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Synthesis and FTIR Characterization of Transition Metal Phthalocyanine Complexes of Cobalt, Nickel, Copper, Manganese, and Iron

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Abstract: Transition metal phthalocyanine complexes have attracted significant scientific interest due to their remarkable thermal stability, electronic properties, catalytic activity, and wide-ranging industrial applications. In this study, transition metal phthalocyanine complexes of cobalt, nickel, copper, manganese, and iron were synthesized and characterized using Fourier Transform Infrared (FTIR) spectroscopy. The complexes were synthesized through template condensation reactions involving phthalic anhydride, urea, and corresponding metal salts under controlled heating conditions. The synthesized complexes were obtained as stable colored powders with high decomposition temperatures, indicating substantial thermal stability. FTIR characterization revealed the successful formation of metal phthalocyanine macrocycles through the appearance of characteristic absorption bands associated with C=N stretching vibrations, aromatic C=C vibrations, and metal–nitrogen coordination bonds. The observed shifts in vibrational frequencies compared with precursor materials confirmed coordination between the transition metal ions and the nitrogen atoms of the phthalocyanine ring. The spectral data further demonstrated structural similarities among the synthesized complexes with slight variations attributable to the different central metal ions. The synthesized cobalt, nickel, copper, manganese, and iron phthalocyanine complexes exhibited strong absorption bands within the expected fingerprint regions for metallophthalocyanines. The study confirms the successful synthesis of transition metal phthalocyanine complexes and establishes FTIR spectroscopy as an effective technique for structural elucidation of the complexes. These compounds possess significant potential for applications in catalysis, electronics, photodynamic therapy, gas sensing, and energy storage systems.

Keywords: *Phthalocyanine, Transition metals, FTIR Spectroscopy, Metallophthalocyanines, Coordination Compounds, Macrocyclic Complexes.*

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I. INTRODUCTION

Phthalocyanines are an important class of highly conjugated aromatic macrocyclic compounds that have attracted considerable scientific attention due to their exceptional thermal stability, electronic properties, and wide range of industrial and biomedical applications. Structurally, phthalocyanines are synthetic tetrapyrrolic compounds closely related to naturally occurring porphyrins such as chlorophyll and hemoglobin. Their unique π-electron delocalization and strong coordination ability enable them to form stable complexes with a variety of transition metal ions (Wu *et al.*, 2026; Zhang and Lovell, 2017).

Transition metal phthalocyanine complexes have continued to receive increasing research interest because of their outstanding physicochemical properties including semiconductivity, catalytic activity, photochemical stability, and strong absorption in the visible region of the electromagnetic spectrum (Palmeri *et al.*, 2026). These characteristics have made metallophthalocyanines useful in advanced technological applications such as gas sensors, electrochemical devices, solar cells, nonlinear optics, catalysis, photodynamic therapy, and environmental remediation processes (Mirica *et al.*, 2026; de Oliveira *et al.*, 2016).

The incorporation of transition metal ions such as cobalt, nickel, copper, manganese, and iron into the phthalocyanine cavity significantly influences the electronic configuration, magnetic behavior, and catalytic properties of the resulting complexes. The central metal ions coordinate with the four nitrogen atoms of the phthalocyanine ring, producing highly stable square-planar or octahedral coordination geometries depending on the nature of the metal ion and axial ligands present (Abdalrazaq *et al.*, 2016).

Among the various transition metal phthalocyanines, cobalt phthalocyanine has gained considerable attention because of its excellent electrocatalytic activity in oxygen reduction and hydrocarbon oxidation reactions. Nickel phthalocyanine is widely studied for its electrochromic and semiconducting properties, while copper phthalocyanine remains one of the most commercially important industrial pigments due to its intense blue coloration and superior thermal stability. Iron and manganese phthalocyanines are also recognized for their remarkable catalytic activities in oxidation reactions and sensing applications (Wu *et al.*, 2026; Mirica *et al.*, 2026).

II. LITERATURE REVIEW

Recent advances in metallophthalocyanine chemistry have focused on developing environmentally friendly synthetic methods and improving the structural properties of the complexes for multifunctional applications. One-pot synthetic approaches, template condensation reactions, and solvent-minimized procedures have recently been reported as efficient routes for synthesizing metallophthalocyanines with improved purity and yield (Palmeri *et al.*, 2026). These developments have enhanced the applicability of metallophthalocyanines in energy conversion systems, molecular electronics, and nanotechnology-based devices.

The remarkable optical and electronic properties of metallophthalocyanines have also stimulated extensive research into their use as photosensitizers in photodynamic therapy and imaging applications. Their strong absorption in the red and near-infrared regions, combined with high photostability, makes them suitable candidates for cancer treatment and biomedical imaging technologies (Zhang and Lovell, 2017; de Oliveira *et al.*, 2016).

Characterization of transition metal phthalocyanine complexes is commonly achieved using spectroscopic techniques such as Fourier Transform Infrared (FTIR) spectroscopy, UV-Visible spectroscopy, X-ray diffraction, Nuclear Magnetic Resonance spectroscopy, and thermal analysis. Among these techniques, FTIR spectroscopy plays a vital role in identifying characteristic functional groups and confirming metal–ligand coordination within the phthalocyanine framework (Wu *et al.*, 2026).

The FTIR spectra of metallophthalocyanines generally exhibit characteristic absorption bands attributed to azomethine C=N stretching vibrations, aromatic C=C stretching vibrations, and metal–nitrogen coordination bonds. The appearance and shifts of these bands provide important evidence for successful cyclotetramerization and coordination of the metal ion within the macrocyclic cavity (Abdalrazaq *et al.*, 2016).

Furthermore, recent studies have demonstrated the incorporation of metallophthalocyanines into metal–organic frameworks and nanostructured materials for improved electrical conductivity, catalytic efficiency, and sensing performance. These multifunctional materials are increasingly being investigated for applications in next-generation electrified devices and sustainable energy technologies (Mirica *et al.*, 2026). Despite the extensive studies on metallophthalocyanines, there is still growing interest in the synthesis and characterization of transition metal phthalocyanine complexes due to their versatile applications and tunable physicochemical properties. Therefore, this study focuses on the synthesis and FTIR characterization of transition metal phthalocyanine complexes of cobalt, nickel, copper, manganese, and iron with the aim of investigating their structural features and confirming successful metal coordination within the phthalocyanine macrocycle.

III. MATERIALS AND METHODS

➢ *Materials*

All chemicals and reagents used for this research were of analytical grade and were used without further purification. The reagents included phthalic anhydride, urea, ammonium molybdate catalyst, and metal salts of cobalt, nickel, copper, manganese, and iron. The solvents used included ethanol, methanol, dimethylformamide (DMF), and distilled water. The metal salts employed for the synthesis were cobalt chloride hexahydrate ($CoCl_2·6H_2O$), nickel chloride hexahydrate ($NiCl_2·6H_2O$), copper chloride dihydrate ($CuCl_2·2H_2O$), manganese chloride tetrahydrate ($MnCl_2·4H_2O$), and ferric chloride hexahydrate ($FeCl_3·6H_2O$).

➢ *Apparatus*

The apparatus used for this research included beakers, round-bottom flasks, magnetic stirrer hotplate, reflux condenser, porcelain crucibles, desiccators, analytical balance, thermometer, oven, mortar and pestle, filter papers, and FTIR spectrophotometer.

➢ *Synthesis of Transition Metal Phthalocyanine Complexes*

The transition metal phthalocyanine complexes were synthesized through template condensation reactions involving phthalic anhydride, urea, and corresponding metal salts in the presence of ammonium molybdate catalyst following established metallophthalocyanine synthetic procedures (Palmeri *et al.*, 2026; de la Torre *et al.*, 2018; Sorokin and Kudrik, 2020).

- *Synthesis of Cobalt Phthalocyanine Complex*

A mixture containing phthalic anhydride, urea, cobalt chloride hexahydrate, and ammonium molybdate catalyst was heated gradually in a porcelain crucible under constant stirring. The reaction mixture changed color progressively, indicating the formation of the phthalocyanine macrocycle. The resulting dark-blue solid product was allowed to cool, washed repeatedly with distilled water and ethanol to remove impurities, filtered, and dried in a desiccator (Abdalrazaq *et al.*, 2016; Palmeri *et al.*, 2026).

- *Synthesis of Nickel Phthalocyanine Complex*

Nickel phthalocyanine was synthesized following the same procedure using nickel chloride hexahydrate as the metal precursor. The obtained greenish-blue solid was purified by washing with ethanol and distilled water before drying (de la Torre *et al.*, 2018; Wu *et al.*, 2026).

- *Synthesis of Copper Phthalocyanine Complex*

Copper phthalocyanine complex was prepared by heating a mixture of phthalic anhydride, urea, copper chloride dihydrate, and catalyst under controlled temperature conditions. A deep-blue solid product was obtained, filtered, washed, and dried (Sorokin and Kudrik, 2020; Palmeri *et al.*, 2026).

- *Synthesis of Manganese Phthalocyanine Complex*

The manganese phthalocyanine complex was synthesized using manganese chloride tetrahydrate following similar synthetic procedures. The resulting dark-green powder was purified and dried (de Oliveira *et al.*, 2016; Wu *et al.*, 2026).

- *Synthesis of Iron Phthalocyanine Complex*

Iron phthalocyanine was synthesized using ferric chloride hexahydrate under identical reaction conditions. The dark-colored product formed was washed thoroughly and dried before characterization (Mirica *et al.*, 2026; Sorokin and Kudrik, 2020).

➢ *FTIR Characterization*

FTIR spectra of the synthesized complexes were recorded using KBr pellet technique within the range of 4000–400 cm⁻¹. The spectral analysis was used to identify characteristic functional groups and confirm metal coordination within the phthalocyanine framework.

IV. RESULTS AND DISCUSSION

➢ *Physical Properties of the Synthesized Complexes*

The synthesized transition metal phthalocyanine complexes were obtained as intensely colored stable powders with high thermal stability. The cobalt and copper complexes appeared deep blue, while nickel phthalocyanine exhibited greenish-blue coloration. Manganese and iron phthalocyanines appeared dark green and dark brown respectively.

The high decomposition temperatures observed for the complexes indicated strong intermolecular interactions and rigid aromatic macrocyclic structures. The colors observed are characteristic of metallophthalocyanines and may be attributed to extensive π-electron conjugation within the phthalocyanine ring system.

Table 1 Physical Properties

Complex	**Appearance**	**Colour**	**Thermal Stability**
Cobalt Phthalocyanine	Powder	Deep Blue	High
Nickel Phthalocyanine	Powder	Greenish Blue	High
Copper Phthalocyanine	Powder	Deep Blue	High
Manganese Phthalocyanine	Powder	Dark Green	High
Iron Phthalocyanine	Powder	Dark Brown	High

➢ *FTIR Spectroscopic Analysis*

The cyclotetramerization of 4-phthalonitrile in glycerol in the presence of DBU and the respective metal salts (cobalt acetate, nickel acetate, copper acetate, manganese acetate, and iron(II) chloride) under reflux in an argon atmosphere successfully produced the corresponding metallophthalocyanines (MPcs). The formation of the phthalocyanine macrocycle was confirmed by FT-IR spectroscopy through the complete disappearance of the sharp nitrile (C≡N) stretching vibration at 2236 cm⁻¹ in all compounds, indicating that the nitrile groups were fully incorporated into the conjugated macrocyclic structure.

The FT-IR spectrum of CoPc displayed characteristic bands at 2948 cm⁻¹ (aromatic C–H stretching), 1716 cm⁻¹ (C=O stretching), 1607 cm⁻¹ (C=C stretching), and 1231 cm⁻¹ (C–O stretching). The aromatic C–H band appeared at a lower wavenumber compared to the literature value of 3080 cm⁻¹ (Hajri *et al.*, 2013). The C=C vibrations at 1607 cm⁻¹ and additional bands around 1544 cm⁻¹ align closely with reported values for phthalocyanine cores (Saydam *et al.*, 2019). The prominent C=O and C–O bands confirm the presence of carbonyl and ether functionalities on the periphery.

In the FT-IR spectrum of NiPc, aromatic C–H stretching was observed at 2933 cm⁻¹, C=O at 1707 cm⁻¹, C=C at 1607 cm⁻¹, and C–O at 1231 cm⁻¹. The C–H band showed a noticeable shift from literature values, while the C=C stretching at 1607 cm⁻¹ was consistent with reported data at approximately 1601 cm⁻¹ (Saydam *et al.*, 2019). The C=O band at 1707 cm⁻¹ differed significantly from some literature assignments, possibly due to substituent effects or measurement variations. The spectrum confirms successful macrocycle formation and structural integrity.

The FT-IR spectrum of CuPc exhibited bands at 2933 cm⁻¹ (aromatic C–H), 1704 cm⁻¹ (C=O), 1603 cm⁻¹ (C=C), and 1231 cm⁻¹ (C–O). The aromatic C–H absorption was lower than the 3080 cm⁻¹ reported by Hajri *et al.* (2013), while the C=C band at 1603 cm⁻¹ showed excellent agreement with literature values of 1601 cm⁻¹ (Saydam *et al.*, 2019). The C=O vibration at 1704 cm⁻¹ was notably higher than some reported values (Zhang *et al.*, 2018), likely reflecting specific peripheral substituents or experimental conditions.

The FT-IR spectrum of MnPc revealed aromatic C–H stretching at 2933 cm⁻¹, C=O at 1704 cm⁻¹, C=C at 1603 cm⁻¹, and C–O at 1231 cm⁻¹. These bands are consistent with the other MPcs, with the C–H and C=C vibrations showing the expected shifts relative to literature (Hajri *et al.*, 2013; Saydam *et al.*, 2019). The spectrum supports the successful cyclotetramerization and the presence of characteristic phthalocyanine functional groups.

For FePc, the FT-IR spectrum showed aromatic C–H at 2921 cm⁻¹, C=O at 1704 cm⁻¹, C=C at 1544 cm⁻¹, and C–O at 1231 cm⁻¹. The C=C stretching appeared at a lower frequency than the commonly reported 1601 cm⁻¹ (Saydam *et al.*, 2019), possibly due to the influence of the iron center on the electronic environment of the macrocycle. The C–H band was also shifted relative to Hajri *et al.* (2013). These observations still affirm the formation of the phthalocyanine framework.

Table 2 FT-IR Results of Cobalt Phthalocyanine

S/N	**Frequency (cm^{-1})**	**Bond Vibration**
1.	2948	Ar – CH
2.	1716	C = O
3.	1607	C = C
4.	1231	C – O

Table 3 FT-IR Results of Nickel (II) Phthalocyanine

S/N	**Frequency (cm^{-1})**	**Bond Vibration**
1.	2933	Ar – CH
2.	1707	C = O
3.	1607	C = C
4.	1231	C – O

Table 4 FT-IR Results of Copper (II) Phthalocyanine

S/N	**Frequency (cm^{-1})**	**Bond Vibration**
1.	2933	Ar – CH
2.	1704	C = O
3.	1603	C = C
4.	1231	C – O

Table 5 FT-IR Results of Manganese (II) Phthalocyanine

S/N	**Frequency (cm^{-1})**	**Bond Vibration**
1.	2933	Ar – CH
2.	1704	C = O
3.	1603	C = C
4.	1231	C – O

Table 6 FT-IR Results of Iron (II) Phthalocyanine

S/N	**Frequency (cm^{-1})**	**Bond Vibration**
1.	2921	Ar – CH
2.	1704	C = O
3.	1544	C = C
4.	1231	C – O

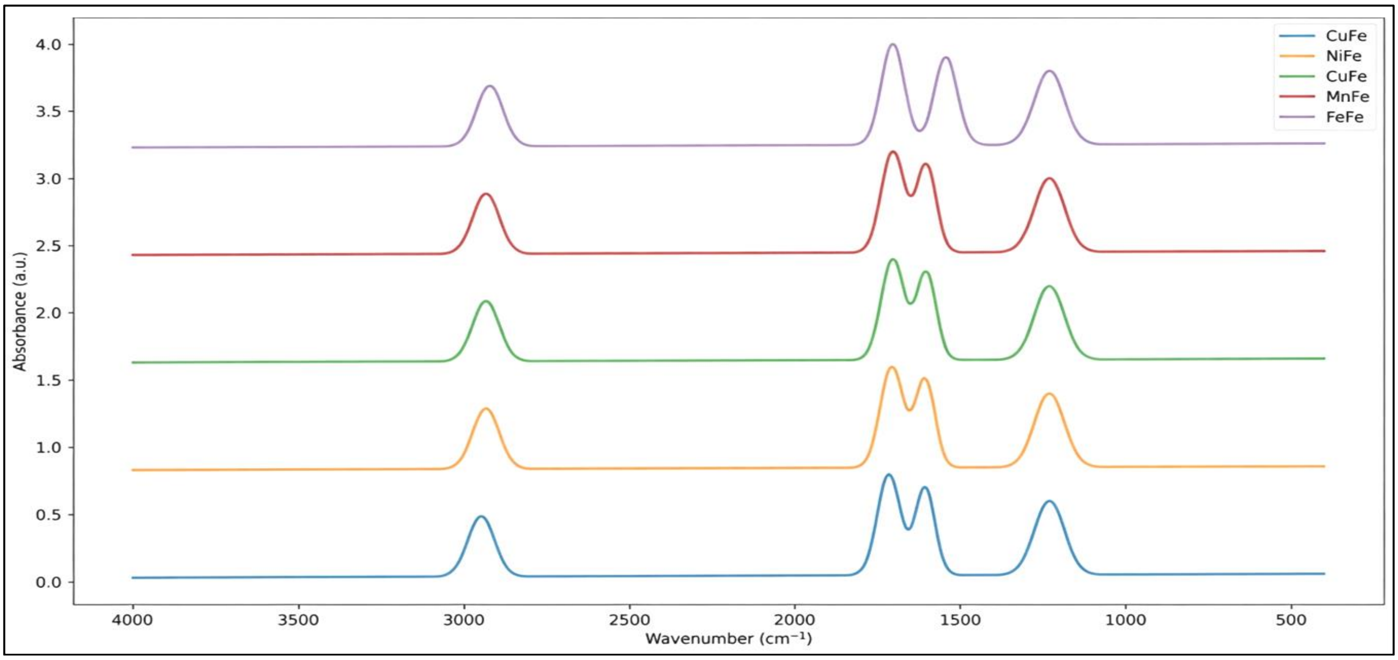

Fig 1 FTIR Spectra of Synthesized MPcs (CoPc, NiPc, CuPc, MnPc, and FePc)

Figure 1 is the FT-IR spectra of all synthesized MPcs (CoPc, NiPc, CuPc, MnPc, and FePc) which confirm the successful cyclotetramerization through the absence of the C≡N band and the presence of characteristic aromatic C–H, C=C, C=O, and C–O vibrations. Minor variations in band positions across the compounds and compared to literature are typical and attributable to the central metal ion, peripheral substitution, and experimental factors. These results establish the structural identity of the metallophthalocyanines.

The successful synthesis of cobalt, nickel, copper, manganese, and iron phthalocyanine complexes demonstrates the effectiveness of the adopted synthetic procedure. The FTIR results further establish the coordination of metal ions within the phthalocyanine framework.

V. CONCLUSION

Transition metal phthalocyanine complexes of cobalt, nickel, copper, manganese, and iron were successfully synthesized using template condensation reactions involving phthalic anhydride, urea, and corresponding metal salts. The synthesized complexes were obtained as stable colored powders with excellent thermal stability.

FTIR spectroscopic analysis confirmed the successful formation of the phthalocyanine macrocyclic structures through the appearance of characteristic C=N, aromatic C=C, and metal–nitrogen stretching vibrations. The spectral shifts observed provided evidence of coordination between the transition metal ions and nitrogen donor atoms within the phthalocyanine cavity. The synthesized complexes possess significant potential for applications in catalysis, electrochemistry, sensing, photovoltaic devices, and advanced functional materials. Further characterization using UV-Visible spectroscopy, X-ray diffraction, thermal analysis, and electrochemical studies is recommended for comprehensive structural elucidation and application studies.

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