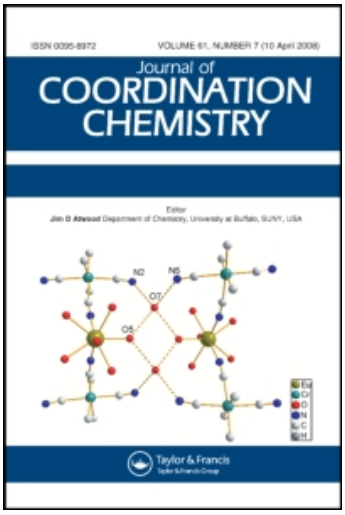

Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713455674

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To cite this Article Sharma, Sangita , Patel, Rasmikant A. , Vora, Jabali and Joshi, J. D.(1999) 'ANTIMICROBIAL ACTIVITY OF CADMIUM(II) CHELATES WITH 2, 2'-BIPYRIDYLAMINE AND PHENOLS', Journal of Coordination Chemistry, 49: 1, 63 — 67
To link to this Article: DOI: 10.1080/00958979908024374
URL: http://dx.doi.org/10.1080/00958979908024374

J. Coord. Chem., 1999, Vol. 49, pp. 63–67
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ANTIMICROBIAL ACTIVITY OF CADMIUM(II) CHELATES WITH 2,2′-BIPYRIDYLAMINE AND PHENOLS

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(Received 4 September 1998; Revised 4 December 1998; In final form 31 March 1999)

The present paper deals with the isolation of mixed ligand chelates of the type CdAL, where A = 2,2′-bipyridylamine and L = phenols such as catechol (catecholato-2) pyrogallol (pyrogallolato-3), 2,3-dihydroxy naphthalene (2,3-dihydroxy naphthalato-2) or protocatechuic acid (protocatechuato-3). The antimicrobial activity of 2,2′-bipyridylamine and the CdAL mixed ligand chelates is described.

Keywords: Cadmium(II); 2,2′-bipyridylamine; phenols; chelates; antimicrobials

INTRODUCTION

The formation and antimicrobial activity of Nickel(II), Copper(II) and Zinc(II) chelates with 2,2′-bipyridylamine and phenols has been studied.[1] The formation constants of the mixed ligand systems (MAL), where A = 2,2′-bipyridyl or 2,2′-bipyridylamine and L = amino acids or polyhydroxy phenols have been studied by earlier workers.[2–6] Dutta and De[7] isolated solid mixed ligand complexes of Cu(II) containing 2,2′-bipyridyl or phenanthroline as primary ligand and glycine or alanine as secondary ligands. Synthesis, characterization and antimicrobial activities of Nickel(II), Zinc(II) and Cadmium(II) with 2,2′-bipyridylamine and thio acids as

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secondary ligands have been studied.[8] Binary and ternary complexes of Cobalt(II) and Nickel(II) with 2,2′,2″-terpyridine as primary ligand and amino acids as secondary ligands have been studied.[9]

In the present communication, the isolation of heterochelates of Cd(II) with 2,2′-bipyridylamine (A) and phenols such as catecholato(-2), pyrogallolato(-3), 2,3-dihydroxy naphthalato(-2) or protocatechuato(-3) is reported in water:ethanol mixtures. The primary ligand 2,2′-bipyridylamine and the metal chelates have been tested for antimicrobial activity.

EXPERIMENTAL

Materials

Catechol, pyrogallol, 2,3-dihydroxy naphthalene and protocatechuic acid were of BDH Analar grade. 2,2′-bipyridylamine was obtained from Fluka and sodium hydroxide from E. Merck. A stock solution of Cd(II) perchlorate was prepared and standardized by the complexometric method.[10] Conductivity water was used in the synthesis of complexes.

Isolation, Analysis and Physical Measurements

The isolation of complexes has been carried out as reported earlier.[1] Elemental analyses were performed with a Coleman CHN analyzer. The metal content was determined[10] by titration with EDTA after decomposing the chelates with a mixture of concentrated nitric acid, perchloric acid and sulfuric acid. Magnetic susceptibilities were measured by the Gouy method. The IR spectra have been recorded on a Perkin-Elmer-983 spectrophotometer as KBr pellets. The UV–visible reflectance spectra were measured on Beckman DK-2A spectrophotometer. Thermogravimetric analysis was carried out using a DuPont Thermal Analyzer.

RESULTS AND DISCUSSION

Analytical data for the complexes indicated 1:1:1 stoichiometry. The decomposition points of all complexes are higher than 270°C. All complexes are non-conducting and diamagnetic as expected. The IR spectra indicate bands corresponding to Cd–N and Cd–O vibrations. All the chelates are partially soluble in ethanol and DMSO but practically insoluble in water

and other common organic solvents. The UV–visible spectra of complexes showed bands due to LMCT transitions and also ligand $n \rightarrow \pi*$ and $\pi \rightarrow \pi*$ transitions or their merger.[11] Thermal analysis showed the presence of water of crystallization, stability of chelates up to 200°C and single-stage thermal decomposition.[12]

Antimicrobial Activity

Bacterial stains (*P. fluorescens*), fungal stains (*A. niger*) and (*R. minuta*) were tested with the ligand and its metal chelates. The effect of the compounds in the growth media were investigated by standard microbiological parameters. The concentration of the compounds was kept at 500 ppm during the experiments. The bacterial culture was maintained on N-agar (N-broth 3% w/v agar). The fungi culture was maintained on PDA 25% (w/v) potato extract, 2% (w/v) dextrose in 3% (w/v) agar agar. The yeast culture was maintained on MGYP in 3% (w/v) agar agar, malt extract 0.3% (w/v), glucose 1.0% (w/v) yeast extract 0.3% (w/v) and peptone 0.5% (w/v) in distilled water and then the pH was adjusted to 6.8. All were subcultured every fortnight and stored at 0–5°C.

Media Composition

For the growth and test of bacteria, fungi and yeast, N-broth, Sabourauds dextrose broth and MGYP media are used, respectively. The composition used is as shown below.

N. broth Peptone 1% (10.0 g) NaCl 0.5% (5.0 g) KH_2PO_4 (2.5 g) and Beef extract 0.3% (3.0 g) were dissolved in 1 L distilled water and the pH was adjusted to 7.2.

Sabouraud's dextrose broth Dextrose (15 g) and peptone (10 g) were dissolved in 1 L distilled water and the pH was adjusted at 5.5.

MGYP Malt extract (3.0 g); glucose (10.0 g) Yeast extract (3.0 g) and peptone (5.0 g) were dissolved in 1 L distilled water and pH was adjusted to 5.5.

Inoculum Preparation

Bacterial and yeast cultures A loopful of cells from pregrown slants was inoculated into sterile N-broth tubes containing 15 mL medium and incubated at 37°C for 24 h to get sufficient cell density (i.e. 1×10^8 cells/mL).

Fungal cultures Well sporulated slants of a fungal culture were used for the preparation of spore suspensions. About 5.0 mL sterile distilled water

containing a few drops of tween 80 solution, was added to the slants and growth was scraped with a sterile nichrome wireloop and collected in a sterile tube. The tween 80 solution acts as a surfactant for better fungal growth. The spore suspension thus obtained was inoculated in the inoculum medium as 5% (v/v) solution and incubated at room temperature on a rotary shaker (200 rpm) for 48 h.

Screening of Compounds for Microbial Activity

The effect of ligands and their metal chelates on microorganisms was judged by the growth parameters for bacterial, fungal and yeast cultures.

Bacterial and yeast cultures Five percent (v/v) inoculum was used to inoculate N-broth (control) and test media (N-broth + 500 ppm compound) and incubated on a rotary shaker (200 rpm) at room temperature. Samples were withdrawn after 24, 48 and 72 h from control and test media. After suitable dilution the optical density per mL was measured at 660 nm. The method is based on the principle that as the growth proceeds, cell number increases which leads to an increase in the optical density of the medium.

Fungal cultures Since fungal cultures show filamentous growth (pellet), their distribution is non-homogenous in the medium, hence an optical method cannot be used to monitor their growth. As a result, gravimetric analysis was carried out to determine dry cell mass.

Ten percent (v/v) inoculum was added to the sterile control medium (without compound) and test medium (control medium + 500 ppm compound). Flasks were incubated at room temperature on a rotary shaker (200 rpm) for 48 h. The contents of the flasks were filtered using cheese cloth and cell pellets were dried to constant weight.

Effects of metal chelates and ligand on the growth of microorganisms selected are presented in Table I. In order to understand the role of the uncomplexed metal ion, a solution of Cd(II) perchlorate having concentration equivalent to that present in 500 ppm chelate was separately tested. This solution showed significant inhibition of growth of all test organisms. The primary ligand 2,2′-bipyridylamine alone showed very high inhibitory activity with only 16–18% growth compared with the control. The chelates of Cd(II) showed moderate activity i.e. (17–53%) growth against *P. fluorescens* and *A. niger*. In case of *R. minuta*, the metal chelates, similar to those of Ni(II), Zn(II), Cu(II) in Ref. [1], did not show much inhibitory activity. Amongst these, protocatechuic acid showed some interesting contrasts. Against *A. niger* [Cd · A · Protocatechuic acid], H_2O showed inhibition almost as high as that shown by the primary ligand 2,2′-bipyridyl

TABLE I Effect of primary ligand 2,2′-bipyridylamine* and its metal chelates on the growth‡ of microorganisms

Compound	*Bacteria P. fluorescens*		*Fungi A. niger*		*Yeast R. minuta*	
	OD† (660 nm)	% *Growth*	*Weight* (g)	% *Growth*	*OD*† (660 nm)	% *Growth*
A	0.150	16.40	0.072	17.42	0.094	16.75
[Cd · A · Catechol], H_2O	0.325	27.08	0.089	41.98	1.001	75.43
[Cd · A · Pyrogallol], H_2O	0.450	37.50	0.112	52.83	1.093	82.36
[Cd · A · 2,3-dihydroxy naphthalene], H_2O	0.443	38.08	0.096	45.24	1.121	84.47
[Cd · A · Protocatechuicacid], H_2O	0.601	50.08	0.038	17.92	1.225	92.31

*A = 2,2′-bipyridylamine, †OD = Optical density, ‡Growth was compared with control and is expressed as %.

amine (18% growth). In the previous work,[1] [Zn · A · Protocatechuic acid] H_2O showed 109% growth of *R. minuta*, indicating the possibility of biodegradation. Thus it may act as a nutrient for *R. minuta*. The investigation of newer chelates of protocatechuic acid is valuable for biochemical reactions. Overall trends suggest that coordination of the ligands cause reduction in antimicrobial action except in a few cases. Phenols in the ternary chelate system have shown trends toward growth acceleration and growth inhibition. Thus further study of different combinations of metal ions and primary ligands with phenols may provide promising and useful outcomes.

Acknowledgements

The authors thank Head, Department of Chemistry, Sardar Patel University, Vallabh Vidyanagar, for providing facilities.

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