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Research paper

Synthesis, characterization, structural study and antibacterial activity of the Schiff bases derived from sulfanilamides and related copper(II) complexes

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ABSTRACT

In the present work, a series of new Schiff base ligands from acetylacetone and salicylaldehyde with sulfonamides derivatives were synthesized and characterized by spectral and analytical techniques. The molecular structures of ligands, *N*-({4-[(E)-(2-hydroxybenzylidene)amino]phenyl}sulfonyle)acetamide (**HL^1**), *N*-({4-[(E)-(2-hydroxybenxiliden)amino]phenyl}sulfonyle)benzamide (**HL^2**) and 4-((4-oxopentan-2ylidene)amino)benzenesulfonamide (**HL^3**) were also determined by the method of X-ray diffraction. Also, new copper(II) complexes [CuL^x](x = 1 for **1**, x = 2 for **2**, x = 3 for **3**) constructed from HL^x were synthesized and characterized by FT-IR, UV–vis spectroscopy and XRD. The copper(II) complexes were screened for their antimicrobial activities against the selected bacteria and compared to the free ligands, using the disc diffusion method.

1. Introduction

Sulfonamide derived Schiff bases are potential class of compounds, which have been found to possess a wide range of medicinal properties [1]. Schiff bases of aromatic or heterocyclic sulfonamides have been used as anti-infectious agents [2,3] and have led to potent inhibitors [4] of several physiologically relevant CA isozymes [5]. They are a potential class of compounds which have been found to possess a wide range of medicinal properties [6,7]. Systems containing both azomethine and sulfanilamide groups are related to biological and pharmaceutical class of compounds which display anticancer [8], antifungal [9], antitubercular [10], diuretic [11], anti-inflammatory [12], antimicrobial [13–15] and several other activities [16,17]. The coordination chemistry of sulphonamide imine complexes has undergone noticeable development in recent years, due to their interesting properties. Some drugs, like sulfamethoxazole, prevent the formation of dihydrofolic acid, a compound synthesized by bacteria for their survival [18,19]. However, not all synthesis of these complexes with different ligands have been reported. There exist only few works on the structure and the applications of sulfanilamide ligands and their related complexes, so, this facilitated the present study [20–23]. In this work, we describe the characterization and applications, as well as the structures of new such ligands. Additionally, the related complexes have been investigated as well. Therefore, the objectives include the synthesis of some new sulfanilamide Schiff base ligands obtained by the condensation of ketone and aldehyde derivations with sulfonamides derivatives, and related copper complexes (Scheme 1). The molecular structures of ligands were determined by X-ray diffraction method and complexes by XRD. The Schiff Bases and the related copper complexes were also tested for their antibacterial activities.

2. Experimental

2.1. Reagents and measurements

All solvents and chemicals were used as received. ^{1}H NMR spectra were obtained on a BRUKER AVANCE DR X400 (400 MHz) spectrometer, using DMSO solvent; FT-IR spectra were obtained as KBr plates using a Bruker FT-IR instrument and UVVis spectra were

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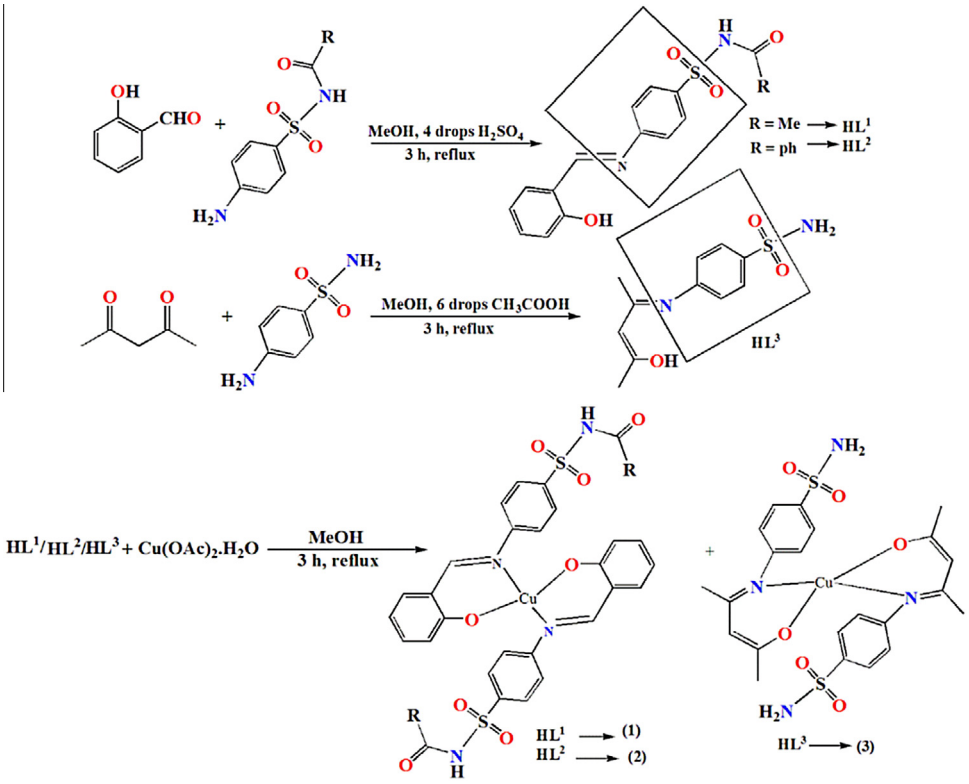

Scheme 1. Syntheses of the Schiff base ligands and related complexes.

obtained on a Shimadzu UV-1650PC spectrophotometer. Melting point was measured by a THERMO CIENTEFIC apparatus.

2.2. Procedure for synthesis of Schiff's bases

2.2.1. Synthesis of HL^1

The Schiff base HL^1 was synthesized with a method different from the one in the literature [24], by refluxing sulfacetamide (1 mmol, 0.214 g) and salicylaldehyde (1 mmol, 0.122 g) in about 15 ml of methanol for 3 h at ca.70 °C, in the presence of 4–5 drops of conc. H_2SO_4. The solid product was collected through filtration and finally air dried. Yield: 58%. M.P. = 215 °C. Anal. Calcd. for $C_{15}H_{14}N_2O_4S$ (%):C, 56.59; H, 4.43; N, 8.80. Found: C, 56.42; H, 4.25; N, 8.75. FT-IR (KBr, cm^{-1}): ν_{max} cm^{-1} (KBr):1645 (s, C=N), 1715 (C=O), 1575, 1616 ($C=C_{aromatic}$), 1093, 1157 (SO_2) 3340 (N—H). UV–vis: λ_{max} (nm) (ε, M^{-1} cm^{-1}) (DMSO-d_6): 350 (40,000), 320 (41,000). ^{1}H NMR (DMSO-d_6, 400 MHz):6–8 (m, 8 H), 9 (s, 1H), 10 (s, 1H), 3 (s, 3H), 11 (s, 1H).

2.2.2. Synthesis of HL^2

HL^2 was synthesized following the same procedure as described for HL^1 except sulfabenzamide was used instead of sulfacetamide. The precipitated ligand was filtered and air dried. The purity of the ligand was checked by thin layered chromatography (TLC) and melting point determination method. Yield: 60%. M.P. = 243 °C. Anal. Calcd. for $C_{20}H_{16}N_2O_4S$ (%):C, 63.14; H, 4.24; N, 7.36. Found: C, 62.52; H, 4.15; N, 7.20. FT-IR: ν_{max} cm^{-1} (KBr):1618 (s, C=N), 1699 (C=O), 1575, 1591 ($C=C_{aromatic}$), 1064, 1164 (SO_2) 3228 (N—H). UV–vis: λ_{max} (nm) (ε, M^{-1} cm^{-1}) (DMSO): 364 (24,000), 326 (27,000). ^{1}H NMR (DMSO-d_6, 400 MHz):7–8 (m, 13H), 9 (s, 1H), 12 (s, 1H), 11 (s, 1H).

2.2.3. Synthesis of HL^3

The Sulphonamide imine Schiff base ligand (HL^3) was prepared by refluxing sulphanilamide (1 mmol, 0.164 g) and acetyl acetone (1 mmol, 0.100 g) in about 15 ml of ethanol for 3 h in the presence of 1–2 drops of CH_3COOH. The precipitated light yellow colored ligand was filtered and air dried. Yield: 45%. M.P. = 210 °C. Anal. Calcd. For $C_{11}H_4N_2O_3S$ (%): C, C, 51.95; H, 5.55; N, 11.02. Found: C, 51.65; H, 5.35; N, 10.95. FT-IR: ν_{max} cm^{-1}(KBr): 1595 (s, C=N), 1625 (C=O), 3128, 3224 (NH_2), 1516, 1566 ($C=C_{arom}$),1163, 1195 (SO_2)., UV–vis: λ_{max} (nm) (ε, M^{-1} cm^{-1}) (DMSO-d_6): 340 (91,000), 330 (22,000). ^{1}H NMR (DMSO-d_6, 400 MHz):12.67 (s, 1H), 7–8.7 (d-d, 4H), 5.5 (s, 1H), 2.8 (s, 6H), 3.3 (s, 2H).

2.3. Procedure for synthesis of complexes (*1–3*)

All the copper(II) complexes were synthesized by the same general procedure from the literature [26]. The appropriate quantity of Schiff base ligand HL^1 (2 mmol, 0.636 g,), HL^2 (2 mmol, 0.76.g) and HL^3 (2 mmol, 0,50 g) were dissolved in 20 ml methanol. These ligands solutions were added dropwise as separation to a 10 ml methanolic solution of $Cu(OAC)_2{\cdot}H_2O$ (1 mmol, 0.199 g). After addition, the reaction mixture was refluxed for 3 h. The metal complexes of Schiff base ligands were separated from the reaction mixture as amorphous solids and washed several times with MeOH, followed by drying under reduced pressure at room temperature. The dark brown (**1**), light green (**2**) and green solid complexes (**3**) were, respectively prepared in a similar manner as described. The precipitate was filtered, washed with methanol and dried under vacuum.

Data analytics for **1**: Yield: 65%. M.P. = 400 °C. Anal. Calcd. for $C_{30}H_{26}CuN_4O_8S_2$, (%): C, 51.61; H, 3.75; N, 8.02. Found: C, 51.05; H, 3.15; N, 7.95. FT-IR: ν_{max} cm^{-1} (KBr): 1587 (s, C=N), 1608 (C=O), 3274 (N—H), 1444, 1533 ($C=C_{arom}$), 1164, 1151 (SO_2).