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Synthesis, Characterization and Antioxidant Activity of Nickel(II) Schiff Base Complexes Derived from 4-(Dimethylamino)benzaldehyde

¹Nighat Nawaz*, ^{2,3}Irshad Ahmad, ⁴Nizam M. Darwesh, ⁵Amjad Wahab, ⁶Sadeeq ur Rahman, ⁶Abdul Sajid,

⁷Farhan A. Khan, ⁷Sher B. Khan, ²Simon G. Patching** and ¹Kamal Uddin

¹Department of Chemistry, Islamia College Peshawar, Peshawar, Pakistan.

²School of Biomedical Sciences and Astbury Centre for Structural Molecular Biology, University of Leeds, Leeds, UK.

³Institute of Basic Medical Sciences, Khyber Medical University, Peshawar, Pakistan.

⁴Institute for Health Research, University of Bedfordshire, Luton, UK.

⁵Department of Chemical Engineering, University of Engineering & Technology, Peshawar, Pakistan.

⁶College of Animal Husbandry and Veterinary Sciences, Abdul Wali Khan University, Mardan, Pakistan.

⁷Department of Animal Health, The University of Agriculture, Peshawar, Pakistan.

nawazicu@gmail.com*; s.g.patching@leeds.ac.uk, simonpatching@yahoo.co.uk**

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Summary: Nickel(II) complexes of the following Schiff base ligands derived from 4-(dimethylamino)benzaldehyde were synthesised: (Z)-1-(4-(dimethylamino)benzylideneamino)propan-1-ol through condensation with 1-amino-propan-1-ol, (N¹E,N²E)-N¹,N²-bis(4-dimethylamino)benzylidene)benzene-1,2-diamine through condensation with benzene-1,2-diamine and 2-((4-dimethylamino)benzylidene)amino)phenol through condensation with 2-aminophenol. The synthesised Schiff bases and the resultant Ni(II) complexes were all isolated as crystals and characterized by melting point, elemental analysis, infrared spectroscopy, NMR spectroscopy, mass spectrometry and by conductance measurements. Antioxidant activity of the Ni(II) complexes was tested by assay with the free radical compound 2,2-diphenyl-1-picrylhydrazyl (DPPH).

Keywords: Antioxidant activity; Compound characterization; Ni(II) complex; Schiff base; Synthesis.

Introduction

Schiff bases are compounds having a carbon to nitrogen double bond (C=N), sometimes referred to as an azomethine bond, and possess the general formula R¹(R²)C=NR³ (R³ ≠ H). They are produced by condensing primary amines and carbonyl compounds (aldehydes and ketones). Schiff bases containing aryl substituents are much more easily synthesized and are more stable than those involving alkyl substituents. Indeed, Schiff bases formed from aliphatic aldehydes are unstable and can polymerize easily. The condensation reaction usually occurs in the presence of acid or base or by heating and goes to completion either by water removal or by product separation or both. Most Schiff bases are reversibly hydrolyzed to their component amine and carbonyl compound by using acid, base or water. The acid-catalyzed formation of Schiff bases involves nucleophilic attack of the primary amine on the carbonyl centre to give an intermediate carbinolamine, which is unstable. Acid-catalyzed dehydration of the carbinolamine results in conversion to an N-substituted imine or Schiff base.

Schiff base compounds often contain many donor atoms (N, O, S), which makes them biologically active and excellent chelators of metal

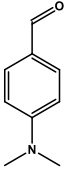
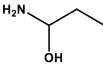
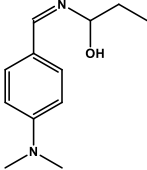
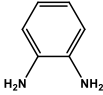
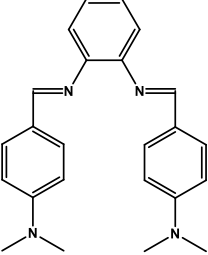
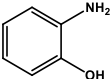
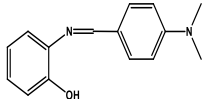
ions. Schiff bases and their metal complexes have a variety of uses in synthetic organic chemistry as intermediates and as catalysts [1-3]. They also make important contributions to pharmaceuticals [4,5] and their other industrial applications include foods, dyes and polymers [1,2,6]. Schiff base compounds have demonstrated antibacterial, antifungal, antimalarial, antiviral and anticancer activities, which are often enhanced when they are in metal complex form [1,7-10].

Another property demonstrated by the metal complexes of Schiff bases is antioxidant activity, whereby they have a high capacity for scavenging free radicals [10-13]. Free radicals are implicated as a major factor in the development of oxidative damage diseases (e.g. atherosclerosis, cancer, liver cirrhosis, diabetes, cardiovascular and neurodegenerative diseases) and in ageing [14-16], which can be promoted by cigarette smoke, drugs and pollution. As a consequence, there is an important need for antioxidants as a defence against free radical attack. In this respect, we have synthesised, characterized and tested the antioxidant properties of three Ni(II) complexes of Schiff bases derived from 4-(dimethylamino)benzaldehyde. The resultant

*To whom all correspondence should be addressed.

bidentate Schiff base ligands are (Z)-1-(4-(dimethylamino)benzylideneamino)-propan-1-ol (**L**₁) through condensation with 1-amino-propan-1-ol, (*N*¹*E,N*²*E*)-*N*^{1,N}²-bis(4-(dimethylamino)benzylidene)benzene-1,2-diamine (**L**₂) through condensation with benzene-1,2-diamine and 2-((4-dimethylamino)benzylidene)amino)phenol (**L**₃) through condensation with 2-aminophenol (Table-1).

Table-1: Synthesised Schiff base ligands and their starting compounds.

Carbonyl compound	Primary amine	Schiff base ligand
		L ₁ 
"		L ₂ 
"		L ₃ 

Experimental

Materials and instrumentation

Chemicals and solvents of analytical grade were obtained from BDH Chemicals or Sigma-Aldrich. All solvents were dried before use by employing reported methods [17]. Melting points were estimated using a Sanyo Gallenkamp apparatus (model MP-D.350MB3.5). Elemental analysis was performed using a Perkin Elmer 2400 Series II elemental analyzer. Infrared (IR) spectra (250-4000 cm⁻¹) were measured using a Thermo Scientific Nicolet 6700 FT-IR instrument. ¹H and ¹³C NMR analyses were performed using Bruker Avance III 300 MHz and 400 MHz spectrometers with samples dissolved in CDCl₃ or CD₃OD containing the internal standard tetramethylsilane (TMS); chemical shifts are given in ppm relative to TMS. Mass spectrometry (EI⁺ mode) was performed using a JEOL JMS-600H instrument. Conductivity measurements were performed using a Eutech CyberScan 500 instrument.

Absorbance measurements for the antioxidant assay were performed using a Hitachi U-2900 UV/visible spectrophotometer.

Synthesis of Schiff base ligands

4-(Dimethylamino)benzaldehyde (1.49 g, 10 mmol) was added to a two-necked round-bottomed flask containing dry toluene (50 ml), followed by addition of the relevant amine [0.75 g (10 mmol) of 1-amino-propan-1-ol for **L**₁, 0.54 g (5 mmol) of benzene-1,2-diamine for **L**₂, 1.09 g (10 mmol) of 2-aminophenol for **L**₃]. The mixture was stirred under reflux for 5-6 hours. In the case of **L**₁ an orange-yellow solution was obtained, from which excess solvent was removed *in vacuo*, then the ligand was isolated by column (silica gel) chromatography and by recrystallisation from various solvents (diethyl ether, ethanol, methanol, chloroform). In the case of **L**₂ a maroon-coloured product was obtained that was insoluble in the solvent; this was filtered off and then washed and recrystallised from various solvents (diethyl ether, ethanol, methanol, chloroform). In the case of **L**₃ a red-coloured product was obtained that was insoluble in the solvent; this was isolated by filtration, then washed with ethanol and subjected to solvent extraction.

L₁: Orange crystals; Yield: 63%; m.p. 108 °C; Analysis Calculated for C₁₂H₁₈N₂O: C, 69.87; H, 8.80; N, 13.58%. Found: C, 69.82; H, 8.84; N, 13.51%; IR (KBr, cm⁻¹) v: 3450-3550 (O-H), 1643 (N=C), 1292 (C-N), 1204 (C-O); ¹H NMR (300 MHz, CDCl₃) δ: 8.24 (1H, s, N=CH), 6.55-7.24 (4H, m, Ar-H), 5.48 (1H, s, OH), 3.94 (1H, t, CH-OH), 3.11 (6H, s, N-CH₃), 1.30 (2H, m, CH₂), 0.91 (3H, t, CH₃); ¹³C NMR (75 MHz, CDCl₃) δ: 160.9 (C=N), 111.4-152.9 (Ar-C), 84.5 (C-OH), 43.0 (N-CH₃), 32.0 (CH₂), 6.7 (CH₃); MS m/z calc. for C₁₂H₁₈N₂O 206.3, found: 205.2 (16%, A), 188.1 (76%, C), 161.1 (20%, D), 144.3 (100%, E), 129.1 (56%, B), 99.8 (44%), 76.1 (41%, G) (Appendix 1); Solubility: H₂O, MeOH, EtOH, DCM.

L₂: Maroon crystals; Yield: 69%; m.p. 184 °C; Analysis Calculated for C₂₄H₂₆N₄: C, 77.80; H, 7.07; N, 15.12%. Found: C, 77.75; H, 7.11; N, 15.19%; IR (KBr, cm⁻¹) v: 3496 (O-H), 1639 (N=C), 1271 (C-N), 1196 (C-O); ¹H NMR (300 MHz, CDCl₃) δ: 8.66 (2H, s, N=CH), 6.38-7.59 (12H, m, Ar-H), 2.91 (12H, s, N-CH₃); ¹³C NMR (75 MHz, CDCl₃) δ: 160.8 (C=N), 110.7-153.0 (Ar-C), 43.2 (N-CH₃); MS m/z calc for C₂₄H₂₆N₄ 370.5, found: 369.7 (77%, A), 354.8 (23%, B), 325.3 (100%, C), 283.1 (13%), 232.3 (36%, D), 168.1 (26%), 120.3 (21%),

104.1 (33%, H), 76.0 (69%, J) (Appendix 2); Solubility: MeOH, EtOH, DCM.

L₃: Grey crystals; Yield: 58%; m.p. 112 °C; Analysis Calculated for C₁₅H₁₆N₂O: C, 74.97; H, 6.71; N, 11.66%. Found: C, 74.89; H, 6.76; N, 11.64%; IR (KBr, cm⁻¹) v: 3500 (O-H), 1658 (N=C), 1277 (C-N), 1199 (C-O); ¹H NMR (300 MHz, CDCl₃) δ: 9.71 (1H, s, OH), 8.48 (1H, s, N=CH), 6.59-7.88 (8H, m, Ar-H), 5.20 (1H, s, OH), 3.06 (6H, s, N-CH₃); ¹³C NMR (75 MHz, CDCl₃) δ: 161.0 (C=N), 110.9-153.2 (Ar-C), 166.0 (C-OH), 42.8 (N-CH₃); MS m/z calc. for C₁₅H₁₆N₂O 240.3, found: 241.5 (100%, A), 224.2 (53%, B), 197.3 (24%, C), 179.9 (33%, G), 120.1 (18%, D), 108.1 (37%, H/I), 93.3 (41%, E), 76.1 (49%, F) (Appendix 3); Solubility: H₂O, MeOH, CHCl₃.

Synthesis of Ni(II) complexes

Nickel(II) chloride (0.65 g, 5 mmol) was dissolved in absolute ethanol (25 ml). A twofold ratio of the relevant Schiff base ligand [2.06 g (10 mmol) of **L₁**, 3.71 g (10 mmol) of **L₂**, 2.40 g (10 mmol) of **L₃**] dissolved in ethanol (25 ml) was then added with constant stirring, resulting in colour change. The mixture was refluxed for 5-6 hours, then excess solvent was removed *in vacuo*. The resultant solid product was filtered off, washed with cold solvents (diethyl ether, ethanol), then allowed to dry and recrystallized using various solvent mixtures (diethyl ether, ethanol, methanol, chloroform).

NiL₁: Pale green crystals; Yield: 54%; m.p. >300 °C; Analysis Calculated for C₂₄H₃₄N₄O₂NiCl₂: C, 53.37; H, 6.34; N, 10.37; Ni 10.87%. Found: C, 53.42; H, 6.37; N, 10.33; Ni, 10.90%; IR (KBr, cm⁻¹) v: O-H disappeared, 1628 (N=C), 1259 (C-N), 1171 (C-O), 622 (Ni-O), 423 (Ni-N); Solubility: H₂O (partial), DMSO. Molar conductance (DMSO, 26 °C) Λ_m: 0.671 S cm² mol⁻¹.

NiL₂: Green crystals; Yield: 57%; m.p. 247 °C; Analysis Calculated for C₄₈H₅₂N₈NiCl₂: C, 66.22; H, 6.02; N, 12.87; Ni 6.74%. Found: C, 66.25; H, 6.02; N, 12.85; Ni, 6.76%; IR (KBr, cm⁻¹) v: 1622 (N=C), 1259 (C-N), 1178 (C-O), 451 (Ni-N); Solubility: H₂O (partial), DCM, DMSO; Molar conductance (DMSO, 26 °C) Λ_m: 0.122 S cm² mol⁻¹.

NiL₃: Red crystals; Yield: 57%; m.p. >300 °C; Analysis Calculated for C₃₀H₃₀N₄O₂NiCl₂: C, 59.25; H, 4.97; N, 9.21; Ni 9.65%. Found: C, 59.21; H, 5.01; N, 9.20; Ni, 9.63%; IR (KBr, cm⁻¹) v: O-H disappeared, 1629 (N=C), 1266 (C-N), 1172 (C-O),

567 (Ni-O), 418 (Ni-N); Solubility: H₂O (partial), DCM, DMSO, MeCN; Molar conductance (DMSO, 26 °C) Λ_m: 0.635 S cm² mol⁻¹.

Antioxidant activity assay

Antioxidant activity of the synthesised Ni(II) Schiff base complexes was tested using the free radical compound 2,2-diphenyl-1-picrylhydrazyl (DPPH) [18,19]. This compound has a violet colour when dissolved in ethanol and a strong absorption maximum centred at around 515-520 nm. DPPH becomes colourless or pale yellow when neutralized. An aliquot of DPPH (0.3 ml) in ethanol (0.5 mM) was added to ethanol (3 ml), followed by addition of the Ni(II) complex dissolved in ethanol (0.5 ml) (total volume 3.8 ml). A UV/visible spectrophotometer was used to monitor the colour change at 517 nm after 100 minutes of reaction. A blank sample contained a mixture of the Ni(II) complex in ethanol (0.5 ml) and ethanol (3.3 ml) (total volume 3.8 ml) without any DPPH (no violet colour). A control sample contained a solution of DPPH (0.3 ml) and ethanol (3.5 ml) (total volume 3.8 ml) without any Ni(II) complex (violet colour remains). The amount of scavenging activity (%) was determined according to the method of Mensor et al [20].

Results and Discussion

Three bidentate Schiff base ligands derived from 4-(dimethylamino)benzaldehyde (**L₁**, **L₂**, **L₃**) (Table 1) were prepared in good yield (58-69%) using the refluxing method followed by product separation. The ligands **L₁**, **L₂** and **L₃** were isolated as crystals having colours of orange, maroon and grey, respectively. A twofold ratio of each Schiff base ligand was reacted with Ni(II) chloride, which produced a colour change. The resultant Ni(II) complexes (**NiL₁**, **NiL₂**, **NiL₃**) were isolated in good yield (54-57%) and as crystals having colours of pale green, green and red, respectively. All compounds showed best solubility in organic solvents at room temperature and variable solubility in water. Based on conductance measurements in DMSO, the Ni(II) complexes are all non-electrolytes. This was expected because the complexes have an overall neutral charge.

The isolated Schiff base ligands and the resultant Ni(II) complexes were characterized by melting point, elemental analysis, IR spectroscopy, NMR spectroscopy and mass spectrometry.

All isolated compounds produced IR spectral bands consistent with those in literature

spectra of similar Schiff base ligands and derived metal complexes. Disappearance of bands for amine (NH_2) and aldehyde (CHO) groups confirmed reaction of the starting compounds. Appearance of a peak in the region $1639\text{-}1658\text{ cm}^{-1}$, coming from an azomethine ($\text{C}=\text{N}$) bond, confirmed Schiff base ligand production and this was shifted to $1622\text{-}1629\text{ cm}^{-1}$ on production of the Ni(II) complexes. This lowering in frequency indicates electron pair donation by the nitrogen atom of the azomethine group to the Ni(II) ion. Disappearance of the O-H frequency ($3450\text{-}3550\text{ cm}^{-1}$) from L_1 and L_3 on formation of the complex confirms deprotonation and co-ordination with the Ni(II) ion. For the Schiff base ligands the C-O peak occurred at a frequency of $1196\text{-}1204\text{ cm}^{-1}$ and for the resultant Ni(II) complexes the C-O frequency range was $1171\text{-}1178\text{ cm}^{-1}$. For the Ni(II) complexes there was appearance of bands for Ni-O ($567\text{-}622\text{ cm}^{-1}$) and Ni-N ($418\text{-}451\text{ cm}^{-1}$) in the far-IR region, which were not present in spectra of the originating Schiff base ligands.

^1H NMR spectra of the synthesised Schiff base ligands were consistent with their expected structures, including signals at 8.24 (1H, s), 8.66 (2H, s) and 8.48 ppm (1H, s) for the proton in the azomethine group of L_1 , L_2 and L_3 , respectively. ^{13}C NMR spectra were also consistent with their expected structures, including signals at 160.9, 160.8 and 161.0 ppm for the azomethine group of L_1 , L_2 and L_3 , respectively.

Synthesis of the Schiff base ligands was further confirmed by mass spectrometry. L_1 produced a molecular ion with mass of 205.2 (expected 206.3) and a fragmentation pattern consistent with its expected structure (Appendix 1). L_2 produced a molecular ion with mass of 369.7 (expected 370.5) and a fragmentation pattern consistent with its expected structure (Appendix 2). L_3 produced a molecular ion with mass of 241.6 (expected 240.3) and a fragmentation pattern consistent with its expected structure (Appendix 3).

All experimental evidence suggests that the general formula for the synthesised Ni(II) complexes is NiL_2Cl_2 or $[\text{NiL}_2\text{Cl}_2]\cdot\text{H}_2\text{O}$. This includes the elemental analysis results, which confirm that the stoichiometry of the complexes is NiL_2Cl_2 in which the ligand acts as a bidentate.

Antioxidant activity of the synthesised Ni(II) Schiff base complexes was tested by DPPH assay, in which there was a decrease in DPPH free radical with increasing concentration of complex (Fig. 1). The results suggest that the Ni(II) complexes

are good scavengers of free radical species and therefore show promise for further investigation to target oxidative damage diseases. Indeed, Schiff base ligand L_3 has previously been tested for radical scavenging activity and its copper and ferrous chelating abilities has been investigated [21]. The organotin(IV) complex of L_3 has also been tested for antibacterial activity [22]. The Ni(II) complex NiL_2 and its complexes with Cu(II), Co(II) and Zn(II) have been tested for DNA binding activity and for antibacterial and antifungal activities [23].

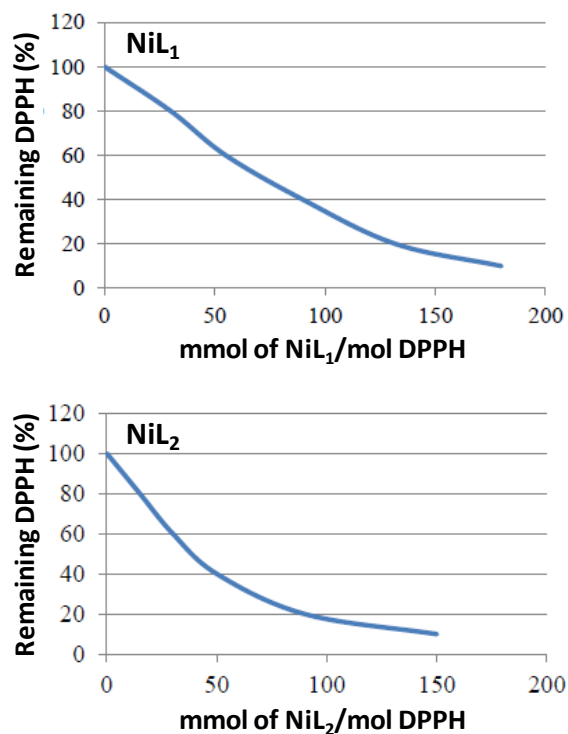


Fig. 1: Antioxidant activity of synthesised Nickel(II) Schiff base complexes. Decrease in DPPH free radical with increasing concentrations of NiL_1 (top) and NiL_2 (bottom), monitored at a wavelength of 517 nm.

Conclusion

Ni(II) complexes of Schiff bases derived from 4-(dimethylamino)benzaldehyde were synthesized in good yields, isolated as crystals and characterized using a range of chemical and physical techniques. Tests of the Ni(II) complexes for antioxidant activity suggest that they are good scavengers of free radical species and therefore show promise for further investigation to target oxidative damage diseases. These and similar Ni(II) Schiff bases could also be tested for cell membrane

permeability and for antibacterial, antifungal, antimalarial, antiviral and anticancer activities.

Acknowledgements

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