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Imam Mohammad ibn Saud Islamic University

College of Science

Department of Chemistry

Synthesis and characterizations of Novel cobalt and zinc complexes of 1-Allylimidazole

A graduation Research Project

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the degree of Bachelor of Science in Chemistry

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Abstract

Two novel organic-inorganic hybrid complexes based on Cobalt(II) and Zinc(II) with 1-allylimidazole ligand were synthesized using two distinct methods: slow-evaporation at room temperature and reflux. $ZnCl_2$(1-allylimidazole)$_2$ has been identified using elemental analysis and infrared spectroscopy. The second complex, $CoCl_2$(1-allylimidazole)$_2$, was identified using single crystal X-Ray diffraction and characterized by UV-Visible spectroscopy.

ملخص

تم تحضير مركبين هجينين عضوي-غير عضوي جديدين يعتمدان على الكوبالت (II) والزنك (II) مع ليجند أليليميدازول باستخدام طريقتين مختلفتين: التبخر البطيء في درجة حرارة الغرفة والتكثيف. تم التعرف على ZnCl2(1-allylimidazole)2 باستخدام تحليل العناصر، التحليل الطيفي للأشعة تحت الحمراء، وفحص الرنين المغناطيسي النووي. تمت دراسة الترتيب الثاني، $CoCl_2$(1-allylimidazole)$_2$، باستخدام حيود الأشعة السينية البلورية الأحادية وتم تحليل بمطياف الاشعة فوق البنفسجية.

List of Abbreviations

°C: Celsius.

OIH: Organic-inorganic hybrid

OLEDs: Organic light-emitting diodes

LECs: Light-emitting electrochemical cells

PBI: Polybenzimidazole

IR: Infrared

M.P.: Melting point.

MW: Microwave.

NMR: Nuclear magnetic resonance.

%: Percent sign.

atm: Atmospheric pressure

Å: Angstrom

mm^3: Millimeter cubic

MLCT: Metal-Ligand Charge Transition

eV: Electron Volt

Introduction

1- Introduction

Organic-inorganic hybrid (OIH) materials combine organic and inorganic components, leveraging the unique properties of both. This synergy allows for tailored materials with enhanced functionalities, often used in electronics [1], sensors [2], and catalysis [3]. The design and synthesis of these hybrids aim to exploit the best attributes of each component, leading to materials with diverse applications and improved performance.

These hybrid systems are divided into two classes: Class I and Class II, based on whether the phases interact weakly or strongly. In fact, the organic and inorganic components are embedded, and only weak bindings (hydrogen, Van der Waals, or ionic bonds) provide cohesion to the entire structure in class I, but in class II hybrids, strong chemical linkages (covalent or iono-covalent bonds) exist between the two parts.

In recent years, OIH has been developed on a massive scale, with several applications in analytical chemistry, bio- and environmental monitoring [4, 5]. One may notice a significant increase in the number of papers and patents connected to the creation of novel OIH. Such combinations can be formed into crystals, thin films [6], nanofibers [7], nanoparticles [8], porous materials [7], and hierarchical nanostructures [9], which is critical when designing effective biosensing systems with fast readout capabilities for biomedical and environmental applications.

1.1. Applications of Imidazole and its derivatives

Various N-heterocyclic frameworks have been recognized as building synthon ligands in the field of (OIH) chemistry due to their capacity to function as donor units, leading to the development of desirable characteristics [10-12]. Researchers with an interest in building hybrid organic-inorganic systems for diverse uses have been intrigued by N-heterocyclic systems, including imidazole and its derivatives. The distinctive characteristics of imidazole-based complexes, such as excellent electrochemical and photoluminescent properties, excellent thermal properties, and facile modification, were considered when designing various host materials such as electron transport, fluorescent, or phosphorescent molecules [13-15].

1.1.1. Optic application [16]

Electroluminescent devices have risen in favor in recent years and improving the efficiency of organic light-emitting diodes (OLEDs) and light-emitting electrochemical cells (LECs) has become a major research focus. Because of their structural diversity, imidazole derivatives have resulted in the development of numerous good electroluminescent devices such as fluorescent and phosphorescent emitters.

1.1.2. Medicinal Application [17]

Imidazole-containing medications have a wider clinical application for treating a number of diseases. Heinrich Debus synthesized imidazole for the first time in 1858, however other imidazole derivatives were discovered as early as the 1840s. He combined glyoxal and formaldehyde with ammonia to make imidazole. Imidazole has anticancer, b-lactamase, carboxypeptidase, hem oxygenase, antiaging, decoagulant, antibacterial, fungicidal, antiviral, antitubercular, antidiabetic, and antimalarial properties.

1.1.3. Industrial application [17]

Imidazole has long been utilized as a corrosion inhibitor for changeover metallic elements like copper. Copper corrosion avoidance is crucial, especially in aquatic environments where copper conductivity decreases due to corrosion.

Imidazole is a key ingredient in many industrial and technical compounds. The thermostable polybenzimidazole (PBI) is a fire retardant that combines imidazole with a benzene ring and bonds to it. Imidazole can be found in various compounds used in photography and electronics.

Imidazole has few explicit functions. It is a pioneer in agricultural chemicals such as enilconazole, climbazole, clotrimazole, prochloraz, and bifonazole.

1.2. Synthesis of OIH materials

There exist multiple techniques for synthesizing OIH materials, and we will discuss the most significant and straightforward ones.

1.2.1. Solvothermal Method [18]

Solvothermal synthesis is the use of a solvent at high temperatures (often 100-1000°C) and pressures (1-10.000 atm). The process is normally carried out in a sealed vessel near or over the boiling point of the reaction medium (for example, in an autoclave). The reaction vessels must be chemically inert. The reaction solution is heated over the boiling point of the solvent employed in the sealed autoclave, resulting in a high pressure. Under such conditions, the solvent is transformed into a supercritical fluid. After the reaction is completed, the autoclave is cooled to room temperature, and the solvents and impurities are removed to yield the desired product (Fig. 1).

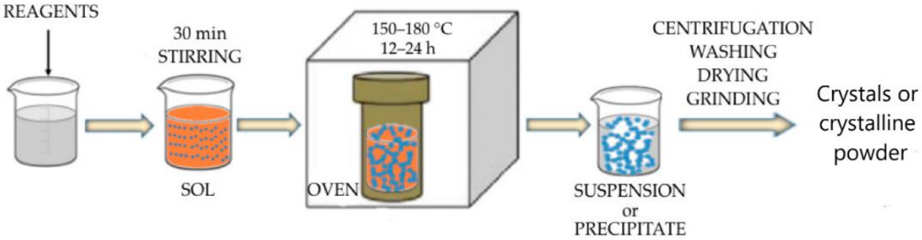

Figure 1. Synthesis of OIH materials by solvothermal method.

1.2.2. Reflux method [19]

To achieve the desired phase and shape, the parameters, the order of precursor addition, the time of reflux, and the cooling rate must be tuned.

The schematic diagram (Fig. 2) illustrates a standard reflux arrangement. The reaction vessel, often a round bottom flask, is equipped with a condenser to prevent the solvent vapors from exiting the system. The condenser is equipped with an external enclosure that allows for the circulation of a coolant, often cooled water. The moving water extracts heat from the solvent vapors, causing them to condense and return to the reaction vessel. Hence, the overall volume of the solution remains constant, allowing for prolonged heating. In the reflux method, the reaction temperature corresponds to the boiling point of the solvent employed. Thus, the selection of the solvent can be adjusted to the reaction's requirements. To achieve higher temperatures, an oil bath or a sand bath is utilized.

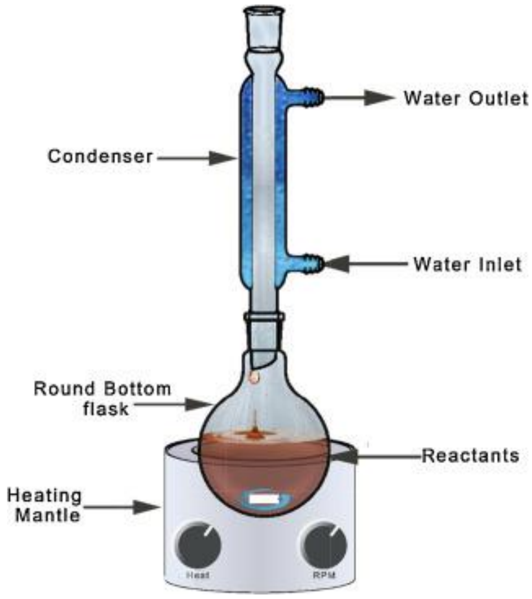

Figure 2. Synthesis of OIH materials by reflux method.

1.2.3. Mechanochemical method [20]

The process of producing inorganic material, co-crystals, metal-ligand complexes, metal organic frameworks, polymers, etc. using energy from motion. Ball-milling, also known as mechano-milling, is an advanced approach derived from classic grinding techniques with a mortar and pestle. Mechano-milling procedures are typically carried out in vibration mills or planetary mills at specific frequencies. The widely employed mechano-milling process has constraints in regulating the reactions for compounds that are susceptible to air and moisture. Typically, mechano-milling procedures include conducting reactions within enclosed containers made of materials such as stainless steel, tungsten carbide, zirconia, agate, and others (Fig. 3).

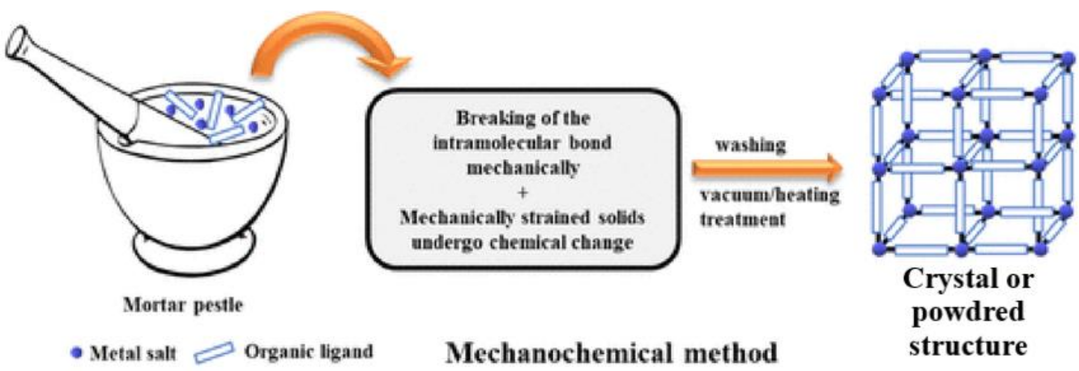

Figure 3. Mechanochemical synthesis of OIH materials

1.2.4. Slow evaporation at room temperature method [21]

This approach is applicable to materials that exhibit air and moisture stability at room temperature. As its name suggests, this process includes the gradual evaporation of the solvent from the solution containing the molecule until it becomes saturated, and crystals start to develop (Fig. 4).

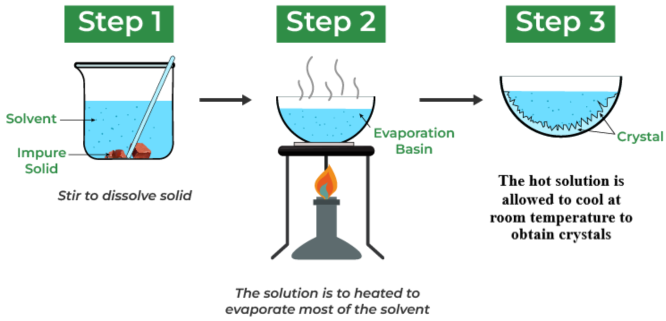

Figure 4. Slow evaporation synthesis method of OIH materials.

Experimental part

2. Synthesis of organic-inorganic complexes

Within this section of the research, we will investigate the procedure for preparing the subjected complexes.

2.1. Materials and Methods

The table below summarizes the reagents utilized in the synthesis of the investigated complexes and provides their respective characteristics. All chemicals were purchased from commercial suppliers and used without further purification.

Table 1: Information about the used chemicals and solvents.

Name of materials	Chemical formula	Solubility	Percentage of purity
Ethanol	C_2H_6O	-	99.8%
Zinc chloride	$ZnCl_2$	H_2O	97%
Cobalt chloride	$CoCl_2$	H_2O	97%
1-Allyimidazole	$C_6H_8N_2$	Ethanol and H_2O	97%

2.2. Synthesis

In this work, assembled from 1-Allylimidazole ligand (which includes imidazole group) and, two Zn and Co-containing complexes, namely, [$ZnCl_2$(1-Allyimidazole)$_2$] and [$CoCl_2$(1-Allyimidazole)$_2$], were synthesized by different methods.

2.2.1. Synthesis of $ZnCl_2$(1-Allylimidazole)$_2$: (Slow evaporation technique)

At room temperature, 1-allylimidazole (0.2 g) and $ZnCl_2$ (0.13 g) were dissolved in 25 mL of ethanol solvent. For three hours, the solution was stirred and heated (Fig. 5a). After one hour of standing and cooling, we noticed the formation of brilliant white powder (Fig. 5b).

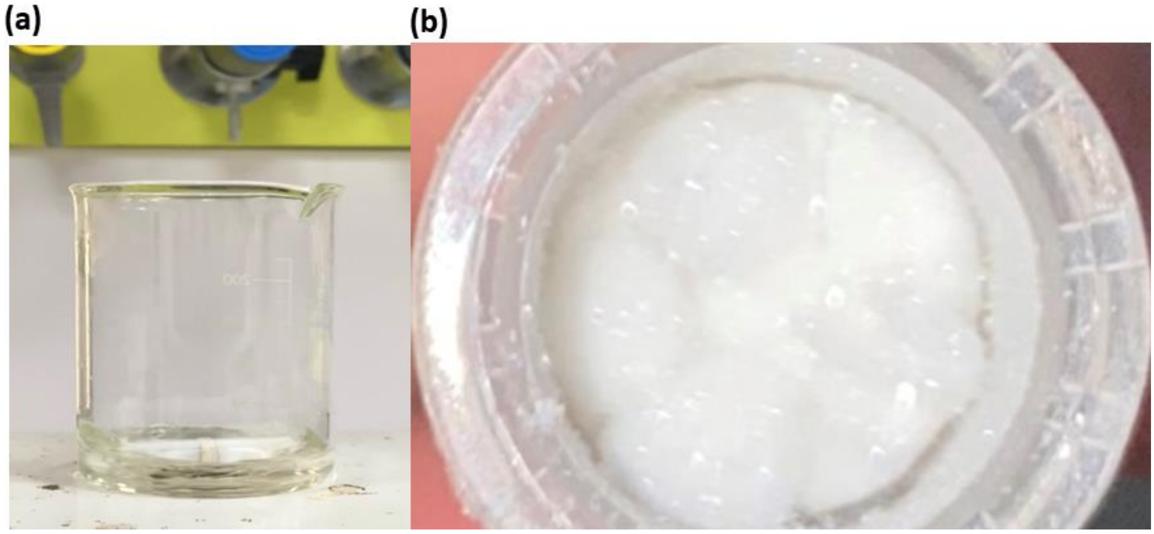

Figure 5. Synthesis of $ZnCl_2$(1-Allylimidazole)$_2$

2.2.2. Synthesis of $CoCl_2$(1-Allylimidazole)$_2$: (Reflux technique)

$CoCl_2.6H_2O$ (1.12 g) and N-allylimidazole (0.2 g) were dissolved in EtOH (25 mL). The reaction mixture was refluxed for 4 hours (Fig. 6a). The precipitated blue powder of 2 was washed with ether and dried under vacuum (Fig. 6b).

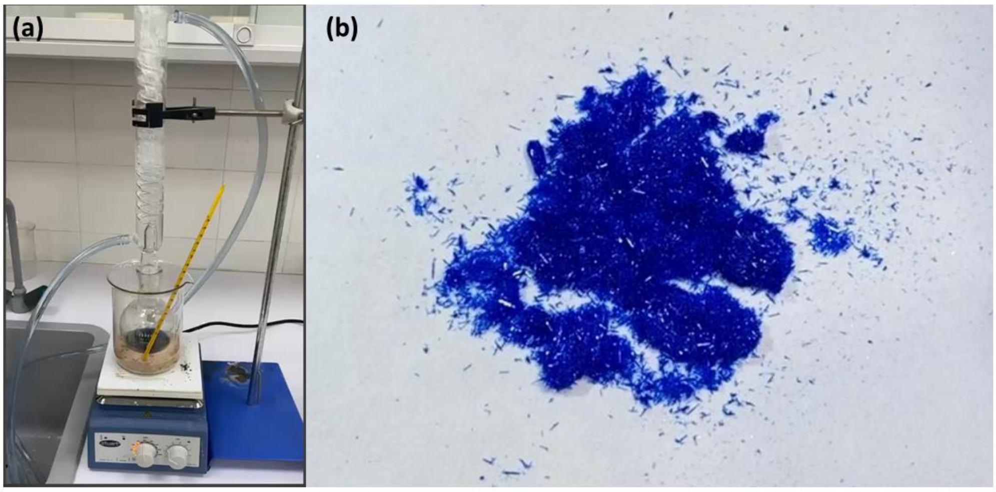

Figure 6. Synthesis of $CoCl_2$(1-Allylimidazole)$_2$

Results and discussion

3. Results and discussions

3.1. Characterization of $ZnCl_2(C_6H_8N_2)_2$ complex

The $ZnCl_2(C_6H_8N_2)_2$ is characterized by elemental analyses and Infrared spectroscopy.

3.1.1. Elemental analysis

Elemental analytical data of 1-Allylimidazole and its Zn-Complex are very close to the theoretical values [22] as shown in Table 2. Elemental analysis shows that the metal to ligand ratio is 1:2 and the composition of metal complex is a tetrahedral complex, $ZnCl_2$(1-allylimidazole)$_2$.

Table 2. Analytical Data of 1-Allylimidazole and its $ZnCl_2(C_6H_8N_2)_2$ Complex.

Compounds	Chemical formula	Color	C (%)	H (%)	N (%)
1-Allylimidazole	$C_6H_8N_2$	Yellow	66.60	7.41	25.89
(Zn-Complex) $_{exp}$	$ZnCl_2(C_6H_8N_2)_2$	White	40.69	4.40	15.54
(Zn-Complex) $_{calculated}$	$ZnCl_2(C_6H_8N_2)_2$	-	40.88	4.57	15.89

3.1.2. Infrared Spectroscopic Analysis

The shape and positions of the IR bands (Fig. 7, table 3) of the complex support the structure suggested. Coordination of the 1-allylimidazole molecules through ‘pyridynic’ nitrogen atoms of the imidazole rings manifests in displacement towards higher energies of the ν(C=C) vibrations and those of ν(C=N) of the imidazole ring. Generally, imidazole has several bands of variable intensity in the region of 1660-1480 cm^{-1} due to C=N and C=C stretching vibrations [23, 24]. In this case, the imidazole is mono-substituted, in which the C=N and C=C stretching vibrations are observed to have strong and medium intensity at 1670 and 1635 cm^{-1}, respectively. In the free ligand, C-N and C-C stretching peaks of are observed at 1504 and 1420 cm^{-1}. Here, strong intensity C-N and C-C stretching peaks were observed at 1525 and 1455 cm^{-1}. The stretching of the coordination Zn-N is attributed to the absorption band at 421-1000 cm^{-1} [25]. Due to the presence of CO_2 in the air, there are two stretching vibration peaks near 2500 cm^{-1} in the IR spectrum [26].

Table 3. IR spectra for 1-Allylimidazole and its $ZnCl_2(C_6H_8N_2)_2$ complex of selected regions (cm^{-1}).

Compound	$C{=}N_{Stretching}$ frequency	$C{=}C_{Stretching}$ frequency	$C\text{-}N/C\text{-}C_{Stretching}$ frequency	$Zn\text{-}N_{Stretching}$ frequency
1-Allylimidazole	1675	1566	1504/1420	-
$ZnCl_2(C_6H_8N_2)_2$	1670	1635	1525/1455	485

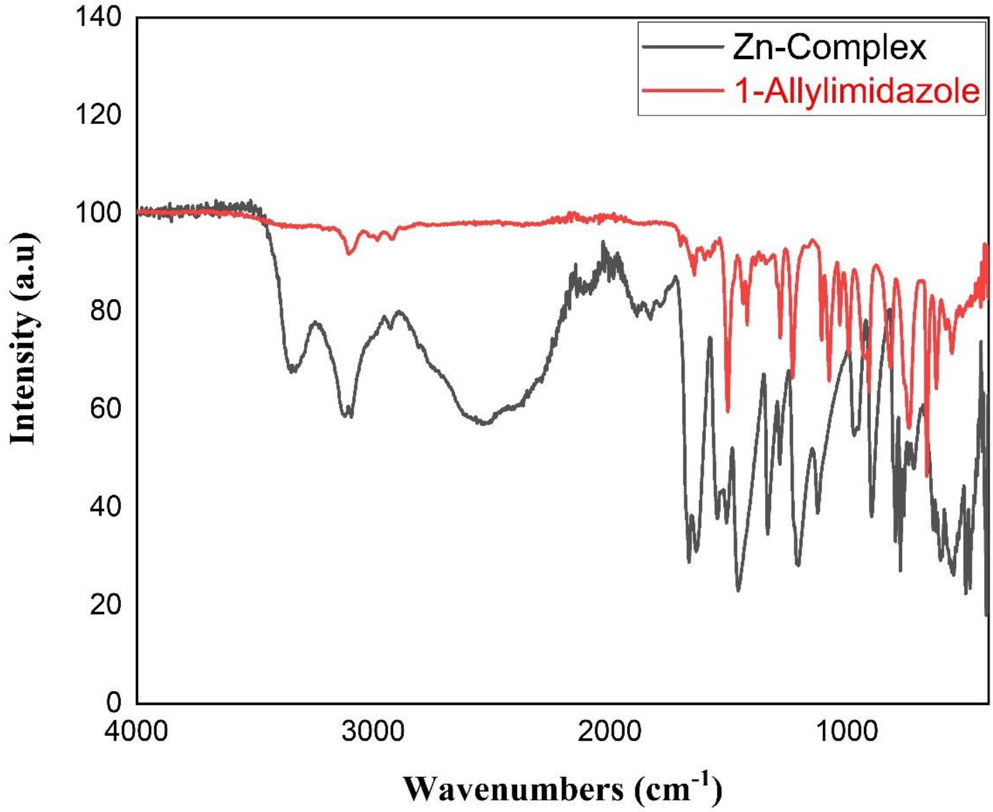

Figure 7. The infrared spectrum of $ZnCl_2(C_6H_8N_2)_2$ recorded at room temperature.

3.2. Characterization of $CoCl_2(C_6H_8N_2)_2$ complex

3.2.1. Single crystal X-Ray diffraction

A Bruker APEX-II CCD diffractometer was used to perform the X-ray measurements. The structures were solved directly using the SHELXS-97 program [27] and refined using full-matrix least-squares methods with the SHELXL-97 program [28]. Table 4 contains details on the crystal data for $CoCl_2(C_6H_8N_2)_2$ complex.

Table 4. Crystal data $CoCl_2(C_6H_8N_2)_2$ Complex.

Chemical formula	$C_{12}H_{16}Cl_2CoN_4$
M_r	346.12 g/mol
Crystal system/space group	Orthorhombic, Pbca
a	8.1083 (13) Å
b	15.348 (2) Å
c	24.982 (4) Å
Volume	V = 3109.0 (8) $Å^3$
Shape and color	Plate, blue
Dimensions	0.3 × 0.1 × 0.05 mm^3

The $CoCl_2(C_6H_8N_2)_2$ complex crystallizes in an orthorhombic system with Pbca space group. The cobalt cation in the complex is tetrahedrally coordinated by four monodentate ligands, which are two N-coordinated imidazole groups and two chloride anions. The molecular structure of monomeric units for this complex is seen in Figure 8. These monomeric units are joined together by intermolecular hydrogen bonds creating a 3-dimenstonal crystalline structure (Fig. 9).

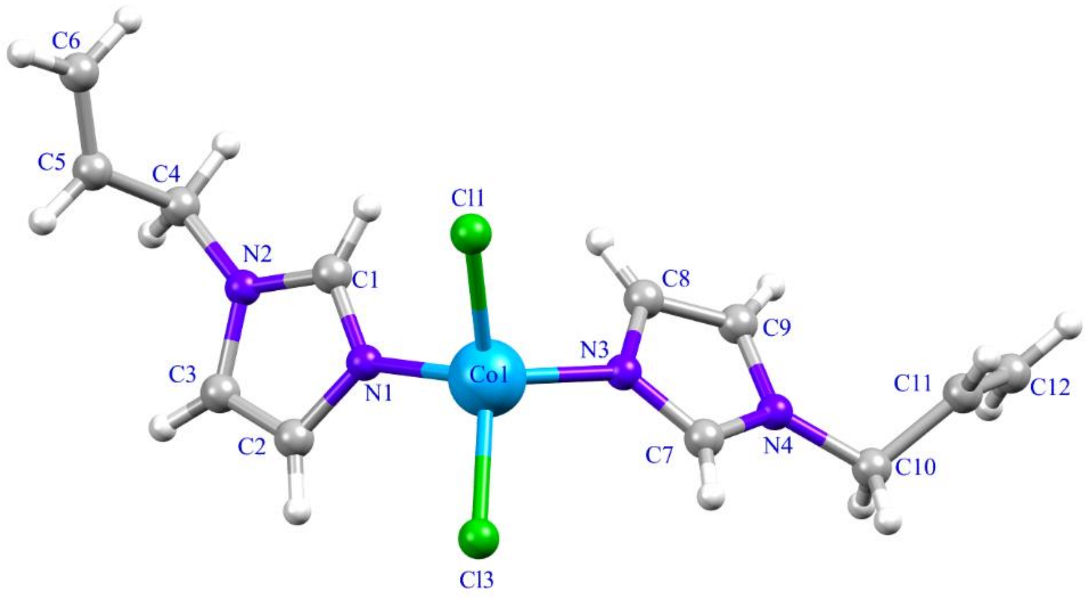

Figure 8. Molecular structure of $CoCl_2(C_6H_8N_2)_2$ complex.

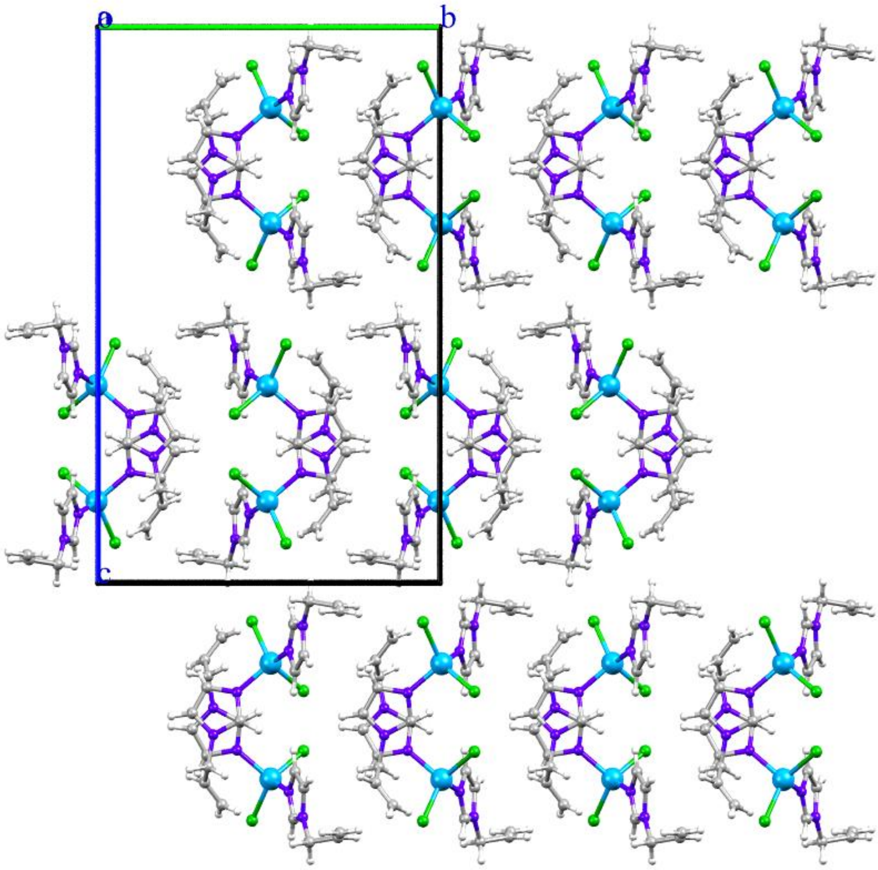

Figure 9. Crystal structure of $CoCl_2(C_6H_8N_2)_2$ complex.

3.2.2. UV–Visible Spectroscopy

Figure 10 displays the absorption spectrum of the $CoCl_2(C_6H_8N_2)_2$ complex. It displays a wide band in the visible area of the spectrum. Within the complex's spectrum, two distinct peaks of absorbance are detected at 268 nm and 395 nm. These peaks correspond to the electron transitions from $\pi \rightarrow \pi^*$ orbitals in 1-Allylimidazole, and to the Metal-Ligand Charge Transition (MLCT) between Cobalt and the ligands.

According to the following equation (which converts the wavelength to energy), the energy gap of this complex was Eg=2.45 eV, indicating that it is a semiconductor.

$$Eg = \frac{1240}{\lambda(nm)}\ \text{eV}$$

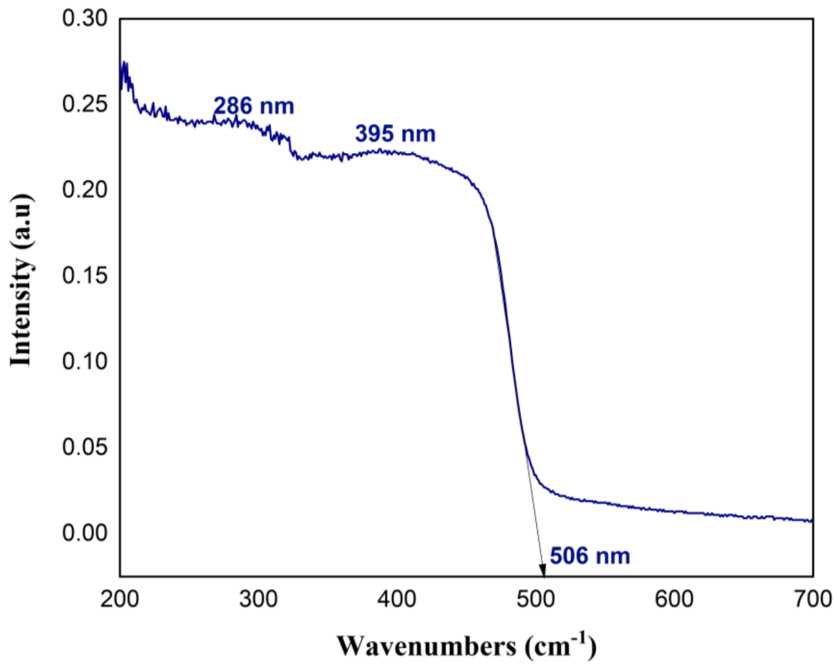

Figure 10. UV-Visible spectrum of $CoCl_2(C_6H_8N_2)_2$ complex in solid state.

Conclusion and recommendation

4.1. Conclusion

In this research project, two complexes of Co(II)/Zn(II)-N-allylimidazole were successfully synthesized. $ZnCl_2(C_6H_8N_2)_2$ expected structure was confirmed by elemental analysis and IR spectroscopy as a tetrahedrally coordinated zinc with four ligands (two chloride anions and two 1-allylimidazole molecules). The coordination compound $CoCl_2(C_6H_8N_2)_2$ was verified using SCXRD analysis, which revealed that the cobalt atom is tetrahedrally coordinated by four monodentate ligands: two imidazole groups coordinated through nitrogen atoms and two chloride anions. The cobalt complex was further investigated by UV-Visible spectroscopy, and it is a semiconductor with an energy gap 2.45 eV.

4.2. Recommendation

1- Recrystallization of $ZnCl_2(C_6H_8N_2)_2$ complex and determination of its structure using single crystal x-ray diffraction

2- Determination of optical and biological properties of synthesized complexes.

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Curriculums

ASAYEL ABDULLAH ALFALEH

GRADUATED GENERAL CHEMISTRY

055 489 8309 AsayelAbdullahh@gmail.com

professional summary

Hardworking, highly motivated professional eager to lend combined knowledge and skills to enhance business performance. Operates well in both individual and team capacities, leveraging seasoned work ethic to quickly adapt to different processes and drive company objectives. Resourceful and results-driven with a passion for growth and efficiency to meet company needs and increase service value.

skills

- Research and Development
- Report Writing
- Microsoft Excel
- Organic synthesis
- Spectroscopy methods
- Environmental chemistry
- Laboratory techniques
- Chemical safety protocols
- Sample Processing
- Collaborative Teamwork
- Data Management
- Nuclear chemistry
- Statistical analysis
- Chromatography techniques
- Problem-solving abilities

education

Expected graduation **Feb 2024**

BACHELOR OF SCIENCE (B.S.) IN GENERAL CHEMISTRY CANDIDATE

Imam Mohammed Ibn Saud Islamic University

languages

ARABIC

Native

ENGLISH

Fluent

KOREAN

Intermediate

Reema Mohammed Munif Al-Shammari

General chemistry

Abaut me

A student at Imam Mohammed Saudi university
Specialty general chemistry

Contact

Phone

0504816401

Email

rreema.2128@gmail.com

ID number

1108381169

Experience

1. Understand the theoretical foundations and basic concepts in general chemistry, including elements, chemical compounds and chemical reactions.

2. The ability to carry out chemical experiments and use various instruments and equipment used in the laboratory.

3. Analysis and interpretation of chemical data using appropriate statistical and mathematical tools.

Languages

- Arabic Advanced
- English Intermediate

University

Imam Mohammed bin Saud University

Courses

OSHA cycle
Computer course

Testimony

There are no certificates

RA

RAGHAD ALMUSALLAM

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Education

Imam Muhammad Ibn Saud Islamic University
Bachelor of Science in CHEMISTRY
2018 - 2024

PERSONAL SKILLS

- Team Work
- Fast training
- Punctuality
- Problem Solving
- Strategic Planning
- Attention to details
- Analytical Thinking
- Time Management

PRACTICAL SKILLS

- Microsoft Office
- Report Writing
- Lab management
- Use of Chemical devices
- knowledge of testing techniques

WORKSHOPS

- Ethics and Work Culture | May 2023
- Soft skills and the art of communication | May 2023

Languages

- Arabic
- English

NUHA HADI OBID AL-RSHEED

CONTACT :

0553957281

nuhahadi.11@gmail.com

SKILLS :

- Work with team
- Communication
- Use Word & PowerPoint
- Good for using computer

COURSE :

- Misk Vocational Preparation Program
- Chemical Crime
- Renewable energy
- The Nano technique

EXPERIENCE :

- Volunteer work in medical laboratories

LANGUAGE :

- Arabic
- English

EDUCATION :

- Students in Imam Mohammed Ibn Saud Islamic University
- Major : Chemistry
- Degree : Bachelor's
- The year of admiss on to university : 2019
- Year of graduate : 2024

Arwa Alkhathami

alkarwa56@gmail.com

0552282353

Riyadh, Saudi Arabia

PROFILE

A highly motivated and hard-working chemistry student at Imam Muhammad Ibn Saud Islamic University who is passionate about using chemistry to solve problems. Proven experience carrying out laboratory tests and interpreting data.

EDUCATION

2019 - 2024 **Imam Muhammad Ibn Saud Islamic University,**
Bachelor of Science in Chemistry

LANGUAGES

Arabic English

SKILLS

Data analysis • Scientific writing • Time management • Analytical thinking

CERTIFICATES

- The Safety Data Sheet Awareness Certification
- The COSHH Risk Assessor Certification