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Full Length Research Article

SYNTHESIS AND CHARACTERISATION OF CU (II) AND FE (II) METAL COMPLEXES WITH SALICYLIC ACID

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ABSTRACT
This study focuses on the synthesis and characterization of copper (II) and iron (II) complexes with salicylic acid as a ligand, using a solvent method. The complexes were prepared by reacting the metal salt solutions with salicylic acid in ethanol. The synthesis was confirmed by color changes and monitored pH levels, with a 79.08% yield for the Cu (II) complex and 60.25% for the Fe (II) complex. The characterization techniques included UV-Vis and FTIR spectroscopy, conductivity measurements, solubility testing, melting point determination, and atomic absorption spectroscopy (AAS). The UV-Vis spectra showed shifts indicative of complex formation, while FTIR spectra confirmed coordination through the hydroxyl and carboxylic groups of salicylic acid. Conductivity results suggested a non-electrolytic nature, and solubility tests indicated the complexes' compatibility with organic solvents. Melting point analysis demonstrated thermal stability, and AAS validated the metal-to-ligand stoichiometry. The findings suggest these complexes' potential for applications in non-aqueous media, catalytic, and indicate a foundation for further exploration of their biological and industrial properties.

INTRODUCTION

Transition metal complexes have become vital in diverse fields such as pharmaceuticals, catalysis, and materials science due to their unique chemical, electronic, and magnetic properties (Advances in Coordination Chemistry, 2023). Their ability to exist in multiple oxidation states and form stable bonds with negatively charged molecules has made them increasingly important in drug development. These complexes are particularly valued for their roles in treating cancer, infections, and inflammatory diseases, as well as in industrial and environmental applications (Wara, 2011).
Copper(II) and iron(II) complexes have been widely studied for their biological and chemical versatility. Salicylic acid, an aromatic carboxylic acid with hydroxyl and carboxyl donor groups, serves as an excellent ligand for these metal ions, forming stable chelates (Advances in Coordination Chemistry, 2023). Cu(II) salicylate complexes have shown anti-inflammatory and antioxidant properties (Praveen and Madhavi, 2016), and Fe(II) salicylates have demonstrated notable antimicrobial and antifungal activity in organic media (Hussaina *et al*., 2021). These complexes have also been found to exhibit enhanced drug potency and reduced toxicity when derived from salicylic acid derivatives like aspirin (Ogodo and Abosede, 2018).
Coordination chemistry explains these behaviors through metal-ligand interactions and geometric configurations. Studies show that Cu(II) and Fe(II) typically form octahedral or distorted octahedral complexes, with spectral bands confirming d–d transitions and charge transfer processes (Lawal *et al*., 2017; Abdel-Latif *et al.,* 2010). Schiff base complexes of these metals have demonstrated improved biological activity, thermal stability, and solubility, further supporting their relevance in therapeutic and material applications (Jabbi *et al*., 2020; Hussaina *et al*., 2021).
This study aims to synthesize and characterize Cu(II) and Fe(II) complexes with salicylic acid, and to evaluate their spectroscopic, thermal, and physicochemical properties, providing a foundation for their potential applications in biomedical and industrial chemistry.

MATERIALS AND METHODS

All chemicals used in this study were of analytical grade, obtained from Sigma-Aldrich, and used without further purification. The materials and reagents were supplied by the Chemistry Laboratory of the Federal University, Birnin Kebbi.

Experiments

The method described by Agwara et al., (2010) was employed in the synthesis of the ligand-metal complexes of salicylic acid. The metal salt 60 mmol (M=$CuNO_3.3H_2O$, $FeSO_4.7H_2O$) were dissolved in 20 ml of appropriate solvent (distilled water) and stirred at room temperature. 60 mmol of the ligands salicylic acid (SA) (8.288 g) were dissolved in 20 ml of ethanol and added in drops to the already stirring metal salt solution at room temperature. After the addition of the ligands in drops, the solution was further stirred for 30 minutes. In this study, a modified work-up procedure was employed to improve the stability and reproducibility of the synthesized metal complexes. Unlike conventional drying at higher temperatures or prolonged periods, the products were oven-dried at a controlled temperature of 75 °C for one hour. This mild drying condition effectively removed residual moisture while minimizing the risk of thermal decomposition of sensitive functional groups. Furthermore, immediate cooling in a desiccator prevented hygroscopic re-absorption of moisture, which is common to transition metal complexes. Repeated washing with distilled water ensured high purity by removing unreacted precursors and side products. Together, these steps constitute a refined adaptation of the protocol by Lawal et al. (2017), offering an optimized and reproducible approach that enhances the quality and stability of the final products.

Product Characterisation

The synthesized Cu(II) and Fe(II) salicylate complexes were characterized using the following analytical techniques; UV-Visible spectroscopy was employed to obtain characteristic absorption peaks between 200–800 nm due to electronic transitions, particularly π-π* and n-π* transitions. The electronic structure provides insight into the types of chromophores present in a molecule, thereby aiding in the interpretation of its spectroscopic properties. FTIR analysis was conducted using KBr pellets within the 650–4000 cm^{-1} range to identify functional groups involved in

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coordination. Conductivity measurements were taken at room temperature using deionized water to assess the electrolytic nature of the complexes, while pH monitoring during synthesis ensured optimal conditions for complex formation. The percentage yield was calculated based on the initial and final weights of the products. Solubility tests were performed by dissolving 0.1 g of each complex in a range of solvents, and thermal properties were evaluated through melting/decomposition point determination using a Gallenkamp apparatus. Water of crystallization was determined by loss on drying, following the procedure described by Bartyzel *et al,* (2020): approximately 2 g of the sample was placed in a pre-weighed crucible, dried in an oven at 110 °C, cooled in a desiccator, and re-weighed repeatedly until the mass change was less than 0.3 mg. The water percentage was calculated from the mass loss, and the number of water molecules per formula unit (n) was obtained by comparing with theoretical values. Finally, Atomic Absorption Spectroscopy (AAS) was used to determine the metal content after acid digestion, following the method described by Aliyu and Bello (2004).

RESULTS AND DISCUSSION

Physical Measurements of the Complexes

The Salicyclic acid and its metal (II) complexes were prepared in good yield, the physical properties of the synthesized metal complexes were analyzed and presented in Table 1. The successful synthesis of Cu(II) and Fe(II) salicylate complexes was confirmed by the formation of light blue and dark brown precipitates, respectively. These characteristic colors of the metal-ligand complexes indicate successful coordination between the metals and salicylic acid. The percentage yield of the Cu(II) complex was 79.08%, while the Fe(II) complex yielded 60.25%, reflecting high efficiency in the synthesis, with minimal loss of materials (Iorungwa *et al*., 2024). The pH during the synthesis of the Cu(II) complex was maintained around 4.80, while for the Fe(II) complex, it was around 5.67. These pH values were optimal for the formation of stable metal-salicylate complexes.

Low molar conductivity values of the Cu(II) (0.001731 µS) and Fe(II) (0.001792 µS) complexes in deionized water further confirm their non-electrolytic nature. This behavior is consistent with coordination compounds where charge neutrality is achieved through covalent or coordinate covalent bonding rather than the presence of free ions (Iorungwa *et al*., 2024). The Cu(II) complex exhibited a melting point of 185°C, while the Fe(II) complex had a melting point of 173°C, indicating their thermal stability (Bashir and Siraj, 2021). Atomic absorption spectroscopy (AAS) verified the stoichiometry of the complexes, with metal-to-ligand ratios of 1:2. The proposed structure for the complexes is $[M(SA)2(H_2O)_2]$, indicating coordination through hydroxyl and carboxyl groups, with two coordinated water molecules (Iorungwa *et al*., 2024). The absence of unbound water molecules in both complexes is evidenced by the IR spectra, which showed no O–H stretching (3200–3600 cm^{-1}) or H–O–H bending (~1600 cm^{-1}) bands typical of lattice water. Gravimetric analysis by oven-drying to constant weight at 110 °C revealed 0.00% water of crystallization, confirming the lack of dehydration in the region usually associated with lattice water loss. This anhydrous nature is further supported by the bidentate coordination of salicylate ligands, which saturates the metal coordination sphere and prevents incorporation of water. These observations are consistent with the findings of Tahier *et al.* (2015), who demonstrated that coordination complexes without weight loss in the low-temperature range are structurally anhydrous.

Table 1. Results of some physical measurements of the complexes

Compound	Color	Conductance(µS)	pH	% Yields	MP (°C)	AAS (mg/kg)	% water of crystallization
SA	White	NA	2.60	NA	157-159	NA	NA
Cu(II)SA	Light Blue	1731	4.80	79.08	164-185	60.33 ± 6.12	0.00
Fe(II)SA	Dark Brown	1792	5.67	60.25	160-173	182.66± 47.14	0.00

Keys: NA= Not Analysed, MP= Melting Point, SA= Salicylic Acid, AAS= Atomic Absorption spectroscopy

Product yield

The % yield and % water of **c**rystallization for both the **C**omplexes was computed using the system of equations in **Eqn. 1 & 3.** (Abubakar Abdullahi *et al*., 2021).

$$\%\ Yield_{\ metal} = \frac{weight\ of\ dried\ Product}{weight\ of\ the\ metal\ salt} \times 100\% \quad \textbf{Eqn. 1}$$

$$\%\ Water\ of\ crystallization = \frac{weight\ loss}{original\ weight} \times 100\% \quad \textbf{Eqn. 2}$$

Equation (2) could be expressed as;

$$\%\ Water\ of\ crystallization = \frac{W2-W3}{W1} \times 100\% \quad \textbf{Eqn. 3}$$

Where w1= original weight of complex(g), W2= Weight of complex before heating + weight of crucible (g), W3= Weight of complex after heating + weight of crucible (g)

Solubility test

Distilled Water and some common organic and inorganic solvents were used to determine the solubility of the Salicyclic acid and its metal (II) complexes. From the results of the Solubility test presented in Table 2. The Solubility tests showed that both the ligand and complexes were insoluble in distilled water, dimethyl ether (DME), ether, and petroleum ether but were soluble in organic solvents such as acetone, methanol, ethanol, and n-hexane. This insolubility in water indicates non-polarity due to the covalent and coordinate covalent bonding within the complexes, as well as the absence of ionic bonds or charged species. The improved solubility in organic solvents may be attributed to the chelation of the ligand with the metal center, which reduces lattice energy and increases lipophilicity of the resulting complex (Ejidike and Ajibade, 2015).

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Table 2. Solubility test of the Salicylic acid and its metal (II) complexes

Solvents	SA	Cu(II)SA	Fe(II)SA
Distilled water	SS	IS	SS
Ether	NA	IS	IS
Petroleum ether	NA	IS	IS
Ammonia	NA	S	SS
n-hexane	NA	S	S
Ethanol	S	S	S
Methanol	S	S	S
DME	NA	IS	IS
Peroxide	NA	S	S
Acetone	S	S	SS

Keys: SA= Salicylic acid, DME= Dimethyl ether, NA= Not Analysed, S= Soluble, SS= Slightly Soluble, IS= Insoluble

Frontier Infrared Spectroscopy (FTIR) Analysis

The FTIR results of the Salicyclic acid metal complexes together with their spectral data are presented in Table 3. The broad band at 3421 cm^{-1} in the IR spectrum of the free ligand (SA) was assigned to v(O-H) stretching vibrations, while a strong absorption band at 1691 cm^{-1} corresponded to v(C=O) asymmetric stretching, and 1460 cm^{-1} to v(C=O) symmetric stretching (Lawal et al., 2017). In the spectra of the Cu (II) and Fe(II) complexes, the v(O-H) stretching vibration shifted to a lower range (3004.2–3237.2 cm^{-1}), indicating coordination via the hydroxyl group. Similarly, v(C=O) stretching bands shifted to lower wavenumbers (1653.1 cm^{-1} in both complexes), accompanied by increased absorption intensity, confirming coordination through the carboxyl group (Abdel-Latif and Issa, 2010). The appearance of v(M-O) stretching vibrations in the range of 402–531 cm^{-1} confirmed metal-oxygen bonding. Additionally, bands in the range of 759–854 cm^{-1} indicated the presence of coordinated water in the complexes (Lawal *et al*., 2017). The band at 1300 cm^{-1} in the free ligand, attributed to phenolic groups, shifted to 1440.6 and 1444.3 cm^{-1} in the Cu(II) and Fe(II) complexes, respectively. This shift confirms the involvement of phenolic oxygen in coordination. The stretching vibrations of saturated v(C-H) bonds were observed at 2844 cm^{-1} (Cu (II)) and 2847.7 cm^{-1} (Fe (II) (Abdel-Latif and Issa, 2010).

Table 3. IR spectra data of the Salicylic acid ligand and it's metal (II) complexes

Compound	*v*(C=O)cm^{-1}	*v*(M-O) cm^{-1}	*v*(H_2O) cm^{-1}	*v*(OH) cm^{-1}	*v*(Ph-OH) cm^{-1}	*v*(C-H) cm^{-1}
SA	1691_{sym} 1460_{asym}	NA	NA	3421	1300	NA
Cu(II)SA	1653.1	402–531	759–854	3004.2–3237.2	1440.6	2844
Fe(II)SA	1653.1	402--531	759–854	3004.2–3237.2	1444.3	2847.7

Keys: SA= Salicylic Acid, Ph= phenol, asym= asymmetric, sym=symmetric, NA= Not Applicable

UV Analysis of Salicylic Acid Ligand and it's Metal (II) Complexes

The electronic spectra data of the Salicyclic acid, and its complexes are given in Table 4. Salicylic acid (SA) exhibits absorption peaks at 230 nm and 320 nm, associated with intra-ligand π-π* transitions. Upon coordination with Cu(II), the absorption peaks shifted to 220 nm and 260 nm, indicating a blue shift. This spectral shift confirms the formation of a new complex (Ogodo *et al*., 2018). Similarly, the broad absorption observed at 320 nm in the Cu(II) complex was assigned to a $^2E\_g \rightarrow {}^2T_2g$ transition, characteristic of distorted octahedral geometry. In contrast, the Fe(II) complex exhibited broad absorption between 500–540 nm, attributed to a $^5T_2g \rightarrow {}^5E\_g$ transition, indicative of octahedral geometry (Lawal *et al*., 2017).

Table 4. UV analysis data of salicylic acid ligand with it metal(II) complexes

Compound	π-π*	n-π*	Geometry
SA	230 nm	320 nm	NA
Cu(II)SA	220-260 nm	320 nm	Distorted Octahedral
Fe(II)SA	200-300 nm	500–540 nm	Octahedral

Keys: SA Salicylic Acid, NA= Not applicable, nm= nanometer

The reaction equations for the synthesized complexes are proposed based on the results obtained from spectroscopic analyses (FT-IR and UV-Visible spectroscopy) and analytical data, including atomic absorption spectroscopy, conductivity measurements, and determination of water of crystallization.

The propose equation of the reaction;

$Cu(NO_3)_2{\cdot}3H_2O + 2C_7H_6O_3 \rightarrow [Cu(C_7H_6O_3)_2(H_2O)_2] + 2HNO_3$

Name of the Copper complex: Diaquabis(salicylato)copper(II)

$FeSO_4{\cdot}7H_2O + 2C_7H_6O_3 \rightarrow [Fe(C_7H_6O_3)_2(H_2O)_2] + H_2SO_4$

Name of the iron complex: Diaquabis(salicylato)iron(II)

Conclusion

The synthesis of copper (II) and iron (II) salicylate complexes was successful, as evidenced by the physical and spectroscopic properties obtained. The observed shifts in FTIR and UV-Vis spectra, along with other measurements, support the coordination of salicylic acid to the metal ions through hydroxyl and carboxylic groups. AAS analysis confirmed the expected metal content, and both complexes lacked water of crystallization but included coordinated water. The stability, non-electrolytic nature, and solubility properties make these complexes suitable for various potential applications, particularly in non-aqueous media.

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Disclosure statement: ***Conflict of Interest****:* The authors declare that there are no conflicts of interest associated with this research.. ***Compliance with Ethical Standards****:* This article does not contain any studies involving human or animal subjects.

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