Preparation, Spectroscopy, Biological Activities and Thermodynamic Studies of New Complexes of Some Metal Ions with 2-[5-(2-Hydroxy-Phenyl)-1,3,4-Thiaziazol-2-Ylimino]-Methyl-Naphthalen-1-OL

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Received 22/5/2021, Accepted 4/7/2021, Published Online First 20/1/2022, Published 1/8/2022

Abstract: This work describes the preparation of new series of tetra-dentate N₅O₃ dinuclear complexes (Cr³⁺, Co²⁺, Cu²⁺) of the Schiff base derived from condensation of 1-Hydroxy-naphthalene-2-carbaldehyde with 2-amino-(2-hydroxy-phenyl)-1,3,4-thiaziazole. The structures of the ligands were identified using IR, UV-Vis, mass, elemental analysis and ¹H-NMR techniques. All prepared complexes have been characterized by conductance measurement, magnetic susceptibility, electronic spectra, infrared spectrum, thermogravimetric analysis (TGA) and metal analysis by atomic absorption. From stoichiometry of metal to ligand and all measurements show a octahedral geometry proposed for all complexes of the (Cr³⁺, Co²⁺, Cu²⁺), conductivity measurement shows that of the prepared (Co²⁺, Cu²⁺) complexes were non electrolyte but (Cr³⁺) complexes were electrolyte. The parameters of thermodynamic, activation energy E_a, enthalpy ΔH, entropy ΔS and Gibbs free energy ΔG were calculated using Coats-Redfern method by the TGA curve. The bioactivity of the prepared (LH₂) and its complexes have been examined with antibacterial activity which shows significant activity against some fungi and bacteria.

Keywords: Biological activities, Thermodynamic parameters, 2-hydroxy-1-naphthaldehyde, 1,3,4-thiaziazole.

Introduction: Multiple or tetra-dentate Schiff bases contain N₅O₃ coordination, and their mineral complexes have gained great interest due to their excellent complexity. They are used in extracting many metallic ions from water due to their ability to form stable metal chelates complexes and are widely studied in coordination chemistry, especially those which contains heterocyclic compounds with the azomethine group, as it has basic properties due to the presence of an electron pair on the nitrogen atom azomethine (-C = N) and often pentagonal or hexagonal rings with the metal ion. Complexes that contain naphthalene compounds were investigated in vitro for their antibacterial and antifungal potentials. The Schiff bases heterocyclic metals complexations have been intensively investigated in recent years in many applications such as in antibiotics and medicine, catalyst, Thiazole compounds are related and have diverse bioactivity activity possibly via N-C-S binding, which is of good importance in many pesticides. The rules have recently gained great importance due to their diverse biochemical properties. The study of the thermal behavior is of great importance in the knowledge of many applications such as structural changes, thermal stability, thermal decomposition and chemical reactivity in the field of polymers, curing and catalysis. The present study describes the coordination behavior of Schiff base (LH₂) towards some transition elements and we report on the results obtained in a study of the biological activities and thermodynamic of (Cr³⁺, Co²⁺ and Cu²⁺) complexes with (LH₂).

Material and Methods: Materials
All chemicals were obtained from (Sigma- Aldrich) companies. 1-Hydroxy-naphthalene-2-carbaldehyde, Salicylic acid, thiosemicarbazide, POCl₃

DOI: http://dx.doi.org/10.21123/bsj.2022.19.4.0829
Instrumentation

The electronic spectra were registered by using Shimadzu 160 A- Spectrophotometer. Mass analysis of ligand was done with LC-Mass 100P Shimadzu. The IR spectra of ligand and complexes were obtained (as a discs of KBr) in the range 4000-4000 cm\(^{-1}\). (Bruker BM6) device was used to conduct magnetic sensitivity measurements at room temperature using the (Faraday Method). Thermal analysis studies of the compounds were performed on Mettler instrument TGA. Conductivity measurements were performed with a conductivity meter Model PCM 3 - JENWAY. CHN analysis was carried out using analyzer model 5500 Carlo-Erba. A.A.S. Spectrophotometer model double-beam atomic absorption spectrometer, model: AA400 Analytic Jeana (made in Germany). Centrifuge model PLC-03, (made in Taiwan), Electro-thermal bath model AA-00267, (made in England).

Preparation of Compound [A] \(^{10-11}\)

The properties M.p. yield and C.H.N.O analysis are shown in Tab. 1.

Syntheses of 2-[5-(2-Hydroxy-phenyl)- 1,3,4-thiadiazol-2-ylimino]-methyl-naphthalen-1-ol \([\text{LH}_2]\) \(^{12}\)

In crucible a stoichiometric (0.02 mol) of compound [A] with same amount of 1-Hydroxy-naphthalene-2-carbaldehyde was put in microwave irradiation 230 W for three minutes, after the completion of the reaction, the obtained solid was recrystallized by absolute ethanol, some of properties are listed in Tab. 1.

Preparation of Metal Complexes

A stoichiometric reaction of the corresponding LH\(_2\) ligand (0.02 mol in 20 ml methanol was added to few drops of Trimethylamine) before mixing in 50ml round bottom flask (0.02 mol) metal (II) chlorides molar ratio (M: L) of 1:1. A mixture was put in ultrasonic bath 60 °C. After 60 minutes, crystalline colored precipitates were formed after cooling at room temperature, the resulting solids were filtered off, washed with distilled water & ether, dried in a desiccator. Some properties are shown in Tab. 1.

Stoichiometric Determination of Complexes: \(^{13}\)

Continuous variation (JOB) method was used make sure to the correlation ratio between ions and ligand in equilibrium media.

| Compound | Yield% | Analysis (calculated) |
|----------|--------|-----------------------|
|          |        | C%                    |
| LH\(_2\)| 67%    | 65.45 (65.69)         |
| C\(_{19}\)H\(_{13}\)N\(_4\)O\(_5\)S | 3.82 (3.77) |
| [Cr\(_2\) (LH\(_2\))\(_2\) (H\(_2\)O)\(_4\)]Cl\(_2\) | %73 |
| [Co\(_2\) (LH\(_2\))\(_2\) (H\(_2\)O)\(_4\)] | %77 |
| [Cu\(_2\) (LH\(_2\))\(_2\) (H\(_2\)O)\(_4\)] | %71 |
| N% | O% | Cl% | M% |
| 12.24 | 19.27 | -- | -- |
| (12.10) | (9.21) | -- | -- |
| 8.83 (8.96) | 13.55 (13.60) | 7.63 (7.56) | 10.97 (11.09) |
| 14.65 (14.53) | -- | 13.23 (13.38) |
| 14.29 (14.38) | -- | 14.45 (14.28) |

Results and Discussion:

FT-IR Spectra of LH\(_2\)

The method for the synthesis of ligand (LH\(_2\)) is illustrated in (Scheme 1). The FT-IR spectra of the ligand showed the disappearance of the bundles of the (C=O) of the aldehyde in the region1645 cm\(^{-1}\) and the amino group (-NH\(_2\)) in the region 3402-3213 cm\(^{-1}\) and the emergence of new beams, which are the bundles of the right group, and the absorption beams of the imine group of the prepared ligand were in the range 1623 cm\(^{-1}\) which belongs to the azomethine group, and the frequencies of the thiadiazole ring appeared at 1053-1239 cm\(^{-1}\). \(^{12}\) Fig.1 and Tab. 2, contain the values of the infrared spectra of the prepared ligand.

Mass Spectral Data and \(^{1}H\)-NMR of Ligand

The mass spectral of Schiff base ligand which appeared at molecular ion peaks, at m/z 348.0 (M+). Fig. 2 which is in good agreement with the expected values m/z=347.39. Fig. 3: \(^{1}H\)-NMR(CDC\(_1\)Cl,400MHz) \(\delta=\) 13.687, 11.506(s,2H, OH), 8.756 (s, H, CH=N), 6.155-7.504 (m,13 H, Ar-H), 3.753 1.584-1.279(solvent+H\(_2\)O)
The all FT-IR spectra bands assignments of the compounds are presented in Tab.2. The band of imine group $\nu$ (C = N) in the LH$_2$ 1623 cm$^{-1}$ complexes were shifted to lower frequencies in all the complexes, that indicates that they are linked by the nitrogen atom, of (C=N) in coordination with the metal $^{14}$. As shown in the tables, the disappearance of the bands in the range 3419 -3481 cm$^{-1}$ belonging to the hydroxyl phenolic (O) complexes is an evidence of its chelation by the phenolic oxygen atom $^{15}$. The bending (wagging and twisting) of the coordination water complexes appear by about 623-757 cm$^{-1}$ $^{16}$. The linked nitrogen atom of thiadiazol ring shows shifted absorption bands of ligand range 1174 -1303 cm$^{-1}$ in complexes which confirms the metal's binding to the group (= N-N =) $^{17}$. For all complexes, a new beam appeared in the range 586-590 cm$^{-1}$ due to the vibrations of the group stretch (M-O) $^{18}$ and showed a stretching of the group (M-N) of the prepared complexes in the bounded region between 458-495cm$^{-1}$, confirming the metal’s association via the (N) atom $^{19}$. All the infrared spectrum values for the complexes are shown in Tab.2.

| Compound               | $\nu$(C=N) | $\nu$(H-O) | $\nu$(C-N=N-C) | $\nu$ (H$_2$O) Wagging | $\nu$ (H$_2$O) Twisting | $\nu$ (H$_2$O) | $\nu$ (M-N) | $\nu$ (M-O) |
|------------------------|------------|------------|----------------|-------------------------|-------------------------|----------------|-------------|-------------|
| LH$_2$                 | 1623(s)    | 3419 -3481 | 1156-1354      | -                       | -                       | 3441           | -           | -           |
| [Cr$_2$(LH)$_2$(H$_2$O)$_2$]Cl$_2$ | 1606(s)   | ---        | 1155-1301      | 623                     | 757                     | 3375           | 493         | 586         |
| [Co$_2$(LH)$_2$(H$_2$O)$_4$] | 1598(s)   | ---        | 1159-1311      | 683                     | 748                     | 3345           | 458         | 590         |
| [Cu$_2$(LH)$_2$(H$_2$O)$_4$] | 1613(s)   | ---        | 1117-1299      | 692                     | 749                     | 3387           | 495         | 587         |

**Electronic Spectra, Magnetic Moments and Molar Conductance of Complexes:**

Most of the complexes of the transition elements show absorbance at certain wavelengths of the spectrum, because most of these complexes are colored. The electron spectrum of the prepared complexes was recorded in the range 200–1100 nm using DMF solvent $^{20}$. The (Vis) spectra of the Chromium complex show two bands at 428- 608 nm Tab.3, attributed to the
allowed transfer \( ^4A_{2g} \rightarrow ^4T_{1g} \) (F) and \( ^4A_{2g} \rightarrow ^4T_{2g} \) (F) respectively\(^21\). It has been observed that the electron spectrum of complex Cobalt (II) is low spin octahedral \( t_{2g}^6e_{g}^1 \), one permissible transition 500 nm, which is \( ^2Eg \rightarrow ^2T_{2g} \) \(^22\). The spectrum of the copper (II) complexes showed an absorption beam at the region 722nm as shown in Tab.3. This is attributed to \( ^2B_{1g} \rightarrow ^2B_{2g} \). It agrees with the published research in this regard \(^23\).

The distinction between the UV -spectra Fig.4 of ligand with complexes shows a displacement that was observed , it ranged between 5-20 nm and there is a difference between the spectra of the solutions of ligand and the metal ion, as well as the clear difference in the colors of the mixing solutions from the solutions of the ligand and the metal ion before mixing, which is clear evidence of a coordination between them \(^24\).Tab.3 gives the electronic spectral, magnetic moments and molar conductance data of the prepared compounds. The results of the magnetic susceptibility gave values for the magnetic moment which correspond to the suggested shape.

### Table 3. Some physical data electronic spectra for \((LH_2)\) and complexes in DMF

| Compound            | Dec. Point \(^{0}\text{C}\) | Conductivity \(\text{ohm}^{-1}\text{cm}^2\text{mol}^{-1}\) 25\(^{\circ}\text{C}\) | Magnetic Moment | Color | Absorption Bands (nm) | Assigned Transition |
|---------------------|------------------------------|-----------------------------------------------------------------|-----------------|-------|------------------------|---------------------|
| \(LH_2\)            | 206-207                      | 8                                                               | -               | yellow | 235                  | \(\pi \rightarrow \pi^*\) |
| \([\text{Cr}_2(LH_2)_2(H_2O)_4]\text{Cl}_2\) | 268d                         | 142                                                             | 3.9             | Violet | 608                  | \(^4A_{2g} \rightarrow ^4T_{2g} \) (F) |
| \([\text{Co}_2(LH_2)_2(H_2O)_4]\)            | 281                          | 19                                                              | 2.39            | Dark   | 500                  | \(^2E_{1g} \rightarrow ^2T_{2g} \) (F) |
| \([\text{Cu}_2(LH_2)_2(H_2O)_4]\)            | 255d                         | 23                                                              | 1.89            | Brown  | 722                  | \(^2B_{1g} \rightarrow ^2B_{2g} \) |

**Figure 4. Ultraviolet spectrum**

**Continuous Variation Method**

The absorbance of the complexes was measured at \(\lambda\) max = 483, 500, and 413nm the stoichiometric ratio between the Cr(III), Co(II), Cu(II), ligand 1:1 the results are shown in Fig. 5.
Thermal Analysis

As shown in Fig. 6, the complex showed three well-defined steps. The first step in the thermal curve that represents the loss of four (H₂O) molecules of Co^{2+} and Cu^{2+} but Cr^{3+} loss (4H₂O+2Cl⁻) this is an evidence of the coordinated water molecules in complexes. The second, third, and fourth steps weight losses are explained in Tab.4. These steps are a loss of mass in the form of gases. In the final step large weight drop can be explained by considering that the residue is a 1:1 mixture of (2MO).

The activation energy (E_a), entropy (∆S), enthalpy (∆H), and Gibbs free energy (∆G) were calculated using the Coats & Redfern equation. All thermodynamic values are given in Tab. 5. We find that the positive values of ∆G indicate that the interactions are non-spontaneous in the transformation state. As for negative values - ∆S, they indicate that ligand (LH₂) has a more ordered structure than the reactants, and that the reactions are more slow than normal or regular. Positive values for ( ΔH ) indicate that the reactions are endothermic. The negative values indicate that the reactions are heat-emitting and the small values of the Arrhenius factor (Z) indicate that the reactions of the decomposition of ligand (LH₂) are fast, while the large and positive values in the state of transition can be classified as slow reactions.

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**Table 5. Thermodynamic & Kinetic factors of the (Cr^{3+}, Co^{2+}, Cu^{2+}) complexes.**

| Sample (step) | T.range °C | N | R² | Ea | ∆H | ZSec⁻¹ x10⁰ | ∆S | ∆G |
|--------------|------------|---|----|----|-----|--------------|-----|-----|
| Cr(1)        | 37-188     | 0.9| 0.99| 129.5| 47.84 | 7.75         | -230.5 | 137.31 |
| Cr(2)        | 188-343    | 0.9| 0.99| 52.199| 122.16 | 0.5445       | -101.14 | 171.11 |
| Cr(3)        | 343-540    | 0.9| 1   | 12.758| -5.856 | 5.01         | -360.81 | 301.9 |
| Co(1)        | 37-178     | 0.9| 0.99| 7.88 | 3.985 | 980.5486     | -94.4712 | 37.71 |
| Co(2)        | 140-272    | 0.9| 0.99| 8.6145| 4.124  | 361.2055     | -105.181 | 61.15 |
| Co(3)        | 272-462    | 0.9| 0.99| 12.2451| 6.9247 | 705.6812     | -100.224 | 73.87 |
| Cu(1)        | 37-178     | 0.9| 0.99| 5.92844| 3.2447 | 145.99       | -109.054 | 40.90 |
| Cu(2)        | 178-325    | 0.9| 0.99| 12.2235| 6.9457 | 480.02       | -103.991 | 74.45 |
| Cu(3)        | 325-449    | 0.9| 0.99| 12.4721| 5.1425 | 977.79       | -100.571 | 69.77 |

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**Table 4. TGA analysis data of complexes**

| Sample (step) | T.range °C | Weight mass loss (calc) found% | Reaction |
|--------------|------------|-------------------------------|----------|
| Cr(1)       | 37-188     | (14.97) 15.26               | (4H₂O+2Cl⁻) |
| Cr(2)       | 188-343    | (29.980) 28.891             | C₁₀H₁₆O₂ |
| Cr(3)       | 343-540    | (41.75) 40.95               | C₁₀H₂₂N₂O₂S₂ |
| Final residual | (14.50) 14.38 |                      | 2CrO¹ |
| Co(1)       | 37-178     | (8.17) 8.60                 | 4H₂O |
| Co(2)       | 140-272    | (31.83) 30.22               | C₂₀H₆O₂ |
| Co(3)       | 272-462    | (46.09) 45.04               | C₁₀H₁₆N₂O₂S₂ |
| Final residual | (17.84) 17.13 |                      | 2CoO |
| Cu(1)       | 37-178     | (8.10) 8.50                 | 4H₂O |
| Cu(2)       | 178-325    | (38.02) 37.42               | C₂₂H₄₄N₂O₂ |
| Cu(3)       | 325-449    | (35.10) 35.65               | C₁₆H₁₄N₂S₄ |
| Final residual | (17.87) 18.36 |                      | 2CuO |

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Figure 5. Continuous Variation method of complex
Figure 6. Thermal Analysis of Complexes
Biological Activity
The drilling method experiment was conducted and the experiment was conducted under aerobic conditions (temperature of 37 °C), four types of pathogenic bacteria were grown: Staphylococcus aureus, Escherichia coli, Pseudomonas aeruginosa and Streptococcus pneumonia. (Two negative for Gram stain and two positive for Gram stain, the compound is effective against positive stain bacteria. Staphylococcus aureus and Streptococcus pneumonia) are effective against Escherichia coil negative bacteria only at 200 ml/mg. All Antibacterial activities of the prepared compounds in Fig.7 and Tab. 6.

Table 6. Antibacterial activity of the prepared compounds.

| Symbol                   | Staphylococcus aureus | Escherishia coli | Pseudomonas aeruginosa | Streptococcus pneumonia |
|--------------------------|-----------------------|------------------|------------------------|-------------------------|
| LH2                      | +                     | +                | +                      | +                       |
| [Cr₂(LH₂)₂(H₂O)₄]Cl₂     | ++                    | +++              | ++                     | +++                     |
| [Co₂(LH₂)₂(H₂O)₄]        | +++                   | +                | +++                    | +++                     |
| [Cu₂(LH₂)₂(H₂O)₄]        | +++                   | +++              | +                      | +++                     |

Note(-)=no inhibition, (+) = (5-10) mm, (+ +)=(11-20) mm, (+ + +) = more than (20)mm

Figure 7. Antibacterial activity of Schiff base ligand

Conclusions:
We have observed new ligand compound and its complexes from the first series transitional metals (studies of their physical properties and various analyses). The collected data demonstrated that the ligand behaves as tetradeutate ligand of N₂O₅; binuclear stable complexes. From the electronic spectra, infrared spectrum and magnetic measurements, it is indicated that most of Cr(III), Co(II), and Cu(II) complexes contain hexa
coordinate and have octahedral geometry Fig.8. Molar conductivity measurements of the prepared complexes indicate that complexes with the formula [M₂(LH₂)₂(H₂O)₄] with M(II) = Co, Cu were neutral (non electrolyte), while the other complexes with the formula [Cr₂(LH₂)₂(H₂O)₄] Cl₂ were electric type (1:2).

![Figure 8. Suggested structure for complexes / M = Co(II) and Cu(II)](image)

**Author's declaration:**
- Conflicts of Interest: None.
- I hereby confirm that all the Figures and Tables in the manuscript are mine. Besides, the Figures and images, which are not mine, have been given the permission for re-publication attached with the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee in University of Baghdad.

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تحضيرودرسة طيفية والفاعليّة الحيوية والكيميائية لمعدقات جديدة لبعض أيونات المعادن مع-[2-(5-هيدروكسي-فينيل)-4,3,1-ثيازول]-2-ريد-نيفينو-2-ثيازول. ناقش شلاع

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الخلاصة:

تم في هذه الدراسة تحضير سلسلة جديدة من المعادن ثنائيّة النواة تحت الحرارة، وتغليف الأشعة تحت الحمراء وتم تقييم نسبة الفئات بطريقة الانتهاء النهائيات، حسب النسبة الحارية (TGA) والأنف طيف الكترون. نظرًا للتحكم في انتاجية القياس وفقًا لقياس، تم انتاج برلم ت يمتلك المعادن المكملة من [Cr(III), Co(II), Cu(I)] كأيضًا، تم تحت ثبت المعادن من [Cr(III), Co(II), Cu(I)] كأيضًا، تم تحت ثبت المعادن من [Cr(III), Co(II), Cu(I)].

الكليّات المفتاحية: الفعاليّة الحيوية، الأسمدة، الهيدروكسي-فينيل، Thiourea, 2,4,6-ثيابوز،-نيفينو-2-ثيابوز.