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In-situ reduction synthesis and antibacterial evaluation of Cu/Ni complexes derived from hexadecylamine dithiocarbamate

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ARTICLE INFORMATION

ABSTRACT

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This work reports on the synthesis of Ni and Cu-dithiocarbamate complexes, prepared via in situ reduction of respective metal salts with long-chain hexadecylamine in the presence of NaBH₄. The as-synthesized metal complexes were obtained in satisfactorily high yields and characterized using various techniques. FTIR results indicate that the bonds between metal ions and sulfur atoms in the complexes are bidentate. SEM images revealed the presence of relatively small, irregular, and spherical particles. EDX spectroscopy confirmed the elemental composition of C, S, Cu, and Ni, supporting the successful synthesis of the metal complexes. XRD analysis shows that the dithiocarbamate (DTC) and Ni complexes are crystalline, while the Cu complex is amorphous. Thermogravimetric analysis (TGA) indicates higher stability of the Cu complex compared to the Ni complex and the DTC ligand. Antimicrobial studies demonstrated significant antibacterial activity of the Ni complex against various pathogenic bacteria, surpassing the effectiveness of the DTC ligand and the Cu complex. These findings suggest potential applications for dithiocarbamate complexes as antimicrobial agents. This research enhances the understanding of the synthesis, characterization, and biological activity of dithiocarbamate complexes, offering insights into their potential applications in various fields such as pharmaceuticals (as antimicrobial agents), agriculture (as fungicides or pesticides), and materials science (as precursors for metal sulfide nanoparticles).

Highlights

- In-situ reduction yielded stable Cu/Ni-hexadecylamine dithiocarbamate complexes
- FTIR confirmed bidentate coordination of sulfur to Cu and Ni centers
- The complexes showed crystalline nature
- Cu complex exhibited higher thermal stability than Ni complex and DTC ligand
- Ni complex displayed superior antibacterial activity over Cu complex and DTC ligand

1. Introduction

From a biological perspective, sulfur-bonding ligands are significant and have drawn interest from inorganic chemists recently. Among the most significant groups of organic ligands containing sulfur are dithiocarbamates. They self-assemble with a transition element, exhibit unpredictable coordination behavior, and have applications in the range of chemical and biological domains (Adeyemi and Onwudiwe, 2020a). Dithiocarbamate functions as a chelating, bidentate, or monodentate ligand; the structural arrangement of the coordination complex is dictated

by numerous binding characteristics (Sarker and Hogarth, 2021). It was discovered that the biological characteristics of dithiocarbamates with extra donor groups, including oxygen and nitrogen atoms, changed dramatically (Prihantono et al., 2021; Pratiwi et al., 2024). Report has it that complexes of dithiocarbamate containing various transition metal ions exhibit antibacterial (Sharma et al., 2022), catalytic (Maurya and Bajpai, 2022), and anticancer (Tella et al., 2022) properties. The majority of dithiocarbamate complexes are employed as nanoprecursors in the nanoparticle manufacturing process (Odularu and Ajibade, 2019; Adeyemi and Onwudiwe, 2020b). Medicinal inorganic chemistry is the

most appealing topic of study since it deals with the application of metal-based complexes to illness detection and therapy (Moustakas, 2021).

The exploration of metal-dithiocarbamate (DTC) complexes has unveiled a significant relationship between their biological activity and various factors such as the metal center, stoichiometry, and especially the substituents on the nitrogen atom (Kaul et al., 2021). These substituents play a crucial role in influencing the electronic and steric characteristics of the ligand and complex, as well as their lipophilicity (Xiao and Cao, 2018). Studies have highlighted that modifications in the aliphatic chain length of the dithiocarbamate ligand can enhance the complex's lipophilicity, correlating with better membrane permeability and increased biological activity, particularly against microorganisms (Andrew and Ajibade, 2017; Adeyemi and Onwudiwe, 2020c). Research shows that the biological properties of metal complexes derived from dithiocarbamates with shorter aliphatic chains (like methyl and ethyl) or cyclic substituents have been widely studied (Torres-Gomez et al., 2014; Adeyemi and Onwudiwe, 2020c), while those with longer chains (especially $>C12$) are comparatively less explored. There is a need for comprehensive investigations into the synthesis and antibacterial effects of these longer-chain complexes, which have not been sufficiently explored.

Copper (Cu) and Nickel (Ni) are transition metals of significant interest in medicinal inorganic chemistry. Copper complexes are well-known for their intrinsic redox activity, which enables the generation of reactive oxygen species (ROS) that can induce oxidative stress and damage bacterial cellular components like DNA, proteins, and lipids (Ismael et al., 2021; Ngece et al., 2025). Nickel complexes, while having different electronic properties, have also demonstrated potent biological activities, including antibacterial and anticancer effects, often acting through distinct mechanisms such as enzyme inhibition or direct interaction with biomolecules (Tanzimjahan et al., 2023; Mañozca-Dosman et al., 2025). Recent research has explored various synthetic routes for these complexes, including one-pot methods and the use of different amine precursors to tailor their properties for specific applications, such as antimicrobial agents or precursors for functional nanomaterials (Al-Obaidy et al., 2020; Ajiboye et al., 2022). The ability to control the size and morphology of these metal complexes, particularly at the nanoscale, is increasingly recognized as a critical factor influencing their biological performance, as smaller nanoparticles often present a larger surface area for interaction with microbial cells, enhancing their antimicrobial potency (Menichetti et al., 2023).

Different studies have indeed been reported on the use of Cu and Ni complexes for various medicinal applications, including antibacterial purposes (Ajiboye et al., 2021; Zavaroni et al., 2025). Studies on metal complexes of dithiocarbamates obtained from RNH_2 ($R = C_6H_{13}^+$, $C_{12}H_{25}^-$ and $C_{18}H_{37}^-$) have also been reported (Torres-Gomez et al., 2014). However, the unique contribution of combining the antibacterial potential of Cu and Ni with the specific lipophilicity and membrane-targeting capability offered by a long-chain hexadecylamine ($C_{16}H_{33}NH_2$) has not been thoroughly investigated in a comparative manner using recent methodological standards. The study aimed to synthesize Copper(II) and Nickel(II) complexes derived from hexadecylamine dithiocarbamate using an *in situ* reduction method. The goal is to provide information on their characterization and antibacterial properties, offering insights into their potential applications in various fields such as pharmaceuticals, agriculture, and materials science.

2. Materials and Methods

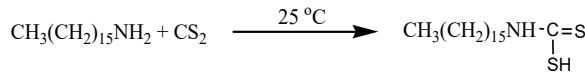
2.1 Materials

Carbon disulphide (CS_2 , $\geq 99.9\%$), hexadecylamine ($CH_3(CH_2)_{15}NH_2$, $\geq 90\%$), Copper sulphate ($CuSO_4$, anhydrous powder $\geq 99.9\%$), nickel sulphate ($NiSO_4 \cdot 7H_2O$, hydrated powder $\geq 99.0\%$), sodium borohydride ($NaBH_4$, powder $\geq 98.0\%$), sodium citrate ($C_6H_5Na_3O_7$, ACS reagent, $\geq 99.0\%$) and deionized water of high purity were purchased from Sigma-Aldrich Pty Ltd. They were used as acquired without any further purification.

2.2 Method

2.2.1 Synthesis of dithiocarbamate (DTC) ligand

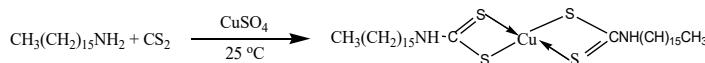
Hexadecylamine dithiocarbamate (DTC) was synthesized following a reported procedure by Zhao et al. (2005), Eqn. 1. Briefly, carbon disulfide (CS_2) was dissolved in 10% ethanol, while a stoichiometric solution of hexadecylamine was prepared separately in a separate ethanol. The ethanolic hexadecylamine solution was added dropwise to the CS_2 solution, and vigorously stirred on a magnetic stirrer for 120 min. The resulting solution was then filtered, and the residue was washed thoroughly with ethanol, dried in desiccators, ground, and stored in a sample bottle, yielding the hexadecylamine dithiocarbamate (DTC) ligand.



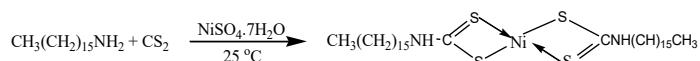
Scheme 1: Representative reaction for synthesis of hexadecylthiocarbamate ligand $C_{16}H_{33}NHCS_2H$.

2.2.2 Synthesis of metal complexes

The Cu/Ni-DTC complex was synthesized following reported procedures outlined by Zhao et al. (2005) and Cao et al. (2013), but with some modifications. Briefly, carbon disulfide (CS_2) was dissolved in 10% ethanol, and a separate solution of hexadecylamine in ethanol was prepared in stoichiometric amounts. The hexadecylamine solution was added dropwise to the CS_2 solution with vigorous stirring for 2 h, resulting in the formation of hexadecylamine dithiocarbamate (DTC). Subsequently, 6 mL of 0.06 M $CuSO_4$ or $NiSO_4 \cdot 7H_2O$ was added, followed by 1.2 mL of 1 mM sodium citrate. After stirring for 5 min, 0.05 M $NaBH_4$ was rapidly added, and the mixture was left to sit at room temperature with continuous stirring for 120 minutes. The resulting mixture was then filtered, and the residue was washed with ethanol, dried in desiccators, ground, and stored in a sample bottle. The solubility of the synthesized ligand and complexes was tested by dissolving them in various solvents.



Scheme 2: Equation of the reaction of the synthesized dithiocarbamate copper complex ($C_{16}H_{33}NHCS_2)_2Cu$



Scheme 3: Equation of the reaction of the synthesized nickel-dithiocarbamate complex ($\text{C}_{16}\text{H}_{33}\text{NHCS}_2\text{Ni}$)

2.3 Characterization of synthesized materials

Transmission electron microscope (TEM) images for the DTC and DTC-metal complexes were obtained using a JEM-ARM200F-G transmission electron microscope at 100 kV accelerating voltage. X-ray powder diffraction patterns of the samples were recorded on a Rigaku MiniFlex X-ray diffractometer system 600. Data were collected in the range from $2\theta = 5^\circ$ to 70° , scanning at 1° min^{-1} with a filter time constant of 2.5 s per step and a slit width of 6.0 mm. The samples were placed on a zero-background silicon wafer slide. Infra-red spectra of the samples were recorded on a Thermo Fisher Scientific FTIR spectrophotometer, using pressed KBr pellets. The surface characteristics of the samples were measured using a JEOL JSM-7600F Scanning Electron Microscope coupled with energy dispersive X-ray spectroscopy (EDS) for surface morphology and elemental composition determination. The X-ray diffraction data were treated using Eva (evaluation curve fitting) software. Baseline correction was performed on each diffraction pattern. The mean crystal size of the synthesized complexes was evaluated by the Debye–Scherrer formula (El Faroudi et al., 2023) (Equation 1).

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (1)$$

where D represents particle diameter, K indicates shape factor (0.9), λ corresponds to the wavelength of the X-ray (0.15419 Å) used, β denotes FWHM (full width at half maximum), and θ represents Bragg angle.

2.3.1 Antimicrobial Activity Determination

Antimicrobial activity was evaluated against a range of bacteria, including *Salmonella typhi*, *Escherichia coli*, *Enterobacter aerogenes*, *Shigella dysenteriae*, *Pseudomonas aeruginosa*, *Streptococcus pneumonia*, *Staphylococcus aureus*, and *Klebsiella pneumonia*. Clinical isolates were sourced from the Medical Laboratory at Ekiti State University Teaching Hospital, Ado-Ekiti. The antibacterial properties of

DTC and its metal complexes were investigated using the Agar well diffusion method as described by Daoud et al. (2015). Wells, each 6 mm in diameter, were created in the agar plates using a sterile cork. DMSO served as a negative control, and known concentrations of DTC and its metal complexes were added to the wells with a micropipette. The plates were allowed to pre-diffuse for 2 h at room temperature and then incubated for 24 h at 37°C . The antibacterial activity was determined by measuring the zones of inhibition in millimeters around the wells.

3. Results and Discussion

3.1 Analytical assessment of dithiocarbamate and its metal complexes

The synthesis of hexadecylamine dithiocarbamate (DTC) ligand was carried out via nucleophilic addition of hexadecylamine to carbon disulfide in an ethanolic medium as outlined in Scheme 1. Subsequently, the synthesis of both copper(II) and nickel(II) complexes, followed by reduction-assisted methods via one-pot synthesis. The chelation reaction involves coordination of two dithiocarbamate ligands to the central metal ion, forming a bidentate metal–ligand complex, as shown in Schemes 2 and 3, respectively. The colour of the DTC ligand, copper(II) and nickel(II) complexes are white, yellow, and gold, respectively (Table 1).

Table 1 presents the proposed structures, colors, and yields of the ligand and its complexes. The analytical data aligns well with this formulation. Each compound exhibits a metal–ligand stoichiometry of 1:2, represented as $[\text{M}(\text{DTC})_2]$, where $\text{M} = \text{Cu}(\text{II})$ or $\text{Ni}(\text{II})$, and DTC = Dithiocarbamate. The solubility of the ligand and metal complexes was assessed in various solvents, including water, DMSO, ethanol, toluene, methanol, and acetic acid. The results of these solubility tests for DTC and its metal complexes in different solvents are summarized in Table 2. The ligand and metal complexes were partially soluble in all the tested solvents but fully soluble in an aqueous DMSO solution (30% DMSO). Dithiocarbamate ligands of ammonium, sodium, or potassium salts have been reported to be water soluble, while their corresponding coordination compounds are insoluble in water (Odularu and Ajibade, 2019). The trend experienced in the ligand solubility might be due to the lack of these sodium/potassium salts in their synthesis. Consequently, a 30% DMSO solution was used to dissolve the ligand and metal complexes for further studies.

Table 1: Analytical data for the synthesized dithiocarbamate and its complexes.

Ligand/Complex	Colour	Yield (%)	Proposed structure
DTC	White	86	
$\text{Cu}(\text{DTC})_2$	Yellow	77	
$\text{Ni}(\text{DTC})_2$	Gold	71	

Table 2: The Solubility of complexes and ligand in different solvents.

Solvents	Water	Methanol	Ethanol	Toluene	DMSO	Acetic acid	30% DMSO
DTC	Partially soluble	Soluble					
CuL₂	Partially soluble	Soluble					
NiL₂	Partially soluble	Soluble					

3.2 FTIR analysis of DTC ligand and DTC-metal complexes

The metal complexes and DTC were found to have identical spectra, indicating that hexadecylamine is the primary component of the backbone layer of the structure of dithiocarbamate. The infrared spectra of the complexes exhibit the characteristic features of the dithiocarbamate structure. Vibrational bands at 2912, 2848, 1361, and 599 cm⁻¹ demonstrate the presence of an alkyl chain, while C-N and C-S vibrations are indicated by bands at 1489 and 928 cm⁻¹, respectively. For the compound Cu(DTC)₂, there is a single strong absorption peak at 3268 cm⁻¹, attributed to N-H stretching. The Cu complex shows two bands: a sharp band at 3268 cm⁻¹ and a slightly broader band at 3167 cm⁻¹. The shape of the latter band suggests interaction with a water molecule or intramolecular hydrogen bonding.

In the FTIR spectrum of the Ni complex, the band at 1471 cm⁻¹ is associated with the C-N stretching of the N-CS₂ group (Amir et al., 2016). Additionally, the FTIR spectrum shows two bands at 996 cm⁻¹ and 953 cm⁻¹, corresponding to the asymmetric and symmetric stretching of CS₂, respectively (Damian and Anthony, 2016). The band at 2848 cm⁻¹ is attributed to C-H stretching (Muthu et al., 2012). The vibrational bands at 1271 cm⁻¹ and 715 cm⁻¹ are assigned to C-N and C-S stretching, respectively (Al-Obaidy et al., 2020).

Vibrational bands in the range of 3219–3180 cm⁻¹ in the DTC complexes are associated with N-H stretching, indicating the absence of nitrogen-metal interactions, suggesting that the bonds in the complexes are between metal ions and sulfur. The stability of these modes suggests that the organic moieties play a role in the formation of the metal complexes. Bands at 996 cm⁻¹ in Cu-DTC and 997 cm⁻¹ in Ni-DTC indicate that the dithiocarbamate moiety in the complexes is bidentate, consistent with findings by Ejelonu (2019) and Ajibade et al. (2013).

3.3 SEM analysis of DTC and its metal complexes

The surface morphology of DTC and its metal complexes was examined using scanning electron microscopy (SEM). The SEM image in Figure 2 reveals that the particles are round, irregular, and relatively small in size. The image clearly shows that the particles tend to strongly agglomerate naturally, with some larger particles forming due to the aggregation or overlap of smaller ones. A similar observation has been reported by Suresh and Jiban (2015).

3.4 EDX spectra of DTC and its metal complexes

EDX spectroscopy was used to determine the elemental compositions of DTC and its metal complexes (Cu-DTC and Ni-DTC). The EDX spectrum of DTC, shown in Figure 3a, confirmed that carbon (C) and sulfur (S) are the primary components of DTC, with peaks at 0.2 and 2.2 keV due to carbon and sulfur, respectively. In addition to C and S peaks, the EDX spectra of Cu-DTC (Figure 3b) and Ni-DTC (Figure 3c) display peaks confirming the presence of copper (Cu) and nickel (Ni), respectively, indicating that the metal complexes were successfully pre-

pared. The percentage compositions of C, S, Cu, and Ni are shown in the insets for each EDX spectrum.

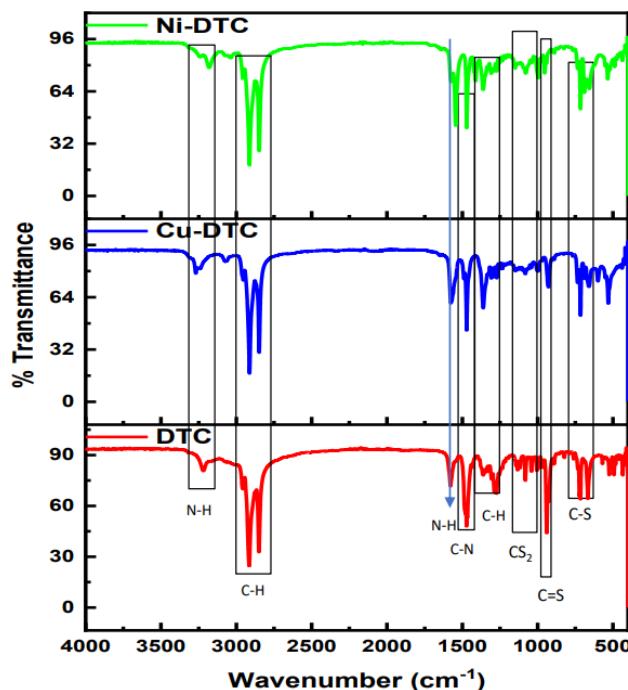


Figure 1: FTIR spectra of DTC ligand and complexes.

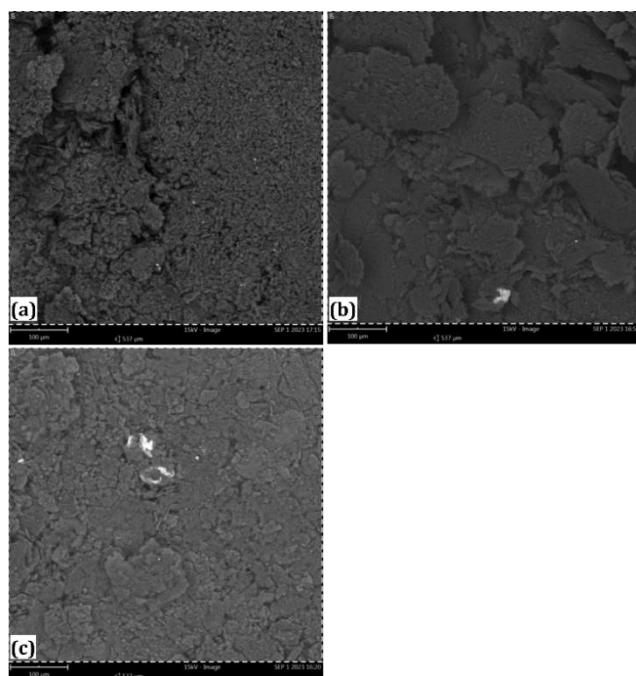


Figure 2: SEM images of (a) DTC, (b) Cu-DTC, and (c) Ni-DTC at 100 μm.

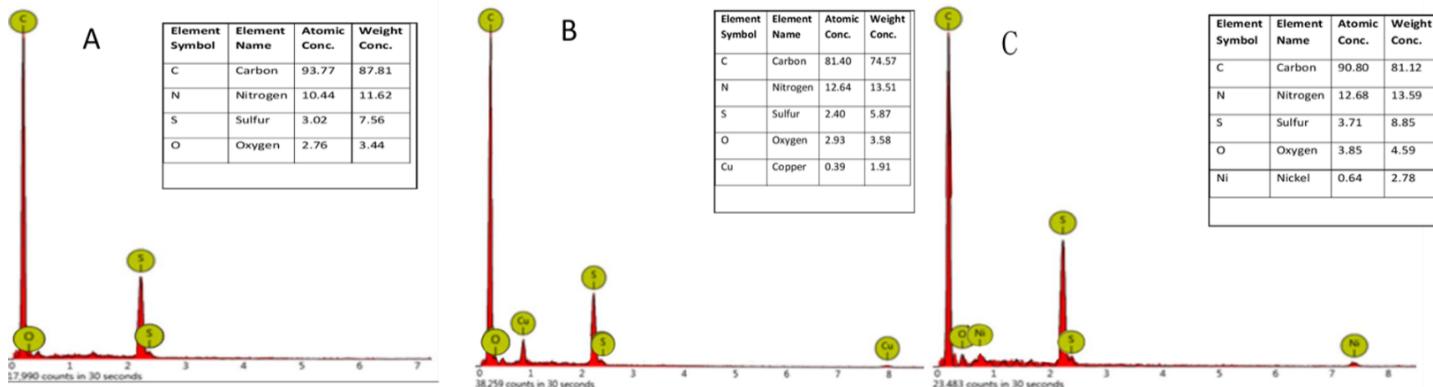


Figure 3: EDX spectra of (a) DTC, (b) Cu-DTC, and (c) Ni-DTC.

3.5 TGA of DTC and its metal complexes

The thermogravimetric profile of the DTC ligand shows three stages: moisture loss above 100 °C, breakdown of ~40.2% of the organic moiety above 300 °C, and loss of amine groups around 400 °C, leaving a final residue of 16.32% sulfur (Figure 4). For the metal-DTC complexes, two stages were observed: moisture loss above 100 °C and final ligand breakdown above 320 °C, with no further weight loss detected above 500 °C. The final residues were 23.85% for Cu-DTC and 19.27% for Ni-DTC, indicating higher stability of the Cu complex compared to the Ni complex and the DTC ligand. This is in accordance with existing literature stating that thermal breakdown is due to decomposition due to dehydration, breakdown of the dithiocarbamate moiety, and decomposition of the organic part of the ligand (Raja et al., 2012).

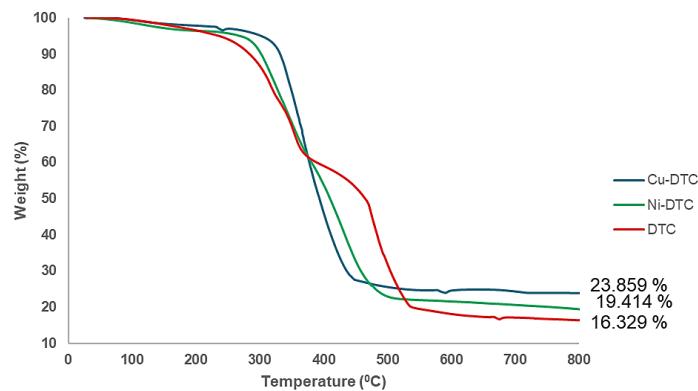


Figure 4: Thermogravimetric analysis (TGA) graph of DTC and its metal complexes.

3.6 XRD analysis of DTC and its metal complexes

Powder X-ray diffraction (XRD) patterns of DTC show peaks at $2\theta = 21.92^\circ$, 34.22° , 37.99° , 44.21° , and 64.49° , corresponding to the (111), (002), (111), (200), and (220) reflection planes, respectively, Figure 5. The XRD pattern of the Cu-DTC complex displays peaks at $2\theta = 19.01^\circ$, 34.18° , 38.15° , 44.24° , and 64.40° , corresponding to the (111), (002), (111), (200), and (220) reflection planes. Using the Debye Scherrer equation $d_{XRD} = 0.9\lambda/\beta\cos\theta$, the average grain sizes were found to be 90.82 nm for DTC, 49.38 nm for Cu-DTC, and 46.23 nm for Ni-DTC, indicating nanosized metal comple-

xes. The powder XRD patterns demonstrate the crystalline nature of DTC, Cu-DTC, and the Ni-DTC complex as shown by their sharp crystalline peaks (Figure 5). Similar findings have been reported by Kumar and Nath (2018).

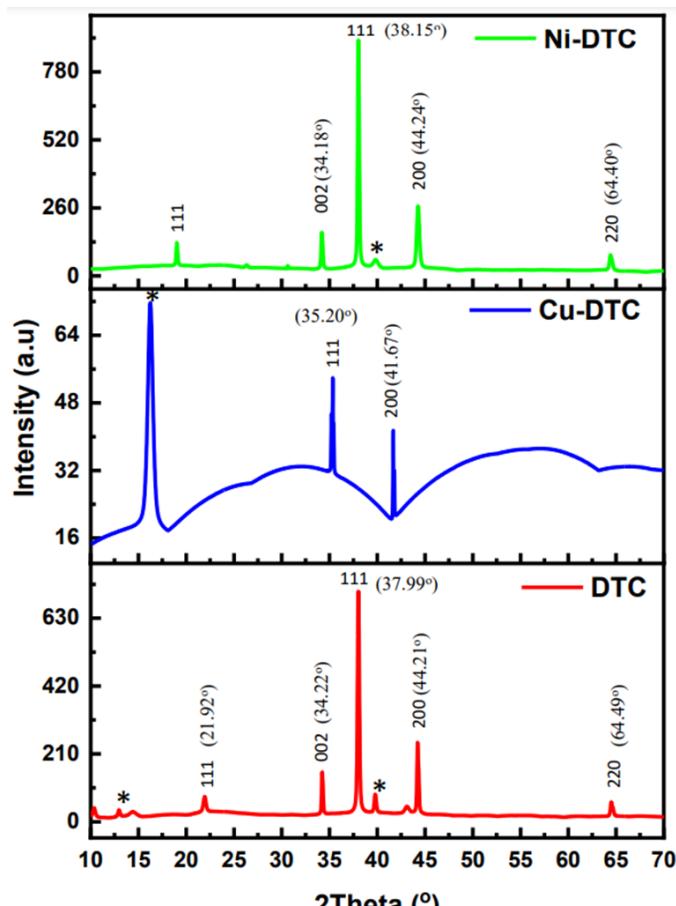


Figure 5: XRD pattern of DTC and its metal complexes.

3.7 Antimicrobial studies

The research investigates the antibacterial qualities of the dithiocarbamate (DTC) and its copper (Cu) and nickel (Ni) complexes synthesized against various pathogenic bacteria. The experiment was done using diffusion via agar well and measured the diameter of zones of

inhibition in millimeters for the concentrations (mg/mL) of each substance against a range of bacteria, including *Salmonella typhi*, *Escherichia coli*, *Enterobacter aerogenes*, *Shigella dysenteriae*, *Pseudomonas aeruginosa*, *Streptococcus pneumonia*, *Staphylococcus aureus*, and *Klebsiella pneumonia*. The results, presented in Table 3, provide valuable insights into the potential effectiveness of these substances as antibacterial agents. The table shows that the nickel complex demonstrates significant antibacterial activity against several tested bacteria, copper shows limited activity, while DTC shows no effectiveness at the tested concentrations. The complexation of the organic ligands to the metal ions significantly increased their activities, as seen by the high antibacterial activities of all the metal complexes surpassing that of the ligand (Abdolmaleki et al., 2023). The table indicates that nickel exhibited a zone of inhibition measuring 46 mm in diameter against *Salmonella typhi*, 42 mm against *Escherichia coli*, 30 mm against *Enterobacter aerogenes* and *Pseudomonas aeruginosa*, 22 mm against *Klebsiella pneumonia*, 30 mm against and 11 mm against *Streptococcus pneumonia*. The release of Cu^{2+} and Ni^{2+} ions from their firmly bonded electron donor groups containing sulfur is responsible for the antibacterial efficiency of the metal complexes, since it leads to the eradication of bacterial species. This is consistent with results from another study published in the literature (Godoy-Gallardo et al., 2021). The current investigation indicates that the nickel dithiocarbamate complex has higher antibacterial efficacy than the copper dithiocarbamate complex. This could be because nickel particles are smaller than copper particles (Section 3.6). Smaller particles have a greater ability to interact with bacterial membranes due to their larger surface area, which enhances their antibacterial activity (Yadav et al., 2016). Smaller nanoparticle size directly correlates with a larger surface area-to-volume ratio (More et al., 2023). This enhanced surface area allows for more extensive and effective contact with the bacterial cell membrane. This increased surface area facilitates more efficient release of higher concentrations of Ni ions, which disrupt and kill the bacteria's cellular processes with greater effectiveness. Smaller NPs also penetrate bacterial cell walls and membranes more easily, which leads to effective internalization and disruption of cellular functions (Duman et al., 2024). This shows that the nickel dithiocarbamate complex appeared to be the most promising antimicrobial agent. There are several uses for dithiocarbamate ligands and complexes. Nevertheless, the complexes seem more effective than the ligands when both are found to be significant in comparable applications. According to Ejelonu et al. (2018), dithiocarbamate complexes have greater antimicrobial activity compared to their ligand sources.

The antibacterial qualities of complexes are attributed to the metal ions released during the oxidative dissolution process. Cu^{2+} and Ni^{2+} ions liberated from the complexes pierced the peptidoglycan, DNA, and protein components of the bacterial cells, stopping the bacterium from replicating further. The bacteria's cell wall ruptures due to the metal ions entering through it, denaturing the protein, and causing the bacteria to die (Ishida, 2018). Positively charged ions have been shown to readily engage in interactions with electron-rich proteins found in the cell wall of bacteria that contain D-amino acids. According to several reports, the mechanism underlying the bacteria's inability to develop is the connection with the positive charge on the nanoparticles and their cell membrane, which has a negative charge (Agnihotri et al., 2014; Balavandy et al., 2014). Elsaesser and Howard (2012) stated that electrostatic contact, adsorption, and nanoparticle penetration within the cell wall of the bacterium are the mechanisms of growth inhibition.

Table 3: Antibacterial effect of DTC and its metal complexes.

Test organisms	Diameter of zones of inhibition (mm)		
	Concentrations (mg/mL)		
	DTC	Cu-DTC	Ni-DTC
<i>Salmonella typhi</i>	-	14	46
<i>Escherichia coli</i>	-	12	42
<i>Enterobacter aerogenes</i>	-	16	30
<i>Shigella dysenteriae</i>	-	14	-
<i>Klebsiella pneumonia</i>	-	-	22
<i>Pseudomonas aeruginosa</i>	-	12	30
<i>Staphylococcus aureus</i>	-	4	-
<i>Streptococcus pneumonia</i>	-	7	11

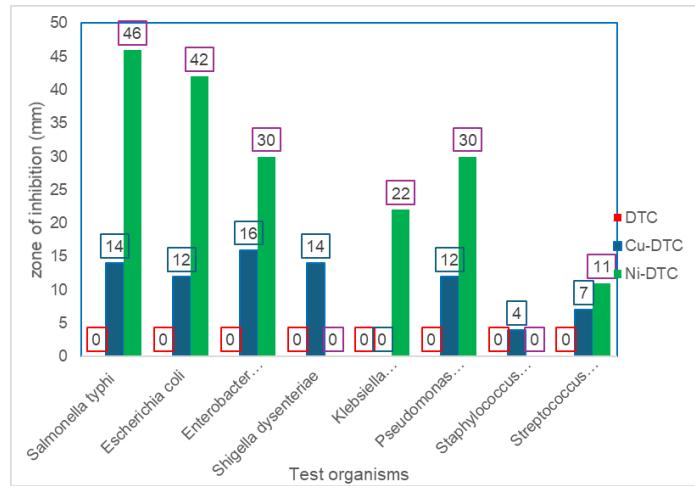


Figure 6: Antimicrobial activities of DTC and its complexes against selected bacterial strains.

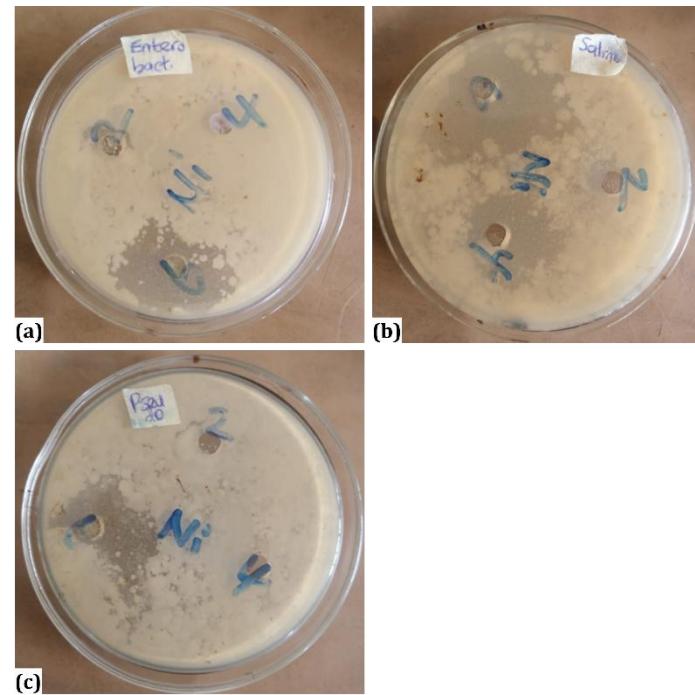


Figure 7: Showing zone of inhibition against pathogenic bacteria (a) *Enterobacter aerogenes*, (b) *Salmonella typhi*, and (c) *Pseudomonas aeruginosa*.

5. Conclusion

This work synthesized and characterized metal complexes of the dithiocarbamate ligand. The band corresponding to the N-H stretching frequency in DTC complexes only signifies the absence of nitrogen to metal connections, meaning that the bonds in these metal complexes are between metal ions and sulfur. It was observed that the bands at 996 and 997 cm⁻¹ in Cu-DTC and Ni-DTC are the complex's dithiocarbamate moiety's distinctive bidentate nature. SEM was used to visualize the morphology of the DTC and its metal complexes and revealed the presence of relatively small, irregular, and spherical particles. EDX spectroscopy confirmed the elemental composition of C, S, Cu, and Ni, supporting successful metal complex synthesis. XRD shows that DTC and the Ni complex are crystalline, while the Cu complex is amorphous in nature. Thermogravimetric analysis showed stepwise decomposition of the ligand and complexes. The antimicrobial activities show that the nickel complex demonstrates significant antibacterial activity against several tested bacteria, copper shows limited activity, while DTC has no effectiveness against the tested organisms. The complexes seem more effective than the ligands when both are found to be significant in comparable applications. This research enhances the understanding of the synthesis, characterization, and biological activity of dithiocarbamate complexes, offering insights into their potential applications in various fields such as pharmaceuticals (as antimicrobial agents), agriculture (as fungicides or pesticides), and materials science (as precursors for metal sulfide nanoparticles).

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Competing Interest

The author affirms that there are no identifiable conflicts of interest, either financial or personal, that could be perceived as influencing the work presented in this paper.

Data Availability Statement

All data generated or analyzed during this study are included in this published article.

Ethics Approval

Not applicable, as no animals were used in this study.

References

Abdolmaleki, S., Khaksar, S., Aliabadi, A., Panjehpour, A., Motieiyan, E., Marabello, D., Faraji, M.H. & Beihaghi, M. (2023). Cytotoxicity and mechanism of action of metal complexes: An overview. *Toxicology*, 492, 153516. <https://doi.org/10.1016/j.tox.2023.153516>.

Adeyemi, J.O. & Onwudiwe, D.C. (2020b). PbS Nanoparticles Prepared Using 1, 10-Phenanthroline Adduct of Lead(II) Bis(N-alkyl-N-phenyl dithiocarbamate) as Single Source Precursors. *Molecules*, 25(9), 2097. <https://doi.org/10.3390/molecules25092097>.

Adeyemi, J.O., & Onwudiwe, D.C. (2020a). The mechanisms of action involving dithiocarbamate complexes in biological systems. *Inorganica Chimica Acta*, 511, 119809. <https://doi.org/10.1016/j.ica.2020.119809>.

Adeyemi, J.O. & Onwudiwe D.C. (2020c). Chemistry and Some Biological Potential of Bismuth and Antimony Dithiocarbamate Complexes. *Molecules*; 25(2), 305. <https://doi.org/10.3390/molecules25020305>.

Agnihotri, S., Mukherji, S. & Mukherji, S. (2014). Size-controlled silver nanoparticles synthesized over the range 5–100 nm using the same protocol and their antibacterial efficacy. *RSC Adv.*, 4(8), 3974–3983. <https://doi.org/10.1039/C3RA44507K>.

Ajibade, P.A., Idemudia, O.G. & Okoh, A.I. (2013). Synthesis, characterization and antibacterial studies of metal complexes of sulfadiazine with N-alkyl-N-phenyldithiocarbamate, *Bull. Chem. Soc. Ethiop.*, 27(1), 77-84. <https://doi.org/10.4314/bcse.v27i1.8>.

Ajiboye, T.O., Ajiboye, T.T., Marzouki, R. & Onwudiwe D.C. (2022). The Versatility in the Applications of Dithiocarbamates. *Int J Mol Sci.*; 23(3), 1317. <https://doi.org/10.3390/ijms23031317>.

Ajiboye, T.O., Oluwarinde, B.O., Montso, P.K., Ateba, C.N. & Onwudiwe, D.C. (2021). Antimicrobial activities of Cu(II), In(III), and Sb(III) complexes of N-methyl-N-phenyl dithiocarbamate complexes, *Results in Chemistry*; 3, 100241. <https://doi.org/10.1016/j.rechem.2021.100241>.

Al-Obaidy, G.S., Ibrahim, K.R. & Mesher, M.F. (2020). Metal Complexes Derived from Dithiocarbamate Ligand: Formation, Spectral Characterization and Biological activity. *Systematic Reviews in Pharmacy*, 11(6), 360-368. <https://doi.org/10.31838/srp.2020.6.57>.

Amir, M.K., Zia-ur-Rehman, Z.R., Hayat, F., Khan, S.Z., Hogarth, G., Kondratyuk, T. & Tahir, M. N. (2016). “Monofunctional platinum(II) dithiocarbamate complexes: synthesis, characterization and anticancer activity”. *RSC Advances*, 6(112), 110517-110524. <https://doi.org/10.1039/C6RA19469A>.

Andrew, F.P. & Ajibade, P.A. (2017). Metal complexes of alkyl-aryl dithiocarbamates: Structural studies, anticancer potentials and applications as precursors for semiconductor nanocrystals. *Journal of Molecular Structure*; 3, 1155. <https://doi.org/10.1016/j.molstruc.2017.10.106>.

Balavandy, S., Shamel, K., Biak, D. R. B. A. & Abidin, Z. (2014). Stirring time effect of silver nanoparticles prepared in glutathione mediated by green method, *Chemistry Central Journal*, 8(1), 11. <https://doi.org/10.1186/1752-153X-8-11>.

Cao H., Minghong, W., Zhaohui C. & Yuming H. (2013). Dithiocarbamate-capped silver nanoparticles as a resonance light scattering probe for simultaneous detection of lead(II) ions and cysteine. *Analyst*, 138, 2420-2426. <https://doi.org/10.1039/C3AN36868H>.

Damian C.O. & Anthony C.E. (2016). Synthesis, characterization, thermal, antimicrobial and antioxidant studies of some transition metal dithiocarbamates: *Springer Science + Business Media Dordrecht*, 43(3), 1465-1485. <https://doi.org/10.1007/s11164-016-2709-2>.

Daoud, A., Malika, D., Bakari, S., Hfaiedh, N., Mnafgui, K. & Kadri, A. (2015). Assessment of polyphenol composition, antioxidant and antimicrobial properties of various extracts of date palm pollen (DPP) from two tunisian cultivars. *Arab. J. Chem.* <https://doi.org/10.1016/j.arabjc.2015.07.014>.

Duman, H.; Eker, F.; Akdaç, E.; Witkowska, A. M.; Bechelany, M.; & Karav, S. (2024). Silver Nanoparticles: A Comprehensive Review of Synthesis Methods and Chemical and Physical Properties. *Nanomaterials*, 14(18), 1527. <https://doi.org/10.3390/nano14181527>.

Ejelonu B.C., Olagboye S.A., Oyeneyin O.E., Ebiesuwa, O.A. & Bada, O.E. (2018). Synthesis, characterization and antimicrobial activities of sulfadiazine Schiff base and phenyl dithiocarbamate mixed ligand metal complexes. *Open J. Appl. Sci.*; 8, 346-354. <https://doi.org/10.4236/ojapps.2018.88026>.

Ejelonu, B. (2019). Synthesis, Characterization and In-Vitro Antimicrobial Studies of M²⁺ Complexes of p-Chlorophenyl-, p-Bromophenyl-Dithiocarbamates. *Open Journal of Applied Sciences*, 9, 587-594. <https://doi.org/10.4236/ojapps.2019.97046>.

El Faroudi, L., Saadi, L., Barakat, A., Mansori, M., Abdelouahdi, K. & Solhy, A. (2023). Facile and Sustainable Synthesis of ZnO Nanoparticles: Effect of Gelling Agents on ZnO Shapes and Their Photocatalytic Performance. *ACS omega*, 8(28), 24952-24963. <https://doi.org/10.1021/acsomega.3c01491>.

Elsaesser, A. & Howard, C. V. (2012). Toxicology of nanoparticles. *Advanced Drug Delivery Reviews*, 64(2): 129–137. <https://doi.org/10.1016/j.addr.2011.09.001>.

Godoy-Gallardo, M., Eckhard, U., Delgado, L.M., de Roo Puente Y.J.D., Hoyos-Nogués, M., Gil, F.J. & Perez R.A. (2021). Antibacterial approaches in tissue engineering using metal ions and nanoparticles: From mechanisms to applications. *Bioact Mater*, 6(12), 4470-4490. <https://doi.org/10.1016/j.bioactmat.2021.04.033>.

Ishida, T. (2018). Antibacterial mechanism of Ag^+ ions for bacteriolytic of bacterial cell walls via peptidoglycan autolysins, and DNA damages. *MOJ Toxicology*, 4(5), 345–350. <https://doi.org/10.15406/mojt.2018.04.00125>.

Ismael, M., Abdel-Mawgoud, A.M.M., Rabia, M.K. & Abdou A. (2021). Synthesis, characterization, molecular modeling and preliminary biochemical evaluation of new copper(II) mixed-ligand complexes. *J. Mol. Struct.*, 1227, 129695. <https://doi.org/10.1016/j.molstruc.2020.129695>.

Kaul, L., Süss, R., Zannettino, A., & Richter, K. (2021). The revival of dithiocarbamates: from pesticides to innovative medical treatments. *iScience*, 24(2), 102092. <https://doi.org/10.1016/j.isci.2021.102092>.

Kumar, L.V. & Nath, G.R. (2018). Synthesis and Characterization Studies of Cobalt(II), Nickel(II), Copper(II) and Zinc(II) Complexes of Carboxymethyl-N-Methyl-N-Phenyl Dithiocarbamate. *Orient J Chem*; 34(6). <http://doi.org/10.13005/ojc/340649>.

Mañozca-Dosman, I.V., Aragón-Muriel, A. & Polo-Cerón, D. (2025). Antibacterial Activity of Metal Complexes of Cu(II) and Ni(II) with the Ligand 2-(Phenylsubstituted) Benzimidazole. *Scientia Pharmaceutica*, 93(2), 22. <https://doi.org/10.3390/scipharm93020022>.

Maurya, C. & Bajpai, S. (2022). Biological Applications of Metal Complexes of Dithiocarbamates. *Journal of Applied Science and Education (JASE)* 2(1), 1–16. <https://doi.org/10.5406/JASE/002.01.002>.

Menichetti, A., Mavridi-Prinetti, A., Mordini, D. & Montalti, M. (2023). Effect of Size, Shape and Surface Functionalization on the Antibacterial Activity of Silver Nanoparticles. *J Funct Biomater*; 14(5):244. <https://doi.org/10.3390/jfb14050244>.

More, P.R., Pandit, S., Filippis, A., Franci, G., Mijakovic, I., & Galdiero, M. (2023). Silver Nanoparticles: Bactericidal and Mechanistic Approach against Drug Resistant Pathogens. *Microorganisms*; 11(2), 369. <https://doi.org/10.3390/microorganisms11020369>.

Moustakas, M. (2021). The Role of Metal Ions in Biology, Biochemistry and Medicine. *Materials (Basel)*. 14(3), 549. <https://doi.org/10.3390/ma14030549>.

Muthu, S., Uma Maheswari, J. & Sundius, T. (2012). “Molecular structural, non-linear optical, second order perturbation and Fukui studies of Indole-3-Aldehyde using density functional calculations”. *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, 106, 299–309. <https://doi.org/10.1016/j.saa.2012.12.080>

Ngece, K., Khwaza, V., Paca, A.M. & Aderibigbe, B.A. (2025). The Antimicrobial Efficacy of Copper Complexes: A Review. *Antibiotics (Basel)*; 14(5), 516. <https://doi.org/10.3390/antibiotics14050516>.

Odularu, A.T. & Ajibade, P.A. (2019). Dithiocarbamates: Challenges, Control, and Approaches to Excellent Yield, Characterization, and Their Biological Applications. *Bioinorg Chem Appl*; 6, 8260496. <https://doi.org/10.1155/2019/8260496>.

Pratiwi, E., Raya, I., Natsir, H., Irfandi, R., Taba, P., Arfah, R., Rasyid, H., Hala, Y., Kasim, S., Khaerunnisa, A.B., Ilham, B., Mazaya, M., Tanzil, Y. & Luthfiana, D. (2024). Investigations of Ni(II)Cysteine-Tyrosine Dithiocarbamate Complex: Synthesis, Characterization, Molecular Docking, Molecular Dynamic, and Anticancer Activity on MCF-7 Breast Cancer Cell Line. *Asian Pac J Cancer Prev*; 25(4), 1301–1313. <https://doi.org/10.31557/APJCP.2024.25.4.1301>.

Prihantono, P., Irfandi, R. & Raya, I. (2021). The comparison of Zn(II) arginine dithiocarbamate cytotoxicity in T47D breast cancer and fibroblast cells. *Breast Dis.*; 40(S1), S55–S61. <https://doi.org/10.3233/BD-219008>.

Raja, D.S., Bhuvanesh, N.S.P. & Natarajan, K. (2012). A novel water soluble ligand bridged cobalt(II) coordination polymer of 2-oxo-1,2-dihydroquinoline-3-carbaldehyde (isonicotinic) hydrazone: evaluation of the DNA binding, protein interaction, radical scavenging and anticancer activity. *Dalton Transactions*, 41, 4365–4377. <https://doi.org/10.1039/C2DT12274J>.

Sarker, J.C. & Hogarth, G. (2021). Dithiocarbamate Complexes as Single Source Precursors to Nanoscale Binary, Ternary and Quaternary Metal Sulfides. *Chem Rev.*; 121(10), 6057–6123. <https://doi.org/10.1021/acs.chemrev.0c01183>.

Sharma, B., Shukla, S., Rattan, R., Fatima, M., Goel, M., Bhat, M., Dutta, S., Ranjan, R.K. & Sharma, M. (2022). Antimicrobial Agents Based on Metal Complexes: Present Situation and Future Prospects. *Int J Biomater*; 6819080. <https://doi.org/10.1155/2022/6819080>.

Suresh S. & Jibin P. 2015. Investigations on structural, optical, morphological and electrical properties of nickel oxide nanoparticles. *International Journal of Nanoparticles*, 8, 289–301. <https://doi.org/10.1504/IJNP.2015.073731>.

Tanzimjahan, A.S., Jerry, O.A., Moganavelli, S., Sunday, N.O. & Damian C.O. (2023). Synthesis, characterization, and biological evaluation of some metal complexes containing N and S donor atoms. *Results in Chemistry*, 6, 101052. <https://doi.org/10.1016/j.rechem.2023.101052>.

Tella, T., Pohl, C.H. & Ayangbenro, A. (2022). A review of the therapeutic properties of dithiocarbamates. *F1000Research*; 11, 243. <https://doi.org/10.12688/f1000research.109553.1>.

Torres-Gomez, N., Vilchis-Nestor, A.R., Gomez-Espinosa, R.M. & Garcia-Orozco, I. (2014). Synthesis and Characterization of Copper(II) Complexes with Long Chain Dithiocarbamates. *Advanced Materials Research*, 976, 164–168. <https://doi.org/10.4028/www.scientific.net/amr.976.164>.

Xiao, Y. & Cao, C. (2018). Influence of substituent effects on the coordination ability of salicylaldehyde Schiff bases. *Journal of Coordination Chemistry*, 71(23): 3836–3846. <https://doi.org/10.1080/00958972.2018.1540780>.

Yadav, L.S.R., Archana, B., Lingaraju, K., Kavitha, C., Suresh, D., Nagabhushana, H. & Nagaraju, G. (2016). Electrochemical sensing, photocatalytic and biological activities of ZnO nanoparticles: Synthesis via green chemistry route. *International Journal of Nanoscience*, 15(4), 1650013. <https://doi.org/10.1142/S0219581X16500137>.

Zavaroni, A., Rigamonti, L., Bisceglie, F., Carcelli, M., Pelosi, G., Gentilomi, G. A., Rogolino, D. & Bonvicini, F. (2025). Antimicrobial Activity of Copper(II), Nickel(II) and Zinc(II) Complexes with Semicarbazone and Thiosemicarbazone Ligands Derived from Substituted Salicylaldehydes. *Molecules*, 30(11), 2329. <https://doi.org/10.3390/molecules30112329>.

Zhao, Y.; Perez-Segarra, W.; Shi, Q. C. & Wei, A. (2005). Dithiocarbamate assembly on gold. *J. Am. Chem. Soc.*, 127: 7328–7329. <https://doi.org/10.1021/ja050432f>.

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