Chemistry Research Journal, 2021, 6(4):111-115

Available online www.chemrj.org

Research Article

ISSN: 2455-8990
CODEN(USA): CRJHA5

Synthesis, Characterization and Antimicrobial Activity of Ni(II)ternary complex of sulfaquinoxaline

Abeer A. Hamed[1*], Zeinab H. Abd El –Wahab[2], Abeer A. Faheim[2], Aida A. Salman[2]

[1]Ministry of Industry, Trade, Small and Medium Enterprises. Industrial Control Authority. Ext. Ramses St. - 6th District – Nasr City.
[2]Chemistry Department, Faculty of Science, Al-Azhar University (Girls branch), Youssif Abbas St., Nasr-City, Cairo, Egypt, P.O. Box 11754.
*Corresponding author email: Abeer1973@yahoo.com

Abstract A ternary complex of sulfaquinoxaline, SQ as primary ligand and *o*-hydroxyacetophenone, HL as secondary ligand is prepared and characterized by elemental analysis, molar conductivity, IR, UV–vis spectroscopy and thermogravimetric analysis. The results reveal that sulfaquinoxaline, SQ coordinates with Ni(II) in bidentate mode through priazine nitrogen atom and secondary amine nitrogen atom. The supporting ligand (HL) uses its two O atoms in coordination with metal ions in bidentate mode. The complex is screened for antibacterial activity against tow gram positive bacteria as antibacterial agents.

Keywords antimicrobial activity, thermogravimetric analysis, UV–vis Spectroscopy, Ternary complexes, sulfaquinoxaline

1. Introduction

Sulfonamides have been used as synthetic antimicrobials since 1937 and are the oldest and most widely employed antimicrobial agents in veteran ary medicine because of their low cost and relatively high efficacy for treating bacterial diseases [1, 2]. Sulfonamides, a series of drugs containing the chemical structure of sulfanillic amide, are one of the most widely administered groups of antibiotics in animal husbandry as preventive and therapeutic agents for bacterial infective diseases [3]. Sulfaquinoxaline is a widely used antimicrobial agent of the sulfonamide class used in veterinary treatment to prevent coccidiosis and bacterial infections [4]. Mixed ligand complexes characterized by the sense that they are having at least two different kinds of ligands associated with the same metal ion in a complex.

The presence of more than one type of ligand in a complex increases chances of variation in properties expected for the complex [5]. The mixed ligand complexes have been used for various essential biological, chemical activities act as an active anti-inflammatory, antimicrobial, antioxidant, antibiotic, also an active catalyst in reactions of hydrogenation and oxidation [6]. In view of these findings, in the work reported here, we synthesized novel mixed ligand complexes based on sulfaquinoxaline and*o*-hydroxy acetophenone. The synthesized complexes were characterized using different techniques; in addition, they were screened for in vitro antimicrobial activity.

2. Experimental part

2.1. Materials and physical measurements

All chemical used in the present work were of highest purity available. $NiCl_2.6H_2O$, *o*-hydroxyacetophenone, are merck, 4-Amino-*N*-2-quinoxalinyl-benzenesulfonamide) (Known as sulfaquinoxaline, abbreviated SQis sigma, DME , diethyl ether and silver nitrate were analytical grade chemical obtained from Merck chemical company.. All materials and solvents were used without further purification.

The elemental analyses of carbon, hydrogen, nitrogen and sulphur were performed using a Perkin-Elmer CHN 2408 CHN elemental analyzer at Micro Analytical Center, Cairo University, Giza, Egypt. The infrared spectra of the ligands and the isolated solid complexes were recorded using KBr discs on a Perkin-Elmer 437 IR spectrophotometer (400-4000 cm^{-1}) at Micro Analytical Center, Cairo University, Giza, Egypt. The ultraviolet and visible spectra were measured at room temperature in the UV-Vis range (200-800 nm) using Shimadzu 3101 PC UV-VIS-NIR made in Japan at Cairo University. The thermogravimetric analyzer TGA-50 SHIMA VZU and DTA, TA50 shimadzu, were used to record simultaneously the TGA curves, at Micro Analytical Center, Cairo University, Giza, Egypt. Magnetic susceptibilities of complexes in solid state were measured by the Gouy method at room temperature using a Johnson Matthey, Alfa product, model MKI magnetic susceptibility balance at Micro Analytical Center, Cairo University, Giza, Egypt.

2.2. Preparation of metal complexes

An ethanoic solution (20 ml) of the nickel chloride salt (0.01 mol) was added dropwise tto a mixture of sulfaquinoxaline (SQ) (0.01 mol) and 2-Hydroxyacetophenone (0.01 mol). The resulting mixture was stirred under reflux for 4hrs on a water bath. The isolated product washed with DMF and diethyl ether then dried in a desiccator over anhydrous $CaCl_2$. These complexes are soluble in DMF and DMSO while insoluble in most organic solvents.

2.2.1. Antimicrobial activity

To evaluate the antimicrobial activity of the investigated compounds; SQ and mixed ligand complexes, a variety of bacterial and fungal species were chosen as follows: two bacterial species ie, *Staphylococcus aureus* (RCMB010010) and *Bacillus subtilis* (RCMB015(1)NRRL B-543 (Gram-positive bacteria); In addition, *Gentamycin* (4µg/mL)was used as standard antibacterial agent. Tow fungi

3. Results and discussion

The prepared ternary complex of Ni(II)was found to be soluble in common organic solvents such as chloroform, DMF, DMSO and insoluble in ethanol, methanol and water.

3.1. Elemental analysis

Microanalysis data obtained for the metal complex confirmed that the complex is mononuclear complex with ligand to metal ratio 1:1:1. The elemental analysis data were found to be in good agreement with the proposed molecular formula (Table 1).

3.2. Conductivity measurements

Molar conductivity measurements of 10^{-3}M concentration of the synthesized mixed ligand metal complex was 18 $\Omega^{-1}cm^2mol^{-1}$. Theis value confirmed the nonelectrolytic nature of the metal complex [7].

Table 1: Analytical and some physical data of the synthesized metal complexes

Metal complexes	**M.Wt. Found (Calcd)**	**Meting point ºC**	**Elemental analysis, Found / (Calcd.) %**						**Λ**
			C	**H**	**S**	**N**	**Cl**	**M**	
$[Ni(HL)(SQ)Cl_2].H_2O$	585.48	230	44.65	3.92	7.77	16.01	11.68	9.02	0.10
$NiC_{22}H_{20}N_4SO_4Cl_2.H_2O$	(584.09)		(45.23)	(3.80)	(5.49)	(9.59)	(12.14)	10.05)	

3.3. IR spectral studies

The main vibration frequencies of the SQ ligand in comparison with its nickel metal complex, Figure (1) showed a sharp band of O=S=O stretching vibration of sulfaquinoxaline is assigned at 1303 cm^{-1} but for Ni(II) complex is assigned at 1303 cm^{-1} range that suggesting that there is no ligand coordination with the metal ions occurs through the oxygen of sulfonyl group. The sharp band of N-H stretching vibration of secondary NH group of sulfaquinoxaline appears at 2865 cm^{-1} while for the complex appears at 2805 cm^{-1} for this large shift mean that there is metal ligand coordination occurred through nitrogen of secondary amino group. The band at 1653 cm^{-1} corresponding to stretching vibration of C=O of acetophenone while Ni(II) complex show this band at1504 cm^{-1}so the shifting is significant so there is a metal ligand coordination through acetophenone carbonyl oxygen atom. The vibrational frequencies due to acetophenone phenolic ―OH group was shifted to lower frequencies in Ni(II) complexes at 3358 cm^{-1} suggesting the involvement of O–H group in coordination [8]. The vibration of pyrazine nucleus, υ(N=C) shifts to lower frequency in the complex with respect to the free ligand, which is suggestive of an interaction with the N-heterocyclic atoms [9].

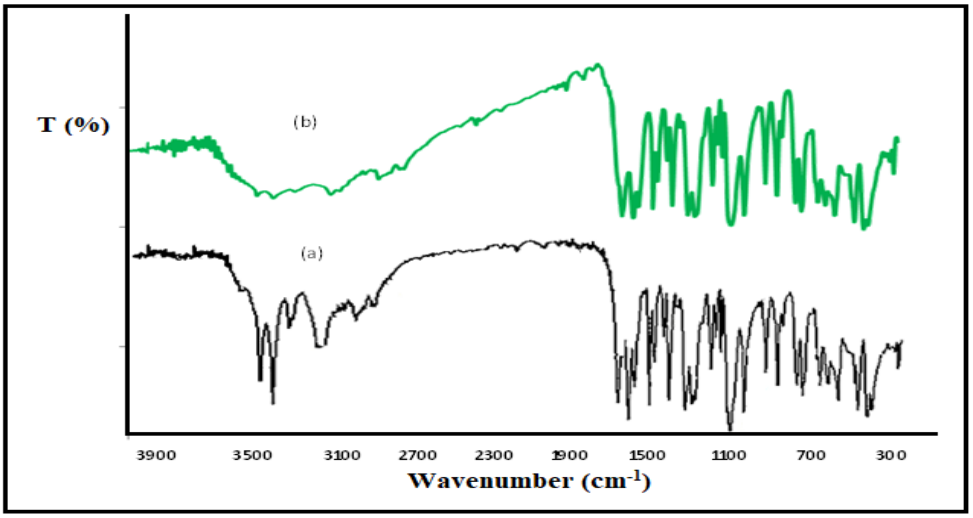

Figure 1: IR spectra of: (a) SQ ligand (b) Ni(II)Complex

3.4. Electronic spectral analysis and Magnetic properties

The electronic spectral measurements of SQ ligand, showed two bands at 225 and 300 nm. The higher energy band is assigned to $\pi–\pi^*$ transitions, while the lower energy band is assigned to $n–\pi^*$ transitions of the SQ ligand.

Upon interaction of the ligand with the selected metal ion and comparison of the spectrum of the free ligand with its Ni(II) complex, the bands of the free ligand were exist in the complex spectra but were slightly shifted to blue or red regions. Also, new bands were observed in the spectra of the complex which are listed in Table (2). This change can be taken as a positive evidence of complex formation. The new band at 450 nm attributed to LMCT.

Ni(II) complex was found to be paramagnetic and its observed magnetic moments value is 2.69 BM indicating presence of two unpaired electrons[10].

Table 2: The magnetic properties and UV-Vis data (nm) for SQ and Ni(II) Complex.

Ligand / Complex	**μ_{eff} [(a)]**	**Absorption bands (nm) / peak assignment**			
		$\pi–\pi^*$	$n–\pi^*$	**CTI[(b)]**	**d-d transitions / peak assignment**
SQ	-	225	300		--
$[Ni(HL)(SQ)Cl_2].H_2O$	3.69	230	285, 325	375	675, $^4A_2(F) \rightarrow {}^4T_1(P)$

[(a)] μ_{eff} is an experimental effective magnetic moment calculated

[(b)] Charge transfer interaction.

3.5. Evaluation of the antimicrobial activity of synthesized ternary complex.

Antimicrobial activity of the ligands SQ and nickel ternary complex were individually tested against tow gram positive bacterial strains namely *Staphylococcus aureus and Bacillus subtilis* in comparison to Gentamycin as control drug Generally, the ligand SQ is biologically active against antibacterial strains and its metal complex showed significantly enhanced antibacterial and antifungal activity against microbial strains in comparison to the free ligand. The results, the measured inhibition zone against the bacterial and fungal strains growth are listed in Table (3) and compared with standards as shown in Figure (2).

The experimental data revealed that:-

- The free ligand, SQ is itself significantly active against all the test bacterial strains. It was found to be ineffective towards *fungai*.
- Ni(ll) complex more potent antibacterial agent than Ligand SQ.

It is obvious from the antimicrobial activity testing result that,the free ligand, SQ as well as its Ni(II) complex can inhibit the growth of tested bacteria with variable degree and showed a remarkable zones of inhibition against the different organisms under investigation. More importantly, the role of metal ions in such activity are clear upon comparison of ligand, metal complexes and standard drugs under identical experimental conditions.

Table 3: Antimicrobial assay of SQ and Ni(II) complex.

Tested organism	**Compounds under study**		
	SQ	Ni	control
	Zones diameter showing complete growth inhibition in mm*		
Gram-positive bacteria:			Gentamycin[(a)]
Staphylococcus aureus (RCMB010010)	10	15	24
Bacillus subtilis (RCMB015(1)NRRL B-543	18	15	26

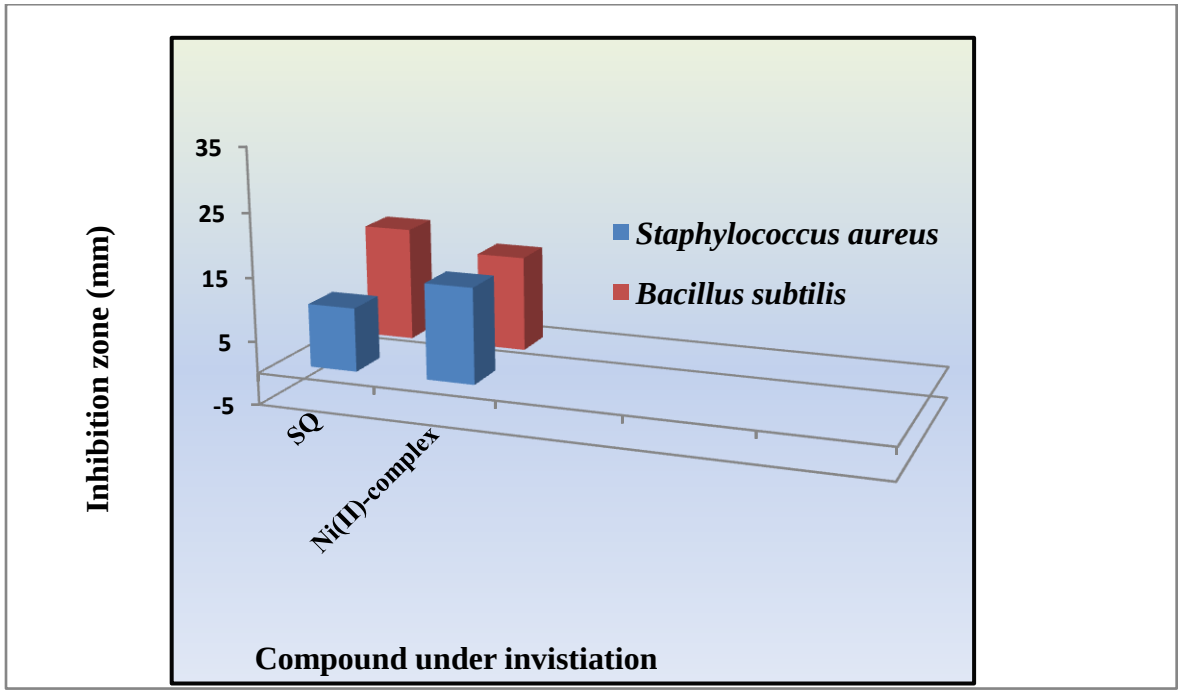

Figure 2: Antibacterial activity of SQ and Ni(II) complex

4. Conclusion

In the present research studies, A ternary complex of Ni(II) with sulfaquinoxaline, SQ as primary ligand and *o*-hydroxyacetophenone, HL as secondary ligand is prepared and characterized by elemental analysis, molar conductivity, IR, UV–vis spectroscopy and thermogravimetric analysis. The results reveal that sulfaquinoxaline, SQ coordinates with Ni(II) in bidentate mode through priazine nitrogen atom and secondary amine nitrogen atom. The supporting ligand (HL) uses its two O atoms in coordination with metal ions in bidentate mode. The general formula of the complex is $[Ni(SQ)(HL)Cl_2]H_2O$. The results of antibacterial activity indicated that the complex have good antibacterial ability for the testing bacterium than that of sulfaquinoxaline, SQ.

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