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SYNTHESIS, SPECTRO-COMPUTATIONAL CHARACTERIZATION AND ANTIBACTERIAL EVALUATION OF BUTANE-2,3-DIONE DIOXIME AND ITS ZINC(II) COMPLEX

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ABSTRACT

This study investigates the coordination behavior, structural changes, and biological activity of butane-2,3-dione dioxime and its zinc(II) complex. FT-IR spectra reveal coordination through oxime nitrogen atoms, evidenced by shifts in O–H, C=N, and N–O stretching vibrations, and the appearance of a Zn–N band at 400–500 cm^{-1}. UV-Vis analysis shows a redshift in absorption bands, indicating ligand-to-metal charge transfer and modified intraligand transitions upon complexation. NMR spectroscopy further confirms coordination, with notable downfield and upfield shifts in proton and carbon signals, respectively. DFT calculations reveal a reduced dipole moment and HOMO–LUMO energy gap in the Zn(II) complex, indicating increased chemical reactivity and structural distortion. MEP mapping supports electron density redistribution upon coordination. Antibacterial screening shows that the free ligand demonstrates superior efficacy against *E. coli* (MIC = 0.08 µg/mL), surpassing even ciprofloxacin, while the Zn(II) complex maintains strong activity (MIC = 0.9 µg/mL). These results highlight the influence of metal coordination on electronic structure and biological performance. The results help accelerate the development of metal-based antimicrobial agents and support efforts aligned with Sustainable Development Goal (SDG) 3: Good Health and Well-being**,** promoting innovative approaches to combat antimicrobial resistance.

Keywords: Butane-2,3-Dione Dioxime, Zinc(II) Complex, Coordination Chemistry, DFT, Antibacterial Activity, FT-IR, NMR

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INTRODUCTION

Antimicrobial resistance is projected to cause 10 million deaths annually by 2050.[1], surpassing cancer—a stark reminder of the urgent need for novel therapeutics aligned with *Sustainable Development Goal SDG-3: Good Health and Well-being*.[2] In this context, metal-based complexes have emerged as promising alternatives to conventional antibiotics.[3] Oxime-based ligands, known for their versatile coordination modes and biological relevance, have gained significant attention in medicinal inorganic chemistry.[4] Among them, butane-2,3-dione dioxime is a vicinal dioxime that readily forms stable complexes with transition metals, particularly Zn(II)**.** Zinc plays a crucial role in biological systems, being involved in enzymatic catalysis, gene expression, and immune modulation.[5,6] Zn(II) complexes exhibit a broad spectrum of biological activities—antibacterial, antifungal, anticancer, and antioxidant—primarily through interactions with biomolecules and disruption of cellular processes.[7,8] Importantly, recent findings suggest that oxime-based Zn(II) complexes can exhibit superior antimicrobial performance compared to conventional antibiotics.[9] However, despite their potential, comprehensive spectroscopic, computational, and biological studies on butane-2,3-dione dioxime and its Zn(II) complex remain limited. The aim of this study is to synthesize and characterize butane-2,3-dione dioxime and its Zn(II) complex, and to investigate their structural, electronic, and biological properties.[10] The compounds were characterized using FT-IR, UV-Vis, ^{1}H and ^{13}C NMR, PXRD, SEM, and mass spectrometry. Density Functional Theory (DFT) was employed to study optimized geometries, electronic structure, HOMO–LUMO gaps, and molecular electrostatic potential (MEP) maps. Additionally, molecular docking

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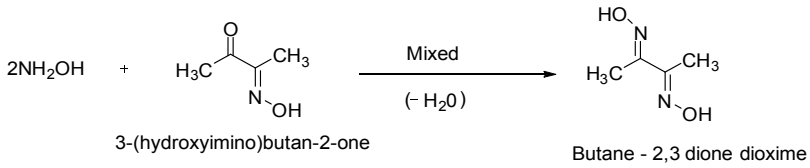
was used to evaluate interactions with biologically relevant proteins.[11,12] Antibacterial activities were tested against *E. coli*, showing that both the ligand and its Zn(II) complex exhibit significant activity, with the free ligand outperforming even standard antibiotics at lower concentrations.[13,14] This multidisciplinary approach bridges experimental and theoretical analyses, offering novel insights into the coordination behavior and therapeutic potential of Zn(II)-oxime systems, thereby supporting the development of advanced metal-based drugs in line with SDG 3.[15]

EXPERIMENTAL

Preparation of Ligand

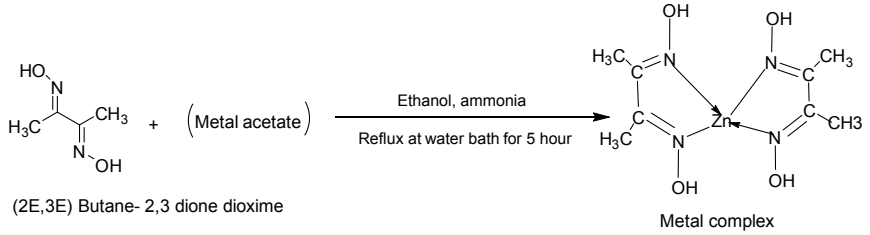
A round-bottom flask was used to mix 1.39 g (20 mmol) of hydroxylamine hydrochloride with 20 mL of distilled water for 20 minutes. Then, a stoichiometric amount of sodium carbonate was added to the solution, and the reaction mixture was gently heated in a water bath at 30–50°C to help the carbon dioxide escape and the water vapor leave. After it cooled to room temperature, 3-(hydroxyimino)butan-2-one (2.32 g, 20 mmol) was added dropwise while stirring with a glass rod. The reaction was done when a white precipitate formed in 5 minutes. We filtered the ligand, rinsed it well with cold distilled water, and then dried it in a vacuum. The result was a white solid with a yield of 78% (1.81 g), as shown in Scheme-1.



Scheme-1: General Synthesis of the Ligand Preparation

Preparation of Metal Complex

A ligand of 2.32 grams (20 mmol) was mixed with 25 mL of methanol and left to stir at room temperature for 10 minutes. Then, a methanolic solution of 4.39 g (20 mmol) Zn(II) acetate dihydrate was added drop by drop to the ligand solution while it was being stirred all the time. The reaction mixture was heated back and forth at 60–70 °C for three hours, which caused a precipitate to slowly form. Then, the mixture was brought to room temperature. The solid part was filtered, washed with cold methanol, and dried in a vacuum. The metal complex Zn(II) was made with a 75% yield (3.62 g), as shown in Scheme-2.



Scheme-2: General Synthesis of the Metal Complex

RESULTS AND DISCUSSION

The FT-IR spectra of butane-2,3-dione dioxime and its Zn(II) complex reveal clear coordination via oxime nitrogen atoms.[16] The O–H stretch shifts from 3300–3400 cm^{-1} (ligand) to 3200–3300 cm^{-1} (complex), indicating hydrogen bonding or electronic environment changes shown in Fig.-1. The C=N and N–O stretches also shift (1620–1650 to 1580–1600 cm^{-1} and 950–1100 to 920–1000 cm^{-1}, respectively), supporting metal-ligand interaction.[17] A new Zn–N band at 400–500 cm^{-1} confirms coordination, with minor changes in C–C and CH_3 vibrations shown in Table-1.UV-Vis spectra of the ligand show bands at 327, 286, and 266 nm ($n\rightarrow\pi^*$, $\pi\rightarrow\pi^*$), whereas the Zn(II) complex exhibits bands at 343 nm (LMCT), 303 nm, and 275 nm, indicating intraligand transitions modified by complexation.[18,19] The complex is diamagnetic, consistent with the d^{10} Zn(II) configuration; measured susceptibility is −95

$\times 10^{-6}$ $cm^3\ mol^{-1}$, as shown in Table-2.[20] ^1H NMR spectra confirm coordination through the oxime nitrogen: the oxime proton shifts from 11.48 ppm to 10.49 ppm, and methyl protons from 1.97 to 2.26 ppm upon complexation.[21,22]

Table 1: FT- I R data of ligand and its Zn metal complex

Vibration Mode	Ligand (cm-1)	Zn(II) Complex (Cm^{-1})	Assignment
ν(OH) (Hydrogen bonded)	3300-3400	3200-3300	Broad due to H-bonding in free ligand and complex
ν (C=N)(Oxime group)	1629-1650	1580-1600	Shift due to coordination of Zn with oxime group
ν(N-O) (Oxime group)	950-1100	920-1000	Slightly reduced in complex after metal binding
ν(Zn-N)	-	400-500	Metal-to-nitrogen bond in the Zn(II) complex
ν(C-C) (Aliphatic)	1350-1450	1330-1430	Slight shift due to complexation
(C-H) (Methyl group)	1350-1400	1340-1380	In-plane bending, slight shift upon coordination

Table 2: UV-Vis data of ligand and its Zn metal complex

Compound	Wavel ength	Absorbance	Transition	Assignment	Magnetic moment	Geometry
Ligand	327	0.04	n→π*	Lone pair Excitation of Nitrogen /Oxygen in oxime group	-	-
	286	0.58	π→π*	Conjugated π-system transition within the ligand Conjugated π-system transition within the ligand (higher energy		
	266	0.53	π→π*			
Zn Metal complex	343	0.55	π→π*	Ligand – to – Metal charge Transfer	0	Tetrahedral geometry
	303	0.52	π→π* IL transition)	Intraligand Conjugated π- system transition		
	75	0.50	π→π* IL transition)	Intraligand Conjugated π- system transition(higher energy)		

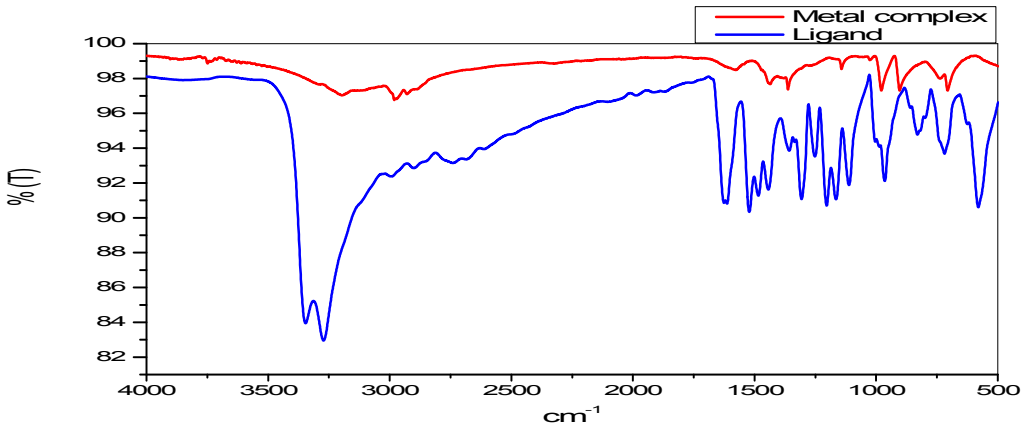


Fig-1: FT-IR spectraof Ligand and its Zn metal complex

In the ^{13}C NMR, the oxime carbon signal shifts from 155.42 ppm to 134.35 ppm, and the methyl carbon from 8.32 ppm to 10.62 ppm, confirming coordination.[23,24] DFT studies using RBLYP/6-31G**(d,p) and LANL2DZ for Zn(II) provided optimized geometries, the free ligand has a total energy of −550.47 Hartree and a dipole moment of 5.20 D, while the Zn(II) complex has −2613.07 Hartree and 1.51 D,[25] indicating reduced polarity and structural distortion upon complexation.[26,27] shown in Fig.-2 and Table-3.

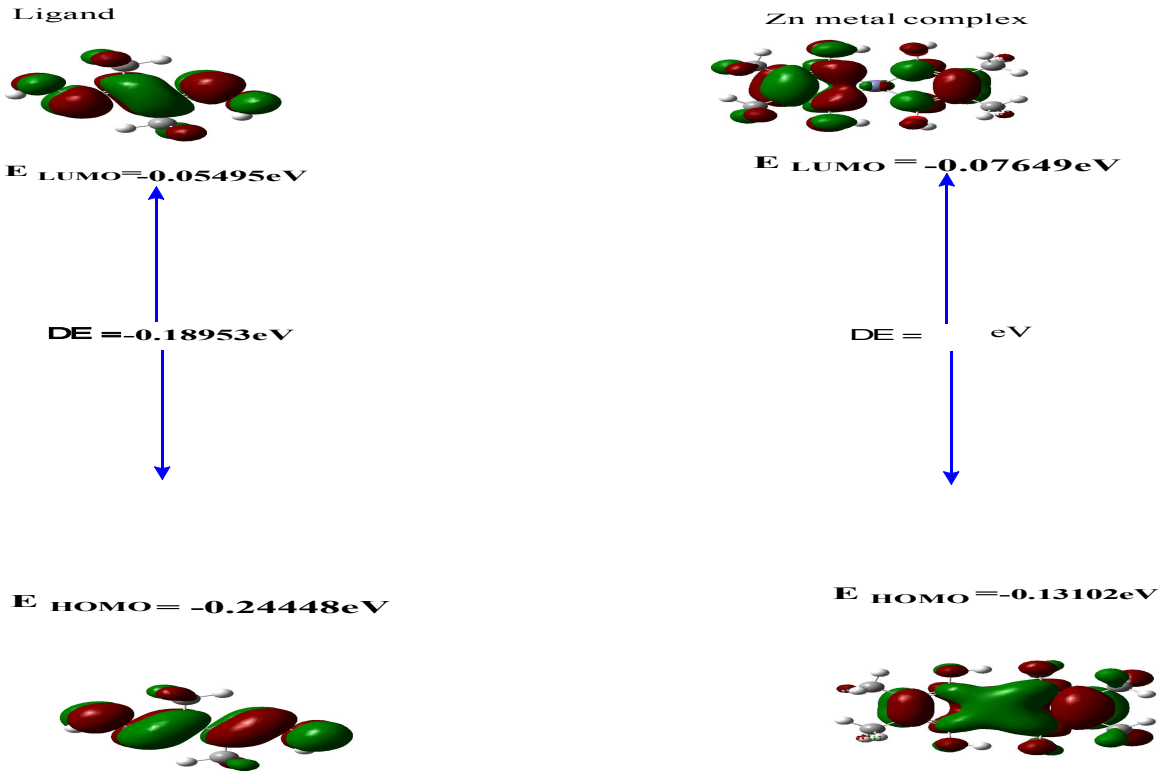


Fig.-2:Energies of HOMO and LUMO of ligand and its Zn metal complexes.

Table 3: Bond angle and bond length of ligand and its Zn metal complex

Ligand and Metalcomplexes	Bond	Bond length (Å)	Angle	Bond Angle
Ligand	C_1-N_9	1.319	N_{10}-C_2-C_1	113.821
	N_{10}-C_2	1.320	Zn_{29}-N_9-O_{13}	126.278
	N_{10}-Zn_{29}	2.039	Zn_{29}-N_{12}-C_6	116.546
	O_{14}-N_{10}	1.420	O_{14}-N_{10}-C_2	117.217
	Zn_{29}-N_9	2.031	N_{10}-C_2-C_4	123.28
	N_9-O_{13}	1.420	N_9-C_1-C_3	122.01
	N_{12}-C_6	1.387	C_5-N_{11}-O_{15}	115.838
	C_5-N_{11}	1.386	C_5-C_6-C_8	126.139
	N_{11}-O_{15}	1.487	C_2-C_1-C_3	123.782
	C_2-C_4	1.500	O_{14}-N_{10}-C_2	117.21
	C_1-C_3	1.498	C_6-C_5-C_7	126.141
Zn metal complex	C_1-N_5	1.301	C_1-N_5-O_7	110.682
	N_5-O_7	0.978	C_2-C_1-C_3	121.102
	C_1-C_3	1.479	C_4-C_3-N_6	124.823
	C_1-C_2	1.502	C_3-N_6-O_8	117.097
	C_3-C_4	1.516		
	C_3-N_6	1.304		
	N_6-O_8	1.422		

FMO analysis shows HOMO–LUMO gap reduction upon complexation (ligand: −0.18953 eV; complex: −0.05453 eV), suggesting increased reactivity shown in Fig.-2.[28] MEP maps reveal redistribution of electron density upon Zn(II) binding, with potential impact on reactivity (ligand: $-6.223e^{-2}$ to $+6.223e^{-2}$; complex: $-5.415e^{-2}$ to $+5.415e^{-2}$) shown in Fig.-3.[29]

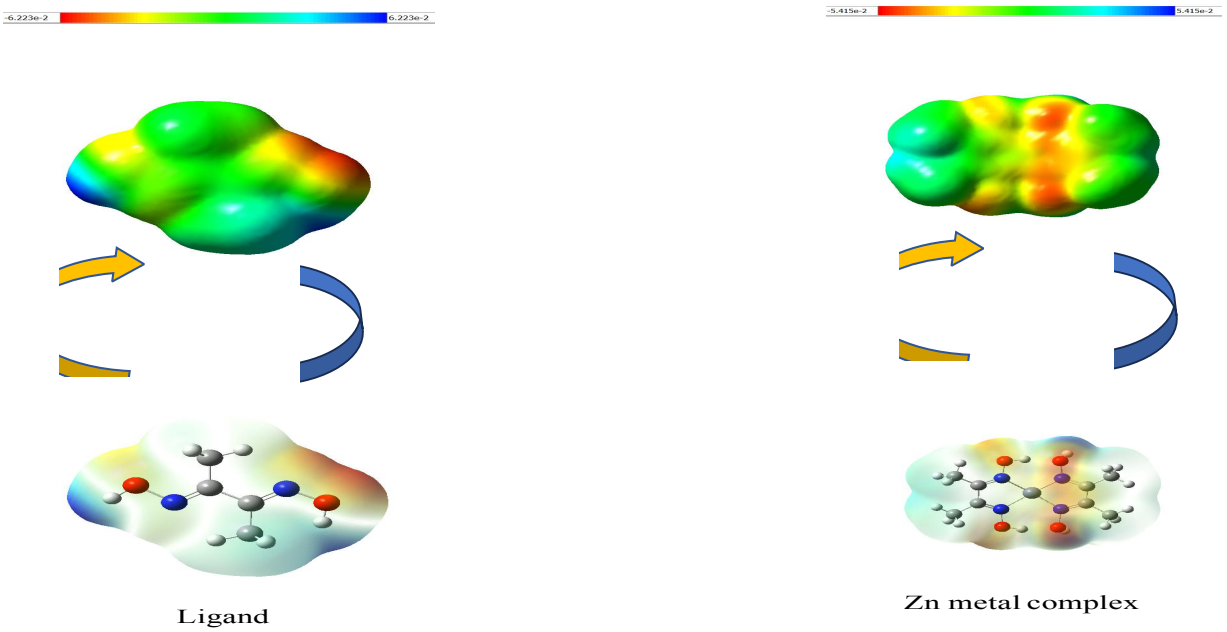


Fig.-3: MEP analysis of ligand and its Zn metal complex

Antibacterial studies against *E. coli* indicate strong activity of the ligand (MIC = 0.08 µg/mL), exceeding that of ciprofloxacin (MIC = 5 µg/mL), while the Zn(II) complex also shows potent, though slightly reduced, activity (MIC = 0.9 µg/mL), as shown in Figs.-4 and 5 and Table-4.

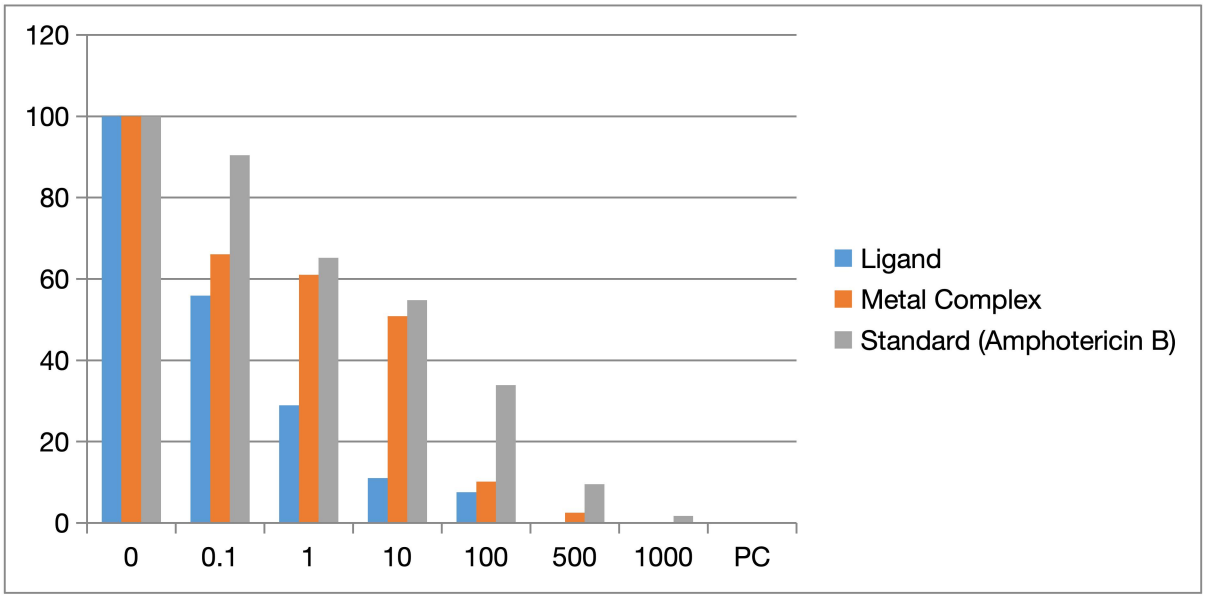


Fig.-4: Antifungal activities of ligand and its Zn metal complex

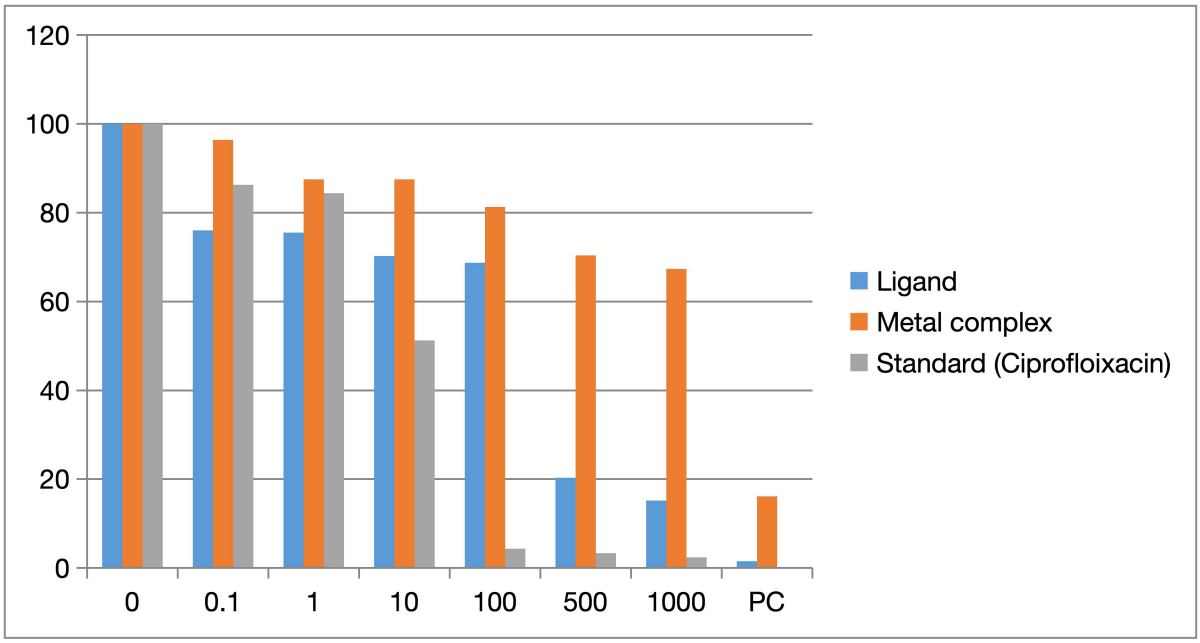


Fig.-5: Antibacterial activities of ligand and its Zn metal complex

Table 4: Antimicrobial activities of ligand and its Zn metal complex

S. No.	Compounds	Bacterial strain *E.coli* MIC (µg/mL)	Fungal strain *A.niger* MIC (µg/mL)
1	Ligand	0.08 µg/mL	0.05 µg/mL
2	Complex(I)	0.9 µg/mL	0.06µg/mL
4	*Ciprofloxacin*	5µg/mL	-
5	*Amphotericin B*	-	0.5µg/mL

Enhanced complex activity at higher concentrations suggests complexation influences bioactivity.[30,31] These results contribute toward UN Sustainable Development Goal 3 (Good Health and Well-being) by demonstrating the potential of metal–oxime complexes as promising candidates fornovel antibacterial agents.[32]

CONCLUSION

The present study confirms the successful synthesis and coordination of butane-2,3-dione dioxime with Zn(II) through oxime nitrogen atoms, as supported by FT-IR, NMR, UV-Vis, and DFT analyses. Spectroscopic and computational studies reveal distinct structural and electronic changes upon complexation, including reduced dipole moment, HOMO–LUMO gap narrowing, and electron density redistribution, suggesting enhanced reactivity. Antibacterial evaluation demonstrates remarkable activity of the free ligand and considerable potency of the Zn(II) complex against *E. coli*, surpassing the standard drug Ciprofloxacin. These findings highlight the potential of metal–oxime complexes as promising antimicrobial agents, warranting further pharmacological and biomedical investigations. In alignment with the UN Sustainable Development Goal 3 (Good Health and Well-being), this work contributes to the search for novel therapeutic alternatives to combat microbial resistance.

CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-3*: *Good Health and Well-being*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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