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Research Article

Characterization of a certain Schiff base Cu(II) Complexes using Spectroscopic techniques

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Abstract — The current work examines the characteristics of the Copper complex, which are significant because of their use in biomedicine. Salicylaldehyde Benzoyl Hydrazine (SBH) **(L)**, $Cu(L^1)_2SO_4.5H_2O$ **(1)**, $Cu(L^2)_2SO_4.5H_2O$ **(2)** and $Cu(L^3)_2SO_4.5H_2O$ **(3)** are the ligands and complexes, where L^1=5-Nitro SBH, L^2=5-Methyl SBH and L^3=5-Bromo SBH. The Cu(II) complexes of Schiff base have been examined using powder X-ray diffraction and various spectroscopic instruments, including FTIR, UV-Vis, and SEM. Following the discovery of the sample's crystalline nature using X-ray diffraction studies, Miller indices, the lattice parameter, and the average crystallite size were computed. The findings verifying Nano powder of copper smaller than 40 nm in size. Based on FT-IR and UV-Visible spectroscopy, salicylaldehyde and benzoyl hydrazine are used in the production of the Cu (II) complex. The metal complexes' morphology is provided via SEM examination.

Keywords — FT-IR, Particle size, SEM, Salicylaldehyde benzoyl hydrazine, UV-Visible XRD.

1. Introduction

Schiff bases' capacity to create stable complexes with various transition metals makes them an important family of chelating agents in coordination chemistry. Schiff bases with the -RC=N- group have become more significant due to the physiological and pharmacological effects they have been linked to. They belong to an intriguing class of chelating compounds that can form both mononuclear and polynuclear metal complexes by coordinating with one or more metal ions [1,2]. These ligands have the ability to coordinate metals because of another group and imine nitrogen, which is typically dependent on the aldehyde. Because of their wide range of applications, a number of Schiff Base ligands and their metal complexes have been studied in the past. Additionally, these ligands and their complexes are essential to our comprehension of the coordination chemistry of transition metal ions [3-6]. Recently, a variety of complexes of metals containing Schiff bases with N, S, and O donors were synthesized along with investigated. This could be as a result of the antiviral and anticarcinogenic qualities demonstrated by these donor ligands and the complexes formed from them [7,8]. There are potential medical applications for Cu(II) complexes in the treatment of several illnesses, namely cancer [9-11]. Salicylaldehyde benzoyl hydrazine's metallic complexes have attracted much of interest recently because of their industrial applications, physicochemical properties, and biological activity. Being able to react with a wide range of transition metal cations and create a huge number of complexes, SBH is another essential Schiff base [12]. In this study, we describe the synthesis and characterization of complexes that include Schiff base ligand. Powder X-ray diffraction, UV-Vis, infrared, and scanning electron microscopy have all been used to analyze L and its Cu(II) complexes.

2. Experimental method

2.1 Starting Material

Transition metal salt $MX_2.nH_2O$, where M = Cu^{2+}, where X = Cl and SO_4.

2.2 Synthesis of the Substituted Salicylaldehyde Benzoyl Hydrazines Ligand

Salicylaldehyde-Substituted Benzoyl Hydrazines were made as follows: A combination of ethanol and water (1:3, v/v, 40 ml) was used to dissolve the required acid hydrazide (20 mmol). After combining the hydrazide solution with a salicylaldehyde solution (2.44 g, 20 mmol) in ethanol (20 ml), the mixture was put in a steam bath for 20 minutes. As the hydrazine cooled to room temperature, crystals formed. After being filtered out, these were left to dry outside [13].

2.3 Synthesis of the Copper Complex

The following basic procedure was used to obtain all of the metal(H) chelates. A mixture of substituted salicylaldehyde

benzoyl hydrazines (1 mmol) into 95% ethanol (20 ml) and a solution containing the salt of metal (1 mmol) into a small amount of ethanol was mixed, and the resultant dark-colored mixture was stirred constantly for two to three hours. Once the steamed mixture had cooled to room temperature, it was left overnight to stand. The solution was separated into dark-colored crystalline solids, which were then passed through filters, cleaned with ethanol, and allowed to dry naturally [14, 15].

2.4 Characterization of the Complex

The morphology of the Ligand and their Copper (II) complexes was shown by scanning electron (SEM) micrographs (Fig. 2). The SEM were acquired at an accelerating voltage of 20 kV, with the magnification set at ×1500. The Ligand and Copper complexes' compressed FTIR spectra were captured using an FTIR Bruker Alpha-II spectrometer in the attenuated mid-infrared range (4000 – 100 cm^{-1}). One method used to characterize the copper complex was UV-visible spectroscopy. The LABTRONICS LT-2203 UV/VIS Spectrophotometer was used to measure UV-Visible Spectroscopy in the 200–800 nm wavelength range. The D8 Advance X-ray diffractometer by Bruker, which is accessible at UGC-DAE-CSR, Indore, was used to perform XRD measurements. A sealed tube was used to create the X-rays, which had a wavelength of 0.154 nm. Using JCPDS software, the obtained samples' X-ray diffraction pattern was acquired by varying the 2θ theta scattering angle from 100 to 800 [16]. Origin was used to evaluate this data on a computer.

3. Results and Discussion

3.1 UV-Visible Analysis

Cu(II) Complex, SBH, Benzoyl Hydrazine, salicylaldehyde complex, and SBH have all had their ultraviolet-visible spectra completed. At 257 nm, the C=O at the π→ π * transition on salicylaldehyde is located. In benzoyl hydrazine, one peak for the C=O on the amide group is observed at 250 nm. Two prominent transitions are seen in the SBH, and they happen at 254 and 296 nm. The 296 nm band belongs to the azomethine C=N bond because C=N has replaced C=O, whereas the band at 254 nm belongs to the benzoyl hydrazine C=O. The absorption bands in all metal complex spectra have typically shifted to longer wavelengths as a result of the ligand's interaction with the metallic ions. The SBH peaks underwent a shift from blue to red with the addition of Cu(II) solution. At 251 and around 320 nm, there were some new peaks. The peak of the SBH, which is positioned at 296 nm, changes to about 320 nm when the locations of the peaks from the Cu(II) complexes and the SBH are compared, exposing the chromophore. This implies a reaction between the salicylaldehyde group and Cu(II) complexes. Because benzoyl hydrazine's bands turn blue at 251 nm, it is involved in the complex's creation.

3.2 FTIR Analysis

Among the most widely utilized spectroscopic methods for compound identification and structural elucidation of metal complexes is infrared spectroscopy. A few of the prominent bands identified within the spectra of the Cu(II) complexes and free XSBH ligands have been ascribed; they are presented in Fig. 1 and Table 2. The OH symmetric and asymmetric stretching bands in free ligands were identified at 3416 and 3453 cm^{-1} in the infrared spectrum, whereas the ν(C=N) + ν(C=O) stretching modes were detected at 1621 cm^{-1}. Free ligands show bands in the infrared spectral area at 3222 and 3363 cm^{-1}, corresponding to ν(N-H) symmetric and ν(N-H) asymmetric stretching, respectively. The CONH-group characteristic amide (I, II, and III) bands are shown in Fig. 1(Ligand) at 1621, 1572, and 1300 cm^{-1}, respectively. The spectra of several Cu(II) complexes showed the stretching and bending vibrations of the phenolic (C-O) at 1550 and 1280–1310 cm^{-1}, respectively. Several complexes, as shown in Table 1, did not exhibit any vibrations at all. Assigning ν(N-N) and ν(Cu-N) to the complexes, respectively, Fig. 1 shows bands at low frequency of 946-976 cm^{-1} and 520-596 cm^{-1}. The sample structure is confirmed by the absence of any distinctive bands of the amide groups in the spectra of any of the Copper complexes.

3.3 SEM Analysis

To assess the surface morphology of ligand and metal complexes, SEM examination was performed (Fig. 2). SEM pictures clearly show that the Ligand's morphology changes significantly following coordination with the copper ion. The coordination of metal ions to the donor sites in the ligand is the reason for the considerable differences between the SEM micrographs of the ligand, L, and metal complexes (a–d). Furthermore, the metal complexes' SEM micrographs showed that altering the metal ions modifies the surface shape of the complexes. Rod-shaped blocks are oriented vertically and horizontally in the SEM image of the ligand (L) in Figure 4 (Ligand). Complex 1 has morphology akin to a tiny needle. Conversely, the micrographs of complexes 2 and 3 exhibit varying sizes of spherical particles, both bigger and smaller.

3.4 X-ray diffraction Studies

In solid state chemistry and materials research, X-ray diffraction is one of the most significant characterization instruments. XRD is a simple method for figuring out the dimensions and form of the unit cell for any given substance. Phase identification is a qualitative investigation, whereas lattice parameter determination and phase fraction analysis are quantitative analyses that can benefit from the application of powder diffraction methods. Peak Positions provide information on the size and shape of the unit cell, whereas Peak Intensities provide information on the electron density inside the cell, or the locations of the atoms. These two types of information are useful in analyzing translational symmetry. If the size has a lower than around 100–200 nm, it also provides information on extended flaws, micro strain, and deviations from a perfect particle from Peak Shapes & Widths [17]. The structural analysis method benefits from the application of X-ray diffraction. Fig. 3 displays the powder XRD pattern of the samples for complexes 1 through 3. The particle size (measured in nm) and lattice parameter (measured in Å) for each sample are shown in Table 1. After indexing the pattern, the particle sizes and lattice characteristics show that complexes 1 and 2 have tetragonal

structures, whereas complex 3 has orthorhombic structure. The produced samples' X-ray diffraction pattern was acquired by the adjustment of the scattering angle 2θ, which was done in steps of 0.01 from 10^0 to 80^0. The Braggs relation has been used to derive the lattice parameters.

$$n\lambda = 2d \sin\theta \qquad (1)$$

The current work has identified the tetragonal structures of two complexes, with sides reported as $a = b \neq c$ and angles $\alpha = \beta = \gamma = 90^0$. Complex 3 has reported sides as $a \neq b \neq c$ with Orthorhombic structure. The Debye-Scherrer formula has been used to determine the particle sizes of these materials.

$$t = {0.9\lambda}/{\beta \cos\theta} \qquad (2)$$

where β = full width half maxima (FWHM) and λ = 1.54 Å.

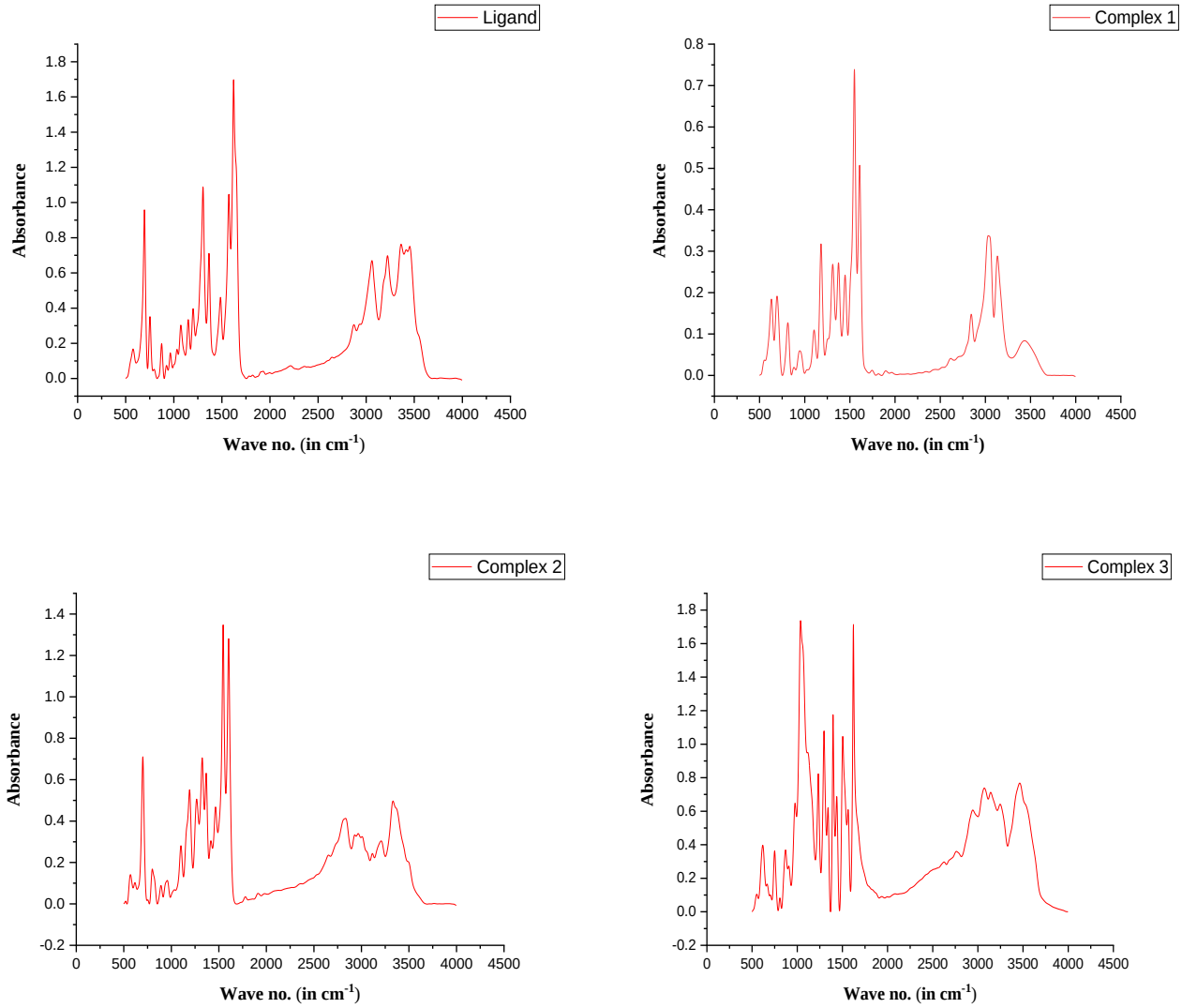


Fig. 1 — IR Spectral Analysis of Salicylaldehyde benzoyl Hydrazine and Copper Complex of Benzoyl Hydrazine in Substitute Salicylaldehyde.

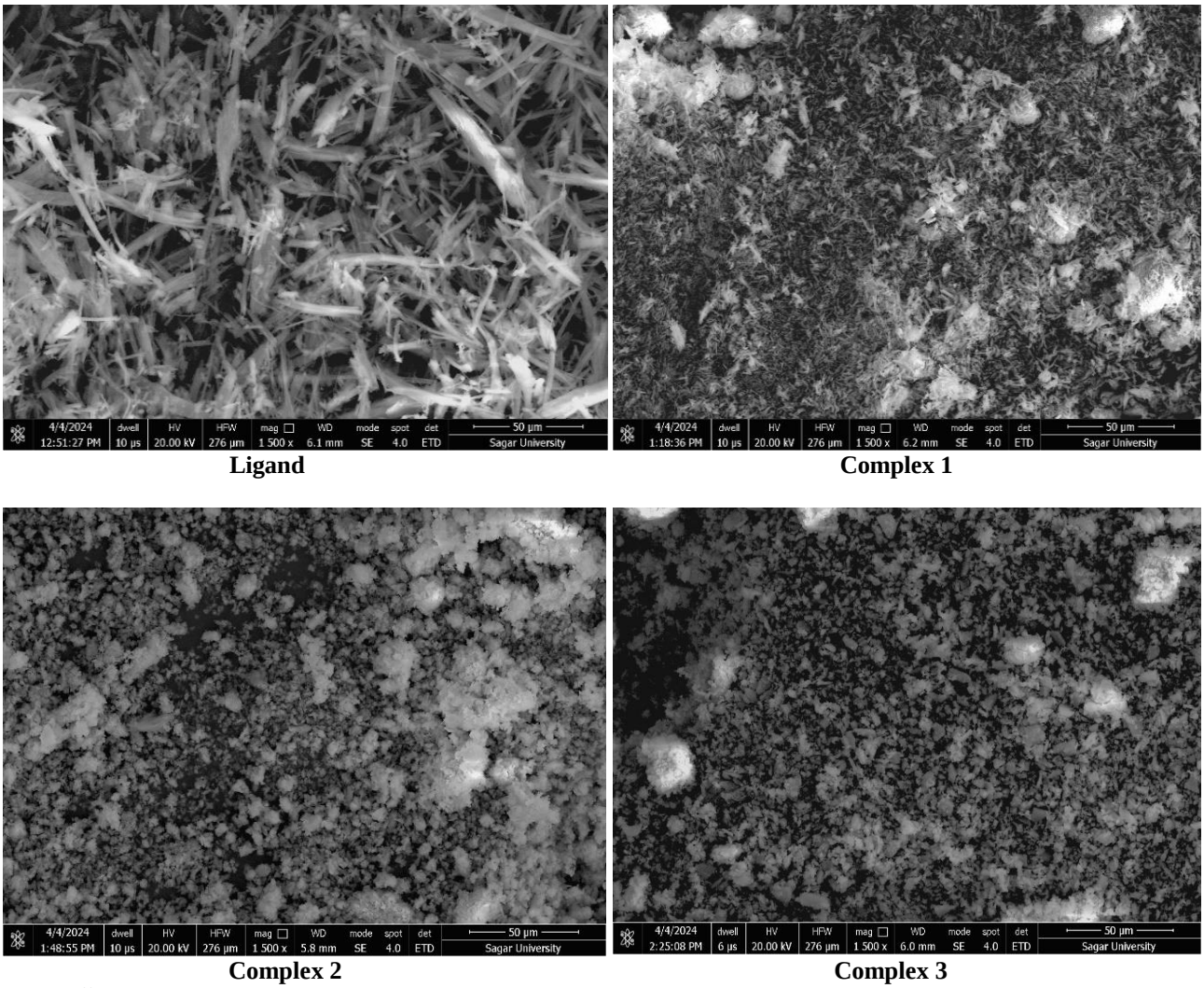
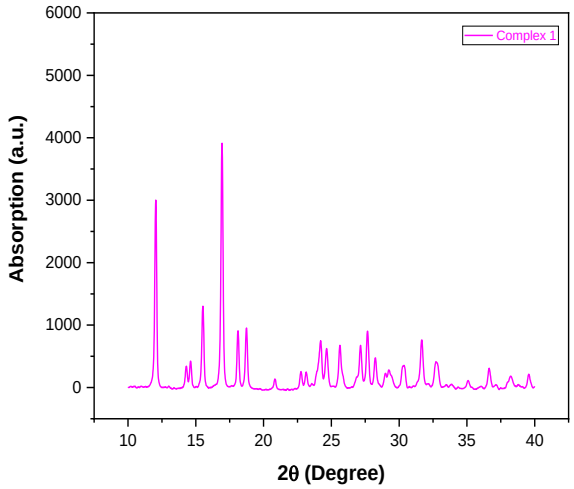
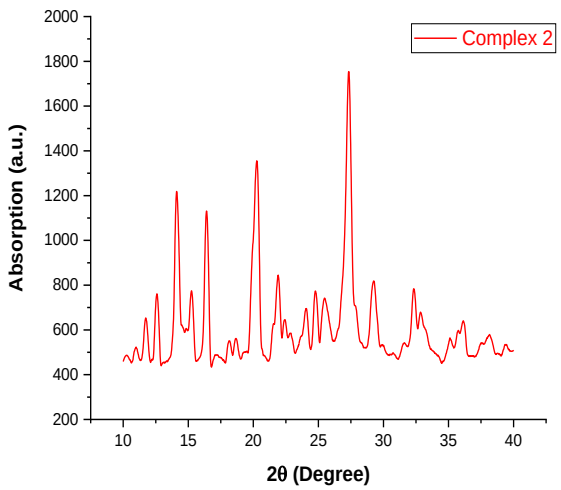


Fig. 2 — Schiff base ligand, L, and its Cu(II) Complex, shown through SEM micrographs.





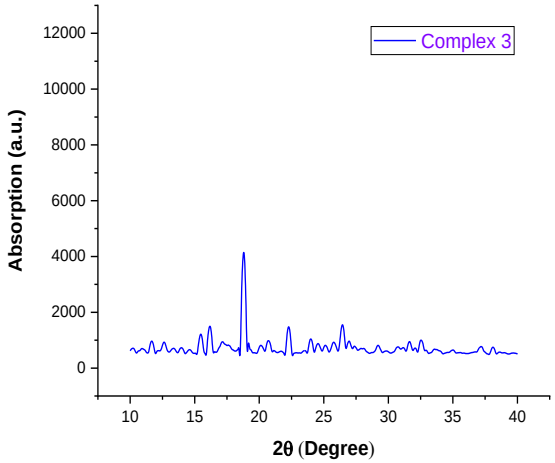


Fig. 3 — Cu(II) Complex X-ray diffractogram pattern.

Table 1 — Cu(II) complexes' lattice parameter, particle size, and (h k l) parameter.

Complex	Lattice Parameter	Particle Size (nm)	(h k l) Parameter
Complex 1	a = b = 7.72 Å, c=29.46 Å	35.34	0 0 4 0 0 6 2 0 0 0 0 8 2 0 3 2 0 4 2 0 5 2 0 6 0 0 10 2 0 7 2 2 1 2 0 8 0 0 12 2 0 11 0 0 14 2 0 13 4 0 0 2 0 14
Complex 2	a = b = 8.65 Å, c = 5.02 Å	16.39	1 1 0 1 0 1 2 2 0 3 1 0 3 0 1 1 1 2 3 2 1 3 3 0 4 2 0 3 1 2 5 1 0 1 0 3 5 0 1 3 3 2 2 1 3 5 3 0 3 0 3 5 1 2 3 2 3 6 1 1 5 3 2 6 0 2 6 3 1

Complex 3	a = 15.03 Å, b = 6.21 Å, c = 19.15 Å	1.62	0 0 2 2 0 1 1 1 0 1 1 1 0 1 2 0 0 4 1 1 3 2 0 4 3 1 0 4 0 1 2 1 4 4 0 5

Table 2 — The ligand and copper complex's infrared spectrum bands (in cm^{-1}).

Assignment	**Ligand**	**Copper Complexes**
ν(OH)	ν_{sym}(OH)3416, ν_{asym}(OH) 3453	ν_{sym}(OH)3416, ν_{asym}(OH) 3453
ν(C=N)+ ν(C=O)	1621	-
ν(N-H)	ν_{sym}(N-H) 3222, ν_{asym}(N-H) 3363	-
Amide (I, II, III)	1621, 1572, 1300	-
ν(C-O)	-	ν_{stre}(C-O) 1550, ν_{bend}(C-O) 1280
ν(N-N)	-	946
ν(Cu-N)	-	520

4. Conclusion

A structural investigation was done on the chemically produced copper complex in the Schiff base ligand. Salicylaldehyde benzoyl hydrazine and its three Cu (II) complexes have been produced and studied using powder X-ray diffraction, FT-IR, UV-visible, and SEM spectroscopic methods in this study. The connection between azomethine nitrogen and enolic oxygen were apparent in the infrared spectrum. The production of Copper complexes involves the Salicylaldehyde benzoyl hydrazine, as indicated by the UV-visible spectroscopy. XRD and SEM were used to determine the crystalline nature, crystallite size, unit cell characteristics, and shape. The average crystallite size in nm was found using XRD.

Future Scope

Many of the mixed-ligand copper complexes that have been discovered recently have demonstrated strong anticancer properties. The development of copper-based anticancer drugs with hydrazone ligands and N-containing co-ligands was made possible by these investigations. Furthermore, pyridine is a frequent prodrug and a building block of several compounds that are vital to biology. Because of their biological significance, pyridine compounds' chemistry and applications have garnered a lot of interest lately.

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Conflict of interest

The authors declare that they have no conflicts of interest.

Data Availability

If you would like more information about this research, the authors are prepared to provide any data.

Authors' Contributions

The study's conception and design were influenced by all authors. Data collection, analysis, and material preparation were completed; the final paper was reviewed and approved by all authors.

References

[1] Sabita Shrestha et al., "Synthesis and Characterization of Copper Complex of Salicylaldehyde Benzoyl Hydrazone", J. Nepal Chem. Soc., Vol. **29**, pp.**11-17**, **2012**.

[2] K. K. Narang et al., "Synthesis, Characterization, Thermal and Electrical Properties of Yttrium(III) and Lanthanide(III) Complexes of Salicylaldehyde Benzoyl Hydrazone" Synth. React. Inorg. Met. Org. Chem. Vol. **30**, pp.**931-954**, **2000**.

[3] V. P. Singh at al., "Synthesis, structural and corrosion inhibition studies on cobalt(II), nickel(II), copper(II) and zinc(II) complexes with 2-acetylthiophene benzoylhydrazone" Inorg. Chim. Acta, Vol. **56**, pp.**379**, **2011**.

[4] T. K. Chaitra at al., "Thermodynamic, electrochemical and quantum chemical evaluation of some triazole Schiff bases as mild steel corrosion inhibitors in acid media" J. Mol. Liq. Vol. **211**, pp.**1026**, **2015**.

[5] P. Mishra et al., Inorg. Met. Org. Chem. Vol. **32**, pp.**1485**, **2002**.

[6] R. C. Maurya et al., "Synthesis, Magnetic, and Spectral Studies of Some Mono- and Binuclear Dioxomolybdenum(VI) Complexes with Chelating Hydrazones Derived from Acid Hydrazides and Furfural or Thiophene-2-aldehyde" Synth. React. Inorg. Met. Org. Chem. Vol. **33**, pp.**309**, **2003**.

[7] Ali at al., "Metal complexes of sulphur-nitrogen chelating agents" Coord. Chem. Rev. Vol. **13**, pp.**101–132**, **1974**.

[8] Williams et al., "Metals, ligands, and cancer" Chem. Rev. Vol. **72**, pp.**203-213**, **1972**.

[9] Cansu Gökçe et al., "Synthesis, characterization and DNA interaction of new copper(II) complexes of Schiff base-aroylhydrazones bearing naphthalene ring" Journal of Photochemistry and Photobiology B: Biology Vol. **122**, pp.**15–23**, **2013**.
[10] J.R.J. Sorenson et al., "Radiation Protection and Radiation Recovery with Essential Metalloelement Chelates
" In Metal Ions in Biological Systems, Marcel Dekker, New York, **1984**.
[11] R.K. Grouch, Adenine Press, Guilderland, New York, **1986**.
[12] Yi-Heng Lu et al., "UV–visible spectroscopic study of the salicyladehyde benzoylhydrazone and its cobalt complexes" Spectrochimica Acta Part A Vol. **65**, pp.**695–701**, **2006**.
[13] Sabita Shrestha et al., "synthesis and characterization of cobalt (iii) complex of salicylaldehyde benzoyl hydrazone" Journal of Institute of Science and Technology Vol. **22**, pp.**132-136**, **2017**.
[14] M. Mohan et al., "Synthesis, Characterization and Antitumour Activity of Iron(II) and Iron(III) Complexes of 3-and 5-Substituted Salicylaldehyde Benzoyl Hydrazones" Inorganica Chimica Acta, Vol. **135**, pp.**167-177**, **1987**.
[15] M. Mohan et al., "Synthesis, Characterization and Antitumor Activity of Manganese(II), Cobalt(II), Nickel(II), Copper(II) Zinc(II) and Platinum(II) Complexes of 3- and Wubstituted Salicylaldehyde Benzoylhydrazones" Inorganica Chimica Acta, Vol. **136**, pp.**65-74**, **1987**.
[16] M. Sharma et al., AIP Conference Proceedings, pp.**1-4**, **2019**.
[17] T. Theivasanthi et al., "Studies of Copper Nanoparticles Effects on Micro-organisms" Archives of Physics Research, Vol. **2**, pp.**112-117**, **2010**.

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