

Synthetic Study of Ni, Cu and Pd Metal Complexes with Antifungal Analysis

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Abstract

By using elemental analysis, molar conductance, IR, NMR, and electronic spectrum data, a number of organic compounds have been created along with their nickel(II), copper(II), and palladium(II) metal complexes. The complexes were tested for their antifungal efficacy against *Penicillium rubrum* and *Aspergillus niger*. All of the produced compounds have demonstrated good antifungal activity, which was generally enhanced upon complexation with metal ions.

Keywords: synthesis, antifungal ligands, *Aspergillus niger* and *Penicillium rubrum*.

Introduction

Antibacterials and antifungals are among the most commonly used drugs. Due to the significance of bacterial and fungal infection resistance, there has been a significant rise in the demand for innovative antibacterial and antifungal compounds. Natural products from fungus are considered to be a substantial source for new antibacterial and antifungal compounds because of the diversity of fungal species, the number of secondary metabolites, and improvements in genetic breeding and fermentation processes. In an effort to discover new antibacterial and antifungal compounds, an expanding variety of fungi, including marine fungi and endophytic fungi on wild plants, are being researched for their antimicrobial activity. In the last ten years, a large number of novel bioactive natural compounds with cytotoxic, anticancer, antiviral, antibacterial, or antifungal characteristics have been discovered in marine fungi.¹⁻⁶

The manufactured 4-amino benzenesulphonamide antimicrobial specialists, also known as sulpha medications, were made possible. Their antibacterial activity is thought to be a result of their structural resemblance to the 4-aminobenzoic corrosive produced by germs to make folic corrosive.⁷ Pyridin-4-carbohydrazide is a recognised authority in tuberculostatics. Metal chelates are constructed using a large number of bivalent particles. The pyridine-4- carbohydrazide structure was confirmed using these structures.⁸ The reason why pyrazine is a more delicate base than pyridine is due to the accepting effect of the subsequent nitrogen. All forms of life, in particular, depend on dihydropyrazines. Members of the pyrazine family have been used to prevent cancer. These concoctions have demonstrated to have significant healing applications.⁹⁻¹³

- As a result, the goal of this effort is to:
1. Produce new complexes, which are cutting-edge alternative medicines.
 2. To analyse the metal drug complexes using IR, NMR, mass, and ESR spectrometry as well as conductivity, solubility, and melting point measurements.
 3. To evaluate the synthesised metal complexes' effectiveness against fungus.

Material and Methods

The following are the various tools, procedures, glassware, solvents, reagents, and techniques utilised in the production of sulfonamide compounds:

- Bruker advance 300 MHz NMR
- Perkin Elmer 100 FT-IR spectrophotometer
- Agilent 1100 MCD trap-5C Mass spectrometer
- Digisun conductivity meter, DI 909 model
- Perkin Elmer UV-Vis spectrophotometer. U.V lamp

Methodology

Synthesis of the Complexes

4-(2-Hydroxybenzylidene) amino)benzenesulfonamide [HBABS]:

To an answer of 1.72 g (0.01 mol) of 4-aminobenzenesulfonamide (Merck) broke up in 100 ml of methanol in a 250 ml round base jar, 1.22 g (0.01 mol) of 2-hydroxy benzaldehyde (SD fine) was included and the substance were refluxed on a water shower for 2 hours. The arrangement, on cooling, gave a yellow hued compound, which was separated and recrystallized from ethanol. Yield (56%), MP:180°C.^{14,15}

4-(Furan-2-ylmethylene)aminobenzenesulfonamide [FMABS]:

An answer of 1.72g (0.01mol) of 4-aminobenzenesulfonamide (Merck) broke down in 100 ml of methanol in a 250 ml round base cup, was included with 0.96g (0.01 mol) of furan-2-carbaldehyde (Fluka) . The arrangement was refluxed on a water shower for 3 hours. The compound isolated was separated and recrystallized from methanol to give a dark hued strong. Yield (62%), MP:130°C.¹⁶

4-(Thiophene-2-ylmethylene)aminobenzenesulfonamide [TMABS]:

To an answer of 1.72g (0.01mol) of 4-aminobenzenesulfonamide (Merck) disintegrated in 100 ml of methanol in a 250 ml round base cup, 1.22 g (0.01 mol) of thiophene-2-carbaldehyde (Fluka) was included. The arrangement was refluxed on a water shower for 3 hours. The compound isolated was separated and recrystallized from methanol to give a light yellow shaded strong. Yield(82%), MP: 140°C.^{17,18}

(Thiophen-2-ylmethylidene) pyridine-4-carbohydrazide [TMPCH]:

To an answer of 1.23g (0.01m) of pyridine-4-carbohydrazide (Finar) disintegrated in 100 ml of methanol in a 250 ml round base cup, 1.22 g (0.01 mol) of thiophene - 2-carbaldehyde (Fluka) was included. The arrangement was refluxed on a water shower for 3

hours. The compound isolated was sifted and recrystallized from methanol to give a light yellow shaded strong. Yield(86%), MP: 130°C.¹⁹

(Thiophen-2-ylmethylidene) pyrazine-2-carboxamide [TMPCA]:

An answer containing 1.24g of pyrazinamide (Hi media) in 100 ml of ethanol in a 250 ml round base carafe was included with 1.12 g (0.01 mol) of thiophene-2-carbaldehyde. The substance were refluxed on a water shower for 2 hours. The compound isolated was separated and recrystallized from methanol to give a light yellow shaded solid Yield (68%), MP:178-180°C.²⁰

Arrangement of the Metal Complexes

The Ni, Cu and Pd buildings with all the ligands were readied utilized.

Ni(II) edifices:

To an answer of the ligand in hot methanol was included gradually, with blending, Ni(OAc)₂.4H₂O (ALDRICH) arrangement in methanol and the blend was refluxed on a high temp water shower. It was concentrated constrained to two-third the first volume and cooled. The strong that isolated out was sifted, washed with water, hot methanol and ether and was vacuum dried over combined CaCl₂.

Cu(II) edifices:

To a methanolic arrangement of copper(II)chloride (CuCl₂.2H₂O), a hot methanolic arrangement of the ligand was included gradually with blending. The blend was refluxed on a heated water shower. It was concentrated constrained to two-third the first volume and cooled. The strong that isolated out was sifted, washed with water, hot methanol and ether and was vacuum dried over intertwined CaCl₂.

Pd(II) buildings:

PdCl₂ (SRL) (1.0g) was broken down in concentrated hydrochloric corrosive and weakened with water to 100 ml to give 0.1N arrangement regarding hydrochloric corrosive. An aliquot of this arrangement was treated with an equivalent volume of methanol and was included drop-wise, under blending, with a hot methanolic arrangement of the ligand. The blend was refluxed on a high temp water shower. It was concentrated constrained to two-third the first volume and cooled. The strong that isolated out was sifted, washed with water, hot methanol and ether and was vacuum dried over combined CaCl₂.

Antifungal movement : Preparation of spore suspension :

From the new societies, spores were gathered and moved to a test tube containing sanitized refined water. The spore suspension subsequently acquired was utilized for testing the antifungal action of the mixes.

Antifungal test:

The antifungal test of the mixes was completed by agar well dispersion strategy as depicted by Magaldi et al²¹. The way of life plates hatched with the test life forms were permitted to set and punched with a sterile stopper borer (5 mm distance across) to make open wells. The wells were loaded up with 100 μ l arrangement at a convergence of 5 mg/ml of the mixes at 30 °C. Following 72 hours, the restraint zones were estimated and contrasted and those of the control DMSO and the standard flucnazole at a convergence of 5 mg/ml. On account of both antibacterial and antifungal measures, the tests were directed in triplicate and the outcomes communicated as mean.

Results and Discussion

In the present study, 4-aminobenzenesulfonamide has been condensed with 2-hydroxybenzaldehyde, furan-2-carbaldehyde and thiophene-2-carbaldehyde; pyridine-4-carbohydrazide with thiophene-2-carbaldehyde and pyrazine-2-carboxamide with thiophene-2-carbaldehyde and the accompanying Schiff base ligands acquired and portrayed.

4-((2-Hydroxybenzylidene)amino)benzenesulfonamide (HBABS) (Fig. 1)

4-((Furan-2-ylmethylene)amino)benzenesulfonamide (FMABS) (Fig. 2)

4-((Thiophen-2-ylmethylene)amino)benzenesulfonamide (TMABS) (Fig. 3)

N'-(Thiophen-2-yl-methylidene)- pyridine-4-carbohydrazide (TMPCH) (Fig. 4)

N-(Thiophen-2-ylmethylidene)- pyrazine-2-carboxamide(TMPCA) (Fig. 5)

The Ni, Cu, and Pd edifices of these Schiff base ligands have been created and generally described on the basis of fundamental research, conductance, warm, attractive, infrared, electronic, and ESR ghostly data. Relevant inferences about the geometry of the structures have been made in light of the knowledge acquired.

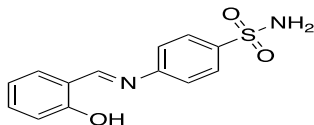


Fig. 1. 4-((2 Hydroxybenzylidene)amino)benzenesulfonamide (HBABS)

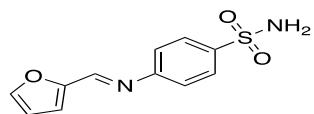


Fig. 2. 4-((Furan-2-ylmethylene)amino)benzenesulfonamide (FMABS)

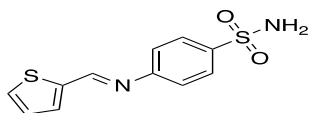


Fig. 3. 4-((Thiophen-2-ylmethylene)amino)benzenesulfonamide (TMABS)

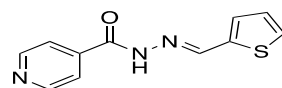


Fig. 4. N'-(Thiophen-2-yl-methylidene)- pyridine-4-carbohydrazide (TMPCH)

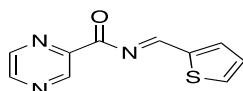


Fig. 5. N-(Thiophen-2-ylmethylidene)- pyrazine-2-carboxamide (TMPCA)

The designer integrated and depicted metal Schiff base structures built of sulfonamide, carbohydrazide, pyrazinamide, and other aldehydes because of the relevance of this class of aggressors. In preparation for the testing, the ligands and a portion of their metal structures that have been created for organic action have been screened. In the current study, thiophene-2-carbaldehyde, pyridine-4-carbohydrazide, pyrazine-2-carboxamide, and the associated Schiff base ligands were condensed using 4-aminobenzene-sulfonamide. These reactions were identified and shown in (figures 1-5).

The Ni, Cu, and Pd structures of these Schiff base ligands have been created and fundamentally depicted based on basic research, conductance, warm, attractive, and infrared data, electronic data, and ghostly ESR results. On the basis of the knowledge gained, pertinent deductions concerning the geometry of the structures have been made. All of the ligands are stable and non-hygroscopic at room temperature. They are somewhat soluble in methanol and (CH₃)₂CO and truly solvent in hot methanol and dimethylformamide. They are insoluble in water. The ligands have been described horrifyingly by investigative, mass, ¹H NMR, and IR data.

Conclusion

The structures of the complexes between five different compounds and Ni, Cu, and Pd have been shown using distinct physico-substance data. Mixed ligand complexes with the transition metals Ni, Cu, and Pd have been developed. Infrared spectroscopy, electric conductivity, melting point, and solubility are used to characterise the complexes in various ways. The complexes were tested for their antifungal efficacy against *Penicillium rubrum* and *Aspergillus niger*.

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