd1-O14	130.1
15	O12-Cd1-O14	113.4
16	Cd1-O11-C11	99.4
17	Cd1-O12-C11	95.8

A deformed octahedral geometry is confirmed by the bond angles surrounding the Cd (II) centre. Because of steric effects and ligand coordination modes, the cis and trans bond angles differ slightly from ideal values. Distortion in the coordination geometry is indicated by the O–Cd–O and C–Cd–O bond angles, which differ from the ideal octahedral values of 90° and 180°. The six-coordinate environment of the Cd (II) ion is confirmed by the trans angles near 180°, the flexibility of the carboxylate binding mode is reflected in the fluctuation in cis angles 90. The suggested structural geometry is further supported by the fact that these bond angle values are similar to those reported for related cadmium (II) coordination polymer[16].

Table-3. List of Bond length

BOND	Length	BOND	Length
Cd1-O11	2.330	C12-C13	1.30(3)
Cd1-O12	2.44	C12-H12	0.850
Cd1-O14	2.31	C13-C14	1.50(2)
Cd1-O11	2.236	C13-H13	0.86
Cd1-O12	2.34	C14-C15	1.27(1)
Cd1-O14	2.30	C14-C19	1.39(3)
O11-C11	1.25(1)	C15-C16	1.36(3)
O12-C11	1.19(1)	C15-H15	0.92
O14-H1	0.765	C16-C17	1.41(3)
O14-H2	0.91	C16-H16	0.84
C11-C12	1.62(3)	C17-C18	1.23(2)
C12-C13	1.31(3)	C17-H17	0.93
C12-H121	0.854	C18-C19	1.36(4)
C13-C14	1.58(2)	C18-C19	1.36(4)
C13-H131	0.86	C17-H17	0.98
C14-C15	1.28(1)	C18-C19	1.43(4)
C14-C19	1.40(3)	C18-H18	0.92
C15-C16	1.44(3)	C19-H19	0.85
C15-H15	0.92	O11-C11	1.24(1)
C16-C17	1.41(3)	O12-C11	1.18(1)
C16-H16	0.85	O14-H1	0.755
C17-C18	1.24(2)	O14-H2	0.94

Fig4.This figure showing bond length

Single crystal X-ray diffraction analysis is used to summarize the chosen bond lengths surrounding the Cd (II) metal core, Stable metal–ligand interactions are confirmed by the Cd–O distances, which are within the typical range reported for comparable Cd (II) coordination polymer. Strong coordination between the metal center and oxygen donors is shown by the Cd–O bond lengths resulting from cinnamic carboxylate oxygen atoms, which are found in the range of 2.20–2.50 Å. The deformed

octahedral geometry surrounding the Cd (II) ion and various coordination modes of the cinnamic ligand are responsible for the common variation in bond lengths. These values are in line with Cd (II) complexes with mixed O- donor ligands that have been previously reported, The Cd (II) complexes crystal structures hydrogen bonding interactions. The packing graphic demonstrates unequivocally that a combination of weak O–C–O interactions and carbon oxygen bonds. There are significant O–C-O carbon oxygen bonding because the carboxylate oxygen atoms of the cinnamic ligands act as hydrogen bond acceptors. These interactions are crucial to the development of the supramolecular architecture because they link nearby coordinating units. Additionally, a number of O–C-O interactions are seen between the oxygen atoms of the carboxylate moieties and aromatic C–H groups. These interactions play a major role in the overall stability of the crystal packing, never the less their weaker character. The illustration illustrates how the hydrogen bonds connect the molecular units into an extended three-dimensional hydrogen-bonded network along the crystallographic b-axis. Strong and weak hydrogen bonding interactions work together to improve the crystal lattice stiffness and structural stability[17].

Fig5.Crystal structure showing molecules arranged along [010]. Hydrogen bonds are shown as blue dotted lines.

Fourier Transform Infrared (FT-IR) Analysis

Fig6. FT-IR spectra of $[Cd(C_6H_5CHCHCOO)_2].2H_2O$

Fourier-transform infrared (FTIR) spectroscopy was used to identify the functional group on the compounds surface. This analysis was conducted at the Motilal Nehru National Institute of Technology, Allahabad Center of Interdisciplinary Research, using a spectrum 300 cm^{-1} FT-IR spectrometer (Perkin Elmer, USA). The observation was conducted in the mid-infrared range of 300–4000 cm^{-1} using the potassium bromide (KBr) Pellet method. FT-IR analysis can be used to identify specific functional groups and chemical bonds in a coordination polymer. FT-IR was used to analyze the coordination polymer in the 4000–300 cm^{-1} range[18]. The obtained spectra are displayed in Fig. 6. Significant alterations in the distinctive bands of cinnamic acid following coordination are visible in the FTIR spectra of the cadmium complex. The free carboxylic groups C=O stretching vibration (~1700 cm^{-1}) shifts or vanishes, and two new absorption bands that correspond to the asymmetric and symmetric stretching vibrations of the carboxylate (COO^-) group, respectively, emerge in the regions ~1550–1600 cm^{-1} and ~1350–1400 cm^{-1}. A stable cadmium–cinnamate complex is formed when the cinnamate ligand coordinates with the Cd(II) ion through oxygen atoms, as further confirmed by the emergence of an extra band in the low-frequency region ~450–550 cm^{-1} that is attributable to Cd–O stretching[19].

Table-3.High frequency region-

	3300-2500cm-1	(Broad) O-H stretching
	3100-3000cm-1	Aromatic =C-H stretching
	2900-2850cm-1	Aliphatic C-H stretching

UV-Visible spectroscopy

Using the proper solvent, the synthesized cadmium (II) complexs UV–visible absorption spectra was measured between 300 and 600 nm. The spectrum displays distinctive absorption bands that correspond to n→π* and ligand-centered (π→π*) electronic transitions[20]. The π→π* transitions of the aromatic rings found in cinnamic acid ligands are responsible for a significant absorption band seen in the 350–480 nm range. The n→π* transitions connected to the carbonyl (C=O) functional groups are responsible for another absorption band that appears in the 480–550 nm range. No d–d transitions are anticipated in the visible area since Cd (II) is a d^{10} metal ion.[21] This behavior is confirmed by the lack of any noticeable absorption band above 400 nm. The electronic transitions are primarily ligand-based, according to the observed spectrum characteristics. Complex formation is confirmed by the minor shift in absorption bands when compared to free ligands, which indicates coordination of cinnamic oxygen atoms with the Cd (II) core[22].

Fig7.UV-Visible spectra of $[Cd(C_6H_5CHCHCOO)_2].2H_2O$

Photoluminescence

Fig.7 displays the photoluminescence (PL) spectra of compounds based on cinnamic acid and cadmium. Their room-temperature fluorescence pattern has been investigated in the solid form. This compound has a noticeable emission band at 550 nm. When Emission energy (E) is 2.25ev. The observed emission pattern indicates that the interaction of ligands with Cd (II) ions increases the stiffness of the structure, hence reducing non-radiative decay pathways. There is also a noticeable rise in light intensity. Because Cd (II) has a d^{10} electronic structure, the emission is mostly controlled by the organic ligands rather than metal-centered transitions[23]-[24]. Organic ligands interact with Cd(II) ions to generate an extended three-dimensional framework that significantly increases the stiffness of the structure. This rigid structure boosts photoluminescence intensity by effectively blocking non-radiative decay paths. The strong and distinct emission peak in the PL spectrum shows that the synthesized MOF has a good luminous quality. These results suggest that the present MOF may be a good choice for usage in illuminating materials, chemical sensing, and optoelectronic devices.[25]

Fig.8Photoluminescence (PL) Spectra of compound

Conclusion

In conclusion, the synthesis and structural characterization of a novel coordination polymer based on Cd (II) have been completed. Hydrogen bonding interactions sustain the framework stable two-dimensional

structure. Strong emission behavior is revealed by photoluminescence experiments, indicating its possible use in luminous materials. The carboxylate oxygen atoms of the cinnamic ligands coordinate the Cd (II) ions to create an extended one-dimensional chain structure, according to single-crystal X-ray diffraction studies (Fig.2). The ligands coordination with the metal core is confirmed by the FT-IR measurements and UV-spectra. Additionally the compound shows significant photoluminescence behavior, which suggests that electronic transitions are controlled by the ligand. These findings show that the synthesized cadmium coordination framework has remarkable luminous characteristics and good structural stability, making it a viable option for luminescent applications.

Declarations

Funding The authors declare that no funding was received for this study.

Ethics Approval and Consent to Participate Not applicable.

Consent for Publication Not applicable.

Data Availability Statement The datasets generated and/or analysed during the current study, including crystallographic and spectroscopic data, are available from the corresponding author upon reasonable request.

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Figures

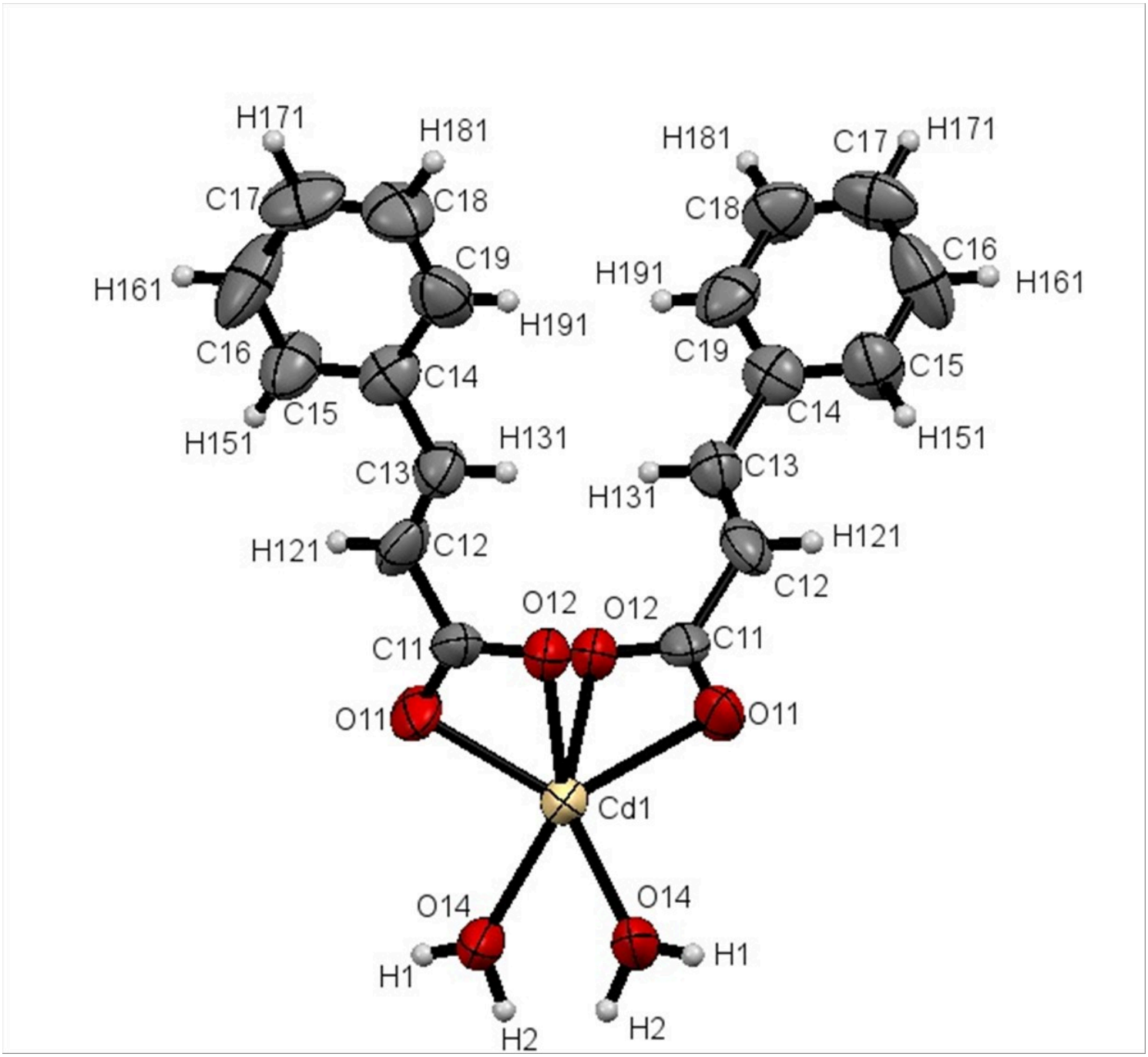


Figure 1

The Single Crystal Structure of $[Cd(C_6H_5CHCHCOO)_2].2H_2O$ Coordination polymer showing Ellipsoid Model

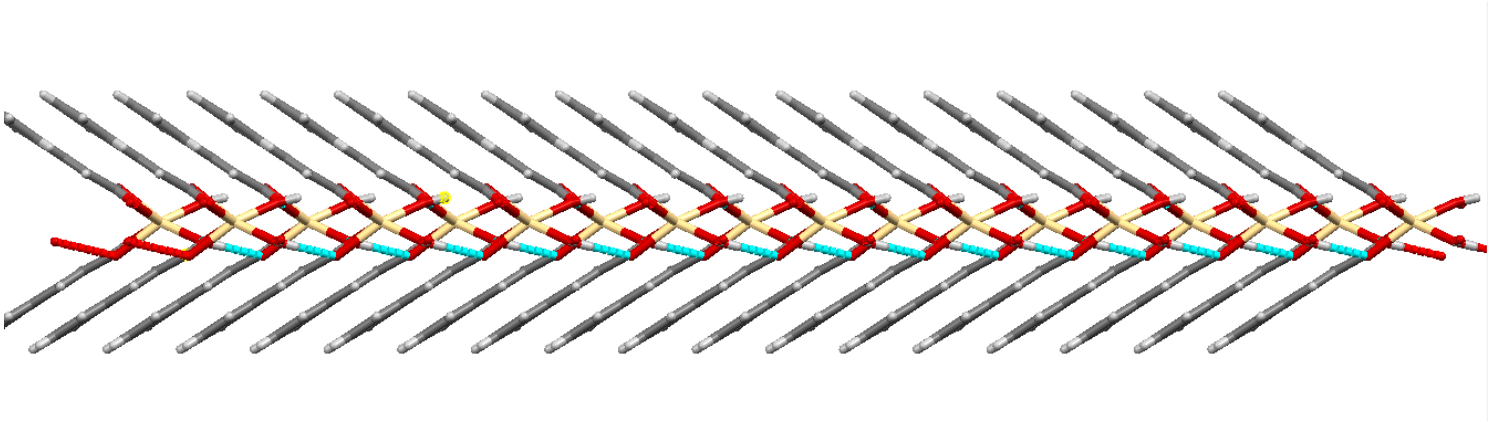


Figure 2

View of the 1D network of Coordination Polymer[$Cd(C_6H_5CHCHCOO)_2$].$2H_2O$

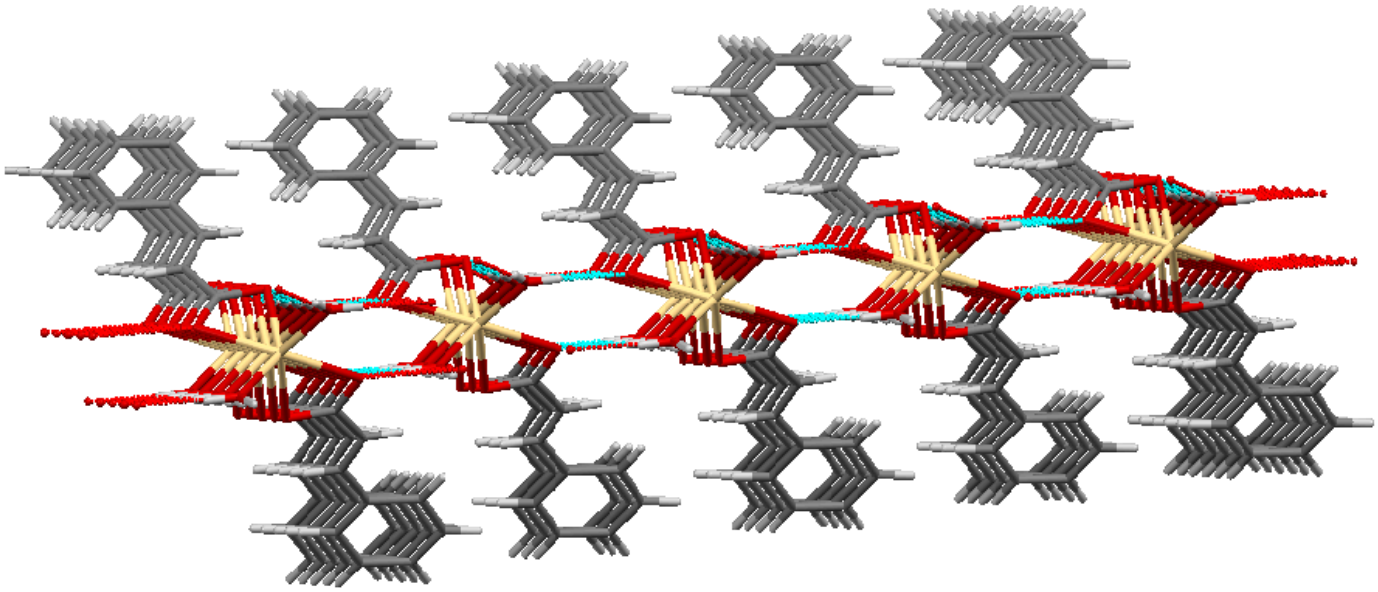


Figure 3

View of the 2D network of Coordination Polymer[$Cd(C_6H_5CHCHCOO)_2$].$2H_2O$

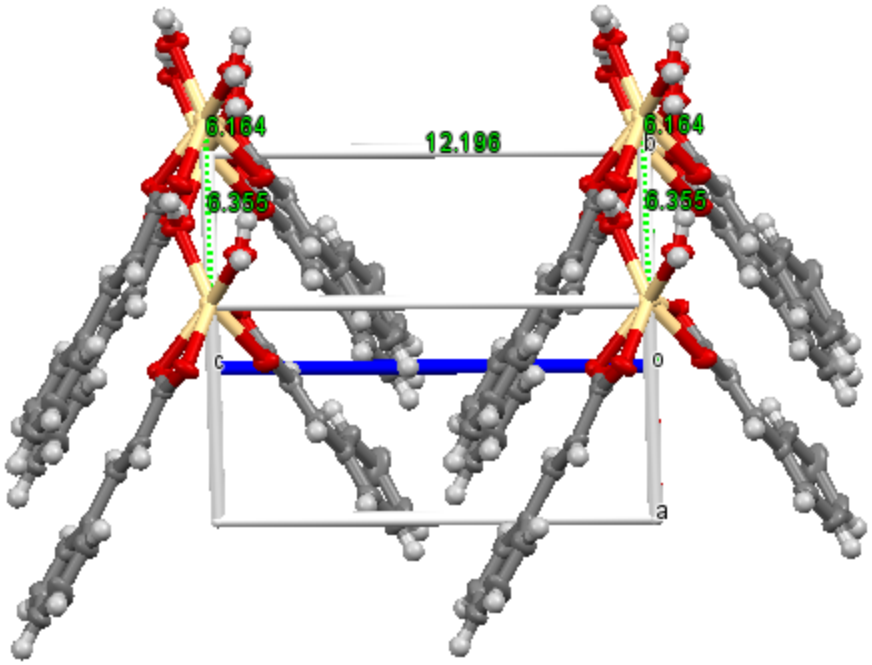


Figure 4

This figure showing bond length

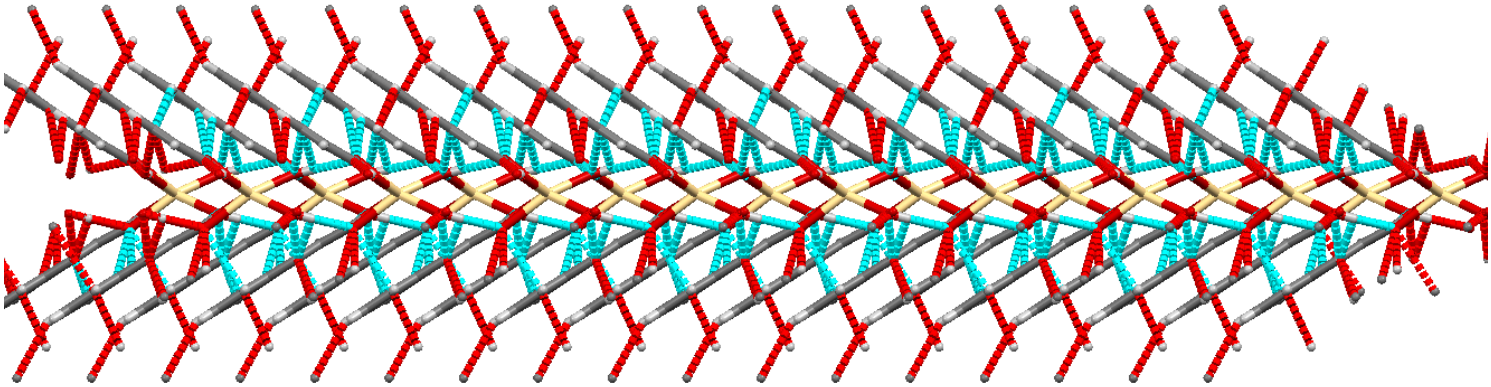


Figure 5

Crystal structure showing molecules arranged along [010]. Hydrogen bonds are shown as blue dotted lines.

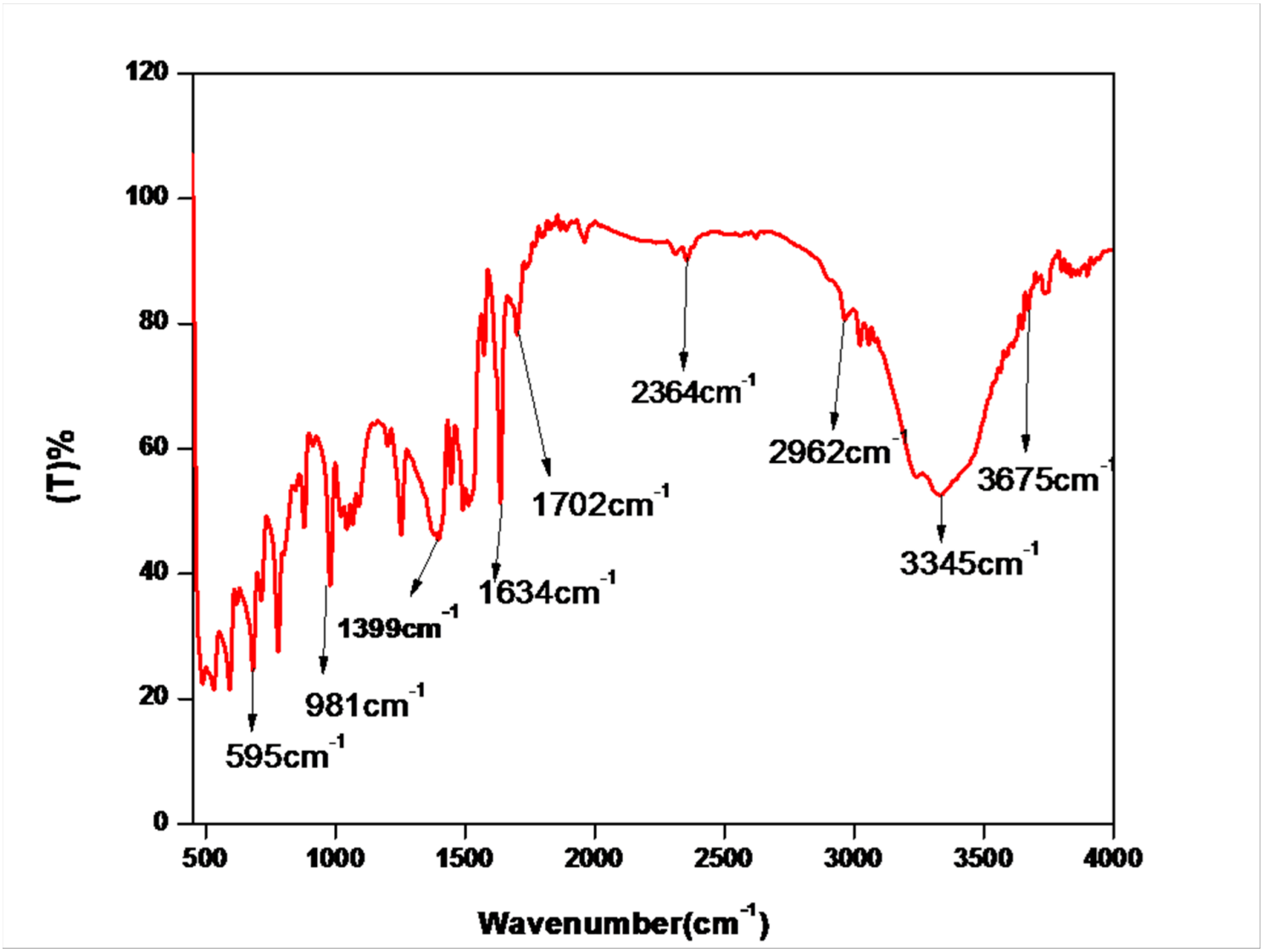


Figure 6

FT-IR spectra of $[Cd(C_6H_5CHCHCOO)_2].2H_2O$

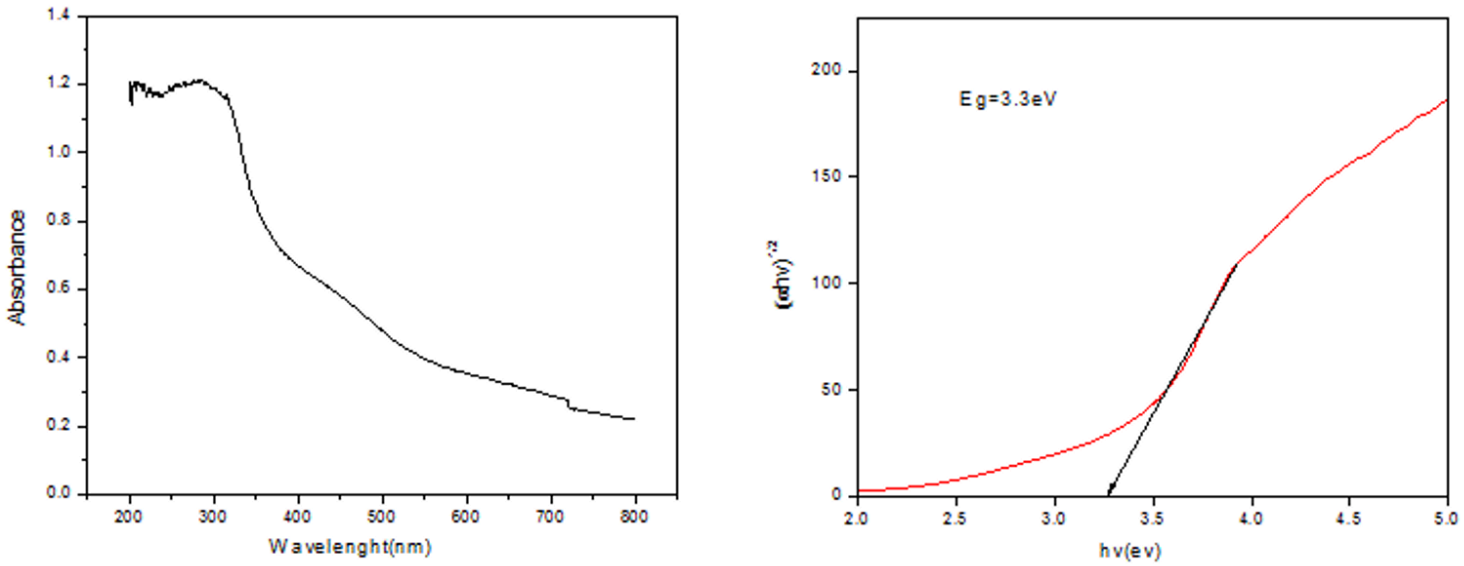


Figure 7

UV-Visible spectra of $[Cd(C_6H_5CHCHCOO)_2].2H_2O$

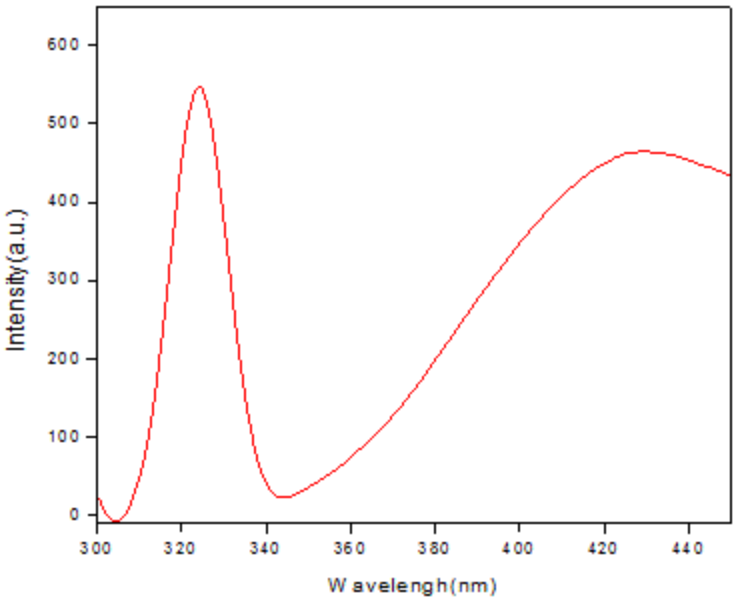


Figure 8

Photoluminescence (PL) Spectra of compound