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Palladium(II) and zinc(II) complexes of neutral $[N_2O_2]$ donor Schiff bases derived from furfuraldehyde: Synthesis, characterization, fluorescence and corrosion inhibitors of ligands



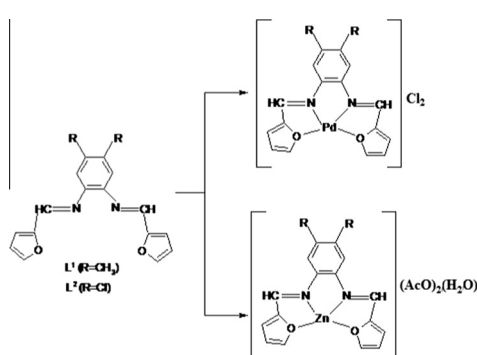
Omyma A.M. Ali*

Chemistry Department, Faculty of Women for Arts, Science and Education, Ain Shams University, Cairo, Egypt

HIGHLIGHTS

- The complexes are characterized by different spectroscopic techniques.
- The Schiff base ligands exhibited efficient corrosion inhibitors.
- Zn(II) complexes may have potential uses as a Zn^{2+} sensor.
- The ligands and Zn(II) complexes exhibited strong fluorescence.

GRAPHICAL ABSTRACT



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ABSTRACT

Metal complexes of Schiff bases derived from furfuraldehyde and 4,5-dimethyl-1,2-phenyldiamine (L^1) or 4,5-dichloro-1,2-phenyldiamine (L^2) have been reported and characterized based on elemental analyses, IR, 1H NMR, UV–Vis, magnetic moment, molar conductance and thermal analysis. The complexes are found to have the formulae $[PdL^{1-2}]Cl_2$ and $[ZnL^{1-2}](AcO)_2 \cdot H_2O$. The molar conductance data reveal that Pd(II) and Zn(II) chelates are ionic in nature and are of the type 2:1 electrolytes. The spectral data are consistent with a square planar and tetrahedral geometry around Pd(II) and Zn(II), respectively, in which the ligands act as tetradentate ligands. The thermal behavior of some chelates is studied and the activation thermodynamic parameters are calculated using Coats–Redfern method. The corrosion inhibition of stainless steel types 410 and 304 in 1 M HCl using the synthesized Schiff bases as inhibitors have been studied by weight loss method. The obtained data considered these ligands as efficient corrosion inhibitors. The ligands and their metal complexes exhibited considerable antibacterial activity against *Staphylococcus aureus*, and *Escherichia coli* and antifungal activity against *Candida albicans*.

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Introduction

The Schiff bases are considered a very important class of ligands, which form complexes with many metals. These complexes have wide applications in some biological aspects [1–4],

* Tel.: +20 22344032.

E-mail address: omaymaahmed92@yahoo.com

analytical fields [5], organic catalysis [6], oxygen carriers [7] and as corrosion inhibitors in especially acidic environments for various alloys and metals [8–10]. Schiff base complexes derived from heterocyclic compounds have found an increased interest in the context of bioinorganic chemistry [11,12]. Various heterocyclic Schiff bases and their complexes having nitrogen and also oxygen donor atoms, have been reported by several scientists [13–15]. The furan ring, as a part in ligand, has been studied and has attracted

great attention for a long time and significant progress has been made in understanding the structure of its complexes [13,16,17]. Complexes of some transition metal ions with Schiff bases derived from 2-furancarboxaldehyde and o-phenylenediamine [18–20], hydrazine hydrate [21], 3,3'-diaminobenzidine [22], 3-aminodibenzofuran [23] and o-tolidine [24] were studied. Furfuraldehyde Schiff bases were found to show the selectivity towards anions and can act as anion-sensitive membrane electrodes [25], they also possess the nitrification inhibitory properties [26]. Moreover, luminescent compounds are attracting much current research interest because of their many applications including emitting materials for organic light emitting diodes, light harvesting materials for photocatalysis and fluorescent sensors for organic or inorganic analytes [27–29]. Indeed, Schiff bases derived from aromatic diamines have received much less attention and are considered. Here we report, the synthesis and characterization of heterocyclic Schiff base ligands, N,N'-bis-(2-furancarboxaldimine)-4,5-dimethyl-1,2-phenylenediamine (L^1), and N,N'-bis-(2-furancarboxaldimine)-4,5-dichloro-1,2-phenylenediamine (L^2) and their complexes $[M(N,N'\text{-bis-(2-furancarboxaldimine)-4,5-dimethyl-1,2-phenylenediamine})Cl_2]$ and $[M(N,N'\text{-bis-(2-furancarboxaldimine)-4,5-dichloro-1,2-phenylenediamine})(AcO)_2 \cdot H_2O]$ ($M = Zn$ and Pd).

Experimental

Materials and instrumentation

$PdCl_2$, $Zn(CH_3COO)_2 \cdot 2H_2O$, 4,5-dimethyl-1,2-phenylenediamine and 4,5-dichloro-1,2-phenylenediamine and furfuraldehyde were supplied from Aldrich. All solvents were of analytical grade. IR measurements (KBr pellets) were carried out on a Shimadzu 8000 FT-IR spectrometer. Magnetic susceptibilities of the chelates were measured at room temperature using a magnetic susceptibility Cambridge England Sherwood Scientific. NMR measurements were performed on a Varian-Mercury 300 MHz spectrometer. Samples were dissolved in $(CD_3)_2SO$ with TMS as internal reference. Mass spectra of the solid complexes (70 eV, EI) were carried out on a Finnigan MAT SSQ 7000 spectrometer. Thermogravimetric analyses (TG and DTG) were carried out under N_2 atmosphere with a heating rate of $10^\circ C/min$. using a Shimadzu DT-50 thermal analyzer. Microanalyses were performed using JEOL JMS-AX500 elemental analyzer. All conductivity measurements were performed in DMF ($1 \times 10^{-3} M$) at $25^\circ C$, by using Jenway 4010 conductivity meter. Ultraviolet spectra were recorded using a Shimadzu UV 1800 Spectrophotometer in the range of 200–800 nm. The photoluminescent properties of all compounds were studied using a Jenway 6270 Fluorimeter.

Preparation of Schiff base ligands

The ligands were obtained from the condensation of furfuraldehyde and 4,5-dimethyl-1,2-phenylenediamine or 4,5-dichloro-1,2-phenylenediamine in 2:1 M ratio, in ethanol for 4 h. The yellow-orange precipitates obtained were filtered, washed with ethanol and dried in a vacuum. Recrystallization from ethanol afforded pure Schiff bases. The infrared and elemental analysis data of the obtained products were consistent very well with their formulae.

Preparation of Pd(II) complexes

A solution of $[PdCl_2]$ (0.5 mmol) in ethanol (10 ml) was added to the solution of ligand (0.5 mmol) in ethanol (10 ml) while stirring. The resulting mixture was allowed to reflux on a water bath for 2 h in the presence of a few drops of concentrated HCl. The formed

solid products were filtered, washing with ethanol and dried in vacuum desiccators. The complexes were purified by crystallization from ethanol.

Preparation of Zn(II) complexes

A solution of hydrated zinc acetate (1.0 mmol) in methanol (10 mL) was mixed with methanolic solution (10 mL) of each ligand (1.0 mmol) and the mixture was left under reflux for 3 h. The product was filtered off, washed with cold methanol and hot petroleum ether, recrystallized from ethanol and finally dried under vacuum.

Corrosion test

Gravimetric corrosion measurements (weight loss method) were carried out according to the ASTM standard procedure described in [30]. In brief, stainless steel specimens in triplicate were immersed for a period of 4 h in 100 ml 1 M HCl containing various concentrations of the studied inhibitors. The mass of the specimens before and after immersion was determined using an analytical balance accurate to 0.1 mg. Before measurements, the specimens were abraded with a sequence of emery papers of different grades (400, 800, 1000 and 1200), followed by washing with double distilled water and finally degreased with ethanol and dried at room temperature. The composition of the stainless steel types 410 and 304 are in wt.% 0.15C, 11Cr, 75Fe, 1Mn, 0.75Ni, 0.04P, 0.03S, 1Si and 0.08C, 18Cr, 66Fe, 2Mn, 8Ni, 0.04P, 0.03S, 1Si, respectively.

Antimicrobial activity

The in vitro activity of ligands and their metal complexes were tested against the bacterial species *Staphylococcus aureus* and *Escherichia coli* in Mueller Hinton-Agar medium. The antifungal activity was tested against the fungi *Aspergillus flavus* and *Candida albicans* cultured on YPD-agar medium. The test compounds were dissolved in DMSO at concentration 2 mg/mL. Antimicrobial activities of each compound were evaluated by the disc-diffusion method. The well (8 mm diameter) was then filled with the test solution and the plates were inoculated at $37^\circ C$ for 48 h (for bacteria) and $30^\circ C$ for 72 h (for fungi). During this period, the growth of the inoculated microorganisms was affected and then the inhibition zones developed on the plates were measured. The effectiveness of an antimicrobial agent was assessed by measuring the zones of inhibition around the well. The diameter of the zone is measured to the nearest millimeter (mm). The antibacterial activity of each compound was compared with that of standard antibiotics such as Streptomycin. The antifungal activity of the test compound was compared that of Chlorometazole as standard antifungal. DMSO was used as a control under the same conditions for each organism and no activity was found. The activity results were calculated as a mean of triplicates.

Results and discussion

The complexes $[PdL^1]Cl_2$, $[PdL^2]Cl_2$, $[ZnL^1](AcO)_2 \cdot H_2O$, $[ZnL^2](AcO)_2 \cdot H_2O$ were synthesized by the reaction of ligands and metal salt in 1:1 M ratio. The results of the elemental analyses of the complexes, which are recorded in Table 1, are in good agreement with those required by the proposed formulae. All complexes were stable at room temperature. The molar conductivity data for 1 mM solutions of complexes (Table 1) suggest that these chelates are ionic in nature and they are of the type 2:1 electrolytes. All the prepared complexes are found in diamagnetic character. The solid

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