



Synthesis, Characterization, and Antioxidant Potential of Nickel (II) Phthalocyanine for Industrial and Biomedical Applications

Mohammed Abbas Al-Silaykhee¹, Saif Ddin Kadhoum Al-Zamili², Ahmed Ghdhban Al-Ziaydi^{3*}

Abstract

Background: Nickel (II) phthalocyanine (Ni-Pc) is a prominent metallophthalocyanine compound known for its unique optical, electronic, and antioxidant properties. Its ability to form stable complexes with metal ions has made it a subject of interest in industrial and biomedical applications, including advanced material development and therapeutic interventions. **Methods:** Ni-Pc was synthesized using commercially obtained chemicals without additional purification. Its structural and physicochemical properties were analyzed through Fourier Transform Infrared (FTIR) spectroscopy, UV-Visible spectroscopy, and proton nuclear magnetic resonance (¹H NMR) spectroscopy. The antioxidant potential of Ni-Pc was evaluated via the DPPH radical scavenging assay, comparing its activity to ascorbic acid. **Results:** The synthesized Ni-Pc exhibited a melting point of 355°C with a yield of 75%. Elemental analysis confirmed its purity and composition (C: 66.17%, H: 19.88%, N: 20.01%). FTIR data revealed characteristic O–H, C=C, C=N, C–N, and Ni–N stretching vibrations, confirming its molecular structure. UV-Visible spectroscopy displayed prominent absorption bands, including a Q-band at ~670 nm, indicative of its

intact aromatic macrocyclic system. ¹H NMR analysis confirmed the successful synthesis of Ni-Pc through characteristic aromatic CH group signals at 7.5–8.04 ppm. The DPPH assay demonstrated its antioxidant efficacy, with an 85% scavenging activity at 25 µg, comparable to ascorbic acid. **Conclusion:** Ni-Pc was successfully synthesized and characterized, exhibiting significant antioxidant potential alongside versatile structural properties. Its robust ability to coordinate with various metal ions underscores its importance in industrial applications, such as carbon nanotube development, and biomedical fields, including antitumor and antibacterial therapies. These findings highlight Ni-Pc's potential as a multifunctional compound for advanced material science and therapeutic advancements

Keywords: Nickel (II) phthalocyanine, antioxidant activity, DPPH assay, biomedical applications, FTIR spectroscopy

Introduction

Nickel (II) phthalocyanine (Ni-Pc) is a coordination complex belonging to the class of phthalocyanines, a group of macrocyclic compounds with diverse industrial, biological, and chemical applications. Structurally, Ni-Pc consists of a central nickel (Ni²⁺) ion coordinated to a large, planar ligand system composed of four isoindole subunits linked by nitrogen atoms. This configuration creates an extensive conjugated π-system, making it structurally analogous to porphyrins, with a robust square-planar geometry that imparts exceptional electronic properties and chemical stability

Significance | This study determined Nickel (II) phthalocyanine's antioxidant efficacy, structural characterization, and versatile applications in industry and therapeutic advancements.

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(Sorokin, 2019). These characteristics contribute to the widespread use of phthalocyanines in various fields, including as pigments, catalysts, sensors, and materials for optoelectronic devices (Chen et al., 2007; Kalyanasundaram & Grätzel, 1998).

The distinctive photochemical and electrochemical properties of Ni-Pc have garnered significant attention in the development of advanced materials and technologies. Its high thermal and chemical stability, combined with its vibrant coloration, has led to applications in paints, inks, and plastics (C. Zhang et al., 2021). Additionally, Ni-Pc plays a vital role in catalytic processes, such as oxygen reduction and hydrogen evolution reactions, underscoring its potential in energy conversion systems (Bottari et al., 2010). The compound's utility extends to chemical and biological sensing, where it is used for detecting gases and biomolecules (Furini et al., 2020). Moreover, emerging applications in organic semiconductors, photovoltaic cells, and medical fields highlight the adaptability of Ni-Pc in addressing contemporary technological challenges (Urbani et al., 2019).

This study focuses on the synthesis of Ni-Pc and the assessment of its antioxidant properties. The synthesis process involves the reaction of p-cyanobenzamide with nickel to produce a high-quality phthalocyanine complex.

2. Materials and Methods

2.1 Chemicals and Reagents

All solvents were distilled and dried before use. Chemicals for synthesis were obtained from Aldrich and BDH Chemical Co. and used without further purification.

2.2 Chemical Synthesis

The synthesis of Nickel (II) phthalocyanine (Ni-Pc) was achieved through a two-step process (Figure 1).

2.2.1 Step 1: Preparation of p-Cyanobenzamide

p-Cyanobenzamide was synthesized by gradually adding 0.08 mL of p-cyano benzoyl chloride to 40 mL of an aqueous amine solution at 60°C in a 100 mL reaction flask, as described by Enokida et al. (1991). The reaction mixture was vigorously stirred for 1 hour and subsequently cooled to room temperature over an additional hour. The resulting precipitate was filtered using a Büchner funnel and thoroughly washed with ice-cold water until the pH of the filtrate reached neutral (pH 7), as confirmed by pH test paper. The crude product was refluxed for 40 minutes and purified through recrystallization from hot water (Gök et al., 2007).

2.2.2 Step 2: Synthesis of Nickel (II) Phthalocyanine (Ni-Pc)

Nickel (II) phthalocyanine was synthesized by reacting 5 g of p-cyanobenzamide with 1.2 g of nickel powder and 3 g of naphthalene as a diluent. The mixture was stirred and heated at 230°C for 1 hour, following Furuyama et al. (2014). The reaction product was treated with 200 mL of boiling acetone for 7 minutes. Excess naphthalene was removed by washing the solid with 1 M sodium hydroxide

(NaOH) in hot water (ARSLAN, 2016). The resulting solid residue was purified by recrystallization from benzene, yielding Ni-Pc with a 50% recovery rate.

2.3 Product Characterization

The synthesized Ni-Pc was characterized using UV-Visible spectroscopy, FTIR, and ¹H NMR spectroscopy (Bruker instruments).

2.4 DPPH Radical Scavenging Activity

To assess the DPPH radical scavenging activity of the synthesized Ni-Pc, methanolic solutions were prepared at a concentration of 1000 ppm. Serial dilutions (25, 20, 15, 10, and 5 µL) of these solutions were added to individual test tubes containing 5 mL of a 0.004% methanolic solution of DPPH, as described by Marinova et al. (2011).

Each reaction mixture was prepared in triplicate, vortexed vigorously, and incubated in the dark for 2 hours to stabilize the reaction. The absorbance of the mixtures was measured at 517 nm using a spectrophotometer, following the protocol by Moreira et al. (2008).

2.5 DPPH Radical Scavenging Calculation

The percentage of DPPH radical scavenging activity was calculated for each sample and standard using the following equation:

$$\text{Percentage of DPPH radical-scavenging-activity} = [1 - (A_t / A_o)] \times 100$$

Where A_t represents the absorbance of the samples, while A_o denotes the absorbance of the controls. The average-values were determined from three standard samples for each compound, using ascorbic acid as the standard reference.

3. Result and discussion

3.1 Synthesis and Characterization

Nickel (II) phthalocyanine (Ni-Pc) was successfully synthesized with a yield of 75% and a melting point of 355°C. Elemental analysis of Ni-Pc showed good agreement with theoretical values: observed C: 66.17%, H: 19.88%, and N: 20.01%, compared to calculated values of C: 67.28%, H: 2.94%, and N: 20.01%. This confirms the chemical purity and accuracy of the synthesis process, aligning with previous studies on metallophthalocyanine compounds (Gök et al., 2007; Aktaş et al., 2013).

3.2 Fourier Transform Infrared (FTIR) Analysis

The FTIR spectrum of Ni-Pc (Figure 2) revealed characteristic peaks, providing insights into its molecular structure:

Broad O–H stretching vibrations were observed at 3000.99–3207.66 cm^{-1} . Medium CH_2 bending vibrations were detected at 980.77–1200.99 cm^{-1} . The benzene ring within the pyrrole unit exhibited C=C stretching at 1607.09–1609.45 cm^{-1} .

Strong C=N stretching vibrations at 1700.03–1768.89 cm^{-1} and C–N stretching at 1333.78–1345.50 cm^{-1} confirmed the coordination between the nitrogen atoms and the nickel ion.

A weak peak at 540.77 cm^{-1} corresponded to Ni–N stretching.

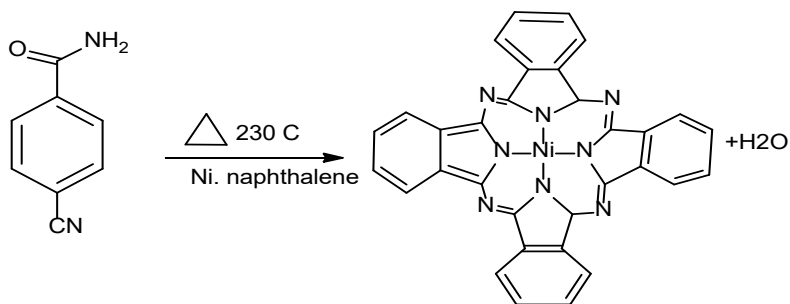


Figure 1. The synthesized of Nickel (II) phthalocyanine.

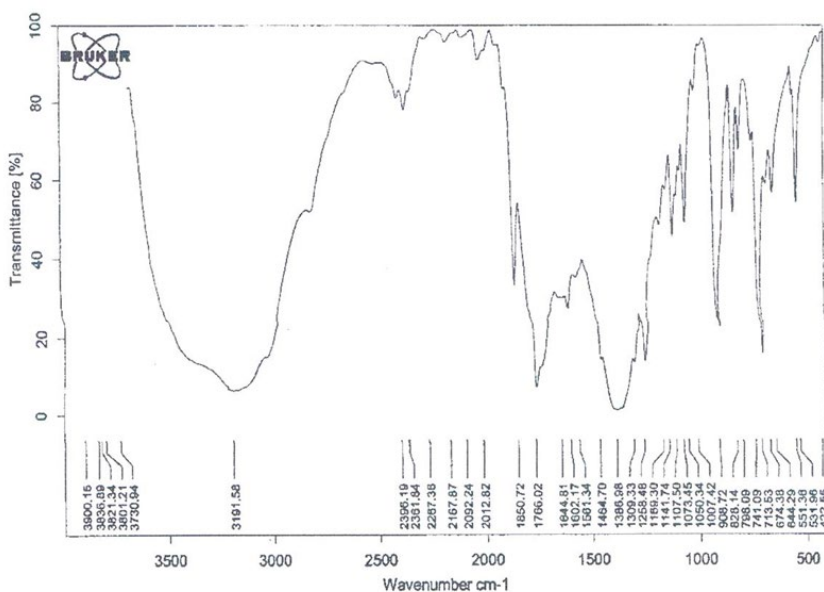


Figure 2. FTIR spectroscopy of synthesis Nickel (II) phthalocyanine compound

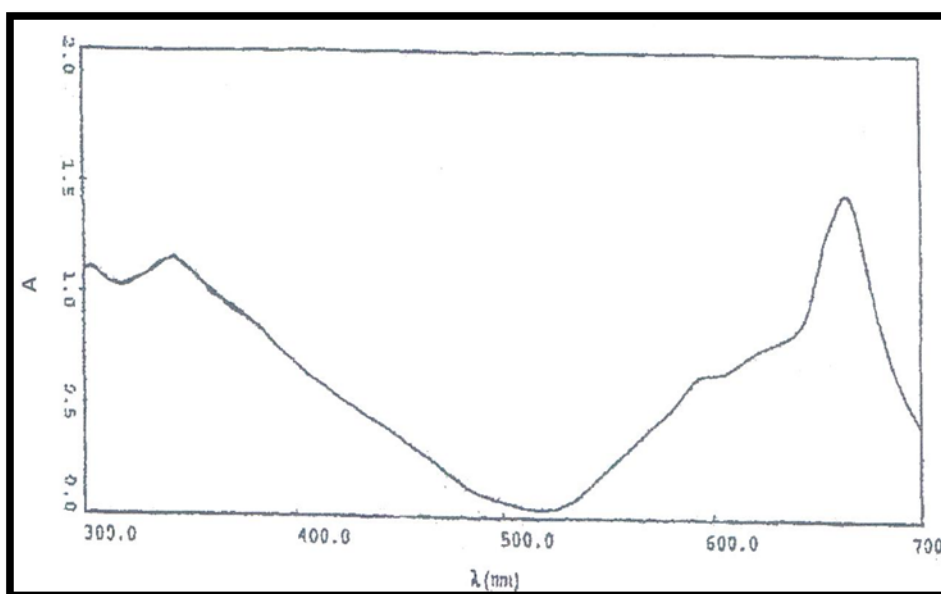


Figure 3. UV/Vis spectrum of Nickel (II) phthalocyanine

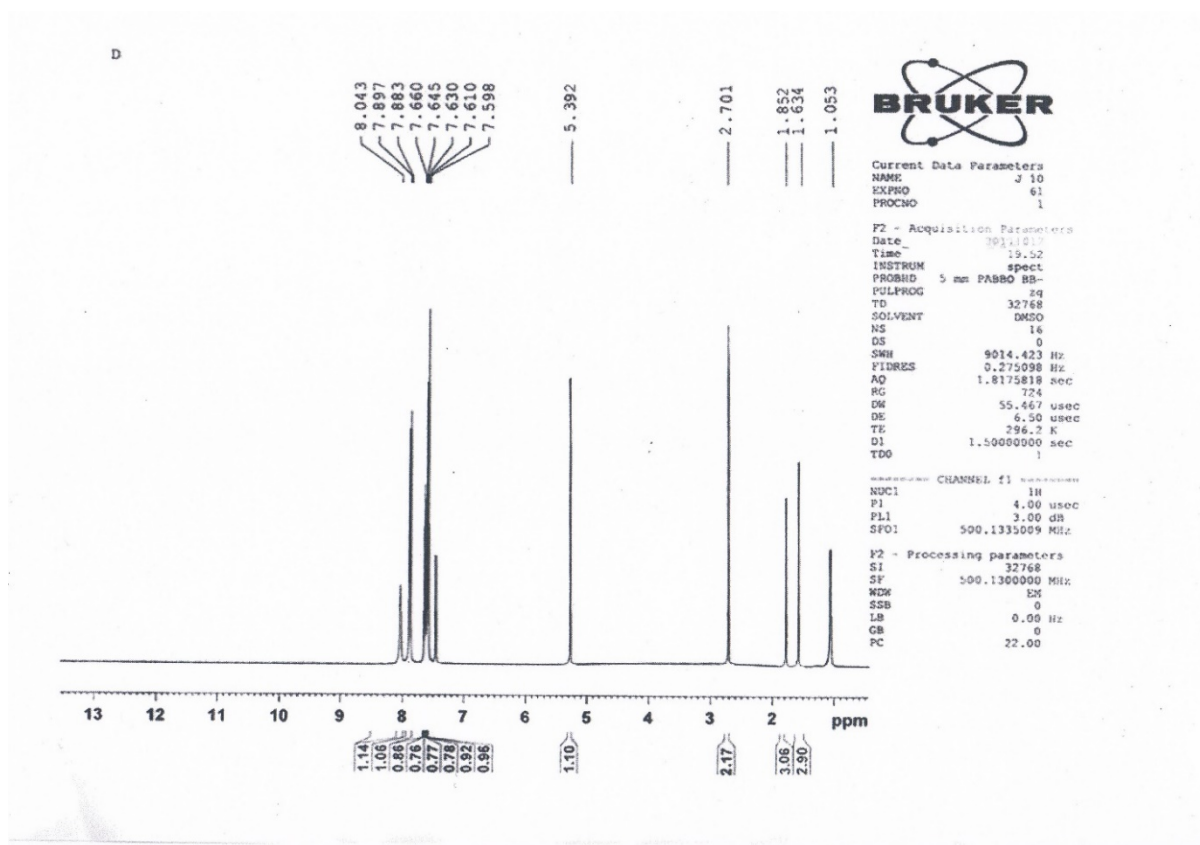


Figure 4: ¹H NMR spectrum of synthesis Nickel (II) phthalocyanine compound

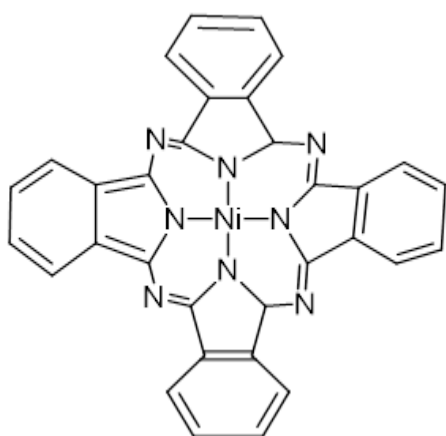


Figure 5. The structure of Nickel (II) phthalocyanine compound

Table 1. DPPH-free radical scavenging action of Nickel (II) phthalocyanine compound on in-vitro assay

Comp	Conc. 1000 ppm														
	5 μ L			10 μ L			15 μ L			20 μ L			25 μ L		
	A _a	A _o	(I%)	A _a	A _o	(I%)	A _a	A _o	(I%)	A _a	A _o	(I%)	A _a	A _o	(I%)
Ni.Pc	0.306	0.655	53	0.252	0.655	62	0.191	0.655	71	0.144	0.655	78	0.101	0.655	85
Vit. C	0.245	0.655	63	0.19	0.655	71	0.121	0.655	82	0.085	0.655	87	0.044	0.655	93

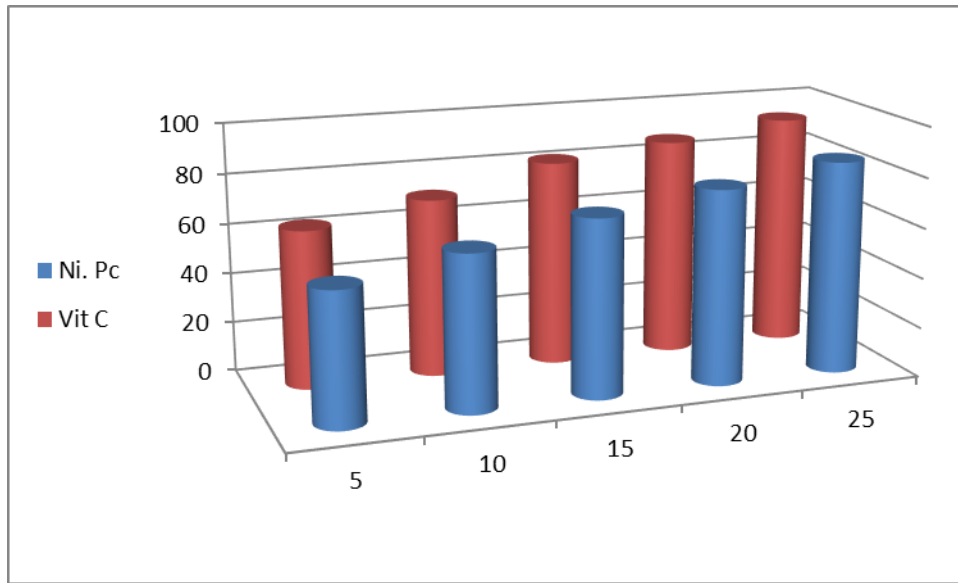


Figure 6. DPPH-free radical scavenging action of Nickel (II) phthalocyanine compound on in-vitro assay

These findings validate the synthesis of Ni-Pc, consistent with reported data for phthalocyanine derivatives (Abed et al., 2019; Lai et al., 2018).

3.3 UV-Visible Spectroscopy

The UV-Visible spectrum of Ni-Pc (Figure 3) showed absorption bands characteristic of phthalocyanine compounds. The prominent Q-band at ~670 nm confirmed the structural integrity of the aromatic macrocyclic system, as observed in similar studies (Aktaş et al., 2013; Furini et al., 2020).

3.4 ¹H NMR Spectroscopy

¹H NMR analysis (Figure 4) of Ni-Pc in DMSO displayed a singlet at 7.5–8.04 ppm, attributed to the 12 protons of the aromatic CH groups. Additionally, a peak at 5.39 ppm was observed, corresponding to the C=C protons. These signals further corroborate the successful synthesis of Ni-Pc, as reported in earlier research (Ağırtaş, 2008; Taher et al., 2024).

3.5 Complexation and Applications

Nickel (II) phthalocyanine demonstrates remarkable versatility due to its ability to coordinate with various metal cations, such as Fe and Co, through its pyrrolic nitrogen atoms. These complexes exhibit stable metal-ligand interactions, enabling applications across diverse fields.

In industrial settings, Ni-Pc plays a critical role in developing advanced materials, such as carbon nanotubes, and contributes to photonic and optoelectronic devices (Kalyanasundaram & Grätzel, 1998; Bottari et al., 2010). In biomedical applications, Ni-Pc complexes show potential as antitumor and antibacterial agents, enhancing the efficacy of therapeutic interventions (Ali et al., 2024; Couto et al., 2020; Y. Zhang & Lovell, 2017).

3.6 Antioxidant Activity

The antioxidant potential of Ni-Pc was evaluated using the DPPH radical scavenging assay. Ni-Pc demonstrated a strong ability to suppress reactive oxygen species (ROS), with an 85% scavenging effect at 25 µg, comparable to ascorbic acid (Figure 6, Table 1). The mechanism involves electron donation, reducing DPPH radicals and diminishing the solution's violet color (Xu & Chang, 2007; Patel & Patel, 2011). This robust activity highlights Ni-Pc's potential in mitigating oxidative stress, making it a promising candidate for therapeutic applications.

4. Conclusion

The synthesis of Ni-Pc was successfully confirmed through comprehensive characterization, including FTIR, UV-Vis, and ¹H NMR spectroscopy. Ni-Pc exhibits significant antioxidant activity and potential for industrial and biomedical applications. These findings support its multifunctionality and pave the way for further exploration in catalysis, materials science, and therapeutic interventions (Sorokin, 2019; Urbani et al., 2019; Zhang et al., 2021).

Author contributions

M.A.A.-S. contributed to the conceptualization, methodology, formal analysis, and drafting of the original manuscript. S.D.K.A.-Z. was responsible for data curation, investigation, validation, and visualization. A.G.A.-Z. supervised the project, administered its execution, and contributed to the review and editing of the manuscript. All authors reviewed and approved the final version of the manuscript.

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Competing financial interests

The authors have no conflict of interest.

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