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Synthesis, Characterization of Nickel (II) Phthalocynine, and Screening of Its Potential Antibacterial Activity

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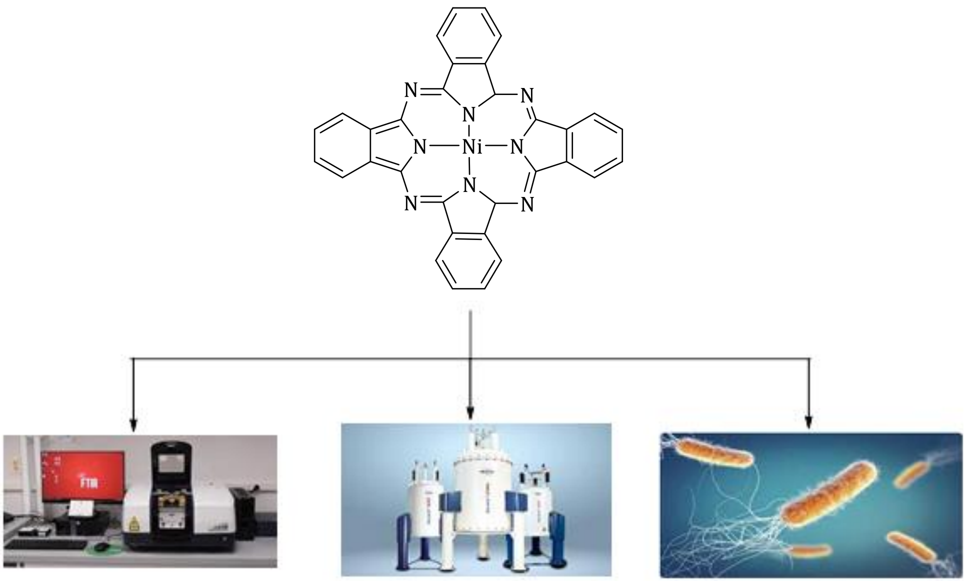
A B S T R A C T

Nickel (II) Phthalocynine (Ni-Pc) is a class of macro-cyclic compounds that has bivalent, tetradentade, planar, and 18 π-conjugated electron aromatic ring systems. Pcs are composed of four pyrrole units linked to four [aza] (—N=C—) groups at the α-carbon of [Pyrrole] unit and they have four aza bridges and four (phenylene) rings. This study aims to synthesize Nickel (II) Phthalocynine. The new synthesized product has been characterized by FT-IR and ^{1}H-NMR spectroscopy. The procedure of this study includes the synthesis of Nickel (II) Phthalocynine (Ni-Pc) by the reaction of (o-cyanobenzamide) with Nickel that was powdered through heating. Different concentrations of Nickel (II) Phthalocynine were studied regarding the effect of antibacterial activity against two species of bacteria *Escherichia coli* and *staphylococcus aureus* by using disk-diffusion methods in *Mueller–Hinton agar.* The results of synthesis of Nickel (II) Phthalocynine shows a good resistance against the selected bacteria with "the minimum inhibitory concentration" (MIC) and "the minimum bactericidal concentration" (MBC) of (Ni-Pc).

G R A P H I C A L   A B S T R A C T



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Introduction

Nickel (II) phthalocyanine (Ni-Pc) is one of Metallo-complex (M.Pc) which is a type of planar-molecule containing four groups of a [Pyrrole] ring linked to a benzene ring [1]. This property gives (Ni-Pc) blue-green color at the region of (640-710 nm). Due to this advantage, Phthalocynine compound is among the important industrial dyes, and also they are related to the biological effective molecule (Chlorophyll) [2]. It has been widely used in thin films because of the special chemical stability, a high electro-catalytic activity, and sensing applications by the supramolecular arrangement which is defined by many attributes of films such as thickness, morphology, and crystallinity. Therefore, it is assumed to be a highly arrangement molecule depends on metallic center atom [3]. The (Ni-Pc) is tested to be applied in the solar cells as a transfer asset mediator [4]. Likewise, it is used in the development of gas detector device due to its excellent thermal-chemical stabilities as a semiconductor property [5]. Many new phthalocyanine derivatives compounds are being developed that characterize the new group on the semiconductor which limited the obtainable efficiency of photosensitizer chemical compounds [6]. The major studies related to the synthesis of the substituted molecules are achieved from the chemical addition of amino, carboxylic, and hydroxyl groups as an extension of the center of macro-cyclic structure (Chenodeoxycholic acid). It has been tested for photovoltaic performance which rendered the highest efficiency of all reported phthalocyanine photosensitizers so far [7].

The Nickel (II) Phthalocynine (Ni-Pc) have the ability to create numbers of compound via collation with cations in the center with nitrogen pyrrolic substance and nickel compounds proved by many studies are very useful in the industry such as carbon nanotube due to the strong metal bonds, and also in the medical field as antitumor and antibacterial activity. Furthermore, it plays an important role in human body by increasing the inhabitation degree of medicine [8,9].

Materials and Methods

All ingredients and solvents were obtained from Aldrich and BDH Chemical Co. Gallen Kamp capillary melting point apparatus was used to measure melting points. (Bruker) tensor M27 spectrometer was used to take the FT-IR measurements. ^{1}H-NMR spectra were collected in a Bruker spectrophotometer Ultra-shield at 400 MHz by using DMSO as an internal standard.

The reaction to get the final compound included two steps, as follow.

Preparation of [o-Cyanobenzamide]

The first step included the preparation of [*o*-Cyanobenzamide] by using 0.07 ml of [*o*-cyano benzoyl chloride] and it was added step by step till the end of amount to 30 ml of amine water solution at 6°C in 100 ml beaker [10]. The solution was kept under a vigorous string for an hour. The mixture was left to be cooled for one hour, and then it was filtered by using [Büchner flask]. The residue was washed continuously with ice water till the pH indicator of washing water turned to 7 according to the pH meter. The solution was reflexed for 30 min and [*o* - Cyanobenzamide] was purified and recrystallized from hot water (1:50) ratio [11,12].

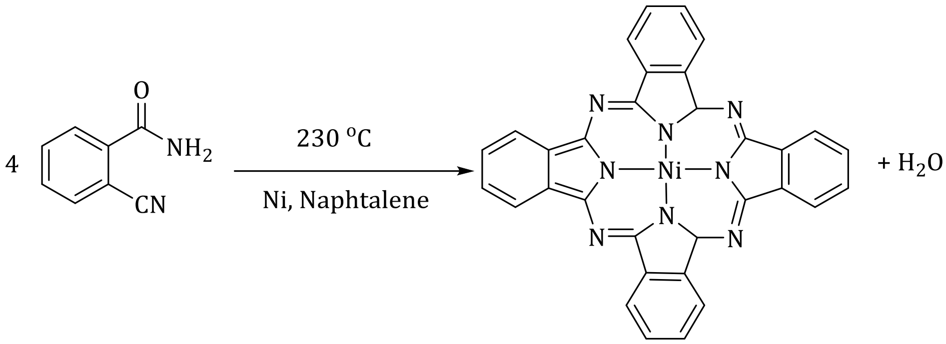
Preparation of Nickel (II) Phthalocynine (Ni-Pc)

The second step included the preparation of (Ni-Pc) by using 4 g of [*o*–Cyanobenzamide] from the first step, 1 g of powdered nickel, and 2.5 g of naphthalene (as diluent) were stirred heating up to 230 °C for one hour [13]. Finally, 100 ml of acetone boiled for 5 min and the final powder product was treated by 1 M of sodium hydroxide diluted in hot water to remove the extra naphthalene [14, 15]. The residue was recrystallized from benzene.

Results and Discussions

The final compounds were synthesized according to mechanism exhibited in Scheme 1. The stretching vibration bands were at 1602.17-1644 cm^{-1} (C=N, C=C) and 3191.58 cm^{-1} (C-H aromatic). The I.R spectrum data and elemental analysis results were summarized in Table 1. The ^{1}HNMR spectrum of [Ni-Pc] indicated the appearance of a broad singlet at the range of (7.5-8.04) P.P.M

(12H, pc-CH), as listed in Table 2. The (Ni-Pc) of this study was tested against microorganisms type of bacteria by assessing of “inhibition zones”, “zone diameter”, and (MIC) values. As exhibited in the Table 3 and Figure 1, the results revealed a good resistance against the selected bacteria with “the minimum inhibitory concentration (MIC) and “the minimum bactericidal concentration” (MBC) [18].



Scheme 1: Mechanism of the (Ni-Pc)

Table 1: Physical properties and FT-IR spectral data cm-1 of synthesized compound

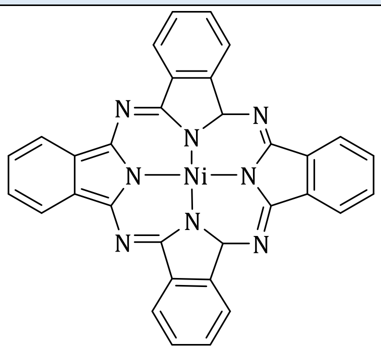
Physical properties				Major FT-IR absorption cm-1			
Structure of compounds	m.p. (°C)	color	Yield (%)	ν (C=N) (cm^{-1})	υ (C-H) Aromatic (cm^{-1})	υ (C=C) (cm^{-1})	Other bands
	355	Blue	75	1602.17 1644	3191.58	1602.17 1644	υ(Ni-N) 908.72

Table 2: ^{1}H-NMR spectral data (δ ppm) for compound

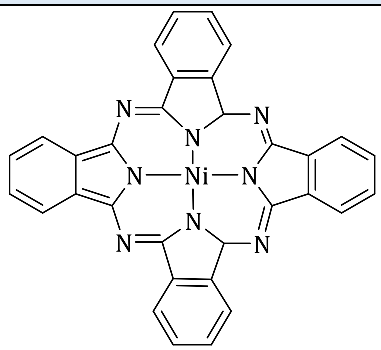
Compound structure	^{1}H-NMR data of (δ-H) in ppm
	s, (7.5-8.04) P.P.M(12H, pc-CH) $C_{33}H_{21}N_8Ni$

Table 3: Antimicrobial activity of Metallo-Phthalocynine

Sample	M.I.C(mg/mL)	M.B.C(mg/ mL)
E. coli	> 13	>29
St. aureus	7	14

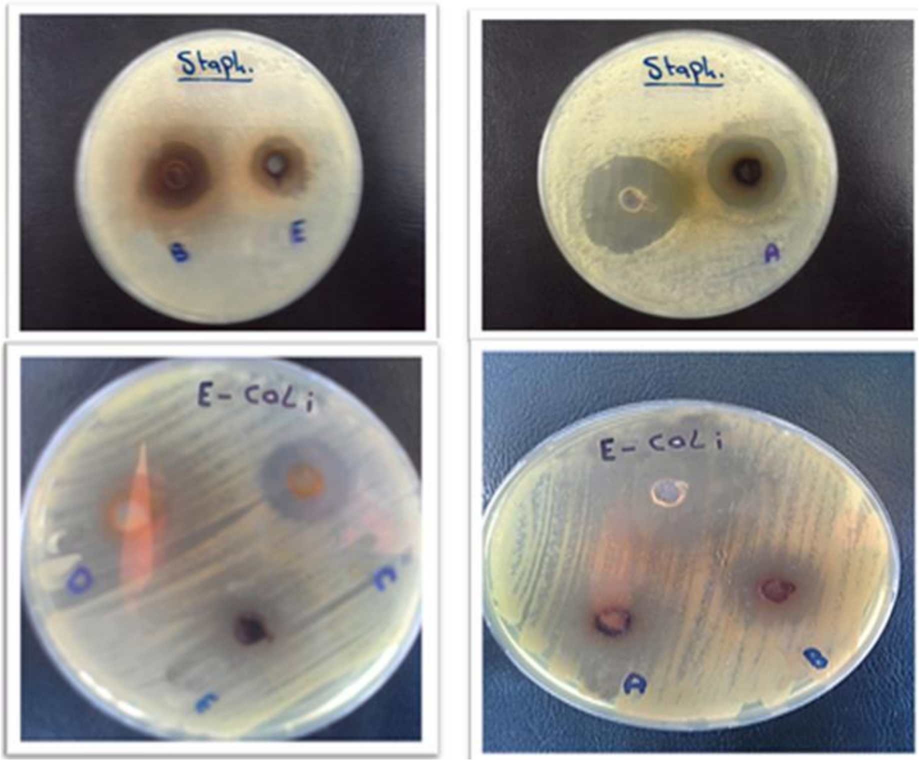


Figure 1: Biological activity for Nickel (II) Phthalocynine

Conclusion

The preparation of Metallo-Phthalocynine from [*o*–Cyanobenzamide] was confirmed by using spectroscopic techniques (FT-IR and ^{1}HNMR). The study of antibacterial activity led to terminate the activity of used types of bacteria.

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Authors' contributions

All authors contributed to data analysis, drafting, and revising of the paper and agreed to be responsible for all the aspects of this work.

Conflict of Interest

There are no conflicts of interest in this study.

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References

[1]. Vicente E., Lima L.M., Bongard E., Charnaud S., Villar R., Solano B., Burguete A., Perez-Silanes S., Aldana I., Vivas L., Monge A., Synthesis and structure–activity relationship of 3-phenylquinoxaline 1,4-di-N-oxide derivatives as antimalarial agents, *European journal of medicinal chemistry*, 2008, **43**:1903 [Crossref], [Google Scholar], [Publisher]

[2]. Abd El-Wahab Z.H., Mashaly M.M., Salman A.A., El-Shetary B.A., Faheim A.A., Co (II), Ce (III) and UO2 (VI) bis-salicylatothiosemicarbazide complexes: binary and ternary complexes, thermal studies and antimicrobial activity, *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, 2004, **60**:2861 [Crossref], [Google Scholar], [Publisher]

[3]. De La Torre G., Vázquez P., Agullo-Lopez F., Torres T., Role of structural factors in the nonlinear optical properties of phthalocyanines and related compounds, *Chemical Reviews*, 2004, **104**:3723 [Crossref], [Google Scholar], [Publisher]

[4]. Luo P.G., Stutzenberger F.J., Nanotechnology in the detection and control of microorganisms, *Advances in applied microbiology*, 2008, **63**:145 [Crossref], [Google Scholar], [Publisher]

[5]. Shahriar G., Mina A., Sajjad S., Preparation and identification of two new Phthalocynine and study of their anti-cancer activity and anti-bacterial properties, *Scientific Research and*

Essays, 2012, **7**:3751 [Google Scholar], [Publisher]

[6]. Enokida T., Hirohashi R., A new synthesis of ε-form nickel phthalocyanine by using DBU at low temperature, *Chemistry Letters*, 1991, **20**:2155 [Crossref], [Google Scholar], [Publisher]

[7]. Kantekin H., Gök Y., Kiliçaslan M.B., Acar I., Synthesis and characterization of new metal-free and nickel (II) phthalocyanines containing hexaazadioxa macrobicyclic moieties, *Journal of Coordination Chemistry*, 2006, **61**:229 [Crossref], [Google Scholar], [Publisher]

[8]. Furuyama T., Satoh K., Kushiya T., Kobayashi N., Design, synthesis, and properties of phthalocyanine complexes with main-group elements showing main absorption and fluorescence beyond 1000 nm, *Journal of the American Chemical Society*, 2014, **136**:765 [Crossref], [Google Scholar], [Publisher]

[9]. Güzel E., Günsel A., Bilgiçli A.T., Atmaca G.Y., Erdoğmuş A., Yarasir M.N., Synthesis and photophysicochemical properties of novel thiadiazole-substituted zinc (II), gallium (III) and silicon (IV) phthalocyanines for photodynamic therapy, *Inorganica Chimica Acta*, 2017, **467**:169 [Crossref], [Google Scholar], [Publisher]

[10]. Nemykina V.N., Lukyanetsb E.A., Synthesis of substituted phthalocyanines. *ARKIVOC: Online Journal of Organic Chemistry*, 2010, 136-208 [Crossref], [Google Scholar], [Publisher]

[11]. Furuyama T., Maeda K., Maeda H., Segi M., Chemoselective Synthesis of Aryloxy-substituted Phthalocyanines, *The Journal of Organic Chemistry*, 2019, **84**:14306 [Crossref], [Google Scholar], [Publisher]

[12]. Bayar Ş., Dinçer H.A., Gonca E., The synthesis of some phthalocyanines derived from bulky substituted phthalonitriles, *Dyes and Pigments*, 2009, **80**:156 [Crossref], [Google Scholar], [Publisher]

[13]. Cabir B., Ağırtaş M.S., Duygulu E., Yuksel F., Synthesis of some metallophthalocyanines bearing 9-phenyl-9H-fluoren-9-yl) oxy functional groups and investigation of their photophysical properties, *Journal of Molecular Structure*, 2017, **1142**:194 [Crossref], [Google Scholar], [Publisher]

[14]. Sairkar P.K., Sharma A., Shukla N.P., Antimicrobial Activity of Guggulsterone E and Z, *International Journal of Current Microbiology and Applied Sciences,* 2016, **5**:20 [Crossref], [Google Scholar], [Publisher]

[15]. Lai Q., Quadir M.Z., Aguey-Zinsou K.-F., LiBH4 Electronic Destabilization with Nickel(II) Phthalocyanine - Leading to a Reversible Hydrogen Storage System, *ACS Applied Energy Materials*, 2018, **1**:6824 [Crossref], [Google Scholar], [Publisher]

[16]. Aktaş A., Acar İ., Koca A., Bıyıklıoğlu Z., Kantekin H., Synthesis, characterization, electrochemical and spectroelectrochemical properties of peripherally tetra-substituted metal-free and metallophthalocyanines, *Dyes and Pigments*, 2013, **99**:613 [Crossref], [Google Scholar], [Publisher]

[17]. Ağırtaş, M.S., Highly soluble phthalocyanines with hexadeca tert-butyl substituents, *Dyes and Pigments*, 2008, **79**:247 [Crossref], [Google Scholar], [Publisher]

[18]. Gaikwad M., Gaikwad S., Kamble R., 'Synthesis of Novel Series of 1-(6-Hydroxy-4-(1H-indol-3-yl)-3,6-dimethyl- 4,5,6,7-tetrahydro-1H-indazol-5-yl)ethan-1-oneas Evaluations of their Antimicrobial Activity with Insilco Docking Study', *Journal of Medicinal and Chemical Sciences*, 2022, **5**:239 [Crossref], [Google Scholar], [Publisher]

[19]. Gaikwad S., Basavanag Unnamatla M., 'Simple, Highly efficient synthesis of 2-amino-4-phenyl-4,5,6,7-tetrahydropyrano[3,2-c]carbazole-3-carbonitrile derivatives using silica supported dodeca-tungstophosphoric acid DTP/SiO2', *Journal of Applied Organometallic Chemistry*, 2022, **2**:24 [Crossref], [Publisher]

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