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Nickel(II) complex based on bis-(1-(pyridin-2-yl-methyl)-benzimidazol-2-yl-methyl) ether and its utilization in the oxidation of 2-amino-4-*tert*-butylphenol

Raghvi Khattar, Anjana Yadav, Kuldeep Mahiya & Pavan Mathur*[†]

Department of Chemistry, University of Delhi, Delhi-110 007, India

Email: pavanmat@yahoo.co.in

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A mononuclear nickel(II) complex $[\text{Ni}(\text{L})_2](\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ is synthesized utilizing a bis-benzimidazolyl ligand (L = bis-(1-(pyridin-2-yl-methyl)-benzimidazol-2-yl-methyl)ether) and characterized by single-crystal X-ray diffraction, elemental analysis, UV-vis and IR spectroscopy. Ni(II) complex crystallizes in the tetragonal system with space group $I4_1/a$ and appears to be propeller-shaped when viewed along the *c*-axis. The $[\text{Ni}(\text{L})_2](\text{NO}_3)_2$ complex has been utilized for the oxidation of 2-amino-4-*tert*-butylphenol to 4-*tert*-butyl-*o*-benzoquinone in the presence of hydrogen peroxide and the average rate of reaction is calculated to be $63 \times 10^{-7} \text{ M min}^{-1}$. The presence of externally added acetate ion tends to inhibit the rate of reaction.

Keywords: Coordination chemistry, Benzimidazoles, N-Picolyl-bis-benzimidazole, Nickel, *o*-Iminobenzosemiquinone, 4-*tert*-Butyl-*o*-benzoquinone.

Benzimidazoles are quite useful intermediates for the development of compounds of pharmaceutical or biological interest¹⁻⁵. Bis-1*H*-benzimidazoles and their derivatives are good coordinating agents that form stable complexes with various transition metals^{6,7}. The redox properties of their metal ion complexes depend on the nature of donor atoms, geometry and flexibility of the chelating ligands⁸. A variety of Ni(II) containing enzymes are known to have the Ni(II) ion in a distorted octahedral environment with the imidazole nitrogen of the histidine amino acid as one of the coordinating atoms^{9,10}. A few Ni(II) coordination compounds with mononucleating tridentate ligands of N–O–N type are reported^{11,12}. Recently, there are reports on the crystal structure of some related complexes with Zn(II), Cu(II), Cd(II), Mn(II) and Co(II) have been reported¹³⁻²². To the best of our knowledge, reports on nickel compounds with N-substituted N–O–N type ligands are limited, though Ni(II) complexes with ligand bis-(2-benzimidazolyl)propane have been reported²³.

Oxidation of phenol has been reported using transition metal ion complexes, in presence of molecular oxygen or hydrogen peroxide²⁴⁻²⁹. The present paper reports the synthesis, spectral and

structural studies on Ni(II) complex with N-picolyl substituted benzimidazolyl ligand. A detailed study of the oxidation of 2-amino-4-*tert*-butylphenol to 4-*tert*-butyl-1,2-benzoquinone in the presence of H_2O_2 is reported using the presently synthesized $[\text{Ni}(\text{L})_2](\text{NO}_3)_2$ complex.

Materials and Methods

Diglycollic acid was obtained from M/S Aldrich (USA) while other chemicals like benzene-1, 2-diamine, 2-(chloromethyl) pyridine and spectroscopic solvents were obtained from commercial sources and used without further purification. Bis-(2-benzimidazolylmethyl) ether (DGB) was prepared as described earlier³⁰. Elemental analysis were obtained using Elemental Analyzer at USIC, University of Delhi, Delhi. X-ray diffraction data were collected on an Oxford Diffraction X'calibur CCD diffractometer with graphite monochromated (Mo/K_α radiation, $\lambda = 0.71073 \text{ \AA}$), temperature of 298(2) K at IIT Bombay, Mumbai, India.

Synthesis of the Ni(II) complex, $[\text{Ni}(\text{L})_2](\text{NO}_3)_2 \cdot \text{H}_2\text{O}$

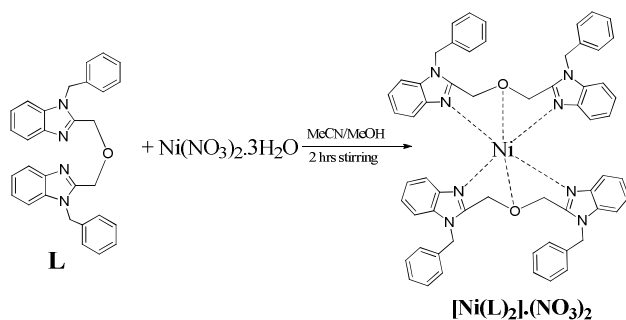
Ligand (L) was synthesized as described earlier³¹. The ligand (100 mg, 0.21 mmol) was dissolved in acetonitrile (15 mL) and a methanolic solution (5 mL) of $\text{Ni}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ (63.2 mg, 0.21 mmol) was added to the ligand solution. The resulting green colored

[†]Retd Professor, Chemistry Department, Delhi University, Delhi, India.

solution was stirred for 2 h. A light green colored powder was formed which was centrifuged, washed with small amounts of methanol:acetonitrile (1:5) and dried over P₂O₅ (Scheme 1). Yield: 45%; Anal. (%):found for C₅₆H₄₈N₁₄O₈Ni. H₂O: C, 59.7; H, 4.2; N, 17.0%. Calcd(%): C, 59.9; H, 4.4; N, 17.4. UV/vis (DMF) λ_{\max} , nm (log ϵ , M⁻¹ cm⁻¹) = 269 (4.39), 280 (4.36), 287 (4.24), 393(1.88), 658(1.30), 750(1.15). IR (KBr pellets, cm⁻¹): ν = 1596 (C=N benzimidazole and pyridine), 1478 (C=N-C=C (benzimidazole)), 755 (C=C benzene), 1340(O-N-O (sym)) and 1458 (O-N-O(asym)).

X-Ray crystal structure determination

X-ray diffraction data were collected on an Oxford diffraction X'calibur CCD diffractometer with graphite monochromated radiation (Mo-K α)



Synthesis of Ni(II) complex
Scheme 1

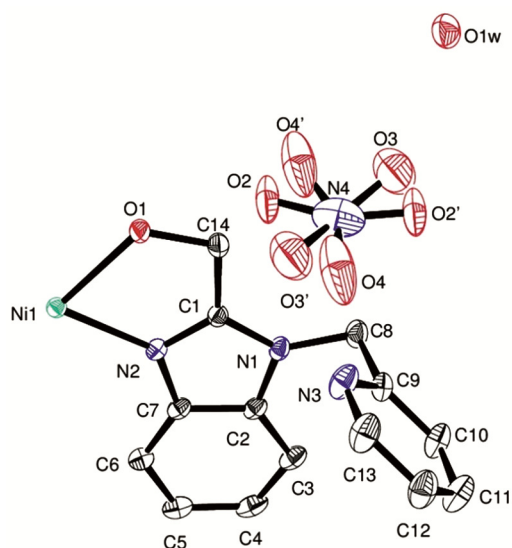


Fig. 1-ORTEP diagram of the asymmetric unit of [Ni(L)₂](NO₃)₂·H₂O complex (showing only 1/4th of the molecule, along with highly disordered nitrate ion) drawn in 20% thermal probability ellipsoids showing atomic numbering schemes.

$\lambda=0.71073$ Å, temp.: 298(2) K at IIT Bombay. The data were corrected for Lorentz and polarization effects. Multi-scan absorption correction was applied. The data reduction was performed using the CrysAlis software package³². The structure was solved by direct methods using SHELXS-97³³ and refined by full-matrix least-squares method on F² (SHELXL-97)³³. All calculations were carried out using the WingX package of the crystallographic programs³⁴. For the molecular graphics, the programs ORTEP-3³⁵ and Mercury³⁶ were used.

Result and Discussion

Crystal structure description of [Ni(L)₂](NO₃)₂·H₂O

The complex was dissolved in HPLC grade methanol and slow evaporation of the methanolic solution at room temperature over a period of 72 h resulted in the formation of needle-shaped green crystals. The asymmetric unit and ORTEP diagram with atom numbering is shown in (Figs 1 and 2). Crystal data collection, refinement parameters and selected bond lengths and bond angles are given in (Tables 1 and 2). A total of 7000 reflections were measured, out of which 3214 were independent and 2282 were observed [$I > 2\sigma(I)$], in the theta range of 2-25°. The reflections were corrected for Lorentz and polarization as well as for the absorption corrections. The refinement shows one highly disordered nitrate ion (high thermal parameters and anomalous bond lengths) and a water molecule (one oxygen). Because

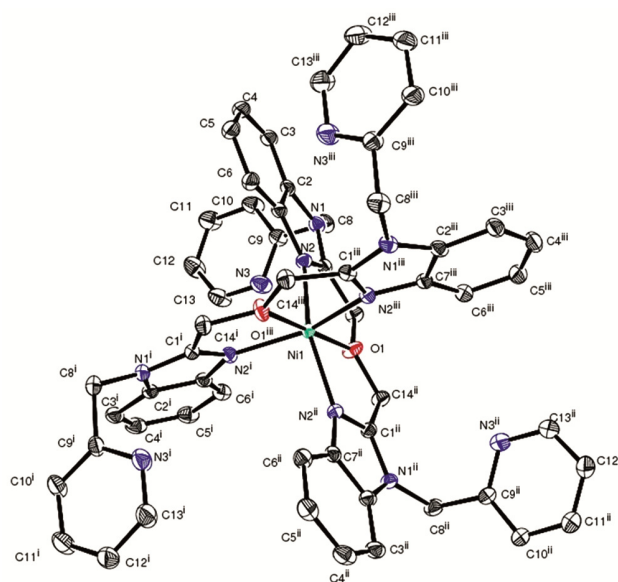


Fig. 2-ORTEP diagram of [Ni(L)₂](NO₃)₂·H₂O complex drawn in 20% thermal probability ellipsoids showing atomic numbering schemes. Nitrate and solvent water have been omitted for clarity.

Table 1—Crystal data and structure refinement of $[\text{Ni}(\text{L})_2] \cdot (\text{NO}_3)_2 \cdot \text{H}_2\text{O}$

Emp. formula	$\text{C}_{56}\text{H}_{50}\text{N}_{14}\text{NiO}_9$
Formula wt	1121.81
Temp.	295(2) K
Wavelength	0.71073 Å
Crystal system	Tetragonal
Space group	$I4_1/a$
Unit cell dimensions	$a = 19.447(4)$ Å, $\alpha = 90.0^\circ$. $b = 19.447(4)$ Å, $\beta = 90.0^\circ$. $c = 14.675(5)$ Å, $\gamma = 90.0^\circ$.
Vol.	$5549.9(3)$ Å ³
Z	4
Density (calc.)	1.343 mg/m ³
Abs. coeff.	0.419 mm ⁻¹
F(000)	2336
Crystal size	$0.19 \times 0.16 \times 0.16$ mm ³
θ range for data collection	2.96 to 29.15° .
Index ranges	$-12 \leq h \leq 25$, $-25 \leq k \leq 24$, $-13 \leq l \leq 20$
Reflections collected	7000
Independent reflections	3214 [$R(\text{int}) = 0.0188$]
Completeness to $\theta = 25.00^\circ$	99.8 %
Abs. correc.	Semi-empirical from equivalents
Max. and min. transmission	0.930 and 0.926
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3214 / 49 / 216
Goodness-of-fit on F^2	1.028
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0616$, $wR2 = 0.1881$
R indices (all data)	$R1 = 0.0816$, $wR2 = 0.1995$
Largest diff. peak and hole	0.780 and -0.376 e.Å ⁻³

Table 2—Selected bond lengths (Å) and bond angles (°) of $[\text{Ni}(\text{L})_2] \cdot (\text{NO}_3)_2 \cdot \text{H}_2\text{O}$

Bond	(Å)
N(2)-Ni(1)	2.081(2)
O(1)-Ni(1)	2.092(3)
Ni(1)-N(2)#2	2.081(2)
Ni(1)-N(2)#3	2.081(2)
Ni(1)-N(2)#1	2.081(2)
Ni(1)-O(1)#3	2.092(3)
Angle	(deg.)
N(2)#3-Ni(1)-O(1)#3	75.85(7)
N(2)#1-Ni(1)-O(1)#3	104.15(7)
N(2)-Ni(1)-O(1)	75.85(7)
N(2)#2-Ni(1)-O(1)	104.15(7)
N(2)#3-Ni(1)-O(1)	104.15(7)
N(2)#1-Ni(1)-O(1)	75.85(7)
O(1)#3-Ni(1)-O(1)	180.0
C(1)-N(2)-Ni(1)	115.22(19)
C(7)-N(2)-Ni(1)	138.4(2)
C(14)-O(1)-Ni(1)	121.29(17)

of the four-fold symmetry of the molecule, the nitrogen of the nitrate group was fixed as 10.5 and each of the three oxygens (showing rotational disorder) was split into two parts and was refined with *sof* of 10.25. The U_{iso} values of each component of the three pairs were kept equal using EADP. These were refined isotropically with fixed N-O distances of $1.242(3)$ Å and O...O non-bonding distances as $2.00(2)$ Å. The water molecule (single oxygen atom) was refined with a *sof* given as a free variable and it showed sensible thermal parameters with a *sof* of 0.25, which due to four-fold symmetry led to a total of one molecule of water per molecule of the complex. All atoms were then subjected to an anisotropic refinement using DELU and SIMU restraints for the oxygens of nitrate group and also making it planar using FLAT. The final model gave $R = 0.0616$ and $wR = 0.1881$ for the observed data and $R = 0.0816$ and $wR = 0.1995$ for all data. The hydrogens (or one hydrogen in the asymmetric unit) belonging to the lattice water could not be modeled although it could be seen in the Fourier map as a close peak near this oxygen. It was also in the right direction to form H-bonds with nitrate O3 on both the sides. This was because refinement of O-H with distance of 0.84 Å always yielded a very short O-H length of ~ 0.5 Å³. Therefore, it could not be included in the model which is now devoid of two hydrogens per molecule. The highest residual intensity is 0.78 eÅ³ only. Now because of the disorder in the nitrate and the lattice water molecule and also due to lack of the hydrogens of this water molecule, which would have shown H-bonds between nitrate and water molecule, there are some A and B level errors in the cif regarding short contacts between them.

Two planes can be passed from each of the tridentate ligands excluding the pyridine rings attached to them. Both these planes are mutually perpendicular to each other (Fig. 3). The two terminal pyridine rings in each of the ligand are in turn perpendicular to the respective planes lying above and below these planes. Looking down along the crystallographic c axis the arrangement gives rise to a propeller shaped conformation to the molecule. The coordination around Ni(II) ion is octahedral where each ligand is coordinating through two benzimidazole nitrogens and one ethereal oxygen atoms in a meridional conformation. The pyridine groups are pointing away from the metal ion and are not coordinating to it. The Ni-N and Ni-O distances

are 2.082(2) and 2.092(3) Å, respectively. The *trans* O1-Ni-O1 angle is 180, but that of N2-N1-N2 is 151.7° only for N2 being part of a rigid five membered ring.

Oxidation of 2-amino-4-*tert*-butylphenol

The oxidation of substituted aminophenol using Ni(II) complex was observed spectrophotometrically. A methanolic solution of Ni(II) complex $[\text{Ni}(\text{L})_2](\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ (2 mL, 1.2 mM) was bubbled with molecular oxygen for 5-10 min and was added to a methanolic solution of 2-amino-4-*tert*-butylphenol (2 mL, 11.9 mM). The ratio of complex:substrate is 1:10. An aliquot of this reaction mixture (0.2 mL) was diluted to make a 2.2 mL solution in methanol and the absorption spectra of this solution were recorded with time in the range 300-1100 nm at room temperature, for a period of 50 min. Figure 4(a), shows a very weak formation of new bands in the region of 380 nm and 700-800 nm, implying a weak oxidation of the

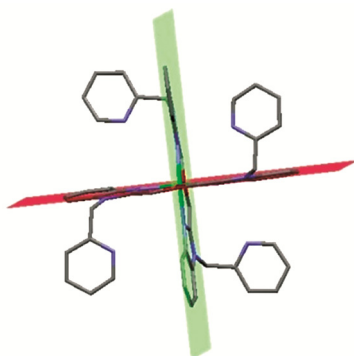


Fig. 3—Two planes mutually perpendicular passing from each of the tridentate ligands excluding the pyridine rings. The two terminal pyridine rings in each of the ligand are perpendicular to the respective planes lying above and below these planes.

aminophenol in presence of molecular oxygen and the Ni(II) complex²⁹. Average rate of reaction was found to be $1.4 \times 10^{-7} \text{ M min}^{-1}$. Since the reaction under molecular oxygen was quite weak, it was carried out in the presence of H_2O_2 .

Methanolic solution of Ni(II) complex $[\text{Ni}(\text{L})_2](\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ (2 mL, 1.2 mM) with 2.4 μL of 5.9 mM of H_2O_2 was added to a methanolic solution of 4-*tert*-butyl-2-aminophenol (2 mL, 11.9 mM). The ratio of catalyst:substrate: H_2O_2 is 1:10:5. This reaction mixture (0.2 mL) was diluted to make a 2.2 mL solution in methanol and the absorption spectra of this solution were recorded with time in the range 300-1100 nm at room temperature, for a period of 50 min. When Ni(II) complex was mixed with 2-amino-4-*tert*-butylphenol, new bands were generated in the region of 380 nm and 700-800 nm. The band at 380 nm suggests the formation of *o*-quinone of 2-amino-4-*tert*-butyl phenol²⁹. The band in the range of 380-400 nm increases in intensity with time. The concentration of product formed was calculated using the reported extinction coefficient of *o*-quinone³⁷. Absorbance versus wavelength plot is shown in Fig. 4(b). The presence of two bands found at 740 nm and 790 nm for the Ni(II) complex with the substrate suggests the formation of new intermediate species. Bands in this region have been earlier identified to be due to *o*-iminobenzosemiquinonato species derived from substituted the *o*-aminophenol³⁸. These bands also increase in intensity in parallel with the 380 nm band.

A blank experiment was also carried out with a methanolic solution of 4-*tert*-butylaminophenol (2 mL, 11.9 mM) mixed with 2 mL of methanolic

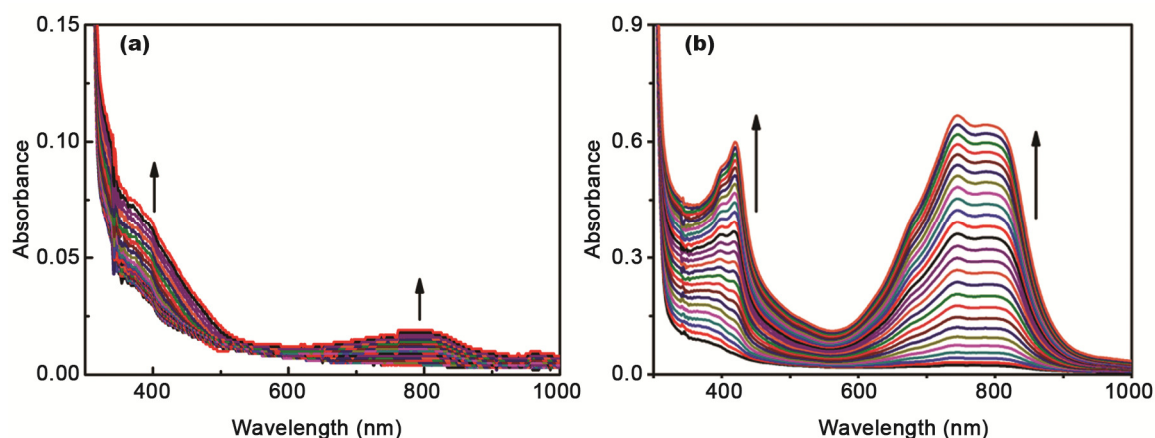


Fig. 4—Time dependant UV-visible spectral changes for the oxidation of 2-amino-4-*tert*-butylphenol catalysed by the Ni(II) complex $[\text{Ni}(\text{L})_2](\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ using (a) molecular oxygen and (b) using H_2O_2 , at room temperature for 50 min.

solution of 5.9 mM of H_2O_2 . The reaction mixture (0.2 mL) was diluted to 2.2 mL with methanol and absorption spectra were recorded in the range of 300-1100 nm at room temperature, for a period of 50 min. No new bands were generated for the blank reaction, implying the role of nickel complex in the oxidation reaction.

An experiment was carried out to check the effect of acetate ion on the rate of formation of *o*-quinone of 2-amino-4-*tert*-butylphenol. For this experiment, the concentrations of substituted aminophenol and Ni(II) complex were kept respectively at 1.2 mM (2 mL) and 11.9 mM (2 mL), acetate anion (3.6 mM, 2 mL) was added so as to keep the ratio of Ni(II) complex: substrate: H_2O_2 :acetate anion as 1:10:5:3 (Figs 5 and 6).

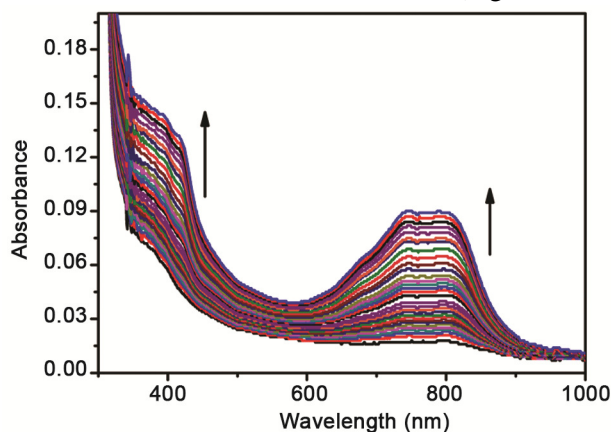


Fig. 5—Time dependant UV-visible spectral changes for the oxidation of 2-amino-4-*tert*-butylphenol catalysed by the Ni(II) complex $[\text{Ni}(\text{L})_2](\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ using H_2O_2 in presence of acetate anion at room temperature for 50 min.

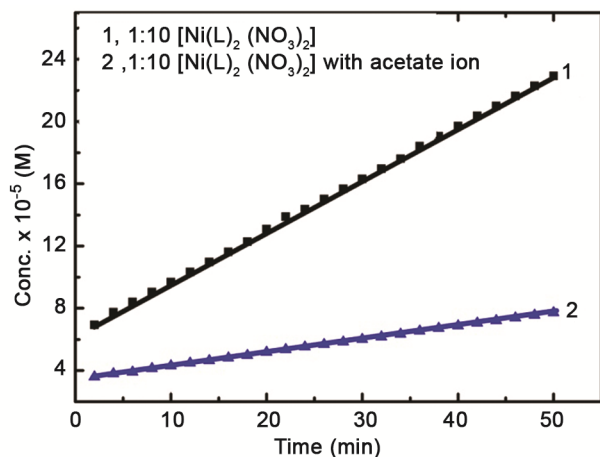


Fig. 6—Formation of 4-*tert*-butyl-*o*-benzoquinone versus time keeping complex concentration:substrate:hydrogen peroxide:: 1:10:5.

The rate of oxidation reaction was found to decrease by approximately four times.

Kinetic studies

The kinetics of the oxidation reaction between 2-amino-4-*tert*-butylphenol in presence of a small amount of Ni(II) complex was carried out in a manner reported earlier^{39,40}.

The amount of substrate (0.53, 0.7, 1.0, 1.5 mM) was varied while keeping the amount of Ni(II) complex fixed at 0.1 mM. Plots of concentration of *o*-quinone ($\lambda_{\text{max}} \sim 380$ nm) formed versus time given in Fig. 7 shows that the average rate of reaction increases as the concentration of the substrate increases at a fixed Ni(II) complex concentration. While keeping the concentration of Ni(II) complex constant a linear plot of logarithm of average rate of reaction plotted against the $\ln[\text{substrate}]$ was obtained with slope 1.0. This suggests a first order dependence on the substrate concentration.

The amount of Ni(II) complex (0.03, 0.07, 0.1 mM) was varied while keeping the amount of 2-amino-4-*tert*-butylphenol (substrate) fixed at 1.5 mM. Plots of concentration of *o*-quinone formed ($\lambda_{\text{max}} \sim 380$ nm) versus time is given in Fig. 8. The average rate was between $16 \times 10^{-7} \text{ M min}^{-1}$ and $63 \times 10^{-7} \text{ M min}^{-1}$. This shows that the rate of reaction increases as the concentration of the Ni(II) complex increases at a fixed substrate concentration. The logarithm of average rate of reaction was plotted against the $\ln[\text{conc. of complex}]$, while keeping the concentration of substrate constant. A linear plot with slope 1.0 was obtained, suggesting a first order dependence on the Ni(II) complex concentration.

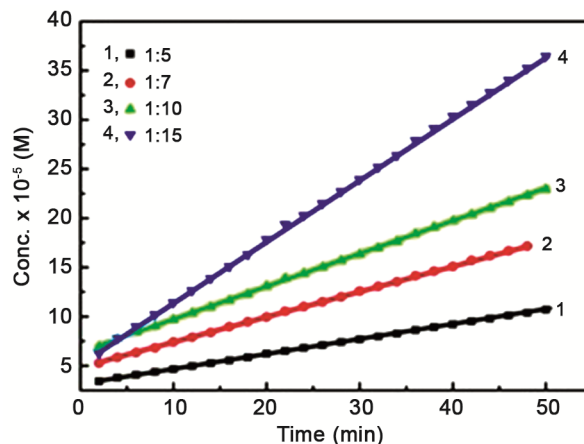


Fig. 7—Formation of 4-*tert*-butyl-1,2-benzoquinone versus time while keeping $[\text{Ni}(\text{L})_2](\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ concentration fixed and varying the substrate concentration.

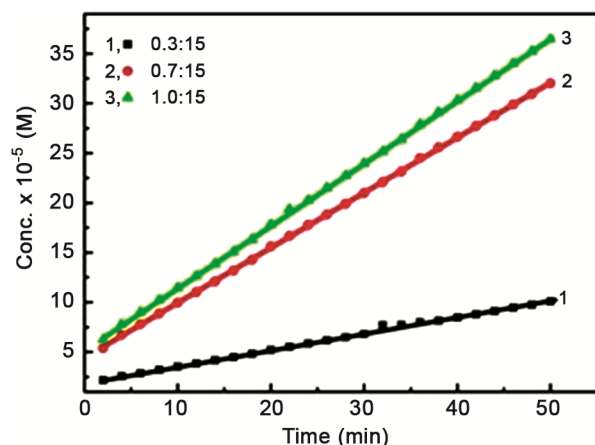


Fig. 8–Formation of 4-*tert*-butyl-1,2-benzoquinone versus time while keeping substrate concentration fixed and varying the $[\text{Ni}(\text{L})_2].(\text{NO}_3)_2.\text{H}_2\text{O}$ concentration.

The intermediate bands generated at $\lambda_{\text{max}} \sim 740$ nm and 790 nm increase in intensity, along with 4-*tert*-butyl-1,2-benzoquinone band ($\lambda_{\text{max}} \sim 380$ nm). An attempt was made to obtain the relative velocity of formation of the intermediate bands and this was compared with the velocity of formation of 4-*tert*-butyl-1,2-benzoquinone band, using the absorption versus time plots (Fig. 4(b)). It is found that the relative velocity of formation of the bands at $\lambda_{\text{max}} \sim 740$ nm and 790 nm are found to be only slightly higher to that for the band at $\lambda_{\text{max}} \sim 380$ nm. This suggests that the species formed at $\lambda_{\text{max}} \sim 740$ nm and 790 nm is likely to be involved in the formation of 4-*tert*-butyl-1,2-benzoquinone. The drop in rate of reaction in the presence of OAc^- ions for the Ni(II) complex, suggests that protons generated during the course of reaction are further required in another catalytic step. The OAc^- anions pick up these released H^+ ions, causing their concentration to deplete and thus impeding an intermediate step.

Conclusions

New Ni(II) complex with an *N*-substituted bis-benzimidazolyl ligand has been synthesized and characterized structurally and spectroscopically. $[\text{Ni}(\text{L})_2].(\text{NO}_3)_2.\text{H}_2\text{O}$ complex is utilized in the oxidation of 2-amino-4-*tert*-butylphenol to the corresponding 4-*tert*-butyl-1,2-benzoquinone in presence of H_2O_2 . Kinetic studies shows that the reaction follows first order dependence with respect to Ni(II) complex and substrate (2-amino-4-*tert*-butylphenol). The presence of acetate anion inhibits the reaction.

Supplementary Data

CCDC 1035680 contains the supplementary crystallographic data for the complex $[\text{Ni}(\text{L})_2].(\text{NO}_3)_2.\text{H}_2\text{O}$. These data can be obtained free of charge from the Cambridge Crystallographic data centre, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: (+44) 1223-336-033; Email: deposit@ccdc.cam.ac.uk) via www.ccdc.cam.ac.uk/datarequest/cif.

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