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SYNTHESIS, CHARACTERIZATION, ANTIBACTERIAL AND ANTIFUNGAL STUDY OF NOVEL NI (II) METAL COMPLEXES OF BIDENTATE 3-FORMYLCHROMONE BASED SCHIFF BASES

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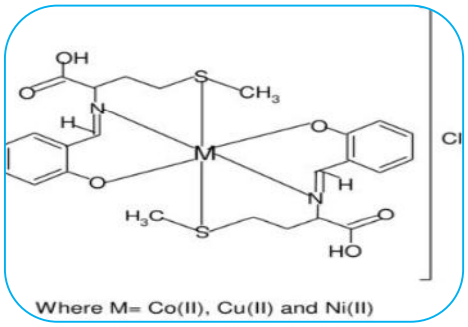
ABSTRACT

The present research work is all about the synthesis of complex compounds of Nickel with already synthesized schiff bases of chromones. The structure conformation of ligand molecule and synthesized complexes is done by using various spectroscopic techniques such as ^1H NMR, IR and elemental analysis. The antibacterial and anti-fungal screening of synthesized compounds were tested according to standard procedure.



KEYWORDS: *Schiff base, chromones, antibacterial activity, anti-fungal activity, transition metal complexes, octahedral, tetrahedral geometry, condensation, H^1NMR, IR, etc.*

INTRODUCTION

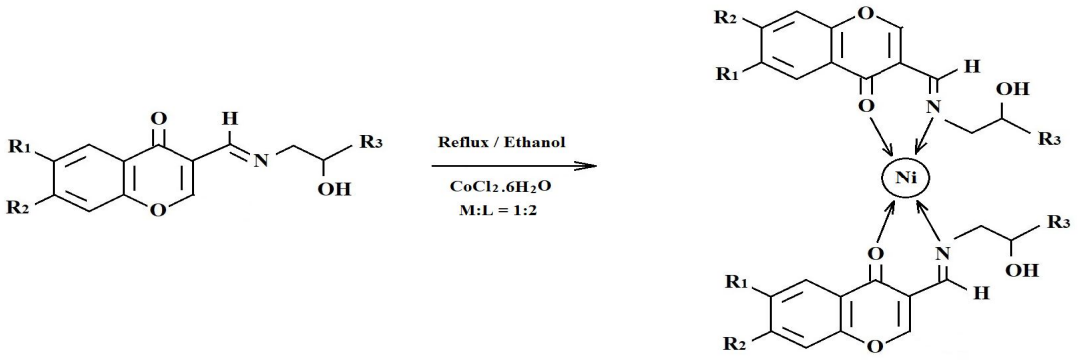
Nickel is a transition metal, found in 10th of periodical table. It has either [Ar]$3d^8 4s^2$ or [Ar]$3d^9 4s^1$ electronic configuration. It shows oxidation state from 0, +1, +3 and +4. The strong oxidants are related as. Ni (IV) is present in only a few species and needs extremely strong oxidants[1]. Ni (II) is also a very strong oxidant with donor-ligands such as thiols and phosphines[2].

Ni (II) complexes with schiff bases of chromone as ligand are typically square planar[4]. However, there are several exceptions to the five coordinated or octahedral geometries in Ni (II) complexes[5]. Generally, Ni (II) complexes, five-coordinate complexes have octahedral geometry. In Six coordinated complexes, sphere is generally completed by solvents such as H_2O, MeOH, through coordination[6-7]. The salen complexes Ni (II) are also found to have a potential catalytic effect[8]. Gibson and his coworkers reported that Ni (II) complexes are used in the preparation of high-density linear polyethylene by ethylene polymerization[9]. During the last decades, researchers have premeditated the relationship of Schiff bases and metal ions pretty thoroughly[10]. In biological systems of human body and animals, metal enzymes containing nickel (II) are an important trace element for nickel[11]. Antibacterial, anti-microbial, antioxidant, and anti-proliferative / cancer activity[12] have also been reported Ni (II) complex. Antiepileptic, anticonvulsant and nickel incidence has also been recorded. Therefore, increasingly attractive and important in the field of bioinorganic substances and coordinated chemicals[13]. Recently reported studies of amino acid basis ligands and nickel complexes (II)[14]. Literature revels that, nickel

complexes covering numerous biological and pharmacological activities, such as antibacterial[15], antifungal[16], anti-proliferative / anti-cancer properties[17] etc.

EXPERIMENTAL: SYNTHESIS OF NI (II) COMPLEXES [1–6]:

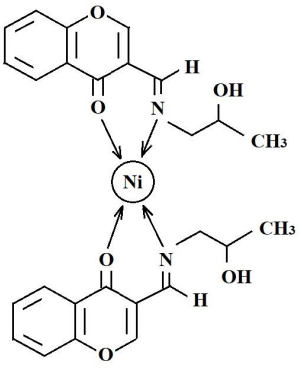
Reported protocol has been used for the synthesis of Nickel (II) complexes by using the 1:2 (M:L) metal to ligands (L_1-L_6) ratio. 1-aminopropen-2-ol, 2-amino-phenylehanol and metal salts ($NiCl_2{\cdot}6H_2O$) are commercially available (Sigma Aldrich) and are used without further purification. The solvents used are purified and dried by standard procedure. After finishing the reaction solid was separated and collected the complexes by washing with different organic solvents and dried, recrystallized and stored in a vacuum desiccator above glued $CaCl_2$. The data of all the complexes were interpreted. Melting points of the ligand and complexes were recorded on open capillaries and are uncorrected. The ligand and complexes were further characterized by using partial elemental analyses, FT-IR, electronic and 1 H NMR spectra. The Schiff base ligands of 3-formyl chromone (L_1-L_6) were synthesized (as per previous chapter).



Scheme-3: Synthesis of Ni (II) metal complexes with 3-formylchromone based Schiff base Ligand L_1

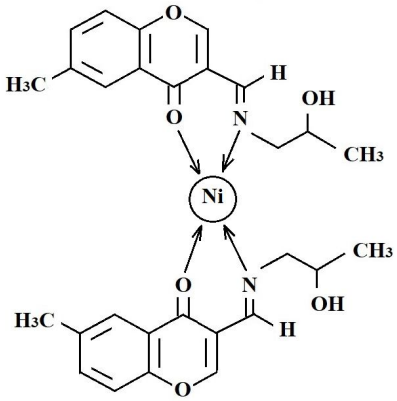
Fig. Structures of ligands

1. **Preparation of $[Ni(L_1)_2Cl_2]$:** 0.01 mol, Schiff base (**L_1**) (231 mg) in methanol was mixed dropwise and a solution 0.005 mol of $CoCl_2{\cdot}6H_2O$ (119 mg) was added and continued refluxing until the solution became green. The solid separated was filtered and resulting violet red solid compound was separated, washed with water followed by methanol, dried and recrystallized with methanol. Finally, light color crystals were obtained.



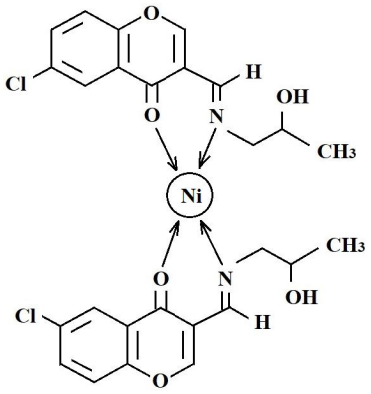
Light green color crystals, yield: 72%, mp: >300 °C, IR υ_{max} (KBr) cm^{-1}: 408 cm^{-1}, 516 cm^{-1}, 854 cm^{-1}, 1622 cm^{-1}, 1640 cm^{-1}, 3494 cm^{-1}; Analytical Calculations (%) for $CoC_{26}H_{26}O_6N_2$: Co-11.31, C- 59.89, H- 4.99, N- 5.38; Found: Co-11.28, C- 58.80, H-5.02, N- 5.92.

2. **Preparation of [Co $(L_2)_2Cl_2$]:** 0.01 mol, Schiff base (L_2) (245 mg) in methanol was mixed dropwise and a solution 0.005 mol of $CoCl_2 \cdot 6H_2O$ (119 mg) was added and continued refluxing until the solution became brown. The solid separated was filtered and resulting violet red solid compound was separated, washed with water followed by methanol, dried and recrystallized with methanol. Finally, dark brown color crystals were obtained.



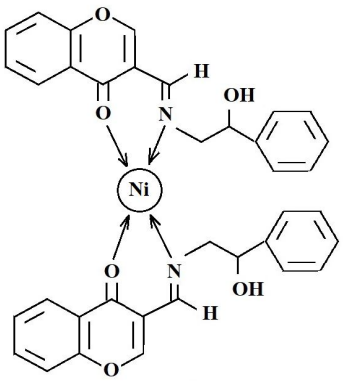
Brown color crystals, yield: 72%, mp: >300 °C, IR υ_{max} (KBr) cm^{-1}: 496 cm^{-1}, 562 cm^{-1}, 854 cm^{-1}, 1582 cm^{-1}, 1640 cm^{-1}; Analytical Calculations (%) for $CoC_{28}H_{30}O_6N_2$: Co-10.73, C- 61.21, H- 5.47, N- 5.10; Found: Co-10.65, C- 61.45, H-5.40, N- 5.08.

3. **Preparation of [Co $(L_3)_2Cl_2$]:** 0.01 mol, Schiff base (L_3) (266 mg) in methanol was mixed dropwise and a solution 0.005 mol of $CoCl_2 \cdot 6H_2O$ (119 mg) was added and continued refluxing until the completion of reaction. Separated solid was filtered, washed with water followed by methanol, dried and recrystallized with methanol. Finally, light brown color crystals were obtained.



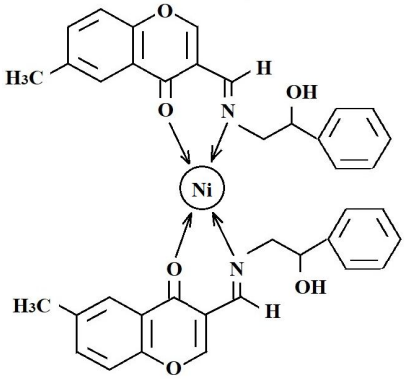
Light-Brown crystals, yield: 70%, mp: >300 °C, IR υ_{max} (KBr) cm^{-1}: 496 cm^{-1}, 562 cm^{-1}, 854 cm^{-1}, 1582 cm^{-1}, 1640 cm^{-1}; Analytical Calculations (%) for $CoC_{26}H_{24}O_6N_2$: Co-9.97, C- 52.80, H- 4.06, N- 4.74; Found: Co-9.98, C-52.76, H-4.04, N- 4.92.

4. **Preparation of [Co $(L_4)_2Cl_2$]:** 0.01 mol, Schiff base (L_4) (293 mg) in alcohol was mixed dropwise and a solution 0.005 mol of $CoCl_2 \cdot 6H_2O$ (119 mg) in ethanol was added and continued refluxing until the completion of reaction. Separated solid was filtered, washed with water followed by methanol, dried and recrystallized with methanol. Finally, light brown color crystals were obtained.



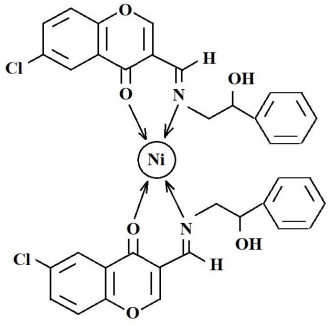
Reddish-Brown crystals, yield: 74%, mp: >300 °C, IR υ_{max} (KBr) cm^{-1}: 496 cm^{-1}, 562 cm^{-1}, 854 cm^{-1}, 1582 cm^{-1}, 1640 cm^{-1}; Analytical Calculations (%) for $CoC_{36}H_{30}O_6N_2$: Co-9.13, C- 66.96, H- 4.65, N- 4.34; Found: Co-9.08, C-66.84, H-4.64, N- 4.30.

5. **Preparation of $[Co\ (L_5)_2Cl_2]$:** 0.01 mol, Schiff base ($\mathbf{L_5}$) (307 mg) in methanol was mixed dropwise and a solution 0.005 mol of $CoCl_2{\cdot}6H_2O$ (119 mg) in ethanol was added and continued refluxing until the completion of reaction. Separated solid was filtered, washed with water followed by methanol, dried and recrystallized with methanol. Finally, dark brown color crystals were obtained.



Dark-Brown crystals, yield: 78%, mp: >300 °C, IR υ_{max} (KBr) cm^{-1}: 496 cm^{-1}, 562 cm^{-1}, 854 cm^{-1}, 1582 cm^{-1}, 1640 cm^{-1}; Analytical Calculations (%) for $CoC_{38}H_{34}O_6N_2$: Co-8.75, C- 67.76, H- 5.05, N- 4.16; Found: Co-8.62, C-67.84, H-5.02, N- 4.08.

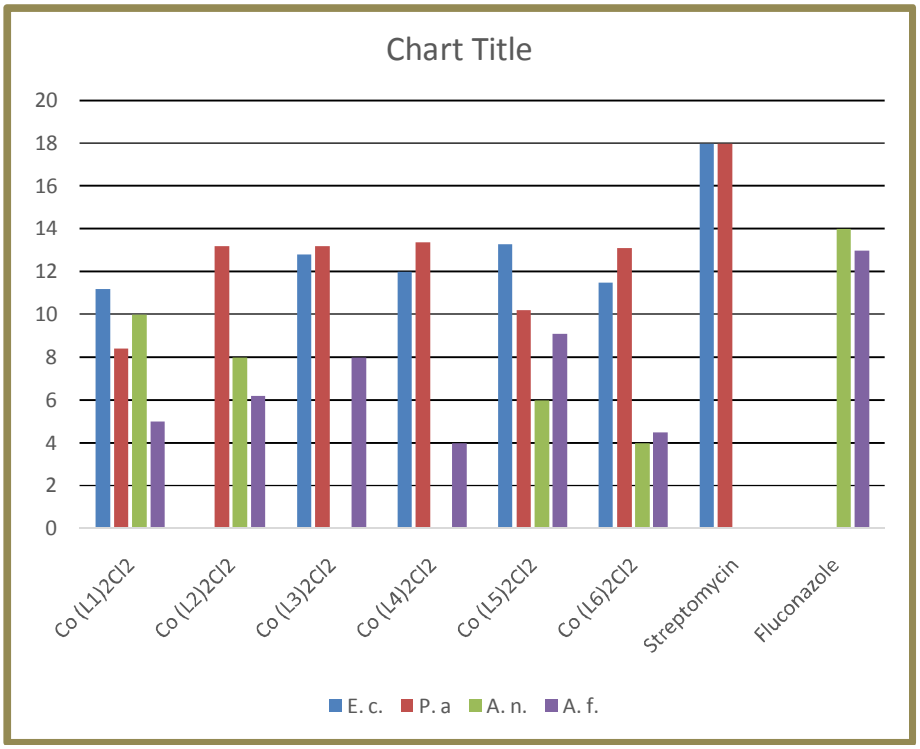
6. **Preparation of $[Co\ (L_4)_2Cl_2]$:** 0.01 mol, Schiff base ($\mathbf{L_4}$) (330 mg) in alcohol was mixed dropwise and a solution 0.005 mol of $CoCl_2{\cdot}6H_2O$ (119 mg) in ethanol was added and continued refluxing until the completion of reaction. Separated solid was filtered, washed with water followed by methanol, dried and recrystallized with methanol. Finally, brown color crystals were obtained.



Brown crystals, yield: 64%, mp: >300 °C, IR υ_{max} (KBr) cm^{-1}: 496 cm^{-1}, 562 cm^{-1}, 854 cm^{-1}, 1582 cm^{-1}, 1640 cm^{-1}; Analytical Calculations (%) for $CoC_{36}H_{28}O_6N_2Cl$: Co-8.67, C- 63.63, H- 4.12, N- 4.12; Found: Co-8.58, C-63.54, H-4.16, N- 4.06.

Antibacterial and Antifungal Activity:

The synthesized compounds were screened for their in vitro antibacterial activity against *Escherichia coli, Pseudomonas aeruginosa* and antifungal activity against *Aspergillus niger, Aspergillus flavus,* by measuring the zone of inhibition in mm. The antimicrobial activity was performed by filter paper disc plate method at concentration 100 µg/mL and reported in Table 7.1. Muller Hinton agar & Sabouroud Dextrose agar were employed as culture medium and DMSO was used as solvent control for antimicrobial activity. Streptomycin and Fluconazole were used as standard for antibacterial and antifungal activities respectively.



*Zone of inhibition was measured in mm. *Escherichia coli (E.c.), Pseudomonas aeruginosa (P.a), Aspergillus niger (A.n.), Aspergillus flavus (A.f.).*

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