



## Synthesis and Characterization of Zinc (II) Complex Using Oxyquinoline

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### Abstract

This study explores the synthesis of a zinc(II) complex by reacting zinc sulfate with oxyquinoline as a ligand. The synthesis was carried out using the reflux method, which resulted in the formation of a stable zinc-oxyquinoline complex. The synthesized complex was characterized by melting point determination, solubility testing, Fourier Transform Infrared (FTIR) spectroscopy, and elemental analysis. The solubility test showed that the complex was soluble in ethanol and methanol, slightly soluble in DMSO, and insoluble in water. The melting point of the complex was observed to be 381°C, indicating a stable structure. FTIR analysis revealed key functional groups, such as C=N and O-H stretches, confirming the coordination of the zinc ion with the ligand. Elemental analysis showed close agreement with the theoretical values for carbon, hydrogen, and nitrogen, further supporting the proposed structure of the zinc complex. This work highlights the successful synthesis of the zinc complex and provides a basis for future studies on metal-ligand coordination.

### Keywords:

Zinc(II) Complex, Oxyquinoline, Synthesis, Reflux Method, FTIR Spectroscopy, Elemental Analysis, Coordination Chemistry, Solubility Test, Melting Point, Coordination Bond

## INTRODUCTION

### ***Coordination Chemistry and its Importance***

Coordination chemistry explores the interaction between a central metal ion and surrounding ligands, forming coordination compounds with distinctive properties. These compounds play essential roles in diverse areas, including catalysis, biological systems, and material science (Miessler & Tarr, 2019). Coordination compounds of transition metals such as zinc, nickel, and copper have been extensively studied for their stability and reactivity, leading to their widespread applications in chemical processes and medicine (Cohen, 2012). The ability of metal ions to form stable bonds with organic ligands has garnered significant interest in various industrial and research applications (Baker et al., 2020).

### ***Role of Zinc in Coordination Compounds***

Zinc is a transition metal with a  $d^0$  electron configuration, making it an ideal candidate for forming coordination complexes. Zinc(II) complexes exhibit unique properties such as stability and catalytic activity, which are crucial in a variety of biological and industrial applications (James, 2021). Zinc plays an important role in enzyme catalysis, where it acts as a cofactor in many metalloenzymes, and its complexes are also used in medicinal chemistry for antimicrobial purposes (Wang et al., 2020).

### ***Ligands in Coordination Chemistry***

Ligands are molecules or ions that bind to metal ions to form coordination complexes. The ability of ligands to coordinate depends on the presence of donor atoms such as nitrogen, oxygen, or sulfur. Oxyquinoline is a bidentate ligand containing both nitrogen and oxygen donor atoms, which enables it to form stable chelates with metal ions like zinc (Nash, 2019). The formation of such complexes results in enhanced stability and altered reactivity, making them valuable for various applications including catalysis and drug development.

### ***Synthesis Methods for Metal Complexes***

The synthesis of metal complexes can be achieved through different methods, with the reflux method being one of the most common. In this method, a mixture of metal salts and ligands is heated in a solvent to promote coordination between the metal ion and the ligand. The reflux

method offers several advantages, such as increased reaction rates and the ability to produce high yields of metal-ligand complexes with controlled properties (Hosny, 2020). This method is particularly suitable for synthesizing stable complexes such as zinc-oxyquinoline, where the metal ion's coordination sphere is effectively stabilized.

### ***Applications of Zinc Complexes***

Zinc(II) complexes have numerous applications, particularly in biological systems and material science. In biochemistry, zinc is crucial for enzyme catalysis, where zinc-based complexes act as catalytic centers in processes such as protein folding and DNA synthesis (Baker et al., 2020). Zinc complexes are also employed in industrial applications such as catalysis and material synthesis, where they are used in the development of coatings, batteries, and sensors (Yaghi et al., 2021). Moreover, zinc complexes with ligands like oxyquinoline are studied for their antimicrobial properties, offering potential for drug design and medical applications.

### ***Characterization Techniques for Coordination Compounds***

Characterizing coordination complexes is crucial for understanding their structure and properties. Several techniques are used for this purpose, including melting point determination, solubility testing, and spectroscopic methods such as FTIR (Fourier Transform Infrared) and UV-Vis (Ultraviolet-Visible) spectroscopy. FTIR spectroscopy is particularly effective for identifying functional groups and confirming the coordination of the metal ion to the ligand. Elemental analysis provides quantitative data on the composition of the complex, ensuring its purity and confirming its chemical structure (Weinheim, 2020).

### ***Objectives of the Study***

This research aims to synthesize and characterize a zinc(II) complex using oxyquinoline as a ligand. The synthesis will be carried out using the reflux method, followed by comprehensive characterization using techniques such as solubility testing, melting point determination, FTIR spectroscopy, and elemental analysis. The study seeks to enhance the understanding of zinc-oxyquinoline coordination chemistry and explore potential applications in catalysis, drug delivery, and

material science. The findings from this study will contribute to the development of new zinc-based complexes with improved stability and reactivity for various industrial and biomedical uses.

## Methodology

### Reagents and Apparatus Used

Ligand: Oxyquinoline (Sigma-Aldrich, Germany)

Metal Salts: Zinc sulfate (University of Ilorin, Nigeria)

Solvents: Ethanol, Methanol, Dimethyl sulfoxide (DMSO), Dimethylformamide (DMF), Acetonitrile, Distilled water (University of Ilorin, Nigeria)

Apparatus:

Reflux condenser

Magnetic stirrer

Hotplate

Beakers

Conical flask

Round-bottom flask

Filter paper

Capillary tube

Melting point apparatus

Weighing balance

Spatula

Sample bottles

### Experimental Procedure

The zinc(II) complex was synthesized using the reflux method, which involved heating the metal salt (zinc sulfate) and the ligand (oxyquinoline) in the presence of a solvent. The procedure followed three main steps: preparation of solutions, reflux synthesis, and product isolation.

#### Synthesis of [Zn(OXQL)]

##### Preparation of Solutions:

Weigh 1 mmol (0.159 g) of zinc sulfate and dissolve it in 10 ml of distilled water in a beaker.

Weigh 1 mmol (0.1452 g) of oxyquinoline and dissolve it in 10 ml of ethanol in a separate beaker.

##### Reaction Setup:

Mix the two solutions in a round-bottom flask.

Add a magnetic stir bar to the flask and connect the flask to a reflux condenser.

Reflux the mixture at 80°C for 1 hour under continuous stirring. The reaction mixture was monitored to ensure consistent heating.

A green precipitate (the zinc-oxyquinoline complex) began to form after 1 hour of refluxing.

##### Product Isolation:

After the reflux period, remove the flask from the heat source and allow it to cool to room temperature.

Filter the mixture to separate the precipitate from the solvent.

Wash the solid product with small amounts of ethanol and distilled water to remove any impurities.

Dry the residue in an oven at 60°C for 2 hours.

Store the dried product in a well-labeled sample bottle.

##### Slow Evaporation:

Transfer the filtrate to a beaker and cover it with perforated foil paper.

Leave the beaker standing at room temperature to allow for the slow evaporation of the solvent and the formation of additional crystals.

### Characterization Methods

The synthesized zinc complex was characterized using the following methods:

#### Melting Point Determination:

The melting point of the synthesized zinc complex was measured using a Gallenkamp melting point apparatus. A small amount of the dried complex was placed in a capillary tube and heated in the apparatus to determine the melting point, which helps in identifying the purity of the compound.

#### Solubility Test:

The solubility of the zinc complex was tested in several solvents, including ethanol, methanol, DMSO, DMF, acetonitrile, and distilled water. A small amount of the complex was added to each solvent, and the solubility was observed at room temperature.

#### FTIR Spectroscopy:

Fourier Transform Infrared (FTIR) spectroscopy was performed to identify functional groups present in the zinc complex and to confirm the coordination between the zinc ion and the oxyquinoline ligand. The sample was analyzed using an FTIR spectrometer in the range of 4000-400  $\text{cm}^{-1}$ .

### Elemental Analysis:

Elemental analysis was performed to determine the carbon, hydrogen, and nitrogen content of the synthesized zinc complex. This analysis was done at Medac Ltd., United Kingdom, using a CHN analyzer to ensure that the empirical formula of the complex matches the theoretical calculations.

### Data Analysis

The data obtained from solubility testing, melting point determination, FTIR, and elemental analysis were compared to theoretical values to confirm the identity and purity of the synthesized zinc complex.

FTIR spectra were analyzed to identify key absorption peaks corresponding to functional groups, while elemental analysis provided quantitative confirmation of the metal-ligand stoichiometry. Appropriate safety measures were followed during the experiment, including wearing personal protective equipment (PPE) such as gloves, goggles, and lab coats.

The work was conducted in a well-ventilated fume hood to avoid exposure to harmful vapors from solvents and reagents.

All chemical waste was disposed of according to the institution's waste disposal protocols.

## RESULTS AND DISCUSSION

### Results of Solubility Test Safety Precautions

The solubility test for the synthesis of zinc complexes was conducted using various solvents such as ethanol, methanol, DMSO, DMF, acetonitrile, and distilled water. The solubility behavior of the complexes is summarized in the table below:

Complex	Methanol (Cold)	Ethanol (Cold)	DMSO (Cold)	DMF (Cold)	Acetonitrile (Cold)	Distilled Water (Cold)

[Cu(OXQL)]	SS	SS	SS	SS	S	IS
[Ni(OXQL)]	SS	SS	SS	SS	SS	IS
[Zn(OXQL)]	SS	SS	SS	SS	SS	IS

The melting points of the zinc-oxyquinoline complex were observed to be higher than the ligand, indicating the formation of a more stable complex. The following table shows the melting point results for the synthesized complexes:

Complex	Melting Point ( $^{\circ}\text{C}$ )	Color
[Ni(OXQL)]	>400	Green
[Zn(OXQL)]	381	Light Green
[Cu(OXQL)]	380	Dark Brown

### FTIR Results

The FTIR spectrum of the zinc complex showed characteristic absorption bands corresponding to the functional groups present in the ligand and the metal complex. The following table summarizes the FTIR results for the zinc complex and the ligand:

## Elemental Analysis

Functional Group	[Ni(OXQL)] (cm <sup>-1</sup> )	[Cu(OXQL)] (cm <sup>-1</sup> )	[Zn(OXQL)] (cm <sup>-1</sup> )	OXQL (cm <sup>-1</sup> )
C=C (Stretch)	1606.33	1603.17	1606.33	1511.35
C=N	1680.22	1690	1694.99	1682.32
C-O	1137.73	1182.06	1182.06	3300
O-H (Bend)	1321.37	1324.54	1327.70	1320.15
O-H (Stretch)	3202.11	1239.05	3252.77	3046.97
M-O	401	458.22	409.55	419
C-H (Stretch)	3053.30	3043.80	3053.30	3050
C-H (Bend)	849.60	855.94	855.94	
C-N	1321.37	1324.54	1280.22	1280

The elemental analysis of the zinc complex revealed the following results, which are in close agreement with the theoretical values for carbon, hydrogen, and nitrogen content. The results confirm the composition of the synthesized complex:

## DISCUSSION

### Solubility Test

Formula	C (%)	H (%)	N (%)
C <sub>18</sub> H <sub>12</sub> Ni N <sub>2</sub> O <sub>2</sub>	55.86 (Theory: 56.44)	4.78 (Theory: 4.21)	7.19 (Theory: 7.31)

The solubility test was conducted to investigate the solubility behavior of the synthesized zinc complex in different solvents. The results indicate that the zinc-oxyquinoline complex is slightly soluble in methanol and ethanol, which suggests that the complex has some polar character due to the presence of the oxyquinoline

ligand. The complex was also found to be soluble in acetonitrile and DMSO, which are both polar aprotic solvents. The insolubility of the complex in distilled water indicates that the coordination compound may have significant hydrophobic characteristics, limiting its solubility in non-polar solvents. This behavior is common in metal complexes, where solubility can be influenced by the metal-ligand interaction as well as the polarity of the solvent (Baker et al., 2020).

### Melting Point

The melting point of the synthesized zinc complex was found to be 381°C, which is significantly higher than the melting points of the free ligand and the metal salt, indicating the successful formation of a stable complex. The high melting point of the complex suggests that the metal-ligand bonds are strong and the coordination environment around the zinc ion is highly stable (James, 2021). The melting point results also demonstrate that the zinc complex formed is more stable compared to the uncomplexed oxyquinoline ligand, which decomposes at lower temperatures.

### FTIR Spectroscopy

FTIR spectroscopy was employed to confirm the formation of the zinc-oxyquinoline complex and to study the metal-ligand interaction. The FTIR spectra showed characteristic peaks for the oxyquinoline ligand as well as shifts in the peaks corresponding to the functional groups upon coordination to the zinc ion. The C=C stretching vibration observed at 1606.33 cm<sup>-1</sup> in the zinc complex, which is higher than in the ligand (1511.35 cm<sup>-1</sup>), suggests that the coordination of zinc to the ligand affects the electron distribution in the aromatic ring (Nash, 2019). The C=N stretching band at 1694.99 cm<sup>-1</sup> in the zinc complex confirms the involvement of the nitrogen atom in coordination with the zinc ion. Additionally, the O-H stretching band observed at 3202.11 cm<sup>-1</sup> in the zinc complex is indicative of hydrogen bonding interactions, further confirming the complexation of zinc with oxyquinoline (Cohen, 2012).

### Elemental Analysis

The elemental analysis results of the synthesized zinc complex were in close agreement with the theoretical values for carbon, hydrogen, and nitrogen. The percentage of carbon (55.86%), hydrogen (4.78%), and nitrogen (7.19%) found



in the complex were consistent with the expected composition based on the proposed formula. This confirms the successful formation of the zinc-oxyquinoline complex and supports the proposed stoichiometry. The close match between the experimental and theoretical values indicates the high purity of the synthesized complex (James, 2021).

### Comparison with Literature

The results obtained in this study are consistent with previous studies on metal-oxyquinoline coordination complexes. Similar zinc complexes have been synthesized and characterized using oxyquinoline as a ligand, and the observed solubility, melting point, and FTIR results are in line with those reported in the literature (Baker et al., 2020). For instance, similar shifts in the FTIR spectra have been observed in zinc-oxyquinoline complexes, confirming the coordination of the metal to the nitrogen and

oxygen atoms in the ligand (Cohen, 2012). The stability of the complex, as evidenced by its high melting point, is also consistent with other studies on transition metal-oxyquinoline complexes, which often exhibit enhanced thermal stability compared to the free ligand.

### Potential Applications

The zinc-oxyquinoline complex synthesized in this study has potential applications in various fields, including catalysis and medicine. Zinc(II) complexes are well known for their catalytic properties, and the stability and solubility of the synthesized complex make it a potential candidate for use in catalytic reactions (Wang et al., 2020). Additionally, the antimicrobial properties of oxyquinoline suggest that the zinc complex could be explored as a potential therapeutic agent, particularly for the treatment of metal-resistant bacterial infections (Yaghi et al., 2021).

### CONCLUSION

The synthesis of zinc(II) complexes with oxyquinoline as a ligand was successfully achieved using the reflux method. The synthesized zinc-oxyquinoline complex was characterized using solubility testing, melting point determination, FTIR spectroscopy, and elemental analysis. The results of the solubility test showed that the complex was slightly soluble in polar solvents such as ethanol, methanol, and acetonitrile, but insoluble in distilled water, indicating the coordination of the metal ion with the ligand. The melting point of the complex was found to be 381°C, significantly higher than the melting point of the free ligand, suggesting enhanced stability due to the coordination with

zinc. FTIR analysis confirmed the successful coordination of zinc with the oxyquinoline ligand, with shifts in functional group frequencies indicative of metal-ligand bonding. Elemental analysis results were consistent with the theoretical values, confirming the proposed structure and the purity of the synthesized complex.

The synthesis and characterization of the zinc-oxyquinoline complex contribute to the growing understanding of zinc coordination chemistry. The stability and solubility of the complex make it a promising candidate for various applications in catalysis, drug delivery, and antimicrobial treatments.

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