



# SYNTHESIS, SPECTROSCOPY AND BIOLOGICAL PROPERTIES OF NICKEL AND PALLADIUM COMPLEXES DERIVED FROM SCHIFF BASE LIGANDS

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## Abstract

This research paper focuses on the synthesis, spectroscopic characterization and biological evaluation of nickel and palladium complexes derived from Schiff base ligands. Schiff base ligands have versatile coordinating abilities and potential biological activity in coordination chemistry. The study involves the synthesis of Schiff base ligands followed by their complexation with nickel and palladium. The resulting complexes are characterized using spectroscopic techniques such as UV-Vis spectroscopy, Fourier-transform infrared spectroscopy (FT-IR), and nuclear magnetic resonance (NMR). Furthermore, the complexes are evaluated for their biological activities against selected microorganisms, and the results are discussed.

**Index Terms-** Antibacterial, Nickel (II), Palladium (II) complexes, IR, UV-Vis, Schiff bases

## 1. Introduction

The synthesis, spectroscopy and biological properties of metal complexes derived from Schiff base ligands have garnered significant attention in the field of coordination chemistry. Schiff base ligands, which are derived from the condensation reaction between an aldehyde or ketone and an amine, possess versatile coordinating abilities and exhibit potential biological activities [1-3]. These ligands serve as excellent chelating agents, forming stable complexes with various transition metal ions.

Nickel and Palladium complexes derived from Schiff base ligands have gained particular interest due to their unique structural features and diverse applications. Nickel complexes have been widely studied for their catalytic, magnetic, and electronic properties, while palladium complexes are known for their applications in catalysis, medicinal chemistry and material science [4-6].

The synthesis of these complexes involves the reaction of the Schiff base ligands with nickel and palladium salts under appropriate conditions. The resulting complexes exhibit distinct spectroscopic properties that can be analyzed using various techniques such as UV-Vis spectroscopy, Fourier-transform infrared spectroscopy (FT-IR), and nuclear magnetic resonance (NMR) spectroscopy. These spectroscopic analyses provide valuable insights into the coordination geometry, electronic transitions, and structural characteristics of the complexes [7-9].

Furthermore, the biological properties of these complexes have been explored due to their potential as antimicrobial and anticancer agents. The presence of metal ions in the complexes, along with the inherent properties of the Schiff base ligands, contributes to their biological activities [10-12]. Evaluating the antimicrobial efficiency of these complexes against selected microorganisms and cancer cell lines provides valuable information for their potential therapeutic applications [13-15].

In this study, we aim to synthesize nickel and palladium complexes derived from Schiff base ligands and investigate their spectroscopic properties and biological activities. The comprehensive characterization of these complexes will contribute to the understanding of their structure-function relationships and shed light their potential applications in various fields.

## 2. Material and Instruments

The chemicals Nickel (II) chloride and Palladium (II) chloride were obtained from reputable suppliers such as Sigma Aldrich and SRL Chemicals, and they were of reagent grade. All reagents purchased were used as received without any further purification.

Infrared (IR) spectra in the range of 400-4000  $\text{cm}^{-1}$  were acquired using a Bruker VI-FTIR Spectrometer. Elemental analysis was conducted using a CHN-932 instrument from Perkin-Elmer 7300 DV elemental analyzers. The determination of content was performed gravimetrically using iodometric titration [16-17]. UV-Vis spectra were recorded using a JASCO V-670 UV-Vis spectrophotometer within the wavelength range of 200-800 nm. High-resolution mass spectrometry (HRMS) measurements were obtained in the ESI positive mode.

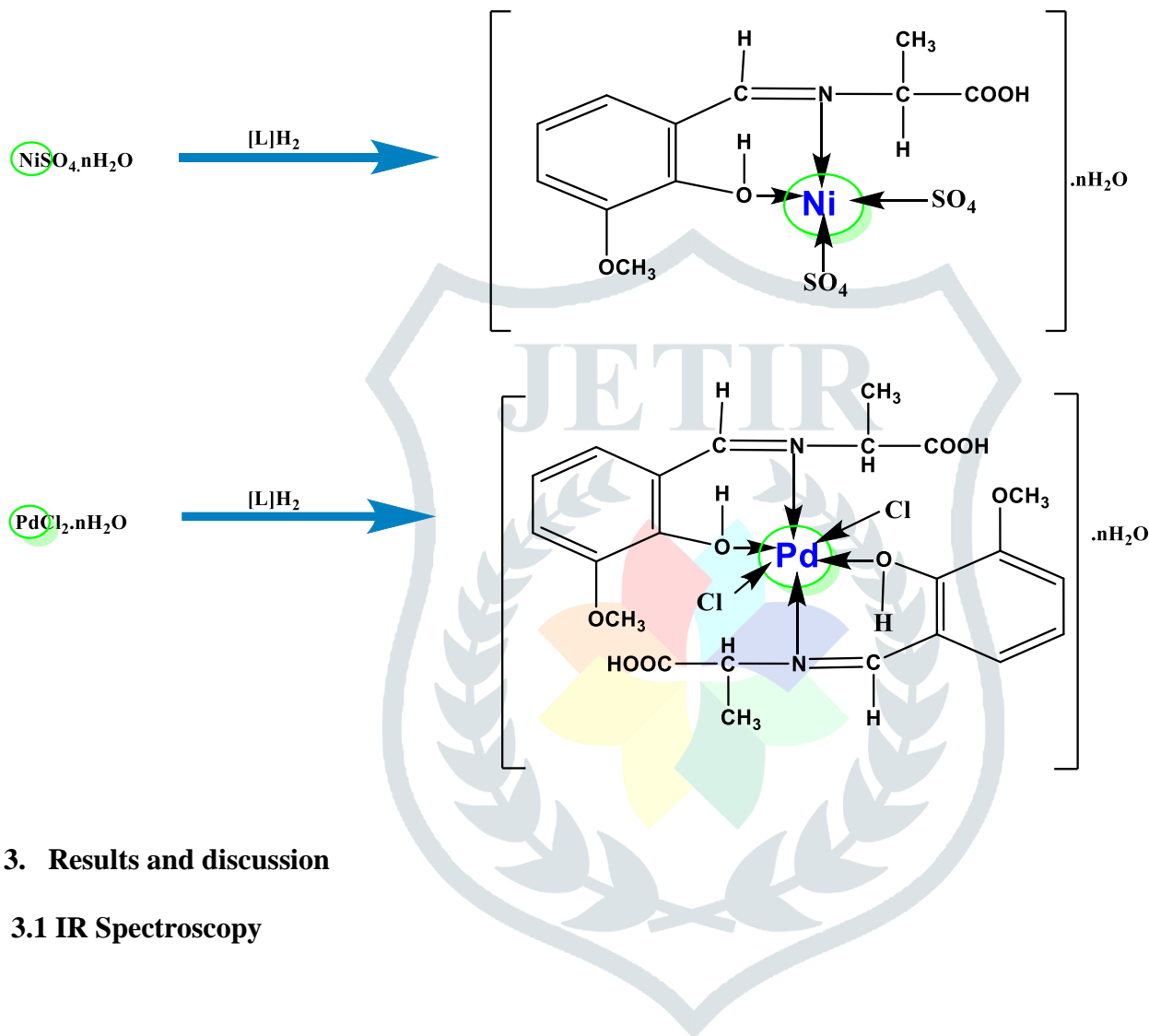
### 2.1 Synthesis of Schiff bases

The Schiff base ligands were synthesized with high yield and purity [18]. In a typical procedure, o-vanillin (1 equiv.) was dissolved in ethanol, and then a solution of alanine (1 equiv.) in ethanol and a small amount of hydrochloric acid were added. The resulting solution exhibited a characteristic color and was refluxed for approximately 6 hours. The solid product was subsequently isolated [19]. The obtained product was filtered, recrystallized using methanol, and the excess solvent was removed under vacuum. Salicylaldehyde yielded a bright yellow-colored product, while o-vanillin resulted in an orange-colored product.

### 2.2 Synthesis of complexes

The metal complexes derived from Schiff base ligands were synthesized using a similar procedure for each complex. To a ethanolic solution of Palladium(II) chloride (0.149 g; 1.254 mmol) or Nickel (II) chloride (0.1255 g; 1.254 mmol), a deprotonated, ethanolic solution of Schiff base ligand was added. Triethylamine was used to deprotonate

the ligand, and the metal salts were slowly added to the ligand solution [20]. The reaction mixture was stirred at room temperature for 1 hour and then heated to reflux at a temperature range of 40°C to 50°C for approximately 6-8 hours. The resulting precipitates were filtered, washed with methanol and ether. The complexes exhibited solubility in common solvents such as methanol and dichloromethane.



### 3. Results and discussion

#### 3.1 IR Spectroscopy

The IR spectroscopy results of the nickel and palladium complexes derived from Schiff base ligands provide valuable information about their structural characteristics and coordination modes. The characteristic vibrational frequencies and their tentative assignments for the complexes [21-22] are summarized in Table 1.

In the IR spectra, a notable shift in the azomethine (C=N) stretching vibration is observed upon complexation. The peak corresponding to the azomethine group in the free ligand appears at a lower wave number, typically in the range of 1556-1650  $\text{cm}^{-1}$ . However, in the metal complexes, this peak undergoes a shift to higher wave numbers, typically in the range of 1630-1650  $\text{cm}^{-1}$ . This shift indicates the coordination of the azomethine nitrogen to the metal ion.

The presence of phenolic (OH) groups in the ligands is evident from the broad absorption bands observed at 3078  $\text{cm}^{-1}$  for the nickel complex and 3432  $\text{cm}^{-1}$  for the palladium complex [23]. However, these bands are absent in the IR

spectra of the mononuclear complexes, suggesting the deprotonation of the phenolic hydrogen and coordination of the oxygen with the metal ion.

The phenolic C-O stretching vibration in the free ligand, typically observed at 1233-1381  $\text{cm}^{-1}$ , undergoes a shift in the metal complexes. The shift can be either to lower or higher wave numbers, indicating the coordination of the oxygen atom to the metal ion.

The appearance of new vibrational bands in the IR spectra of the complexes compared to the ligands indicates the formation of metal-ligand bonds. These include M-O and M-N vibrational frequencies in the ranges of 450-624  $\text{cm}^{-1}$  and 535-613  $\text{cm}^{-1}$ , respectively. These bands are absent in the IR spectra of the Schiff base ligands, further supporting their involvement in metal coordination.

Additionally, the IR spectra of the complexes exhibit characteristic bands corresponding to other functional groups present in the ligands, such as (-NH),  $\nu$  (SO<sub>2</sub>),  $\nu$  (S-N), and  $\nu$  (C-S) groups. These bands remain relatively unchanged in the complexes, indicating that these functional groups do not directly participate in metal coordination [24].

Overall, the IR spectroscopy analysis of the nickel and palladium complexes provides evidence of their coordination with the Schiff base ligands and offers insights into the structural features of the complexes.

**Table 1. IR spectral data of Schiff base ligand [L]H<sub>2</sub> and its complexes ( $\text{cm}^{-1}$ )**

Compounds	$\nu$ (OH)	$\nu$ (C=N)	$\nu$ (C-O)	$\nu$ (M-N)	$\nu$ (COO)	$\nu$ (M-O)
[L]H <sub>2</sub>	3403	1648	1241	-	1357	-
[NiLX]	3078	1631	1181	541	1361	453
[PdLX]	3432	1631	1258	595	1393	614

### 3.2 UV-Visible Spectroscopy

The UV spectra analysis of the ligands and their metal complexes provides insights into their electronic transitions and molecular properties. The spectra were measured at a concentration of  $10^{-4}$  mol/L in a DMSO solvent at room temperature [25].

The ligand exhibits two main absorption bands at 271 and 380 nm. The absorption bands at 271nm and 380nm are assigned to  $\pi \rightarrow \pi^*$  and  $n \rightarrow \pi^*$  transitions.

In the metal complexes, the electronic transitions due to the organic ligands are observed, showing absorption bands related to  $\pi \rightarrow \pi^*$  and  $n \rightarrow \pi^*$  transitions. The absorption bands in the range of 260–299 nm correspond to the azomethine (CH=N) transitions, while the bands in the range of 320–417 nm are associated with the carbonyl

(C=O) transitions. These transitions indicate the involvement of the ligand's functional groups in the electronic transitions within the complexes [26].

The absorption spectra of the complexes exhibit similarities, indicating resemblances in the structures of the complexes. Notably, an intense absorption band is observed in the high-energy region at approximately 372 nm, which can be attributed to charge transfer (MLCT) transitions.

The UV-visible spectra of the complexes, along with their respective ligands, are depicted and the specific absorption values are listed in Table 2.

**Table 2. UV Spectral data of ligand and complexes (nm)**

Compounds	$\pi \rightarrow \pi^*$	$n \rightarrow \pi^*$
[L]H <sub>2</sub>	271	380
[NiLX]	282	328
[PdLX]	265	366

### 3.3 Mass spectrometry

The Time-of-Flight Mass Spectrometry (TOF-MS) Electrospray Ionization positive mode was utilized to record the spectral data of the ligands and their complexes. The resulting mass spectra and their corresponding fragmentation patterns with m/z values. In the ligand spectra, the peaks corresponding to the molecular ions were observed at m/z values.

### 3.4 Antibacterial activity

The synthesized Schiff base ligands and their corresponding Nickel and Palladium complexes were evaluated for their biological activity against both gram-negative and gram-positive pathogens, specifically *E. coli* MTCC 1687 and *S. aureus* MTCC 902. The antibiotic Sensitivity Test or Agar Well Diffusion method was employed for the screening. The bacterial strains were maintained in a refrigerator and sub-cultured on agar medium. Filter paper discs of 5 mm diameter and 1 mm thickness were impregnated with the complexes dissolved in methanol. These discs were placed on the agar plates, and the Petri dishes were then incubated at 37°C for 24 hours. Gentamicin was used as the standard antibiotic for the respective bacteria [29-30]. The zone of inhibition, measured in millimeters, for various compounds against the bacteria is presented in Table 3.

The Schiff base ligands demonstrated inherent biological activity, which can be attributed to the presence of the imine group, known for its involvement in biological transformation reactions. However, the Nickel and

Palladium complexes exhibited significant antibacterial activity, surpassing that of the free Schiff base ligand, owing to the synergistic effect resulting from the metal-ligand chelation [40]. The antibacterial activity of the Nickel and Palladium Schiff base complexes was found to be superior to that of the ligands against the same microorganisms under identical experimental conditions, consistent with previous findings [41]. The ligands and their metal complexes were systematically evaluated for their antibacterial properties, revealing that the metal complexes possess more potent bactericidal activity compared to the ligands themselves.

**Table 3. Antibacterial and antifungal activity data of ligand and its complexes**

Complexes	Antifungal PN	Antifungal AN	Antibacterial PA	Antibacterial BC
[L]H <sub>2</sub>	-	-	+	+
[NiLX]	-	-	++	++
[PdLX]	+	+	++	++

## Conclusion

In conclusion, the synthesized Schiff base ligands and their metal complexes exhibited distinct spectroscopic features, crystalline nature, and improved antibacterial activity. These findings highlight the significance of metal coordination in enhancing the biological properties of the ligands. Further studies to explore the mechanism of action and evaluate the potential of these compounds for broader therapeutic applications.

In this study, we have successfully synthesized Nickel (II) and Palladium (II) complexes using Schiff base ligand. The complexes were subjected to comprehensive spectral analysis, which played a crucial role in characterizing their structure and composition. UV and IR spectra provided valuable insights into the complexation process between the metal ions and the ligands. Mass spectra confirmed a strong 2:1 binding between the ligands, nickel (II), and palladium (II).

In addition to the spectroscopic characterization, we evaluated the antibacterial activity of these complexes. The complexes exhibited moderate antibacterial activity against bacterial species, namely *E. coli* and *S. aureus*, with Gentamicin used as a standard reference. These findings suggest the potential of the synthesized complexes as antibacterial agents, although further investigations are necessary to understand their mechanism of action and explore their broader therapeutic applications.

Overall, our study contributes to the understanding of the synthesis, spectroscopic properties, and antibacterial activity of nickel (II) and palladium (II) complexes with Schiff base ligands. These findings lay the foundation for future research and development of novel metal complexes with enhanced antibacterial properties.

## Acknowledgements

The author (S. Agrawal) would like to express their gratitude to the Department of Chemistry, CMP degree college (constituent college of University of Allahabad), for providing the necessary laboratory facilities.

Special thanks are extended to Department of Energy & Environmental Engineering, the CSIR-Indian Institute of Chemical Technology, Hyderabad, for providing I.R., UV, and NMR spectra, Delhi University for the mass spectrometry, and MNNIT Allahabad for conducting the antibacterial studies.

The contributions and support of these institutions are greatly appreciated and have significantly contributed to the successful completion of this research project.

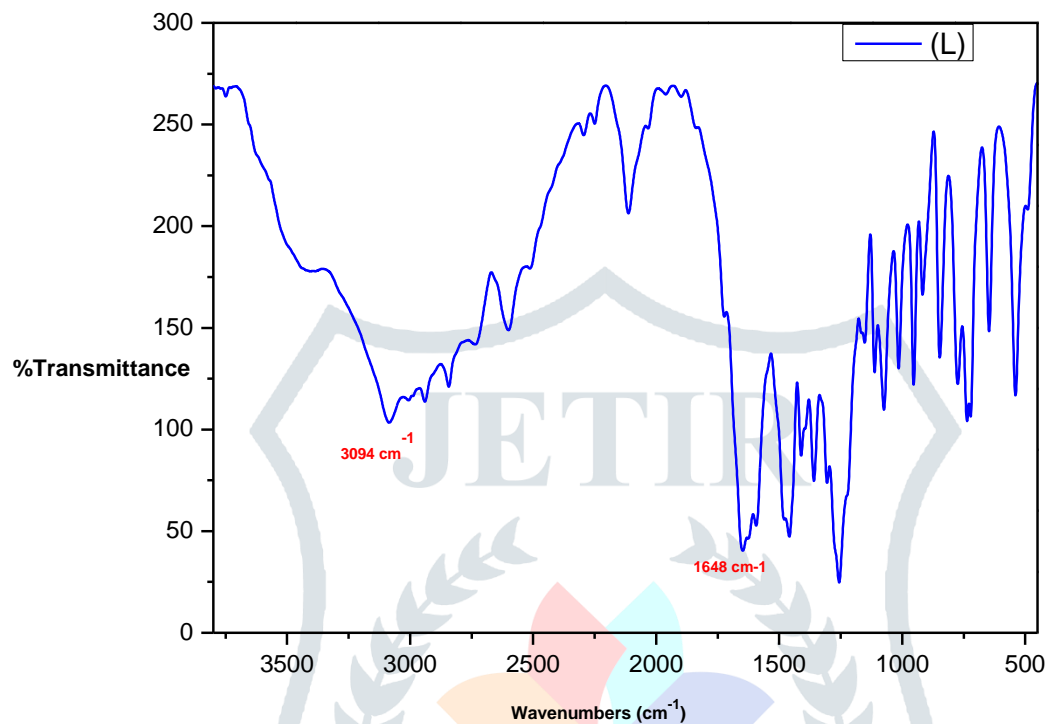
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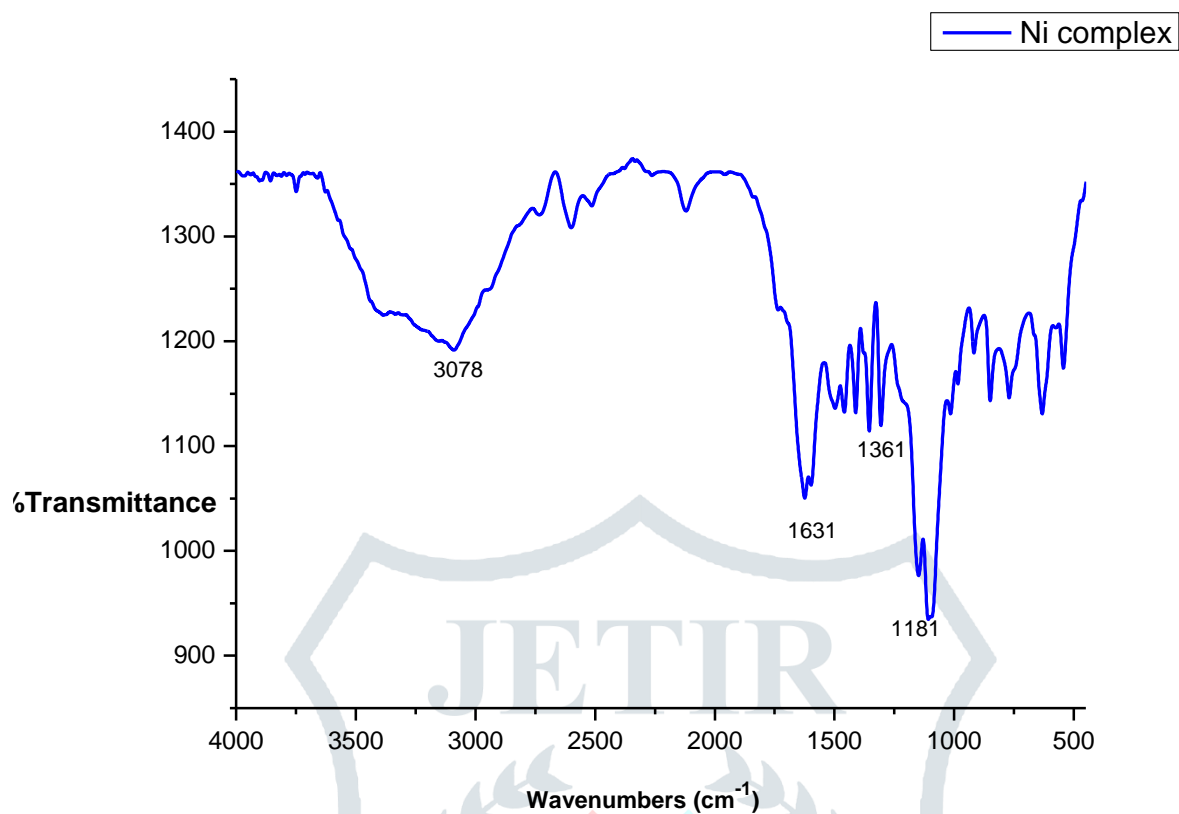
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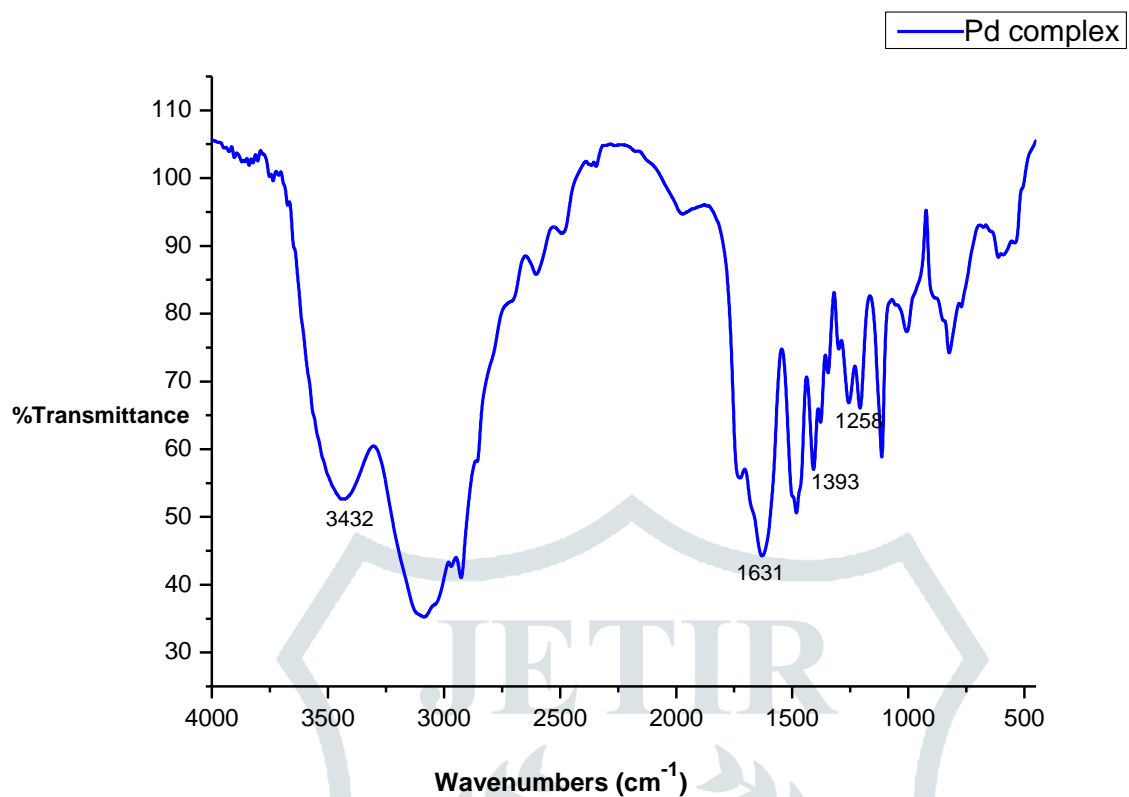
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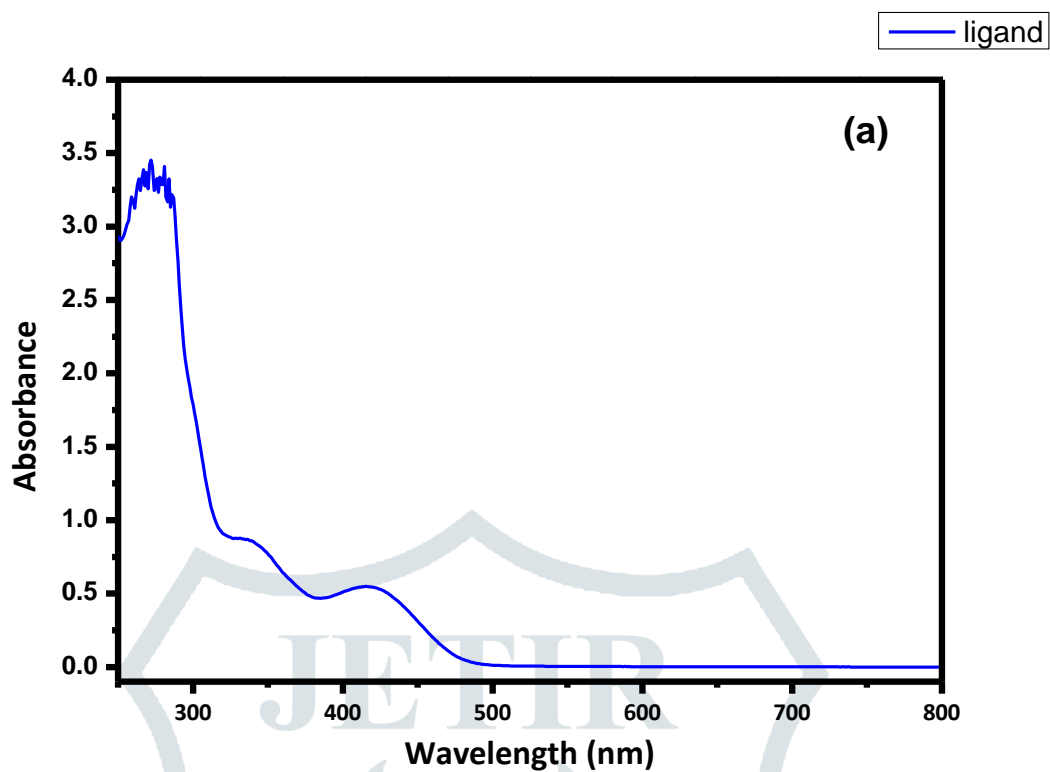
**Fig. IR Graph of Schiff Base Ligand**



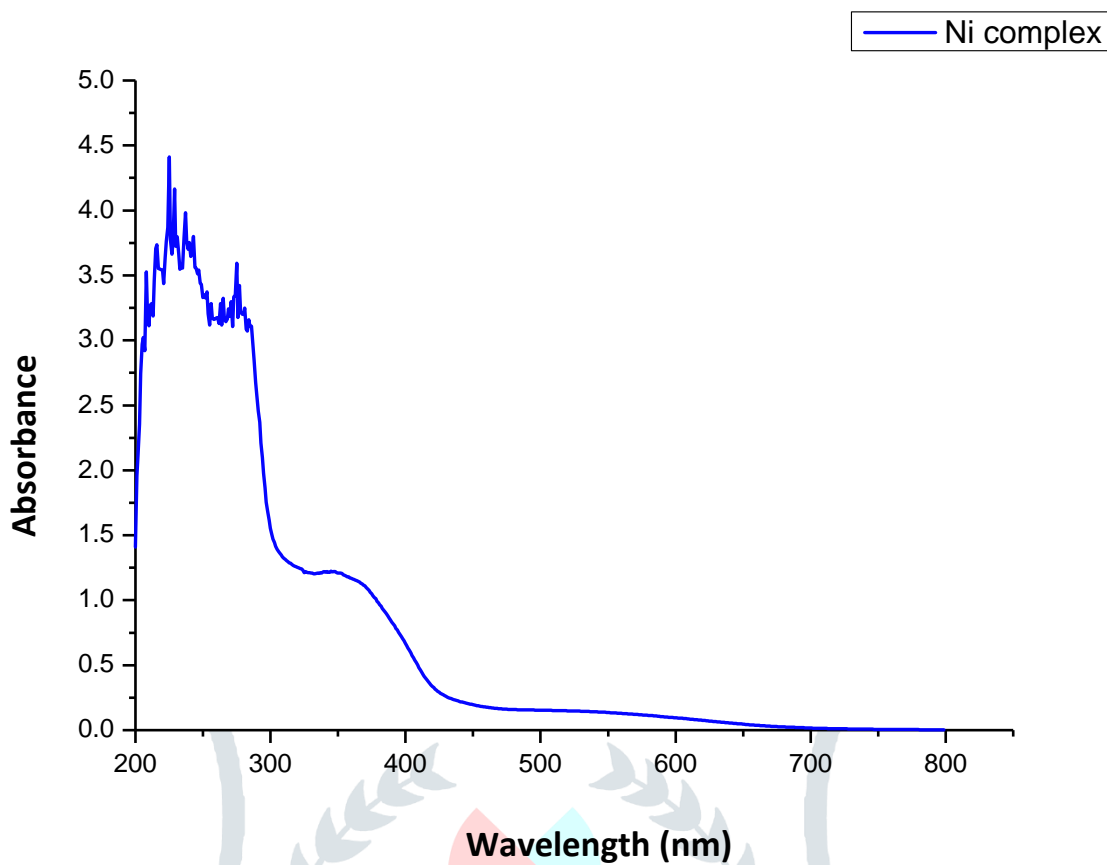
**Fig. IR Graph of Ni complex**



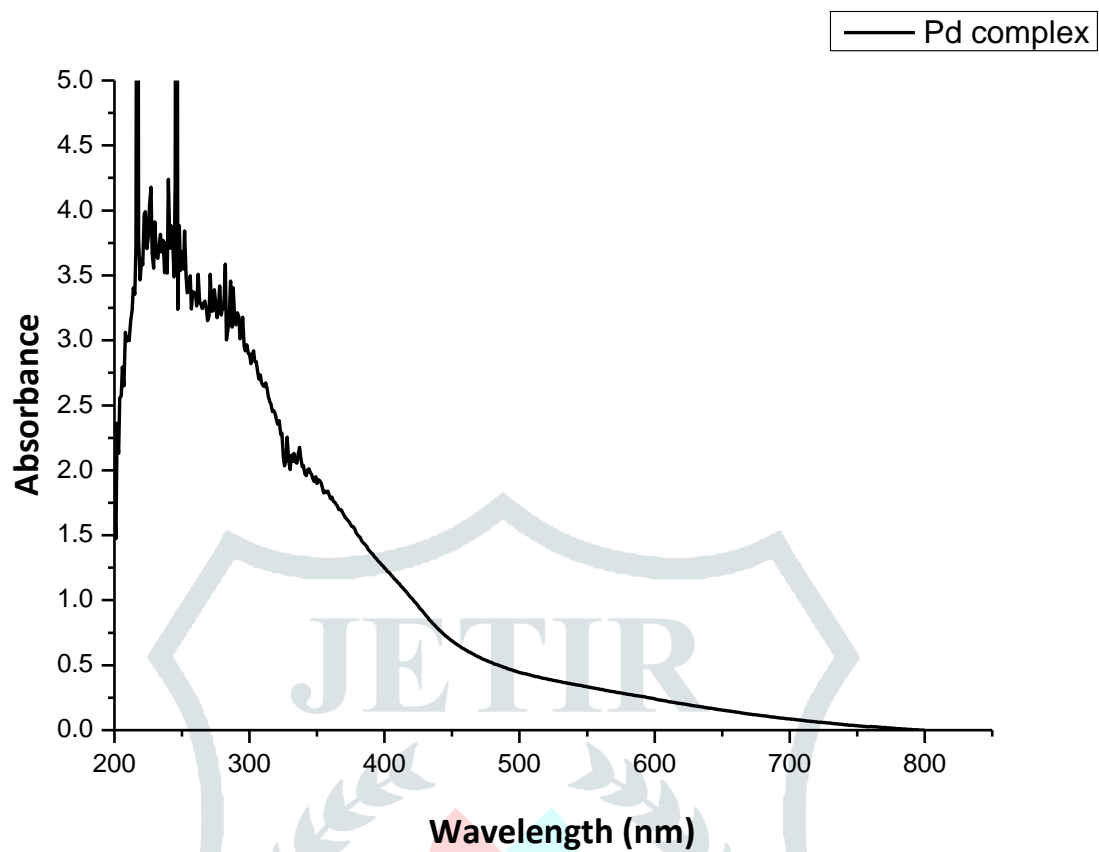
**Fig. IR Graph of Pd complex**



**Fig. UV-Vis Graph of Schiff Base Ligand**



**Fig. UV-Vis Graph of Ni Complex**



**Fig. UV-Vis Graph of Pd Complex**