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A MICROWAVE ASSISTED GREEN SYNTHESIS, CHARACTERIZATION, AND ANTIMICROBIAL SCREENING OF Co(II) AND VO(IV) SCHIFF BASE COMPLEXES DERIVED WITH 2 FURFURALDEHYDE

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ABSTRACT

Cobalt(II) and Vanadium(IV) Schiff base complexes have gained significant research attention due to their structural versatility, varied catalysis, material, and biomedical applications. This study produced Co(II) and VO(IV) metal complexes utilizing a Schiff base from 2-furfuraldehyde and 3,4-dichloroaniline (FCA) using reflux and microwave-assisted techniques. The produced compounds were evaluated using physicochemical and spectroscopic methods, *viz.* elemental analysis. IR, UV–Visible, electron paramagnetic resonance (EPR), molar conductance, magnetic susceptibility, and TGA. The obtained complexes were found to be air- and moisture-stable, crystalline, and intensely colored. Analytical results confirmed a 1:2 (metal-to-ligand) stoichiometry in all complexes. Thermogravimetric studies revealed multi-step decomposition, indicating high thermal stability. The X-ray diffraction (XRD) data provided information on lattice parameters, crystal system, and unit cell dimensions, confirming product crystallisation. The antimicrobial evaluation revealed that the metal complexes exhibited enhanced activity against both bacterial strains (*Escherichia coli, Staphylococcus aureus, Streptococcus faecalis*) and also against fungal (*Trichoderma polysporum, Aspergillus niger, Candida albicans*) strains; thus, reflecting their potential biomedical relevance. This research aligns with UN Sustainable Development Goal (SDG) 3: Good Health and Well-Being, by contributing to the development of new bioactive compounds with potential therapeutic applications. Furthermore, the use of microwave-assisted green synthesis demonstrates a more efficient and environmentally sustainable approach, supporting SDG 9: Industry, Innovation, and Infrastructure. Overall, the study provides a foundation for exploring new entities of Schiff base metal complexes for pharmaceutical and environmental applications.

Keywords: Biological Screening, Microwave Synthesis, Thermal Analyses, XRD Analyses, 2-Furfuraldehyde.
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INTRODUCTION

Coordination chemistry still studies Schiff base metal complexes. Due to their structural flexibility, simple synthetic routes, and a wide spectrum of potential applications. The presence of donor atoms such as nitrogen and oxygen in these ligands enhances their chelating capability, enabling the formation of stable and geometrically diverse transition metal complexes. Numerous pharmaceutical compounds currently in use contain metal centres, to be used as new therapeutic molecules based on metal-chelates.

The self-assembly behaviour of organic ligands coordinated to metal ions or organometallic fragments has also been widely investigated, offering valuable insights into molecular design and supramolecular architecture. Importantly, coordination of bioactive ligands with metal ions often leads to modified pharmacological and toxicological properties, improving their biological effectiveness. Moreover, metal chelation therapy may improve therapeutic efficacy against bacteria, fungi, and viruses.[1-5] Microwave-assisted synthesis, a green chemistry branch, has gained much interest for its environmental and practical advantages. Reactions under microwave irradiation offer reduced pollution, lower cost, higher yields, and simplified processing and handling. This technique significantly shortens reaction times and often results in higher yields compared to reflux heating, indicating that the effect of microwave irradiation is not purely thermal. Microwave energy promotes molecular polarization under irradiation, thereby accelerating reaction rates. The key advantages of microwave-assisted synthesis include: milder reaction conditions; shorter reaction times; and

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enhanced yields.[6-10]

In this study, a new FCA-Schiff base ligand was synthesised from 2-furfuraldehyde with 3,4-dichloroaniline (FCA). It was coordinated with VO(IV) and Co(II) ions using conventional reflux and microwave techniques to assess efficiency, yield, and structure. The FCA-ligand and its corresponding metal complexes were thoroughly characterised to verify their molecular structures and coordination behaviour. The antimicrobial potential of these compounds was evaluated against selected bacterial strains-*Staphylococcus aureus, Escherichia coli, and Streptococcus faecalis*—as well as fungal species including *Trichoderma polysporum, Aspergillus niger, and Candida albicans*. Antimicrobial effectiveness was higher in metal complexes than free ligand. Synthesized complexes have interesting pharmacological relevance, environmental and biological applications. This research aligns with the United Nations Sustainable Development Goal SDG-3: Good Health and Well-Being, by targeting bioactive compounds with antimicrobial potential. Additionally, the adoption of microwave-assisted synthesis promotes SDG-9: Industry, Innovation, and Infrastructure, demonstrating an energy-efficient and sustainable approach in material synthesis.

EXPERIMENTAL

Materials and Methods

Analytical and reagent-grade chemicals used in Synthesis were purchased from HI Media and CDH Chemie. C, H, N percentage calculated on Heroes' elemental CDRI Lucknow's advanced analytical instrument facilities analyser. At Dr. Harisingh Gour University, Sagar (M.P.), a Perkin Elmer Lambda-2B spectrophotometer recorded the UV–Visible spectra in methanol. The room-temperature Elico CM-82 Conductivity Bridge has been used for molar conductance measurements of 10^{-3} M methanolic solutions of the complexes. Magnetic susceptibility data were obtained at room temperature using a Gouy balance, with $CuSO_4 \cdot 5H_2O$ as the standard. Fourier Transform Infrared (FTIR) KBr pellet spectra were acquired at SAIF, CDRI, Lucknow (U.P.) using a Perkin Elmer RX1 spectrophotometer in the 4000–400 cm^{-1} range. Powder XRD patterns were taken using a RINT2000 wide-angle goniometer at 40 kV, 30 mA, and CuKα radiation (λ = 1.54056 Å) at 25°C, between 5°–70°. The Varian E-112 spectrometer at SAIF, IIT Mumbai, was used to record X-band EPR spectra at room temperature using TCNE as the internal reference. Thermal analyses were carried out by Q500 universal V4.5A TA instrument; heating at 10°C min^{-1} in air. Using a custom-designed microwave oven (Model 2001 ETB, 230 V) with a rotating tray, the complexes were synthesized in an open glass vessel**.** At 2450 MHz, 800W of microwave energy was generated. The microwave device contained a thermometer to monitor temperature.

Antimicrobial Activity

Biological efficacy of a novel synthesized complex was examined against a panel of microbial strains, including *Escherichia coli, Staphylococcus aureus,* and *Streptococcus fecalis, Trichoderma polysporum, Aspergillus niger,* and *Candida albicans*. Three doses used in the bioassays: 25µg/mL, 50µg/mL, and 100µg/mL. The results were shown in terms of the inhibition zone (mm). Streptomycin and Nystatin were used as normal examples to compare the killing extents of bacteria and fungi.

FCA (Ligand) was Synthesized by Reflux Heating

Equimolar amounts of 2-furfuraldehyde (0.08 mol) and 3,4-dichloroaniline (0.08 mol) (Fig.-1) in methanol were taken. It was heated over a water bath after which the product was filtered, washed with ether and ethanol, re-crystallised, and dried in a vacuum. A thin-layer chromatography test with silica gel G (yield: 68%) was used to check the purity and reaction completeness of the light brown product.

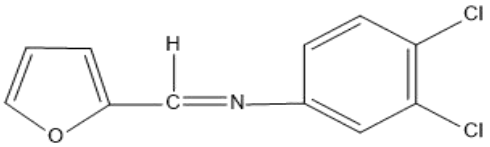

Fig.-1: Structure of FCA (Ligand)

FCA (Ligand) was Synthesized by a Microwave-Assisted Method

In a microwave grinder of microwave 1:1 ratio of 2-furfuraldehyde and 3,4-dichloroaniline was mixed. A microwave oven heated the reaction product after adding 3 mL of solvent. It took a short reaction

time, 6 minutes. This solid was recrystallized and dried. It was vacuumed. TLC tracked the reaction (yield: 82%).

FCA Schiff Base Metal Complexes Synthesized by the Conventional Method

A 50 ml methanolic solution of $VOSO_4 \cdot 5H_2O/CoCl_2 \cdot 6H_2O$ was mixed with a Schiff base (FCA) solution to prepare metal complexes, by refluxing 1:2 metal-to-ligand ratio. The product was heated in a water bath for 12–14 hours. The product has been filtered, washed with ether and ethanol, re-crystallized, and dried in a vacuum. The yield was 65-70%.

FCA Schiff Base Complexes synthesized by the Microwave Method

In a grinder, the Schiff base ligand and $VOSO_4 \cdot 5H_2O/CoCl_2 \cdot 6H_2O$ were mixed and irradiated in an oven by adding 4 ml of solvent. Short reaction time was 8-12 min. The obtained products were recrystallized, washed with ethanol, and dried under reduced pressure. TLC on silica gel G assessed the reaction progress; yield 78-80%.

RESULTS AND DISCUSSION

With microwave-assisted synthesis, the process was completed faster, giving a yield more than the thermal approach. It was also checked that the results were correct via repetition. Microwave-aided synthesis method was compared to the traditional heating method. Some processes that took 6-14 hours with the conventional method were completed in 6-12 minutes with Microwave irradiation, improving yield results from 65-70% to 78-82%. At ambient temperature, all synthesized metal complexes were obtained as colored, crystalline solids that exhibited good stability toward air and moisture. Upon heating, the complexes decomposed at elevated temperatures, indicating their thermal robustness. They were soluble in common organic solvents. Elemental analyses confirmed that all the complexes possess a 1:2 metal-to-ligand stoichiometric ratio, consistent with the proposed molecular formulations. The molar conductance, carried out in 10^{-3} M methanol, revealed values of 48.7 S cm^2 mol^{-1} for the Co(II) complex and 86.3 S cm^2 mol^{-1} for the VO(IV) complex. These results suggest a non-electrolytic nature for the cobalt complex, while the vanadyl complex exhibits univalent electrolytic behaviour.[11,12] The comparative data on reaction duration, yield percentage, and the corresponding C, H, and N elemental analyses for both conventional (CM) and microwave-assisted (MM) syntheses are presented in Table-1.

Table-1: The Molar Conductance Values; C, H, N Percentage Values; Reaction Period and Yield Percentage of the Compounds {Conventional Method (CM); Microwave Method (MM)}

Comp./Molecular Formulae Molecular Wt. [Colour]	Reaction Time		Yield (%)		Elemental analysis Found (Calculated)			*Λ_m
	CM (hours)	MM (minutes)	CM	MM	%C	%H	%N	
FCA ($C_{11}H_7NOCl_2$) 240.0 [Light Brown]	6.1	6.2	68	82	55.3 (55.0)	3.0 (2.9)	6.1 (5.8)	-
$[VO(C_{11}H_7NOCl_2)_2]SO_4.2H_2O$ 678.9 [Black]	13.9	14.2	65	78	38.5 (38.8)	2.2 (2.6)	4.5 (4.1)	86.3
$[Co(C_{11}H_7NOCl_2)_2]2H_2O$ 645.9 [Red Brown]	12.0	12.1	70	80	40.4 (40.8)	2.9 (2.7)	4.5 (4.3)	48.7

*Λ_m = (S cm^2mol^{-1})

Fourier Transform Infrared (FTIR) Spectroscopy

To identify metal–ligand bonding coordination sites of the complexes under investigation, the IR spectra were compared to the free FCA-ligand. Characteristic vibrational peaks in both ligands and their complexes were examined to identify the functional groups participating in coordination. The free FCA Schiff base displays a medium-intensity band at 1620 cm^{-1}, attributed to the ν(C=N) stretching vibration of the azomethine group. In the metal complexes, band shifts by 10-15 cm^{-1}, indicating azomethine nitrogen atom coordination with the metal core. This downward shift reflects a reduction in electron density over the C=N bond upon chelation.

A band appearing at 1132cm^{-1} in the ligand has been assigned to $\nu(C-O-C)$ streching; it shifts downwards by about 25-30 cm^{-1} in the complexes, suggesting coordination through the phenolic or ether oxygen atom. The broad absorption bands around 3378 ±3cm^{-1} correspond to O-H streching vibrations, suggesting the presence of coordinated or lattice water molecules. Additional bands at

571±7cm^{-1} and 450±17cm^{-1} indicate ν(M–O) and ν(M–N) streching vibrations, indicating metal-oxygen and metal-nitrogen bond formation. A sharp absorption band at 981cm^{-1} indicates the terminal ν(V=O)vibration. Absorption bands at 1100 cm^{-1}(ν_3) and 1100 cm^{-1}(ν_3)support the presence of an ionic sulphate group in the VO. The absence of the band ν_2 and the lack of splitting in the ν_3 mode indicate that the sulphate group retains its tetrahedral symmetry (T_d) in the complex.[13-16]

Magnetic Moment and Electronic Spectral Values of FCA-complex

The electronic absorption spectra, taken at ambient temperature in methanol, revealed the metal ions' ligand field (Table-2). Two absorption bands dominate the Co–FCA complex's electronic spectra at 12,500 cm^{-1} and 19,231 cm^{-1}, attributable to the transitions $^4T_1g(F) \rightarrow {}^4A_2g(F)(\nu_2)$, and $^4T_1g(F) \rightarrow {}^4T_1g(P)(\nu_3)$, respectively. The calculated ligand field parameters, including 10Dq, B, β, β%, λ, ν_2/ν_1, and LFSE, are found to be 6944 cm^{-1}, 1004 cm^{-1}, 0.89, 10.3%, –513, 2.2, and 66.37 kJ·mol^{-1}, respectively. The observed magnetic moment (5.04 B.M.) supports an octahedral geometry around the Co(II) centre with considerable covalent character. The first transition band (ν_1) was determined theoretically. The oxovanadium(IV) complex shows two absorption bands at 20,408 cm^{-1} and 12,345 cm^{-1}, which correspond to the transitions $^2B_2 \rightarrow {}^2B_1$ (ν_2) and $^2B^2 \rightarrow {}^2E$ (ν_1). The measured magnetic moment (1.79 B.M.) is consistent with a trigonal bipyramidal or square pyramidal geometry around the VO(IV) centre.[17-20]

Table-2: Magnetic Moment Values and Electric Spectra of FCA-complexes

S.No.	Comp.	Transitions	Bands (cm^{-1})	Magnetic Moment (B.M.)
1.	Co(II)-FCA	$^4T_1g(F) \rightarrow {}^4A_2g(F)(\nu_2)$	12500	5.04
		$^4T_1g(F) \rightarrow {}^4T_1g(P)(\nu_3)$	19231	
2.	VO(IV)-FCA	$^2B_2 \rightarrow {}^2E$ (ν_1)	12345	1.79
		$^2B_2 \rightarrow {}^2B_1$ (ν_2)	20408	
		$^2B_2 \rightarrow {}^2A_1$ (ν_3)	-	

Electron Spin Resonance Spectra

The values of ESR parameters of VO(IV) complex-FCA (Table-3), *viz.* $g^{\|}$, $g^{\wedge}$, g_{av}, and Dg are as 1.9429, 1.9844, 1.9666 and 0.0406, respectively. The parameter g_{av} was calculated by the equation [$(g_{av}) = 1/3(2g^{\wedge} + g^{\|})$].[21,22]

Table-3: Electron Spin Resonance Values of VO(IV) Complex

Complex	$g^{\|}$	$g^{\wedge}$	g_{av}	Dg
$[VO(FCA)_2].SO_4.2H_2O$	1.9429	1.9844	1.9666	0.415

Thermal Analyses

Thermal analyses reveal the degradation patterns of the complexes, showing that initial water loss was followed by ligand breakdown in hydrated compounds, in the next stages. Non-isothermal solid-state kinetic data are summarized in Table-4. A detailed examination of the thermogram of the $[VO(FCA)_2]\cdot SO_4\cdot 2H_2O$ complex indicates that the compound remains thermally stable up to approximately 60 °C. The first weight-loss step observed between 60–160 °C is suggested to correspond with the release of two lattice water molecules, as per experimental and calculated residual weights of 89% and 94%, respectively. Beyond this temperature, decomposition of the dehydrated complex occurs gradually, corresponding to the breakdown of the coordinated ligand moieties. A constant weight plateau was observed after 500 °C, indicating the formation of a stable metal oxide residue.

Table-4: Non-isothermal Kinetic Parameters of the Oxovanadium(IV) Complex

S. No.	Complexes	Methods	Dec. stage/temp. (K)	E (J mol^{-1})	Z (S^{-1})	ΔS* (J mol^{-1})
1.	$[VO(FCA)_2]SO_4.$ $2H_2O$	P-N	Ist/498	11.48	1.71x10^{-13}	-493.42
		C-R		14.74	3.69x10^{-13}	-487.13

The experimental and theoretical remaining weights (26% and 21%) are in good agreement, confirming the formation of vanadium oxide as the final decomposition product. These results

demonstrate the high thermal stability of the synthesized complex, which further supports its structural robustness and coordination integrity.[23-25]

XRD Analyses

XRD data show the crystalline nature of the compounds. The X-ray crystal system was calculated by the error and trial method. The X-ray powder diffraction (XRD) pattern of the VO–FCA complex suggests that the compound crystallizes in a tetragonal crystal system. The diffractogram exhibited 29 prominent reflections, with the most intense peak observed at a d-spacing of 17.3301 Å. The corresponding $\sin^2\theta$ and hkl values for various lattice planes were calculated to determine the structural parameters. The crystal data for the complex were found to be: a = b = 0.001975 Å, c = 0.00032 Å, V = 12,935.50 Å³, Z = 14, D_obs = 1.2202 g·cm⁻³, and D_cal = 1.2719 g·cm⁻³. The close agreement between the observed and calculated density values, as well as between the observed and theoretical $\sin^2\theta$ values, supports the accuracy of the proposed structure. The estimated unit cell volume and average crystallite size were calculated to be 12,935.50 Å³ and 4.665 nm, respectively, confirming the nanocrystalline nature of the VO–FCA complex.[26,27]

Antimicrobial Activity

The microbial resistance ability of the synthesized compounds illustrated that the free ligand (FCA) reflects only moderate activity against all tested organisms. However, upon coordination with transition metal ions, the antimicrobial activity was significantly enhanced. The enhanced activity of these metal complexes, especially at higher concentrations, reflects a dose-dependent response and suggests improved bioavailability upon metal coordination. Antibacterial and antifungal data are summarized in Table-5. The enhanced antimicrobial potential is due to the chelation effect; metal coordination compounds can tailor lipophilicity and hydrophilicity. The change in polarity in such bioactive entities makes them more compatible for the ADM (absorption, distribution, metabolism) mechanism than free ligands; thus enhancing their ability to penetrate microbial cell membranes. Such chemotherapeutics may be used as active topical pharmacophores against bacteria and fungi.[28-32] The microbial activity aligns with the United Nations Sustainable Development Goal SDG-3: Good Health and Well-Being, by targeting bioactive compounds with antimicrobial potential.

Table-5: Antibacterial and Antifungal Data of Synthesized Compounds (at MIC)

Compound	Antibacterial									Antifungal								
	Escherichia coli			*Staphylococcus aureus*			*Streptococcus fecalis*			*Aspergillus niger*			*Trichoderma polysporum*			*Candida albicans*		
	25#	50#	100#	25#	50#	100#	25#	50#	100#	25#	50#	100#	25#	50#	100#	25#	50#	100#
FCA	12	12	16	-	10	20	-	-	-	13	13	15	12	15	19	11	14	18
VO(IV)	18	22	26	14	15	22	12	16	19	16	18	20	15	18	24	18	22	25
Co(II)	16	18	22	15	17	20	11	15	17	14	17	21	17	22	25	16	22	26
Nystatin	-	-	-	-	-	-	-	-	-	18	22	25	22	26	28	20	24	30
Gentamycin	20	24	30	18	22	25	17	22	24	-	-	-	-	-	-	-	-	-

#ppm

CONCLUSION

The present study demonstrates the successful preparation and characterization of VO(IV) and Cobalt(II) coordination compounds, obtained from 2-furfuraldehyde with 3,4-dichloroaniline (FCA-ligand), by both traditional methods and microwave methods. Enhancing yield and improving process is a very important issue in industry. The analytical and spectroscopic data confirmed the formation of stable, air- and moisture-resistant complexes with a 1:2 metal-to-ligand stoichiometry. Thermal studies demonstrated the high stability of complexes. The biological screening (Antimicrobial studies) revealed that metal complexation significantly enhanced the bioactivity of the ligand, highlighting their potential biomedical relevance.

This research aligns with Sustainable Development Goal SDG-3: Good Health and Well-Being, by contributing to the development of new bioactive compounds with potential therapeutic applications. Furthermore, the use of microwave-assisted synthesis demonstrates a more efficient and environmentally sustainable approach, supporting SDG-9: Industry, Innovation, and Infrastructure. Overall, the study provides a foundation for further exploration of Schiff base metal complexes in chemical, pharmaceutical and environmental applications.

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CONFLICT OF INTERESTS

The authors declare that there is no academic, personal or financial conflicts.

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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