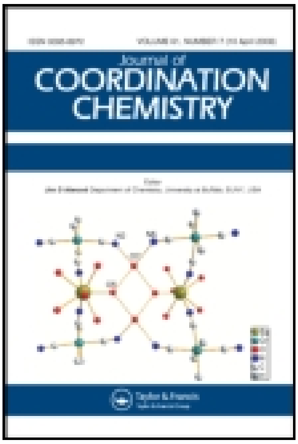

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Synthesis, characterization, NLO study, and antimicrobial activities of metal complexes derived from 3-(3-(2-hydroxyphenyl)-3-oxoprop-1-enyl)-4H-chromen-4-one and sulfanilamide

S. Sumathi [a] , P. Tharmaraj [a] , C.D. Sheela [b] , R. Ebenezer [a] & P. Saravana Bhava [c]

[a] Department of Chemistry , Thiagarajar College , Madurai - 625 009, India

[b] Department of Chemistry , The American College , Madurai - 625 002, India

[c] Department of Chemistry , Vellaichami Nadar College , Virudhunagar - 626 001, India

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Synthesis, characterization, NLO study, and antimicrobial activities of metal complexes derived from 3-(3-(2-hydroxyphenyl)-3-oxoprop-1-enyl)-4H-chromen-4-one and sulfanilamide

S. SUMATHI†, P. THARMARAJ*†, C.D. SHEELA‡, R. EBENEZER†
and P. SARAVANA BHAVA§

†Department of Chemistry, Thiagarajar College, Madurai – 625 009, India
‡Department of Chemistry, The American College, Madurai – 625 002, India
§Department of Chemistry, Vellaichami Nadar College, Virudhunagar – 626 001, India

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A series of metal(II) complexes ML (where M = Cu(II), Co(II), Ni(II), Zn(II), Mn(II), Cd(II), and VO(II)) have been prepared from 3-(3-(2-hydroxyphenyl)-3-oxoprop-1-enyl)-4H-chromen-4-one and sulfanilamide. The structures of the complexes have been investigated by elemental analysis, magnetic susceptibility, molar conductance, IR, UV-Vis, NMR, mass, and ESR spectral studies. Conductivity measurements reveal that the complexes are non-electrolytes, except the oxovanadium complex. Spectral and other data show square pyramidal geometry for oxovanadium and octahedral for the other complexes. The redox behaviors of the copper and vanadyl complexes have been studied through cyclic voltammetry. Antimicrobial activities of the compounds against several microorganisms indicate that some complexes have higher activity than free ligand. The compounds may serve as photoactive materials as indicated from their characteristic fluorescence properties. The second harmonic generation efficiency of the ligand was found to be higher than that of urea and KDP.

Keywords: 3-(2-Hydroxyphenyl)-3-oxoprop-1-enyl-4H-chromen-4-one; Sulfanilamide; Metal complexes; Schiff base; Fluorescence; SHG

1. Introduction

The pronounced biological activity of metal(II) complexes of Schiff bases derived from sulfa drugs has led to considerable interest in their coordination chemistry [1–5]. Sulfanilamide is a potent antibiotic because these drugs undergo metabolic alterations in tissues [6–10]. Their Schiff bases and complexes were investigated for physiologically relevant carbonic anhydrase and superoxide dismutase activities [11, 12]. Similarly, chromones are a widespread class of naturally occurring oxygen-containing heterocyclic compounds. The pharmaceutical and chemical significance of these compounds offers interesting possibilities in exploring more pharmacological activities [13–19]. Condensation products of sulfa drugs with carbonyl groups are biologically active and also have good complexing ability. Many drugs possess modified pharmacological

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*Corresponding author. Email: ptharma@rediffmail.com

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and toxicological properties when administered in the form of metal complexes. 3-Formylchromone readily reacts with primary amines in alcoholic medium yielding an enamine-adduct which reacts further to give the corresponding Schiff base [20]. The role of 3-formylchromones as versatile synthons in heterocyclic chemistry as well as their pharmaceutical importance is well-known. These compounds also have interesting photochemical properties [20]. Nonlinear optical (NLO) materials play an increasingly important role in a wide range of photonic applications [21, 22]. In general, chalcone belongs to the class of conjugated charge transfer compounds that show NLO properties [23]. Several metal complexes derived from chalcones of chromone derivatives [24] are reported, but no work has been reported on the above-mentioned Schiff bases. Interesting structural features, NLO properties, and biological activities of such compounds were key factors to carry out this investigation. In this study, we present the characterization, biological screening, NLO properties, and fluorescence of 3-(2-hydroxyphenyl)-3-oxoprop-1-enyl-4H-chromen-4-imine-(bis-sulfanilamide) (HPOCS) and its metal complexes.

2. Experimental

All chemicals and solvents were purchased from commercial sources. Phosphorous oxychloride, DMF, o-hydroxyacetophenone, and sulfanilamide were Merck India products and used as supplied. Solvents were doubly distilled and stored in molecular sieves (4Å) before use. For voltammetric experiments, tetrabutylammonium perchlorate (TBAP, Sigma) was used as supporting electrolyte. NMR measurement was made on a BRUKER 300 MHz spectrometer (Madurai Kamaraj University, Madurai). UV-Vis spectra were recorded on a THERMO SPECTRONIC 6 HEXIOS α; fluorescence spectra were performed on an ELICO SL174 spectrofluorometer (Lady Doak College, Madurai) using DMSO as solvent. EI mass was recorded by TEOL-GC MATE-2 (IIT, Madras, Chennai). IR spectra were obtained on a SHIMADZU FTIR 8400 S (Standard Fire Work College, Sivakasi). ESR spectra of the copper(II) and oxovanadium complexes were done at 300 and 77 K (IIT, Mumbai) using TCNE (tetracyanoethylene) as the g-marker. Magnetic susceptibilities of the complexes were measured on a Sherwood Magnetic susceptibility balance Mk1 (Thiagarajar College, Madurai). Cyclic voltammetric studies in DMSO were carried out on a CH Instrument for basic electrochemical system using a platinum disc working electrode, platinum wire counter electrode, and Ag/AgCl reference electrode for the oxovanadium complex and for copper(II) complex, platinum working electrode was replaced by glassy carbon electrode. Molar conductances of the complexes were measured in DMSO at room temperature using a Systronic Conductivity Bridge 304. All the complexes were screened for biological activity using the well diffusion method. The second harmonic generation (SHG) conversion efficiency of the ligand was determined by modified version of the powder technique (IISc, Bangalore).

2.1. *Synthesis of Schiff base (HPOCS)*

A mixture of 3-(3-(2-hydroxyphenyl)-3-oxoprop-1-enyl)-4H-chromen-4-one (HPOC) (2.9 g, 1 mmol) [25] and sulfanilamide (3.4 g, 2 mmol) in ethanol was stirred for 6 h at

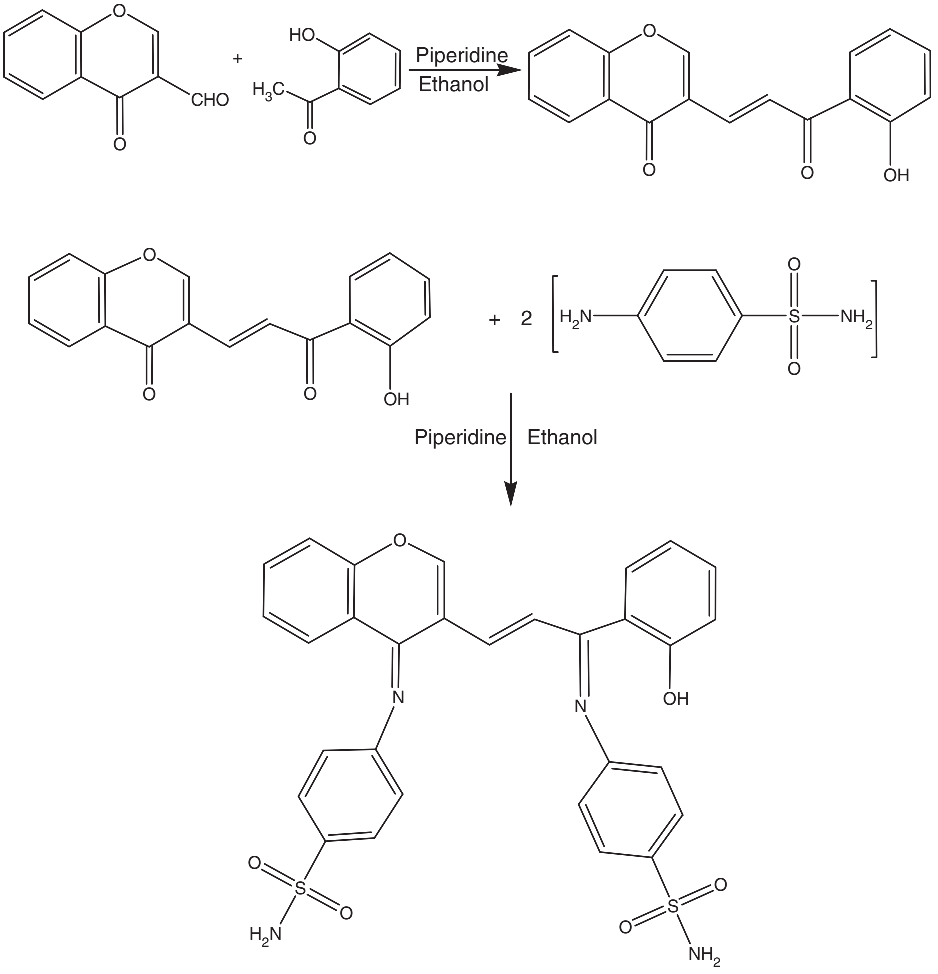


Figure 1. Synthesis of the Schiff base (HPOCS).

room temperature in the presence of piperidine [26]. The solution was concentrated through rotary evaporation and the resulting residue was refrigerated overnight. Reddish yellow solid was collected and recrystallized from ethanol. Yield: 68%, m.p.: 98°C [^{1}H-NMR, 14.25 δ (s, 1H, –OH), 5.86–8.42 δ (m, aromatic proton), 7.691 and 8.229 δ (d, 15.3 Hz, olefinic *trans* coupling signals), 12.09 δ (s, 2H, $-SO_2NH_2$)]. The EI mass of the ligand (HPOCS) gives a molecular ion peak m/z at 600 (Supplementary material). Figure 1 shows the synthesis.

2.2. *Synthesis of metal(II) complexes*

A solution of metal(II) chloride in ethanol (1 mmol) was stirred with an ethanolic solution of HPOCS (0.5 g, 1 mmol) on a magnetic stirrer. Upon concentration, the solid complex precipitated, was filtered, washed thoroughly with ethanol, and dried in vacuum. The oxovanadium complex was synthesized by stirring under reflux and the precipitated complex was collected by filtration, washed with ethanol, and dried under vacuum.

2.3. *NLO properties of ligand*

The SHG efficiencies of HPOC and HPOCS were determined by modified version of the powder technique developed by Kurtz and Perry [27]. The compound was ground into powder and packed between two transparent glass slides. A Nd:YAG laser beam of wavelength 1064 nm radiated the sample cell. The transmitted fundamental wave was absorbed by a copper sulfate solution, which removes the incident 1064 nm light and Filter BG-38 removed any residual 1064 nm light. Interference filter bandwidth is 4 nm for a central wavelength of 532 nm. The green light is finally detected by the photomultiplier and displayed on the oscilloscope. The second harmonic signal was detected by a photomultiplier tube and displayed on a storage oscilloscope. The efficiency of the sample was compared with microcrystalline powder of KDP and urea. The input energy used in this particular setup is 2.2 mJ pulse^{-1}.

2.4. *Biological activity*

Antimicrobial activities of the compounds were tested *in vitro* by the well diffusion method [28] against *Pseudomonas aeruginosa*, *Escherichia coli*, and *Staphylococcus aureus* using agar nutrient as the medium; antifungal activity against the fungus *Candida albicans* cultured on potato dextrose agar as medium was also tested. Stock solution (10^{-3} mol L^{-1}) was prepared by dissolving the compounds in DMSO and the solutions were serially diluted to find the zone of inhibition. A well was made on the agar medium inoculated with microorganisms and filled with the test solution. The plate was incubated for 24 h for bacteria and 72 h for fungi at 35°C. During this period, the test solution diffused, and the growth of the inoculated microorganisms was affected. The inhibition zone at each concentration was noted.

3. Results and discussion

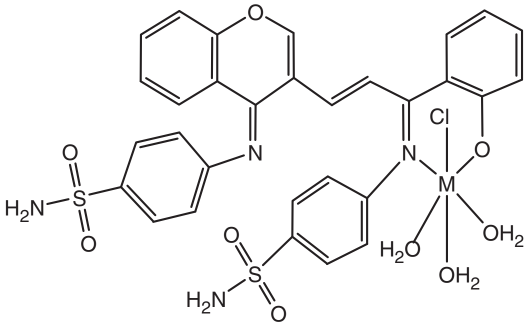
The analytical data and physical properties of HPOCS and metal complexes are presented in table 1. The molar conductances of the complexes imply that the complexes are non-electrolytes except oxovanadium, which is a 1 : 1 electrolyte. The analytical data of the complexes are in good agreement with the general formula [MLX $\cdot$ 3H_2O], where M = Cu(II), Ni(II), Co(II), Zn(II), Mn(II), Cd(II); X = Cl^- and for VO(II) [ML $\cdot$ 2H_2O]X where X = SO_4^{2-}. The magnetic moments show paramagnetic Cu(II), Co(II), and Ni(II) and are in agreement with six-coordinate octahedral metal (figure 2) and the VO(II) complex shows square-pyramidal geometry.

3.1. *^{1}H-NMR spectra*

In comparison with ^{1}H-NMR spectrum of HPOCS, phenolic –OH at 14.25 δ disappeared in the spectrum of the Zn(II) complex, indicating deprotonation upon

Table 1. Analytical, molar conductance, and magnetic susceptibility data of Schiff base (HPOCS) and metal(II) complexes.

		Calculated (found), %						
Compound	m.p. (°C)	M	C	H	N	S	Conductance ($S\,cm^2\,m^{-1}$)	μ_{eff} (BM)
Schiff base	98	–	60.0(59.5)	4.0(3.9)	9.3(9.0)	10.6(10.2)	–	–
$[CuLCl_2{\cdot}2H_2O]$	110	8.1(7.8)	46.8(46.2)	3.6(4.5)	7.2(6.8)	8.3(7.9)	13	1.92
$[CoLCl_2{\cdot}2H_2O]$	130	7.7(7.0)	47.1(46.6)	3.7(3.3)	7.3(7.0)	8.4(8.0)	10	4.73
$[NiLCl_2{\cdot}2H_2O]$	149	7.6(7.8)	47.0(46.8)	3.7(3.4)	7.2(6.9)	8.4(8.2)	18	3.23
$[ZnLCl_2{\cdot}2H_2O]$	120	8.5(8.0)	46.6(46.1)	3.6(3.5)	7.3(7.2)	8.2(7.9)	11	–
$[MnLCl_2{\cdot}2H_2O]$	128	8.1(7.7)	46.5(46.1)	3.7(3.0)	7.2(6.7)	8.3(7.8)	19	5.89
$[CdLCl_2{\cdot}2H_2O]$	150	7.5(7.0)	44.1(43.6)	3.4(3.1)	6.7(6.5)	7.8(7.2)	20	–
$[VOL{\cdot}2H_2O]SO_4$	125	6.3(6.0)	45.0(44.7)	3.6(3.1)	7.0(6.5)	12.0(11.7)	63	1.78



M=Cu(II), Co(II), Ni(II), Zn(II), Mn(II), Cd(II)

Figure 2. Proposed structure of the metal(II) complexes.

complex formation. Shifts of olefinic trans proton signal downfield (7.8 and 8.3 δ) compared to the free ligand suggest zinc coordinated through azomethine. The signal at 3.49 δ is assigned to coordinated water [29]. No appreciable change is observed in the other signals of this complex.

3.2. *Mass spectra*

The mass spectra of HPOCS and Cu(II) and Co(II) complexes have been recorded (Supplementary material). The molecular ion peak for HPOCS was observed at 600 m/z, whereas for Cu(II) and Co(II), the peaks appeared at 752 and 748 m/z, respectively, corresponding to $[ML(H_2O)_3Cl]$.

3.3. *IR spectra*

The ligand shows a strong band at 3382 cm^{-1} (table 2) characteristic of hydroxyl which shifted to 3371–3348 cm^{-1} upon complexation, indicating coordination [30]. The band at 1649 cm^{-1} characteristic of the azomethine group shifted to 1643–1625 cm^{-1}, indicating that the azomethine nitrogen is coordinated [30]. Bands at 1323 and

Table 2. IR spectral data (in cm^{-1}) of HPOCS and metal(II) complexes.

Compound	Coordinated water $\nu(OH)$	$\nu(OH)$	$\nu(C=N)$	$\nu(M–N)$
Schiff base	–	3382	1649	–
$[CuLCl_2{\cdot}2H_2O]$	3444	3346	1641	425
$[CoLCl_2{\cdot}2H_2O]$	3464	3348	1643	428
$[NiLCl_2{\cdot}2H_2O]$	3464	3348	1641	454
$[ZnLCl_2{\cdot}2H_2O]$	3458	3350	1641	464
$[MnLCl_2{\cdot}2H_2O]$	3462	3371	1624	460
$[CdLCl_2{\cdot}2H_2O]$	3462	3367	1629	459
$[VOL{\cdot}2H_2O]SO_4$	3471	3346	1639	432

Table 3. Electronic spectral data of metal(II) complexes.

Compound	Frequency (cm^{-1})	Transition	Geometry
$[CuLCl_2 \cdot 2H_2O]$	22271 17152	INCT $^2E_g \rightarrow {}^2T_{2g}$	Distorted octahedral
$[CoLCl_2 \cdot 2H_2O]$	9606 15847 23148	$^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$ $^4T_{1g} \rightarrow {}^4A_{2g}$ $^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$	Distorted octahedral
$[NiLCl_2 \cdot 2H_2O]$	10869 13440 24038	$^3A_{2g} \rightarrow {}^3T_{2g}$ $^3A_{2g} \rightarrow {}^3T_{1g}(F)$ $^3A_{2g} \rightarrow {}^3T_{1g}(P)$	Distorted octahedral
$[VOL \cdot 2H_2O]SO_4$	13386 12376 22988	$^2B_2 \rightarrow {}^2E$ $^2B_2 \rightarrow {}^2B_1$ $^2B_2 \rightarrow {}^2A_1$	Square pyramidal

1153 cm^{-1} are attributed to $\nu_{as}(SO_2)$ and $\nu_s(SO_2)$, respectively. These remain almost unchanged in the complexes, indicating no coordination [31]. The IR spectrum of HPOCS exhibits bands at 3251 cm^{-1} assignable to $\nu(NH_2)$. The appearance of this mode at almost the same frequency in all the complexes suggests no coordination. In all the metal complexes, there are new bands at 420–464 cm^{-1} assigned to $\nu(M–N)$ [32]. In the vanadyl complex, a band at 974 cm^{-1} is attributed to $V=O$ frequency [33]. The coordinated waters in the complexes are indicated by a broad band at 3460–3577 cm^{-1} and the two weaker bands at 750–800 and 694–732 cm^{-1} are due to $\nu(–OH)$ rocking and wagging, respectively [34, 35].

3.4. *Electronic spectra*

Electronic absorption spectral data of the ligand and complexes recorded in DMSO are given in table 3. The copper(II) complex shows bands assigned as an intraligand charge transfer band (INCT) at 22,271 cm^{-1} and a d–d band at 17,152 cm^{-1} ($^2E_g \rightarrow {}^2T_{2g}$ transition) [36]. This band strongly favors an octahedral geometry, supported by the magnetic susceptibility value (1.92 BM) [37]. The nickel(II) complex exhibits three d–d bands [38] at 10,869, 13,440, and 24,038 cm^{-1} assigned to $^3A_{2g} \rightarrow {}^3T_{2g}$, $^3A_{2g} \rightarrow {}^3T_{1g}(F)$,

Table 4. The spin Hamiltonian parameters of copper and vanadyl complexes.

	($10^{-4}\,cm^{-1}$)				($10^{-4}\,cm^{-1}$)				(cm)
Complex	A_{iso}	g_{iso}	$g_{\parallel}$	$g_{\perp}$	$A_{\parallel}$	$A_{\perp}$	α^2	β^2	$g_{\parallel}/A_{\parallel}$
$[CuLCl_2 \cdot 2H_2O]$	143	2.28	2.34	2.16	173.4	34.2	0.48	2.3	134
$[VOL \cdot 2H_2O]SO_4$	100	2.11	2.12	2.01	91.1	26.8	1.02	1.12	232

and ${}^3A_{2g} \rightarrow {}^3T_{1g}(P)$, respectively. The magnetic moment of Ni(II) complex is 3.23 BM [39], characteristic of octahedral geometry. The cobalt(II) complex has three absorptions [40] at 9606, 15,847, and 23,148 cm^{-1}, which are assigned as ${}^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$, ${}^4T_{1g}(F) \rightarrow {}^4A_{2g}(F)$, and ${}^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$ transitions, respectively. The band at 9606 cm^{-1} confirms the octahedral geometry, which is also consistent with its magnetic moment of 4.73 BM [41]. For the vanadyl(II) complex, three bands at 13,386, 12,376, and 17,513 cm^{-1} are assigned to ${}^2B_2 \rightarrow {}^2E$, ${}^2B_2 \rightarrow {}^2B_1$, and ${}^2B_2 \rightarrow {}^2A_1$, respectively. A square pyramidal geometry [3] is proposed for this system, because it shows a band at 12,376 cm^{-1} and its magnetic moment value is 1.78 BM [33]. Structurally related work exists in the noninvolvement in coordination of hetero NH_2 group para to $-SO_2NH$-group [42], but this study consists of complexes coordinated through hetero NH_2 group para to $-SO_2NH_2$.

3.5. *Electrochemical behavior*

Cyclic voltammograms of the copper(II) complex in DMSO recorded at room temperature using TBAP as the supporting electrolyte from 0.8 to −0.5 V show that the peak current varies with scan rate and the E_{pa}–E_{pc} values are greater than 200 mV, indicating irreversible reduction processes [43]. The ratio of anodic to cathodic peak currents corresponding to the chemical change occurs with the electron transfer as Cu(II) → Cu(III) → Cu(II) → Cu(I) at $E_{pa} = 0.211$ V, and $E_{pc} = 0.554$ V. The oxovanadium complex exhibited two reduction peaks [44] at 0.463 and 0.296 V and one oxidation peak at 0.537 V. The redox process involves V(IV) → V(III) → V(II) → V(IV).

3.6. *EPR spectra*

EPR spectra of copper and oxovanadium complexes are important in studying the metal ion environment. The complexes are recorded in DMSO at 300 and 77 K and the spin Hamiltonian parameters of the complexes are listed in table 4. The observed spectral parameters show $g_{\parallel} > g_{\perp}$ characteristic of an axially elongated octahedral geometry [45]. The g_{iso} value is 2.28 indicating covalent character of the metal ligand bond, supported from the fact that the unpaired electrons lies predominantly in the $d_{x^2-y^2}$ orbital, as was evident from the value of the exchange interaction term G, estimated from the expression $G = (g_{\parallel} - 2.0023)/(g_{\perp} - 2.0023)$. The observed value of G for copper complex (2.14) suggests that an exchange coupling is present and misalignment is appreciable. The α^2 value (0.48) indicates appreciable in-plane covalency in the metal ligand bonding. The calculated value $(g_{\parallel}/A_{\parallel})$ 134.9 cm for the complex indicates a slightly distorted structure. The poor in-plane π bonding is reflected

Table 5. Fluorescence parameters of HPOCS and its metal(II) complexes in DMSO.

Compound	Emission maximum (nm)
Schiff base	455, 520, 893
$[CuLCl_2 \cdot 2H_2O]$	457
$[CoLCl_2 \cdot 2H_2O]$	457, 523
$[NiLCl_2 \cdot 2H_2O]$	460, 529
$[ZnLCl_2 \cdot 2H_2O]$	457, 523
$[MnLCl_2 \cdot 2H_2O]$	511
$[CdLCl_2 \cdot 2H_2O]$	525
$[VOL \cdot 2H_2O]SO_4$	456

in β^2 values. The molecular orbital coefficients α^2, β^2 were calculated using the following equations:

$$\alpha^2_{\mathrm{Cu}} = A_{||}/p + (g_{||} - 2.0023) + 3/7\,(g_{\perp} - 2.0023) + 0.04,$$
$$\beta^2_{\mathrm{Cu}} = (g_{||} - 2.0023)E/(-8\lambda\alpha^2).$$

The EPR spectrum of vanadyl complex shows a single vanadium is present. The $g_{||}$, $g_{\perp}$, and $A_{||}$ values suggest square pyramidal geometry. The molecular orbital coefficients α^2, β^2 were also calculated [46]; the lower α^2 compared to β^2 indicates that the in-plane σ-bonding is more covalent than the in-plane π-bonding.

$$\alpha^2 = (2.0023 - g_{||})E/8\lambda\beta^2,$$
$$\beta^2 = 7/6(-A_{||}/p + A_{\perp}/p + g_{||} - 5/14 g_{\perp} - 9/14\, g_{\mathrm{iso}}).$$

3.7. *Fluorescence*

Spectral studies show that HPOCS and its complexes are fluorescent. HPOCS exhibits a broad and strong emission band with maximum wavelengths 893, 520, and 455 nm upon photo-excitation at 451 nm. Similarly, the above complexes were characterized by emission bands at 457–529 nm. The emission band at 893 nm disappeared because of the interaction of the ligand with metal ion (table 5). Significant differences in the positions of emission maxima of Schiff base and its complexes establish the complexation process [47].

3.8. *NLO property*

From the experimental data, HPOC and HPOCS show better second harmonic efficiencies than urea and KDP, HPOC showing 1.6 and 6.9 times more activity than urea and KDP, respectively, while HPOCS shows 1.1 and 4.6 times more activity than urea and KDP, respectively.

3.9. *Biological activity*

The zone of inhibition values of the compounds against the bacteria and fungus are summarized in table 6. Amikacin and ketoconazole were used as reference drugs

Table 6. Antimicrobial activity of HPOCS and its metal complexes (zone of inhibition in mm[a]; concentration in $10^{-3}\,\mathrm{mol\,L^{-1}}$).

Compound	*P. aeruginosa*	*S. aureus*	*E. coli*	*C. albicans*
Schiff base	7	16	16	16
$[\mathrm{CuLCl_2 \cdot 2H_2O}]$	9	8	15	13
$[\mathrm{CoLCl_2 \cdot 2H_2O}]$	8	R	10	R
$[\mathrm{NiLCl_2 \cdot 2H_2O}]$	8	8	12	12
$[\mathrm{ZnLCl_2 \cdot 2H_2O}]$	7	12	15	12
$[\mathrm{MnLCl_2 \cdot 2H_2O}]$	12	9	16	R
$[\mathrm{CdLCl_2 \cdot 2H_2O}]$	15	16	20	18
$[\mathrm{VOL \cdot 2H_2O}]\mathrm{SO_4}$	12	7	16	9
Standard	18	18	17	R

R = resistant.

[a] Its value observed is within the error limits of ±1.

for antibacterial and antifungal activities, respectively. The observed values indicate that most of the complexes possess higher activity than the free ligand. The antibacterial activity of Cd(II) complex is more active than HPOCS and the standard drug amikacin against *E. coli*. Similar studies [32, 42] reveal that Cu(II) and Ni(II) complexes show relatively higher antimicrobial activities on bacteria and fungi. Such an increased activity of the complexes can be explained on the basis of chelation theory [48] and size of the metal ion. The mode of action of compounds may involve the formation of a hydrogen bond with the active center of cell constituents resulting in interference with normal cell process.

4. Conclusion

The Schiff base (HPOCS) complexes [ML] of Cu(II), Co(II), Ni(II), VO(II), Zn(II), Mn(II), and Cd(II) derived from 3-(3-(2-hydroxyphenyl)-3-oxoprop-1-enyl)-4H-chromen-4-one and sulfanilamide were synthesized and characterized by spectral and analytical data. HPOC and HPOCS exhibit appreciable nonlinear properties in comparison with KDP and urea, and can be utilized for further NLO investigations. Most metal chelates show better antimicrobial activity than HPOCS, particularly Cd(II) has higher activity than the standard one. Such increased activity of the metal complex is of tremendous importance in the pharmaceutical field. Ligand and its metal complexes are fluorescent.

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