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Transition metal complexes of 5-bromosalicylidene-4-amino-3-mercapto-1,2,4-triazine-5-one: Synthesis, characterization, catalytic and antibacterial studies

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Abstract: Transition metal complexes of 5-bromosalicylidene-4-amino-3-mercapto-1,2,4-triazine-5-one with metal precursors, such as Cu(II), Ni(II), Co(II) and Pd(II), were synthesized and characterized by physico-chemical and spectroscopic techniques. All the complexes are of the ML type. Based on analytical, spectral data and magnetic moments, the Co(II) and Ni(II) complexes were assigned octahedral geometries, while the Cu(II) and Pd(II) complexes square planar. A study on the catalytic oxidation of benzyl alcohol, cyclohexanol, cinamyl alcohol, 2-propanol and 2-methyl-1-propanol was performed with N-methylmorpholine-*N*-oxide (NMO) as co-oxidant. All the complexes and their parent organic moiety were screened for their biological activity on several pathogenic bacteria and were found to possess appreciable bactericidal properties.

Keywords: triazine; Schiff base; catalytic oxidation; antibacterial studies.

INTRODUCTION

Catalytic oxidation of alcohols is an important industrial process due to the wide variety of products synthesized by this route. Many systems have been reported for the oxidation of alcohols, mainly involving catalysts containing transition metals such as bivalent Co,¹ Pd,² Ni³ and Ru.⁴ The ability of transition metals to assume a wide range of oxidation states and coordination geometries provides unique opportunities for catalysis. The development of highly efficient transition metals-based catalysts is also driven by their tolerance towards functional groups, their ease of synthesis and their versatility. Schiff bases and their transition metal complexes are still found to be of great interest in inorganic

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chemistry, although this subject has been studied extensively.^{5,6} Schiff bases and their metal complexes show biological activity as antibiotics, antiviral and anti-tumour agents because of their specific structures.^{7,8}

Triazole chemistry is becoming more important due to its excellent biological activity. The triazole antifungal drugs include fluconazole, isavuconazole, itraconazole, voriconazole, pramiconazole and posaconazole. The triazole plant protection fungicides include epoxiconazole, triadimenol, propiconazole, metconazole, cyproconazole, tebuconazole, flusilazole and paclobutrazol. Studies have been reported on the synthesis and characterization of metal complexes of Schiff base involving 1,2,4-mercapto triazoles with salicylaldehyde, *p*-nitrobenzaldehyde, *o*-nitrobenzaldehyde, *p*-toluylaldehyde and cinnamaldehyde.⁹⁻¹¹ Transition metal complexes of polydentate ligands involving five donor atoms have also been reported.¹² These ligands were reported to coordinate through deprotonated oxygen, thio-enolic sulphur and azomethine nitrogen atoms.

In the present study, the synthesis and characterization of transition metal complexes of 5-bromosalicylidene-4-amino-3-mercapto-1,2,4-triazine-5-one (BrSAMT) were undertaken and their applications as catalysts for the oxidation of benzyl alcohol, cyclohexanol, cinnamyl alcohol, 2-propanol and 2-methyl-1-propanol. Their possible anti-bacterial activity was also explored.

EXPERIMENTAL

Methods

The metal content of these complexes were estimated using a Varian AA 55B atomic absorption spectrometer. The infrared spectrum was recorded on an ABB BOMEM FT-IR spectrophotometer. Carbon, hydrogen, nitrogen and sulphur were analyzed using a Thermo Flash EA 1112 series CHN analyzer. The magnetic susceptibilities were determined using a Sherwood Scientific magnetic susceptibility meter (Cambridge, UK). The electronic spectra of the complexes in DMF were recorded on a GBC model UV-Vis spectrophotometer. Thermogravimetric analysis was realised using an SII Exstar 6000 model TGA analyzer. Gas chromatographic analysis was performed on a Shimadzu 2014 model instrument. The ¹H-NMR spectra were recorded on a Bruker AV 400 instrument using TMS as the internal standard. The conductivity measurements were performed on an Elico 32 model instrument.

Synthesis of the ligand

The ligand was synthesized in two steps. In the first step, 4-amino-3-mercapto-1,2,4-triazine-5-one was synthesized according to a literature procedure.¹³ In the second step, a solution of 0.10 mol of 4-amino-3-mercapto-1,2,4-triazine-5-one in 10 ml of ethanol and 0.10 mol of 5-bromo salicylaldehyde was refluxed for 4 h, cooled, filtered, washed with water, dried and then recrystallized from absolute alcohol. The yellow coloured 5-bromosalicylidene-4-amino-3-mercapto-1,2,4-triazine-5-one (BrSAMT) was dried in a desiccator.

Synthesis of the complexes

To a hot solution of 40 mmol of BrSAMT in 20 ml alcohol, 0.002 mol of the respective transition metal chlorides in water (minimum quantity 2 ml) was added and the mixture refluxed for 2 h and then cooled. The different coloured complexes were filtered, washed with

water, ethanol, diethyl ether and dried in a desiccator. The resultant complexes were brown coloured for the Cu(II) and Co(II) complexes, orange brown for the Pd(II) complex and reddish orange for the Ni(II) complex.

Catalytic oxidation experiments with N-methyl morpholine-N-oxide (NMO)

To a solution of the required alcohol (0.060–0.12 mL, 1.0 mmol) in dichloromethane (20 mL), NMO (0.35 g, 3.0 mmol) and the required metal-complex (0.01 mmol) were added and the solution was heated under reflux for 2 h. The mixture was then filtered and the filtrate was dried over anhydrous Na_2SO_4 . It was then evaporated to dryness and extracted with diethyl ether. The diethyl ether extract was filtered and evaporated to give the corresponding aldehyde.^{14,15} The products were identified using gas chromatography by comparing their retention time with those of authentic samples.

Biological tests

The synthesized complexes of BrSAMT were screened for their *in vitro* antibacterial activity against pathogenic strains of gram-negative bacteria, *i.e.*, *Escherichia Coli*, *Pseudomonas aeruginosa*, *Salmonella typhi* and *Shigella flexneri* using the plate technique. The bacteria were cultured in previously sterilized Mueller Hinton agar medium in a petri dish. Solutions of the ligand and its complexes in DMF were plated onto the cultured agar medium and incubated for a period of 24 h at 37 °C. Solutions of the ligand and its complexes in DMF were plated on cultured agar medium and incubated for a period of 24 h at 37 °C. The components to be tested were dissolved in DMF to a final concentration of 0.5 and 1% and soaked in filter paper discs of 5 mm diameter and 1 mm thickness. Nicotinic acid was used as reference material. After the incubation period the plates were observed for zones of inhibition (in cm).

RESULTS AND DISCUSSION

Analytic and spectral data

Ligand. Yield: 60 %; Anal. Calcd. for $\text{C}_{10}\text{H}_6\text{BrN}_4\text{O}_2\text{S}$: C, 36.4; H 2.1; N, 17.0; S 9.7 %. Found: C, 36.5, H, 2.1, N, 17.2, S, 9.7 %. IR (KBr, cm^{-1}): 1690 s ($-\text{C}=\text{O}$ stretching of $-\text{CO}$ in the triazole group), 1650 ($\text{C}=\text{N}-$ stretching of the azomethine group), 1455 (thioamide group). $^1\text{H-NMR}$ (200 MHz, $\text{DMSO-}d_6$, δ / ppm): 11.20 (1H, s, $-\text{SH}$), 8.7 (1H, s, $-\text{CH}=\text{N}-$), 6.24–8.76 (3H, m, aromatic, $J = 7.8$ Hz).

Ni(II) complex. Yield: 60 %; Anal. Calcd. for $\text{C}_{10}\text{H}_{10}\text{BrClNi}_4\text{O}_4\text{S}$: C, 26.29; H, 2.15; N, 12.15; S, 7.01 %. Found: C, 26.32, H, 2.15, N, 12.28, S, 7.03 %. IR (KBr, cm^{-1}): 3500, 900 ($-\text{OH}$ stretching of H_2O), 1730 s ($-\text{C}=\text{O}$ stretching of $-\text{CO}$ in triazole group), 1620 ($\text{C}=\text{N}-$ stretching of the azomethine group), 1450 (thioamide group), 490 (Ni–O), 440 (Ni–N). $^1\text{H-NMR}$ (200 MHz, $\text{DMSO-}d_6$, δ / ppm): 11.24 (1H, s, $-\text{SH}$), 8.5 (1H, s, $-\text{CH}=\text{N}-$), 6.24–8.76 (3H, m, aromatic, $J = 7.8$ Hz). Magnetic moment, $\mu_{\text{eff}} / \mu_{\text{B}}$: 2.73.

Co(II) complex. Yield: 60 %; Anal. Calcd. for $\text{C}_{10}\text{H}_{10}\text{BrClCo}_4\text{O}_4\text{S}$: C, 26.25; H, 2.20; N, 12.20; S, 7.01 %. Found: C, 26.31, H, 2.21, N, 12.27, S, 7.02 %. IR (KBr, cm^{-1}): 3500, 905 ($-\text{OH}$ stretching of H_2O), 1740 s ($-\text{C}=\text{O}$ stretching of $-\text{CO}$ in the triazole group), 1610 ($\text{C}=\text{N}-$ stretching of the azomethine group),

1445 (thioamide group), 495 (Co–O), 445 (Co–N). ¹H-NMR (200 MHz, DMSO-*d*₆, δ / ppm): 11.10 (1H, *s*, –SH), 8.55 (1H, *s*, –CH=N–), 6.24–8.76 (3H, *m*, aromatic, *J* = 7.8 Hz). Magnetic moment, μ_{eff} / μ_B: 3.72.

Cu(II) complex. Yield: 60 %; Anal. Calcd. for C₁₀H₆BrClCuN₄O₂S: C, 28.15; H, 1.36; N, 13.09; S, 7.41 %. Found: C, 28.25, H, 1.42, N, 13.18, S, 7.54 %. IR (KBr, cm⁻¹): 1745 *s* (–C=O stretching of –CO in the triazole group), 1620 (C=N– stretching of the azomethine group), 1452 (thioamide group), 490 (Cu–O), 440 (Cu–N). ¹H-NMR (200 MHz, DMSO-*d*₆, δ / ppm): 11.55 (1H, *s*, –SH), (1H, *s*, –CH=N–), 6.24–8.76 (3H, *m*, aromatic, *J* = 7.8 Hz). Magnetic moment, μ_{eff} / μ_B: 1.73.

Pd(II) complex. Yield: 60 %; Anal. Calcd. for C₁₀H₆BrClCuN₄O₂S: C, 25.58; H, 1.28; N, 11.75; S, 7.01 %. Found: C, 25.66, H, 1.29, N, 11.97, S, 7.02 %. IR (KBr, cm⁻¹): 1744 *s* (–C=O stretching of –CO in the triazole group), 1615 (C=N– stretching of the azomethine group), 1448 (thioamide group), 491 (Pd–O), 446 (Pd–N). ¹H-NMR (200 MHz, DMSO-*d*₆, δ / ppm): 11.35 (1H, *s*, –SH), 8.45 (1H, *s*, –CH=N–), 6.24–8.76 (3H, *m*, aromatic, *J* = 7.8 Hz).

Infrared spectra

The ligands showed an infrared band at 2900 cm⁻¹, indicating the absorptions ν(C–H). The ligand also showed the presence of four thioamide bands I, II, III and IV at 1500–1600, 1270, 900, and 800 cm⁻¹, respectively, indicating the presence of the thioamide moiety in the ligand molecule. No systematic shifts of these bands in the complexes were observed. The band at around 1700 cm⁻¹, corresponding to ν(C=O) of the carbonyl group in the ligand, was altered in the complexes. This shows the possibility of carbonyl group participation in the bonding. The bands at 1650 and 1600 cm⁻¹ in the ligand and the complexes correspond to ν(C=N) of the azomethine linkage¹⁶ and the shift in frequency to lower wave numbers indicates the coordination of the nitrogen of this group with the metal ions. In the complexes, the band at 1300 cm⁻¹, due to the phenolic group, was shifted to higher wave numbers, indicating the involvement of the phenolic oxygen in the coordination with the metal. The broad band at around 3500 and 890 cm⁻¹, corresponding to ν(O–H) in cases of the Co(II) and Ni(II) complexes indicates the presence of coordinated water molecules in these complexes. The presence of a band at 669 cm⁻¹ in the spectrum of the BrSAMT ligand and its complexes, due to C–Br stretching, remained unchanged, indicating that the bromo group was not involved in the bonding.

Electronic spectra

In the case of six coordinated octahedral and pseudo-octahedral Ni(II) complexes, the three spin allowed transitions from ³A_{2g}(F) to ³T_{2g}(F), ³T_{1g}(F) and ³T_{1g}(P) generally fall within the regions 7000–13000 cm⁻¹, 11000–20000 cm⁻¹

and 19000–27000 cm^{-1} , respectively. The ratio of the frequencies of the second to the first transitions would be around 1.8.¹⁷ The first transition, corresponding to ${}^3A_{2g}(F)$ to ${}^3T_{2g}(F)$ was not recorded in the electronic spectra. For high spin octahedral complexes of Co(II), three transitions are expected in the electronic spectra and they are ${}^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$, ${}^4T_{1g}(F) \rightarrow {}^4A_{2g}$ and ${}^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$ in order of increasing energy. The first transition was not recorded but the position was calculated from the second transition. For the complexes of Cu (II), only one transition was observed, assigned to ${}^2E_{1g}(F) \rightarrow {}^2T_{2g}(F)$.

¹H-NMR Spectra

The ¹H-NMR spectra of the free ligand and the complexes were recorded in DMSO. The ligand exhibit a singlet at 11.24. This signal in the spectra of the complexes remained as such, suggesting the non-participation of sulphur in the coordination. The singlet at 8.7 ppm in the ligand due to the azomethine proton was shifted to 8.5 ppm in the complexes. In the spectrum of the ligand, multiplets due to aromatic protons appeared in the range δ 6.24–8.76 ppm. These resonance signals remain unchanged in the spectra of the complexes, suggesting their non-involvement in bonding. The peak corresponding to –OH at 10.8 ppm in the free ligand was not observed in the spectra of the complexes. This clearly shows deprotonation of –OH during complex formation.

Magnetic susceptibility

The magnetic susceptibility results indicate that the complexes of Co(II), Ni(II) and Cu(II) are paramagnetic, while the Pd(II) complex is diamagnetic in nature. Ni(II) being a d^8 in an octahedral field contains two unpaired electrons and hence shows paramagnetism. The value of $2.73 \mu_B$ is close to the theoretical value of 2.83 for octahedral Ni(II) complexes. Co(II) is a d^7 system, thus its complexes show paramagnetism equivalent to three unpaired electrons in octahedral geometry. Since the orbital contribution of tetrahedral Co(II) is much less than that of octahedral complexes, tetrahedral Co(II) complexes generally have lower values of magnetic moments, as compared to that of octahedral complexes. This high degree of orbital contribution for octahedral complexes is due to the three-fold degeneracy of the ${}^4T_{1g}$ ground state. The observed value of $3.72 \mu_B$ is close to the theoretically calculated values of $3.83 \mu_B$ for octahedral Co(II) complexes. Cu(II), being a d^9 system, shows paramagnetism in square planar complexes. The value of $1.74 \mu_B$ is close to theoretical value of 1.73 BM for Cu(II) square planar complexes.

Molar conductance

The results of molar conductance, which were less than $30 \mu\text{S m}^2 \text{mol}^{-1}$ in DMF solution, indicate that all the formed complexes were non-electrolytes, electrically neutral and non-ionic in nature.¹⁸

Thermogravimetric analysis

It was observed that the complexes of Ni(II) and Co(II) show a loss in weight between 150–200 °C, indicating that crystal water molecules and coordinated water molecules are present in the complexes. The TG curves in the 200–310 °C range suggest that the loss in weight for all complexes corresponds to decomposition of the ligand. In the range above 310 °C, the loss in weight corresponded to the remaining organic ligand molecules. In all cases, the remaining residues were the corresponding metal oxide. These results are in accordance with the composition of the complexes.

Catalytic studies

The catalytic activity of the newly synthesized complexes was examined in the presence of NMO as a co-oxidant for the oxidation of primary and secondary alcohols in CH₂Cl₂. The results are presented in Table I. The reaction was initially conducted using molecular oxygen. Results of the present investigation suggest that the complexes are able to react more efficiently with NMO than with molecular oxygen, as indicated by the low product yield when molecular oxygen was employed as the co-oxidant. This is in accordance with a previous observation¹⁹. *N*-Methylmorpholine and water were the by-products during the course of the reactions. An important characteristic of the transition metal–NMO system

TABLE I. Oxidation of alcohols by the synthesised metal(II) complexes

Complex	Substrate	Product	Yield ^a , %	TON ^b
Ni(II) BrSAMT	Benzyl alcohol	Benzaldehyde	85	87
	Cyclohexanol	Cyclohexanone	80	82
	Cinnamyl alcohol	Cinnamaldehyde	88	89
	2-Propanol	Acetone	73	75
	2-Methyl-1-propanol	2-Methylpropanal	76	77
Co(II) BrSAMT	Benzyl alcohol	Benzaldehyde	87	89
	Cyclohexanol	Cyclohexanone	92	93
	Cinnamyl alcohol	Cinnamaldehyde	89	90
	2-Propanol	Acetone	76	78
	2-Methyl-1-propanol	2-Methylpropanal	72	74
Pd(II) BrSAMT	Benzyl alcohol	Benzaldehyde	90	92
	Cyclohexanol	Cyclohexanone	91	92
	Cinnamyl alcohol	Cinnamaldehyde	85	87
	2-Propanol	Acetone	74	76
	2-Methyl-1-propanol	2-Methylpropanal	79	81
Cu(II) BrSAMT	Benzyl alcohol	Benzaldehyde	86	88
	Cyclohexanol	Cyclohexanone	88	89
	Cinnamyl alcohol	Cinnamaldehyde	93	94
	2-Propanol	Acetone	77	78
	2-Methyl-1-propanol	2-Methylpropanal	78	79

^aYield based on substrate; ^bturn-over number, moles of product per mole of catalyst

results in the selective oxidation at the alcoholic group of the unsaturated cinnamyl alcohol to cinnamaldehyde, while the double bond remained unaffected. Saturated aliphatic alcohols, such as 2-propanol and 2-methyl-1-propanol, were converted to the corresponding aldehydes with high conversion.

Biological tests

The Cu(II), Co(II) and Ni(II) complexes of BrSAMT showed a greater zone of inhibition compared to the uncomplexed Schiff base, indicating that these complexes possess more antibacterial activity against pathogenic strains of gram negative bacteria, such as *E. Coli*, *P. aeruginosa*, *S. typhi* and *S. flexneri*.^{20,21} The results are given in Table II.

TABLE II. Antibacterial activity of the synthesised metal(II) complexes (Zone inhibition in cm)

Compound	<i>S. Typhi</i>		<i>P. aeruginosa</i>		<i>E. Coli</i>		<i>S. Flexneri</i>	
	Amount, μg							
	30	50	30	50	30	50	30	50
Ligand	–	0.9	0.8	1.2	0.7	1.2	0.7	0.9
Ni(II) BrSAMT	1.3	1.7	1.4	1.9	1.3	1.8	0.9	1.4
Co(II) BrSAMT	1.0	1.2	0.8	1.1	0.9	1.4	0.8	1.2
Cu(II) BrSAMT	1.2	1.6	1.3	1.8	0.9	1.1	0.9	1.4
Pd(II) BrSAMT	–	–	–	–	–	–	–	–
Nicotinic acid	1.8	2.0	2.4	2.6	2.4	2.7	1.3	1.8
DMF	–	–	–	–	–	–	–	–

CONCLUSIONS

Based on the above characterization data and keeping in mind the preferred geometries, octahedral structures are proposed for the Co(II) and Ni(II) complexes (Fig. 1) and square planar geometries for the Cu(II) and Pd(II) complexes (Fig. 2). The newly synthesized complexes are good catalysts for the oxidation of alcohols to the corresponding carbonyl compounds. The catalytic efficiency was high when NMO was employed as the co-oxidant. The Cu(II), Ni(II) and Co(II) complexes exhibited antibacterial activity against gram-negative bacteria, such as *E. Coli*, *P. aeruginosa*, *S. typhi* and *S. flexneri*. Further studies on the catalytic and biological applications are in progress.

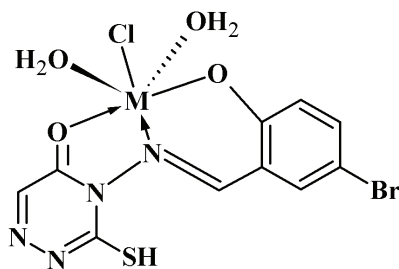


Fig. 1. Proposed structure for the Ni(II) and Co(II) complexes.

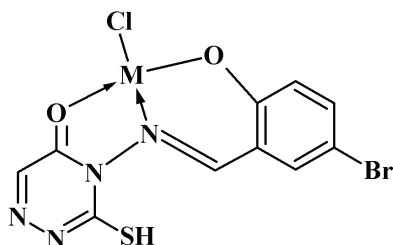


Fig. 2. Proposed structure for the Cu(II) and Pd(II) complexes.

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ИЗВОД

КОМПЛЕКСИ ПРЕЛАЗНИХ МЕТАЛА СА 5-БРОМОСАЛИЦИЛИДЕН-4-АМИНО-3-МЕРКАПТО-1,2,4-ТРИАЗИН-5-ОН ЛИГАНДОМ: СИНТЕЗА, КАРАКТЕРИЗАЦИЈА, КАТАЛИТИЧКА И АНТИБАКТЕРИЈСКА ИСПИТИВАЊА

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Описана је синтеза, физичко-хемијска и спектроскопска карактеризација комплекса Cu(II), Ni(II), Co(II) и Pd(II) са 5-бромосалицилиден-4-амино-3-меркапто-1,2,4-тризин-5-он лигандом. Нађено је да су сви добивени комплекси ML-типа. На основу аналитичких података, IR спектроскопије и магнетних мерења закључено је да комплекси Co(II) и Ni(II) имају октаедарску, а комплекси Cu (II) и Pd(II) квадратно-планарне геометрију. Помоћу *N*-метилформолин-*N*-оксида и молекулског кисеоника као оксиданаса, у присуству наведених комплекса као каталитичких агенаса, испитивана је оксидација бензил-алкохола, циклохексанола, цинамил алкохола, 2-пропанола и изобутил-алкохола. Нађено је да неки од добијених комплекса показују антибактеријске особине.

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