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Synthesis and Characterization of Ni(II) Complexes with Bidentate (NO) Schiff Bases

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ABSTRACT

New complexes of nickel(II) have been prepared in the reaction mixture of nickel(II) acetate, ethanolamine and 5-X salicylaldehyde (X= Br, Cl). Diaquabis(2-hydroxyethylimino)methyl)-4-bromophenolato)nickel(II) and Diaquabis(2-hydroxyethylimino)methyl)-4-chlorophenolato)-nickel(II) were characterized by elemental analysis, IR spectroscopy and ESI mass spectrometry. Elemental analysis and mass spectrometry data of the complexes suggests the stoichiometry is 1:2 (metal-ligand).

Keywords: Schiff base, nickel complexes, IR spectroscopy, mass spectrometry.

INTRODUCTION

Schiff bases are types of ligands which have many applications like a free ligand[1-5] and also in the complexes form with transit metals. The studies of transit metals complexes with the Schiff bases are shown in the previous works[6-12]. In general the azomethine group (C=N) which is the functional group of Schiff bases is aided in forming a stable complex by either a second group. Schiff base derived from salicylaldehyde and ethanolamine can be used as bidentat ligands, in wich the two hydroxyl groups of the ethanolamine moieties are free from coordination [13].

MATERIAL AND METHODS

All reagents and chemicals were used as obtained from Aldrich and Acros. The infrared spectra were determined on a Perkin-Elmer System 2000 FT-IR spectrometer with KBr pellets. The mass spectra were measured on an FT-ICR-MS Bruker-Daltonics ESI spectrometer (APEX II, 7 Tesla). Elemental analyses were performed on a VARIO EL microanalyzer (Heraeus).

Preparation of complex (1)

Measured 1mmol $Ni(OAc)_2 \cdot 4H_2O$ and solved in mixture of 10 ml ethanol + 5ml H_2O at 50°C. Also, measured 2mmol of 2-aminoethanol and 2mmol of 5-bromosalicyladehyde and solved in 15 ml absolute ethanol at 50°C. Then the second mixture added in the nickel(II) acetate solution and refluxed for 20 minute. Precipitated green complexes were filtered, washed with acetone and diethyl ether. Yield: 80%; IR(KBr, cm^{-1}): 3451, 1638, 1462, 1383, 1309, 1172, 1089, 827, 691; ESI-MS *m/z* 578.8 (M^-); Elemental analysis: Anal. Calc. for $C_{18}H_{22}Br_2N_2NiO_6$: C, 37.22%; H, 3.82%; N, 4.82%. Found: C, 37.36%; H, 3.56%; N, 4.60%, Mp: 250°C(decomp).

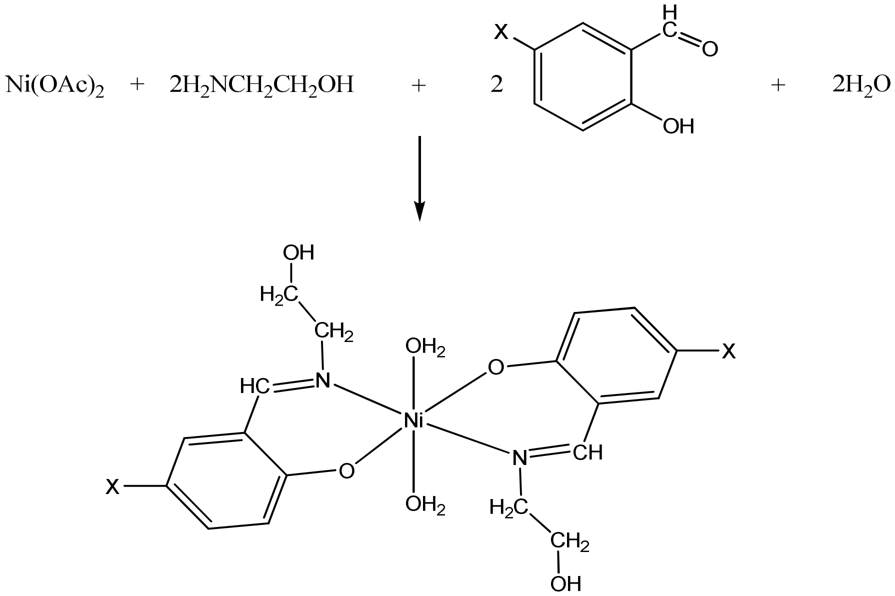
Preparation of complex (2)

Measured 1mmol $Ni(OAc)_2 \cdot 4H_2O$ and solved in mixture of 10 ml ethanol + 5ml H_2O at 50°C. Also, measured 2mmol of 2-aminoethanol and 2mmol of 5-chlorosalicyladehyde and solved in 15 ml absolute ethanol at 50°C. Then the second mixture added in the nickel(II) acetate solution and refluxed for 20 minute. Precipitated green complexes were filtered, washed with acetone and diethyl ether. Yield: 78%; IR(KBr, cm^{-1}): 3436, 1635, 1461, 1384, 1311, 1176, 1088, 828, 710; ESI-MS *m/z* 490.9 (M^-); Elemental analysis: Anal. Calc. for $C_{18}H_{22}Cl_2N_2NiO_6$: C, 43.94; H, 4.51; N, 5.69. Found: C, 44.05%; H, 4.32%; N, 5.48%, Mp: 250°C(decomp).

RESULTS AND DISCUSSION

In this paper we have shown the forming of two Ni(II) complexes in the "in situ" reaction of nickel(II) acetate, ethanolamine and 5-bromosalicyladehyde or 5-clorosalicyladehyde. Elemental analysis and mass spectra suggests that water is coordinated with nickel and the complexes have1:2 (Ni-ligand) stoichiometry. Based on the elemental analysis and mass spectra the formula, $[NiL_2(H_2O)_2]$ (L= Schiff base), was suggested for the both complexes. The general equation for synthesis and suggested structure of complexes is shown in scheme 1.

Scheme 1.



X=Br (1); X=Cl (2)

(1)*Diaquabis(2-hydroxyethylimino)methyl)-4-bromophenolato)nickel(II)* ;***(2)****Diaquabis(2-hydroxyethylimino)methyl)-4-chlorophenolato)nickel(II)*

Elemental analysis

The elemental analysis data match very well with those calculated and they show that nickel is coordinated with water and Schiff bases in 1:2 ratio.

Table 1. Elemental analysis data of Ni (II) complexes

	Calc. %			**Found %**		
	C	**H**	**N**	**C**	**H**	**N**
(1) $C_{18}H_{22}Br_2N_2NiO_6$	37.22	3.82	4.82	37.36	3.56	4.60
(2) $C_{18}H_{22}Cl_2N_2NiO_6$	43.94	4.51	5.69	44.05	4.32	5.48

IR spectra

The IR spectra of complexes are very clear and the characteristic absorptions of O-H and C=N bands which are present in complexes are easily identified.

The complexes show a broad band of hydroxyl groups at 3600-3200 cm^{-1}[14,15] with maximum absorption in the 3451cm^{-1} **(1)** and 3436 cm^{-1} **(2)**. These bands show the free hydroxyl groups and coordinated water molecules to the Ni(II) ion in the complexes [10]. Also the bands at 691cm^{-1} and 710cm^{-1} assign to Ni–OH_2 bonds [10]. The azomethine groups (C=N) at the both of complexes appear with a strong stretching absorption band at 1638 cm^{-1} **(1)** respectively 1635cm^{-1} **(2)** [14,15]. The IR specter of complex **(2)** are shown in the fig. 1.

Fig. 1. IR specter of Ni complex (2)

Mass spectra

The *m/z* values of complexes prove that complexes were formed in the above mentioned ratio. The experimental values of ESI-MS are almost the same with theoretically calculated (table 2). The mass specter of complex **(2)** are shown in the fig. 2.

Table 2. Mass spectra of Co(II) complexes

		(1)	**(2)**
		$C_{18}H_{22}Br_2N_2NiO_6$	$C_{18}H_{22}Br_2N_2NiO_6$
m/z	Found (M-H^+)	578.8	490.9
	Calc. (M)	579.9	492.0

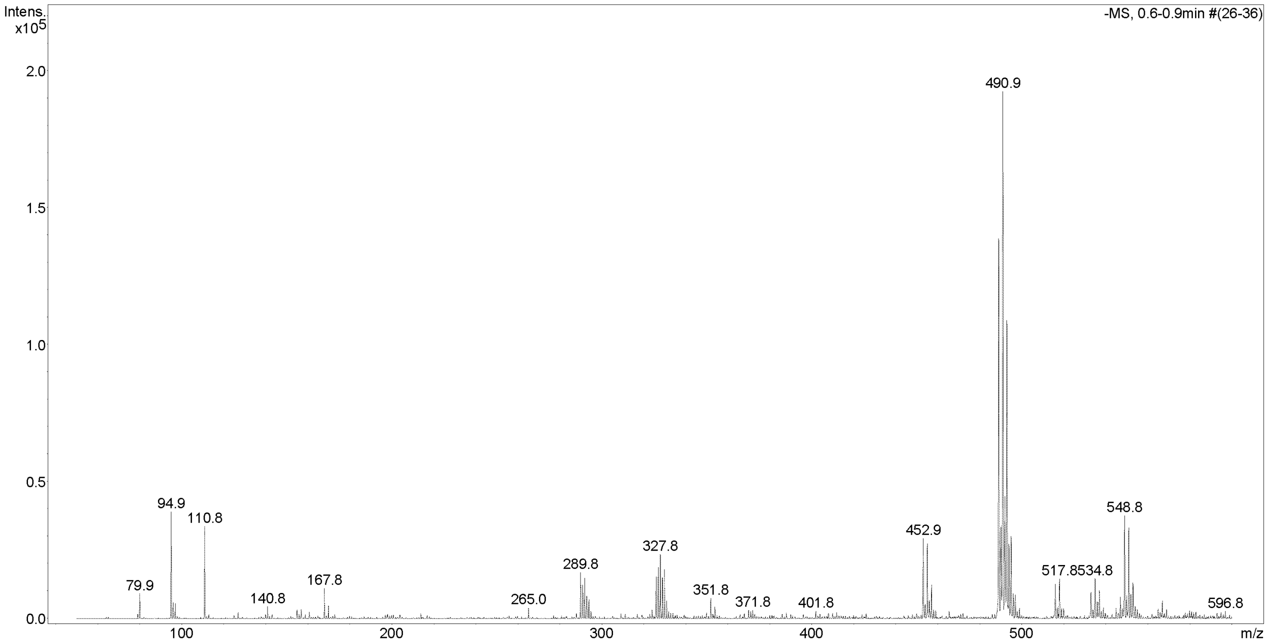


Figure 2. Mass specter of Ni complex (2)

CONCLUSIONS

The data of elemental analysis, IR spectra and mass spectra show that Schiff bases synthesized "in situ" reaction are coordinated with nickel (II) ion and form Diaquabis(2-hydroxyethylimino)methyl)-4-bromophenolato)nickel(II) and Diaquabis(2-hydroxyethylimino)methyl)-4-chlorophenolato)nickel(II) complexes. The Schiff bases are coordinated with Ni(II) ion through phenolic oxygen and azomethine nitrogen.

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