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Synthesis, characterization and structural determination of some nickel(II) complexes containing imido Schiff bases and substituted phosphine ligands

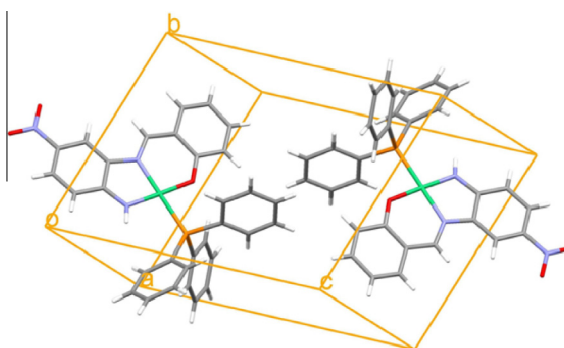
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HIGHLIGHTS

- The new complexes were synthesized by a simple method.
- The article contains some scopes like amido –NH complexes.
- This paper is a general study and contains inorganic, organic (Schiff bases) and physical chemistry (TG and DTA).
- The structure of the complexes is square planar.

GRAPHICAL ABSTRACT

The new amido tridentate ONN Schiff base complexes of $[\text{NiL}(\text{PR}_3)]$ were synthesized and characterized by IR, UV–Vis, NMR and elemental analysis. Coordination geometry of $[\text{NiL}(\text{PPh}_3)]$ was determined by X-ray crystallography. The thermogravimetry of the complexes were carried out in the range of 20–600 °C.



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ABSTRACT

Some new tridentate ONN Schiff base complexes of $[\text{NiL}(\text{PR}_3)]$ (where L = Salicylidene2-amino4-nitrobenzene (L^1), 5-BrSalicylidene2-amino4-nitrobenzene (L^2), 5- NO_2 Salicylidene2-amino4-nitrobenzene (L^3), 5-MeOSalicylidene2-amino4-nitrobenzene (L^4) and 3-MeOSalicylidene2-amino4-nitrobenzene (L^5), R = Bu and Ph (with L^1)) were synthesized and characterized by IR, UV–Vis, ^1H NMR spectroscopy and elemental analysis. The geometry of $[\text{NiL}^1(\text{PPh}_3)]$ was determined by X-ray crystallography. It indicated that the complex had a planar structure and four coordinates in the solid state. The thermogravimetry (TG) and differential thermoanalysis (DTA) of the synthesized complexes were carried out in the range of 20–600 °C, leading to the decomposition of L^1 – L^3 type in three stages and of L^4 – L^5 and $[\text{NiL}^1(\text{PPh}_3)]$ type in four stages. Thermal decomposition of the complexes was closely dependent upon the nature of the Schiff base ligands and proceeded via the first order kinetics.

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Introduction

Metal Schiff base complexes have been studied because of their interesting properties such as their catalytic activity of hydrogenation of olefins [1], reversible binding to oxygen [2], amino group transfer [3], modification of nanostructure compounds [4], and collection of some toxic metals [5].

In this area, the tridentate Schiff base complexes were applied as effective corrosion inhibitors for some metals in acidic media [6]. Some Ni(II), Cu(II), Ru(II) and Pd(II) complexes containing tridentate Schiff bases and phosphine were studied and their

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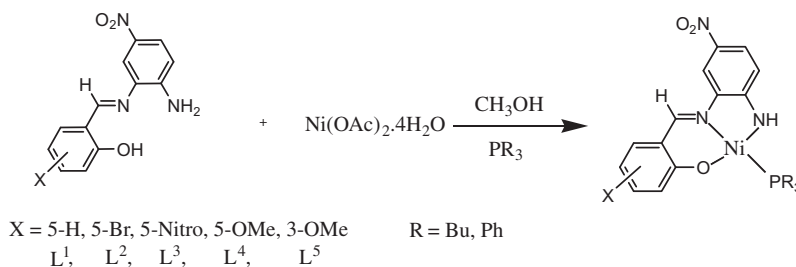


Fig. 1. The structure of nickel complexes.

coordination geometries were identified and applied as catalyst for oxidation of alcohols and Suzuki–Miyaura cross coupling reactions [7–11].

Continuing our studies on tridentate Schiff base complexes [12–14], herein we report the synthesis of novel Ni(II) complexes containing ONN amido tridentate Schiff bases and phosphines (Fig. 1). The synthesized complexes were identified by IR, NMR, UV–Vis spectroscopy and elemental analysis. The structure of $[\text{NiL}^1(\text{PPh}_3)]$ was determined by X-ray crystallography. The thermogravimetry (TG) and differential thermoanalysis (DTA) of the nickel(II) complexes were carried out in the range of 20–600 °C. The thermal decomposition kinetic parameters were also calculated and discussed using Coats and Redfern method [15].

Experimental

Chemicals and apparatus

All chemicals and solvents, being of reagent quality, were used without further purification. Infrared spectra (KBr disks) were recorded on a FT-IR JASCO-680 spectrophotometer in the 4000–400 cm^{-1} . The elemental analyses were determined on a CHN-O-Heraeus elemental analyzer. UV–Vis spectra were recorded on a JASCO V-570 spectrophotometer in the 190–900 nm. The ^1H NMR spectra were recorded in CDCl_3 on DPX-400 MHz FT-NMR. Thermogravimetry (TG) and differential thermoanalysis (DTA) were carried out on a PL-1500. The measurements were performed in air atmosphere and the heating rate was kept at 10 °C min^{-1} .

Synthesis

According to the literature [14], the tridentate Schiff base ligands, L^1 – L^5 , were prepared by condensation of aldehyde and 4-nitro, 1,2-diaminobenzene by 1:1 mol ratio. The reactants were mixed in methanol and refluxed for 2 h. The yellow precipitates were appeared during the reaction. The precipitates were filtered and washed with methanol and recrystallized by dichloromethane/methanol mixed solvent through the partial evaporation of dichloromethane.

The $[\text{NiLPR}_3]$ complexes were synthesized by the following procedure. One mmol of the Schiff base ligand was added to a methanolic solution containing nickel(II) acetate and phosphine (1 mmol from which one). The solution was refluxed for 2 h during which the color of solution was changed to red. The solution was filtered and the red crystals were collected after 24–48 h, washed with methanol and recrystallized from dichloromethane/methanol.

$\text{NiL}^1(\text{PBu}_3)$: Yield (80%). Anal. calc. for $\text{C}_{25}\text{H}_{36}\text{N}_3\text{O}_3\text{PNi}$: C, 57.14%; H, 7.02%; N, 8.13%. Found; C, 57.32%; H, 7.21%; N, 8.46%. FT-IR (KBr cm^{-1}) ν_{max} , 3433 (N–H), 1609 (C=N), 1445 (C=C). UV–Vis, λ_{max} (nm) (Ethanol): 244, 267, 458. δ (400 MHz, CDCl_3 , Me_4Si) 0.889–1.984 (m, 27H, CH of PBu_3), 2.317 (s, 1H, NH), 6.403 (s, 1H, Aromatic), 6.741 (s, 1H, Aromatic), 6.871 (s, 1H, Aromatic), 7.516 (s, 1H, Aromatic), 7.881 (s, 1H, Aromatic), 8.611 (s, 1H, Aromatic) and 8.610 (s, 1H, HC=N).

$\text{NiL}^2(\text{PBu}_3)$: Yield (80%). Anal. calc. for $\text{C}_{25}\text{H}_{35}\text{N}_3\text{O}_3\text{PBrNi}$: C, 50.42%; H, 5.93%; N, 7.06%. Found; C, 50.13%; H, 6.11%; N, 7.49%. FT-IR (KBr cm^{-1}) ν_{max} , 3387 (N–H), 1596 (C=N), 1479 (C=C). UV–Vis, λ_{max} (nm) (Ethanol): 247, 459. δ (400 MHz, CDCl_3 , Me_4Si) 0.955–0.991 (t, 9H, CH_3 , $J = 14.4$), 1.499–1.751 (m, 18H, CH_2), 2.315 (s, 1H, NH), 6.413–6.435 (d, 1H, Aromatic, $J = 8.8$), 6.736–6.759 (d, 1H, Aromatic, $J = 9.2$), 7.295–7.301 (d, 1H, Aromatic, $J = 2.4$), 7.610–7.616 (d, 1H, Aromatic, $J = 2.4$), 7.870–7.893 (d, 1H, Aromatic, $J = 9.2$), 8.592 (s, 1H, Aromatic) and 8.742–8.768 (d, 1H, HC=N, $J = 10.4$).

$\text{NiL}^3(\text{PBu}_3)$: Yield (80%). Anal. calc. for $\text{C}_{25}\text{H}_{35}\text{N}_4\text{O}_5\text{PNi}$: C, 53.51%; H, 6.29%; N, 9.98%. Found; C, 53.73%; H, 6.48%; N, 10.43%. FT-IR (KBr cm^{-1}) ν_{max} , 3370 (N–H), 1599 (C=N), 1459 (C=C). UV–Vis, λ_{max} (nm) (Ethanol): 234, 268, 445.

$\text{NiL}^4(\text{PBu}_3)$: Yield (80%). Anal. calc. for $\text{C}_{26}\text{H}_{38}\text{N}_3\text{O}_4\text{PNi}$: C, 58.90%; H, 7.22%; N, 7.69%. Found; C, 58.63%; H, 7.52%; N, 8.02%. FT-IR (KBr cm^{-1}) ν_{max} , 3367 (N–H), 1580 (C=N), 1491 (C=C). UV–Vis, λ_{max} (nm) (Ethanol): 247, 466. δ (400 MHz, CDCl_3 , Me_4Si) 0.945 (s, 9H, CH_3), 1.546–1.69 (m, 18H, CH_2), 2.332 (s, 1H, NH), 3.8 (s, 3H, O– CH_3), 6.405 (s, 1H, Aromatic), 6.739–7.013 (m, 3H, Aromatic), 7.850 (s, 1H, Aromatic), 8.814 (s, 1H, Aromatic), 8.939 (s, 1H, HC=N).

$\text{NiL}^5(\text{PBu}_3)$: Yield (80%). Anal. calc. for $\text{C}_{26}\text{H}_{38}\text{N}_3\text{O}_4\text{PNi}$: C, 58.90%; H, 7.22%; N, 7.69%. Found; C, 59.32%; H, 7.63%; N, 7.92%. FT-IR (KBr cm^{-1}) ν_{max} , 3397 (N–H), 1594 (C=N), 1435 (C=C). UV–Vis, λ_{max} (nm) (Ethanol): 245, 270, 461. δ (400 MHz, CDCl_3 , Me_4Si) 2.412 (s, 1H, NH), 0.948–0.984 (t, 9H, CH_3 , $J = 14.4$), 1.521–1.744 (m, 18H, CH_2), 3.777 (s, 3H, O– CH_3), 6.418–6.440 (d, 1H, Aromatic, $J = 8.8$), 6.446–6.685 (m, 1H, Aromatic), 6.752–6.770 (d, 1H, Aromatic, $J = 2.2$), 7.121–7.141 (d, 1H, Aromatic, $J = 7.6$), 7.871–7.896 (d, 1H, Aromatic, $J = 7.2$), 8.615 (s, 1H, Aromatic) 8.839–8.867 (d, 1H, HC=N, $J = 11.2$).

$\text{NiL}^1(\text{PPh}_3)$: Yield (80%). $\text{C}_{31}\text{H}_{24}\text{N}_3\text{O}_3\text{PNi}$: C, 64.64%; H, 4.20%; N, 7.29%. Found; C, 65.12%; H, 4.38%; N, 7.53%. FT-IR (KBr cm^{-1}) ν_{max} , 3392 (N–H), 1607 (C=N), 1433 (C=C). UV–Vis, λ_{max} (nm) (Ethanol): 249, 445. δ (400 MHz, CDCl_3 , Me_4Si) 5.306 (s, 1H, NH), 6.118–6.141 (d, 1H, Aromatic, $J = 9.2$), 6.561–6.583 (d, 1H, Aromatic, $J = 8.8$), 6.728–6.745 (d, 1H, Aromatic, $J = 6.8$), 7.158–7.866 (m, 17H, Aromatic), 8.647 (s, 1H, Aromatic), 8.841–8.870 (d, 1H, HC=N, $J = 11.6$).

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Crystal data collection and processing of $[\text{NiL}^1(\text{PPh}_3)]$

The X-ray diffraction measurements were made on an STOE IPDS-2T diffractometer with graphite monochromated Mo $\text{K}\alpha$ radiation. For this complex, a red plate crystal with a dimension of $0.50 \times 0.20 \times 0.15$ mm was chosen and mounted on a glass fiber and used for data collection. Cell constants and an orientation matrix for data collection were obtained by least-squares refinement of diffraction data from 14,584 unique reflections. Data were collected at a temperature of 298(2) K to a maximum 2θ value of

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