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Research article

Nicotine sensing behavior of nickel(II) complexes catalyzed oxidation and coupling reactions

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ABSTRACT

One of the main source of demise during the next ten years will be coronary heart disease and stroke, which are brought on by smoking (nicotine). To identify the percentage (%) of nicotine consumption by electrocatalytic sensor towards nicotine for target-specific prevent stroke, four uninuclear Ni^{2+} complexes of substituted butanimidamide Schiff base ligands [H_2L_{1-4}] was prepared. All the complexes were thoroughly analyzed by using several spectroscopic techniques such as CHNS analysis, FT-IR, NMR (^1H & ^{13}C) UV–Vis and NMR. The analyses showed tetradentate binding mode of ligand around nickel(II) metal ion leads to the structure of square planar with N_2X_2 (X = O, S) donor fashion. In addition, the well-defined nickel(II) complexes were utilized for oxidation of various alcohols such as cyclohexanol, and benzyl alcohol were produced to the assorted oxidized products with high yield respectively using greener co-oxidant (molecular oxygen). In addition, Nickel(II) complexes was further utilized as catalyst for aryl-aryl coupling reaction *via* Suzuki-Mayura method to obtain biphenyl compound. Furthermore, nickel(II) complexes were exploited for electrochemical detection of nicotine sensing in μM concentration.

1. Introduction

Schiff base transition metal complexes with the ligands bearing S,N or S,N,O donors are known to have outstanding stereo structure, redox and electron excitation activities [1–3]. This interest is quietly expanding, as off a significant exploration is now being adoring to the evolution of new transition metal ions with Schiff base complexes, in order to further development of applications in the focus of pharmaceutical and material chemistry [4–6]. Especially the 3d metal complexes [7–9] utilized for catalysts in variety of

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organic transformations such as isomerization [10], hydroformylation [11], coupling [12], hydrogenation [13], oxidation [14] and multiple sensor applications [15,16] etc. Deriving the carbonyl compound from the alcohols through the oxidation plays a key role in the pharmaceutical and chemicals industries. Metal complexes have been shown to mediate alcohol oxidation utilizing a number of co-oxidants including TEMPO [9], and O_2 or air [17], t-BuOOH [18], $2SO_8$ [19]. BrO^{3-} [20], NMO [21], PhIO [22],

Nicotine is a poisonous substance (alkaloid) found in tobacco plants that is frequently consumed [23,24]. Furthermore, neuroscience research has revealed that central nervous system will be harmed by the cigarette addiction, leading to neuro-degenerative illnesses and lung cancer including Parkinson's and Alzheimer's disease [25,26]. The World Health Organization (WHO) estimates that tobacco use accounts for 1.35 million deaths annually in India, making it one of the leading causes of disease and death there as well. Active or passive smoking kills more than 500,000 Americans, which was identified by the Centre for Disease Control and Prevention (CDCP) [27]. Tobacco smokers who inhale dried tobacco leaves suffer from a diverse range of health issues, including stroke, lung disease, asthma, heart attack and a harmful influence on the brain of human [25,26]. Tobacco has a nicotine content ranging from 1% to 3%, which defines its quality [28–30]. Furthermore, medically perilous quantity of 500–800 nm nicotine isolated in human saliva [31]. As a result, reliable nicotine content detection is critical in forensic science, toxicology tests, pharmaceuticals human bodily fluids and many other domains.

Influenced by the above facts, it is considered worthwhile to undertake the synthesis, and spectral investigation of synthesized nickel(II) complexes encompassing tetradentate Schiff base ligands as shown in Scheme 1. The efficacy of nickel (II) complexes assessed by reduction-oxidation behavior and electrocatalytic sensing behavior towards nicotine.

2. Experimental part

2.1. Materials and methods

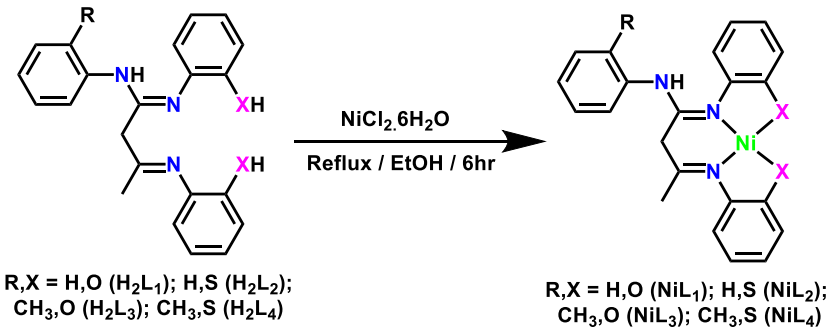
Every reagent that was utilized bought from Merck and was used as such. Melting points were measured using an uncorrected Veego VMP-DS melting point equipment. The CHNS analysis was done in Vario EL III CHNS analyzer at Cochin University, Kerala, India. PerkinElmer spectro-photometer is used to analyse the IR spectra having a good refinement of 4 cm^{-1} in transmittance mode at Saveetha School of Engineering, Chennai. In quartz cells, dichloromethane solution is utilized for the data collection of electronic spectra of all the complexes. The spectra were taken at ambient temperature using a Systronics twin beam UV–Vis Spectro-photometer 2202. NMR (^1H & ^{13}C) spectra were acquired in $CDCl_3$ at room temperature using a Bruker 500 MHz apparatus at Gandhigram Rural University in Dindigul. Standard calomel electrode is used as a potential reference for recording the electrochemical data by equipping glassy-carbon working electrode in acetonitrile medium.

2.2. Synthetic procedure for new Nickel(II) schiff base complexes

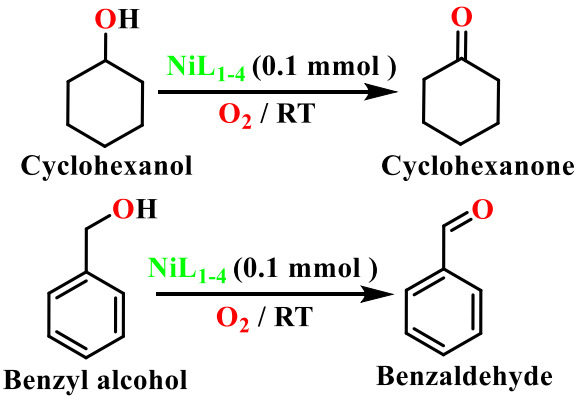
An ethanolic solution of metal salt [$NiCl_2{\cdot}6H_2O$] (0.023 g; 0.1 mmol) was added to the respective ligands (0.035–0.037 g; 0.1 mmol) in equal proportion and the wholesome content was heated for 6 h (Scheme 2) until the color changes. The final solution was kept aside for cooling after reducing the quantity to 3 mL. After four days crystalline colored precipitate was obtained dried under *vacuum*. All the attempts made to grow the single crystals failed.

2.3. Catalytic oxidation

In 20 mL of dichloromethane, substrate alcohols (0.07–0.13 mL; 1 mmol) were added and further nickel(II) complex (0.007–0.008 g; 0.01 mmol) solution is mixed through mild agitation added by maintaining the ambient temperature with oxygen environment for 6 h. Petroleum ether (60–80 °C) is used for the extraction of dried extract. For isolating the suitable carbonyl product 2,4-dinitrophenylhydrazene was added and converted to respective hydrazone derivative then the extracts were filtered and evaporated [33]. The ratio of substrate to catalyst is 100:1. The alcohols used as substrate were benzyl alcohol and cyclohexanol and their oxidized products were benzaldehyde, cyclohexanone and respectively.



Scheme 1. Preparation of nickel(II) Schiff base complexes (**$NiL_{1\text{-}4}$**).



Scheme 2. Conversion to alcohols to ketones/aldehydes by **NiL_{1-4}** Schiff base complexes.

2.4. C_6H_5–H_5C_6 coupling reactions

Turnings of magnesium (0.3g) was transferred into a glassware fixed with a sentinel tube containing fused $CaCl_2$. To activate the magnesium, an iodine crystal was added. In anhydrous Et_2O (5 mL), phenyl bromide [0.7 mL of total 1.8 mL] is dissolved and mixed with the above mixture and refluxed. After adding drops of the remaining bromobenzene in Et_2O (5 mL), the wholesome solution was refluxed for 40 min. This mixture was refluxed for 6 h with 1 mL (0.01mol) of phenylbromide in an-hydrous diethylether (5 mL) and the Ni^{2+} complex (0.05 mmol) chosen for study. The ether extract produced a crude biphenyl product after chilling and hydrolyzing with a saturated ammoniumchloride. This product was then isolated to get pure C_6H_5–H_5C_6 and equated favorably with an actual specimen [27]. The ratio of substrate to catalyst is 200:1.

2.5. Nicotine sensor

To begin, to get a shiny surface over the glassy carbon electrode (GCE, area = 0.07 cm^2), an usual cleaning procedure was adopted. Second, 6 μL of nickel complex dispersion was drop casted on the GCE surface and left to dry naturally. After that, nickel complexes were gently placed in de-ionised water to remove unbound materials from the electrode surface.

3. Results and discussion

A condensation type of reaction is carried out between equimolar ratio of acetoacetotoludide/actetoacetanilide with *o*-amino-thiophenol/o-aminophenol to get the Schiff base ligands (Scheme 1). The synthesis of Nickel(II) Schiff base complexes were achieved in good yields in which is prepared by the reacting metal salts [$NiCl_2{\cdot}6H_2O$] with selected ligands [H_2L_{1-4}] in benzene in equal proportion. In this reaction, H_2L_{1-4} acted as a dibasic tetradentate ligand in N_2S_2 fashion. The novel compounds exhibit remarkable stability and solubility in all typical organic media, while also being crystalline and non-hygroscopic. TLC on silica gel was used to check their purity. The analytical results acquired for novel complexes accorded well with the molecular formulas proposed (Table 1).

3.1. IR data interpretations

The accurate binding mode of the ligands and Ni^{2+} complexes were identified by comparing the IR spectra (Table 1 and Fig. 1). There has been no attempt is made to link a particular vibration to each and every band. The uncoordinated and CH stretching was appeared around 3000 cm^{-1}. The independent Schiff base ligands displayed a prominent peak in the area 1603-1627 cm^{-1}, which is the azomethine (>C═N-) group's characteristic frequency [31]. In all the complexes, the $\nu_{(C═N)}$ band is migrated to a lesser frequency 1495-1605 cm^{-1} (Fig. S1-S3), signifying the coordination of Schiff base ligand through the azomethine nitrogen atom. A medium intensity band found at 3000 cm^{-1} owing to ν(OH) in the autonomous Schiff base ligand $H_2L_{1,3.}$ A band that emerged at 1355 cm^{-1}

Table 1
Microanalytical informations of nickel(II) Schiff base complexes.

Complex	Color	Molecular formula	Molecular weight (Calculated)	M.P (°C)	Found (calculated) (%)			
					C	H	N	S
NiL_1	Green	$C_{22}H_{19}N_3NiO_2$	416.1	186	63.50 (63.3)	4.6 (4.49)	10.10 (10.02)	–
NiL_2	Brown	$C_{22}H_{19}N_3NiS_2$	447.04	234	58.95 (58.46)	4.27 (4.12)	9.37 (9.21)	14.31 (14.24)
NiL_3	Black	$C_{23}H_{21}N_3NiO_2$	430.13	210	64.22 (64.1)	4.92 (4.79)	9.77 (9.68)	–
NiL_4	Brown	$C_{23}H_{21}N_3NiS_2$	462.26	208	59.76 (59.71)	4.58 (4.49)	9.09 (8.98)	13.87 (13.69)

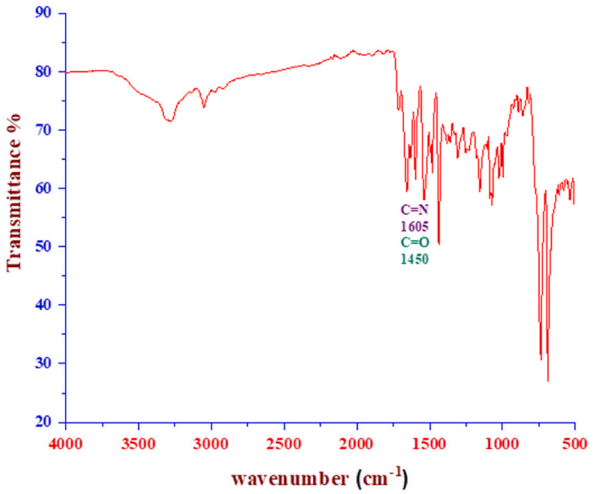


Fig. 1. FT-IR Spectrum of the NiL_1 Schiff base complex.

owing to phenolic-C-O elongation in the autonomous Schiff base ligand $H_2L_{1,3}$ has been moved to a greater frequency around 1450-1460 cm^{-1}, signifying the coordination amongst phenolic oxygen atoms [32]. In complexes containing H_2L_2 and H_2L_4 ligands, the peak belongs to the thiophenolic-SH is disappeared. Furthermore, in complexes [NiL_2] and [NiL_4], the absorption owing to v(C–S) of H_2L_2 and H_2L_4 at 1240 cm^{-1} is moved to a greater frequency range at 1230-1260 cm^{-1}, showing that metal ion coordinating thiophenolic sulphur atom [31–33].

3.2. *Electronic spectral interpretations*

Three strong bands were noticed in the electronic spectra of complexes recorded in dichloromethane solvent in the range 275–559 nm (Table 2 and Fig. 2). The electronic spectra of free ligands [33] appeared in the range 275–291 nm which can be designated to π-π* transitions due to the molecular orbitals available on the thiophenolic and phenolic chromophore. The coordination of lone pair electrons from the thiophenolic sulphur and phenolic oxygen atoms bounded to the Ni^{2+} ion has displaced these crests in the spectra of the complexes. Furthermore, the transitions at 352–559 nm attributed to *n*-π* transition due to aryl rings and azomethine moieties from the ligands are displaced in the spectra of complexes, representing the participation of imine group nitrogen coupled with the central metal atom. All of the compounds exhibited absorbance in the 248–472 nm range is assigned for the ligand-to-metal charge transfer followed by intra-ligand transformations. The distinctive sort of visible bands in the 572–614 nm range can be ascribed to d-d transformations concerning the orbitals of Ni^{2+} ion [34].

3.3. *^1H and ^{13}C NMR spectral studies*

The clearest characterization of the synthesized nickel(II) complexes was through their ^1H and ^{13}C NMR spectra (Table 3 and Figs. 3 and 4). From these complexes; protons of the phenyl rings were found as multiplets/triplets/doublets around 5.4–8.0 ppm whereas –NH group were observed at 8.03–8.8 ppm [21] (Fig. S4-S6). In all the complexes, CH_2, CH_3 and Ar-CH_3 groups were observed at around 3.82–4.19, 1.42–1.80 and 2.327–3.84 ppm respectively. During complexation, the phenolic –OH and thiolic –SH were disappeared. The ^{13}C NMR spectra of complexes NiL_1-NiL_4 showed C═N resonances at 149.15–149.86 ppm whereas aromatic carbon appeared in the region at 117–134 ppm which is comparable with the previously reported nickel(II) complexes. In addition, one sharp singlet in the range 40.07–44.11, 14.18–20.86 and 30.45–31.61 ppm range corresponded to methyl and methylene groups (Fig. S7-S9).

Table 2
FT-IR and UV–Visible spectral informations of nickel(II) Schiff base complexes.

Complex	C═N	C–O	C–S	UV–vis
NiL_1	1605	1450	–	252, 304, 564
NiL_2	1595	–	1230	261, 562
NiL_3	1597	1460	–	230, 265, 292, 447
NiL_4	1497	–	1260	262, 303, 562

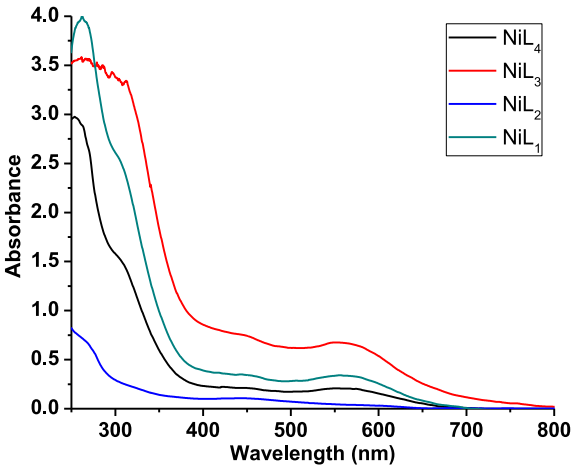


Fig. 2. UV–Visible spectra of the NiL_{1-4} Schiff base complexes.

Table 3
NMR spectral informations of nickel(II) Schiff base complexes.

Complexes	^{1}H - NMR	^{13}C - NMR
NiL_1	6.1–8.0 (m,ar), 8.03 (s,NH), 3.82 (s,>CH_2), 1.71 (s, -Me)	120-134 (aromatic C), 40.07 (>CH_2), 149.86 (–NH–C═N-Ph), 17.0(-Me), 136.34(–CH_3–C═N-Ph)
NiL_2	6.0–7.9 (m,ar), 8.06 (s,NH), 3.858 (s,>CH_2), 1.8 (s, -Me)	120-134 (aromatic C), 44 (>CH_2), 149.15 (–NH–C═N-Ph), 20.01 (-Me), 140.66 (–CH_3–C═N-Ph-)
NiL_3	5.4–7.9 (m,ar), 8.026 (s,NH), 3.86 (s,>CH_2), 1.8 (s, -Me), Ar-Me (s, 3.84)	120-134 (aromatic C), 44.11 (>CH_2), 30.45 (Ar-CH_3) 149.26 (–NH–C═N-Ph), 20.86 (-Me), 140.86 (–CH_3–C═N-Ph-)
NiL_4	7.25–7.79 (m,ar), 8.8 (s, –NH), 4.19 (s,>CH_2), 1.42 (s, -Me), Ar-Me(s, 2.327)	117-132 (aromatic C), 43.1 (>CH_2), 149.86 (–NH–C═N-Ph), 14.18 (-Me), 136.34 (–CH_3–C═N-Ph-), 31.61 (Ar-CH_3)

3.4. Catalytic applications

3.4.1. Oxidation of alcohols

Various substrates, such as benzyl alcohol and cyclohexanol, were oxidized. At room temperature, **NiL_{1-4}** complexes act as catalysts in dichloromethane with molecular oxygen as a co-oxidant. In this oxidation reaction, there is no detectable oxidized product without catalyst as nickel complexes in presence of co-oxidant. According to the below reaction conditions, a variety of alcohols such as cyclohexanol and benzyl alcohol were produced to the assorted oxidized products whereas the activity is tremendously improved to the new nickel(II) complexes. The oxidized products are summarized in Table 4 (Scheme 2). The aldehyde/ketone derivates were measured as 2,4-dinitrophenylhydrazones. All of the catalysts, such as nickel complexes, were discovered to catalyze the conversion of alcohols to their oxidized products like ketones/aldehydes, but the yields and turnover rates varied with each catalyst. Notably, the greater yield reported for benzyl alcohol oxidation over cyclohexanol is owing to the α-CH molecule of $C_6H_5CH_2OH$ being more acidic than cyclo-hexanol [33,35,36].

3.4.2. C_6H_5–H_5C_6 coupling reactions

C_6H_5–H_5C_6 reactions are cutting-edge techniques for the synthesis of numerous significant chemicals. Formation of C_6H_5–H_5C_6 by the newly prepared Ni^{2+} complexes utilized as catalysts (Table 4 and Scheme 3). The schematic method involves the reaction of phenylmagnesiumbromide with phenylbromide to produce biphenyl. Phenylbromide was initially transformed into the RMgX. The aforementioned reagent was then treated with phenylbromide, followed by the catalyst, and the wholesome quantity was heated for 6 h. After completing the reaction, the product biphenyl was isolated from the reaction mixture. There was very little biphenyl generated when the Ni^{2+}catalyst was not used in the reaction. When equated to the quantity of biphenyl derived by nickel complexes a catalyst vs those obtained without catalyst, this is greater in quantity [33]. The product yields were depicted in Table 4.

3.5. Nicotine sensing behavior

The electro-catalytic response of nickel complexes in PBS (0.1 M, pH 7.3) was investigated using cyclic voltammetry (CV) with 0.08 mM nicotine (Fig. 5 and S10-S12). The strong electrocatalytic character of the nickel complexes led to the discovery of a particularly nicotine's high oxidation peak at +900 mV. Concurrently, nickel complexes were used to investigate nicotine (0.08 mM) oxidation. On

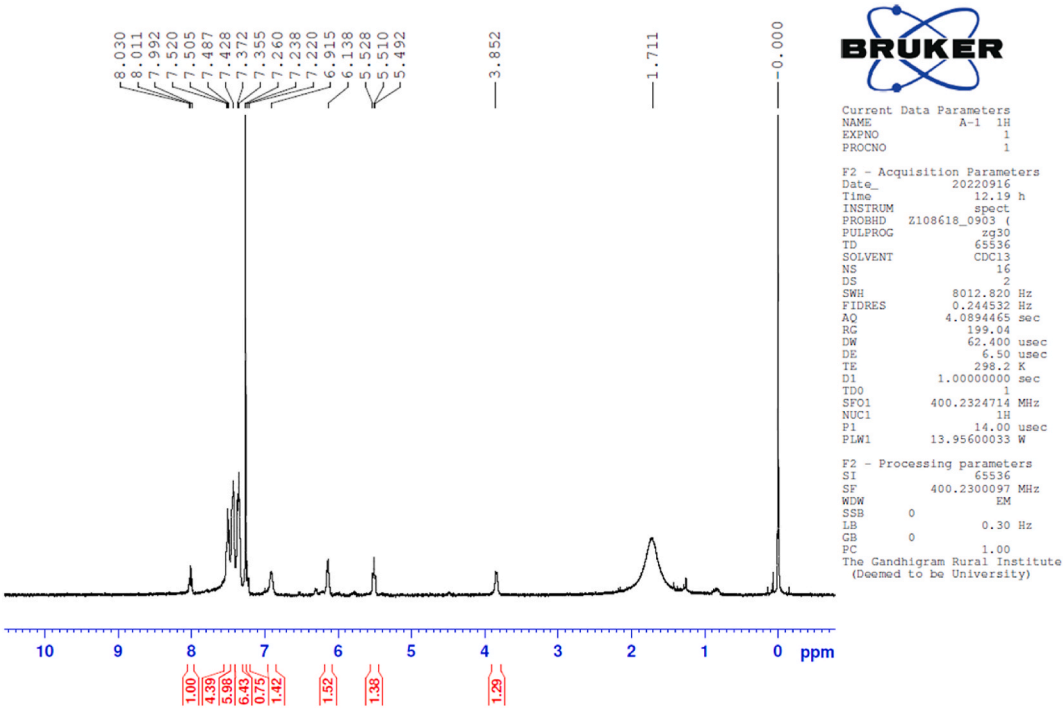


Fig. 3. ^{1}H NMR spectrum of the [NiL$_1$] Schiff base complex.

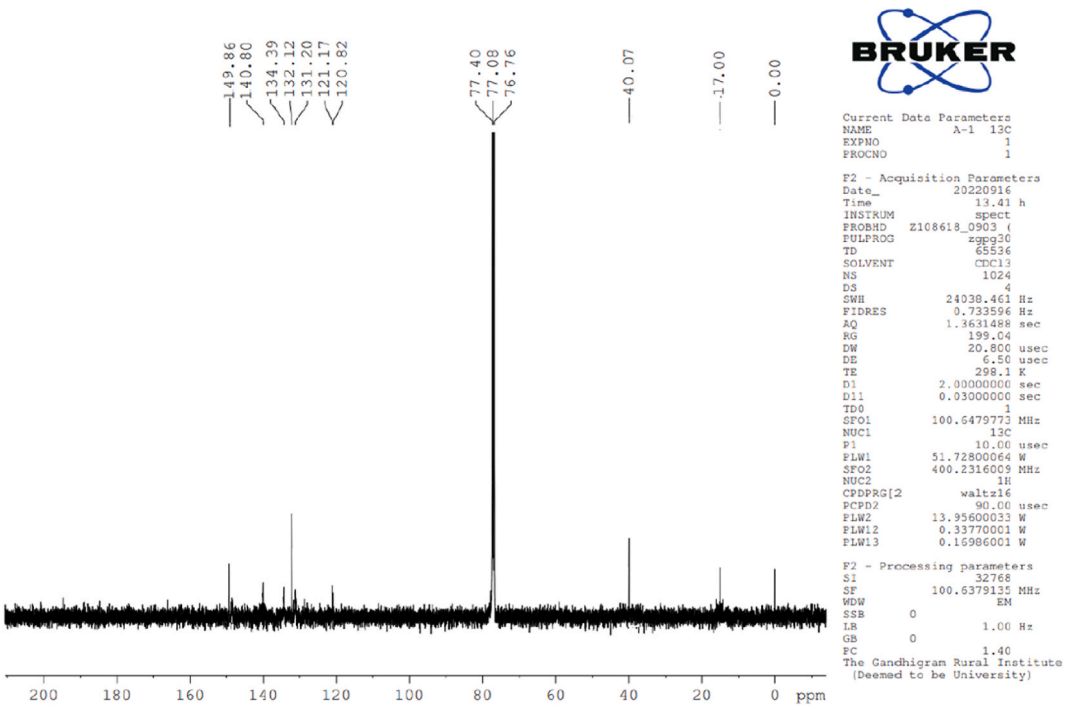
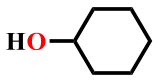
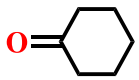
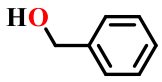
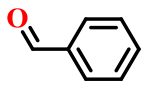
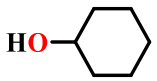
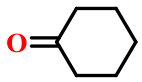
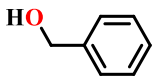
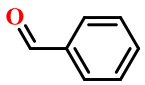
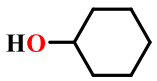
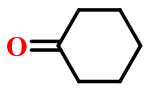
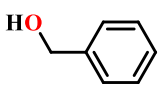
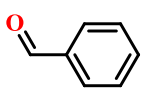
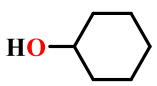
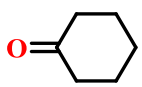
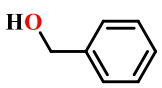
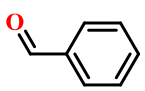


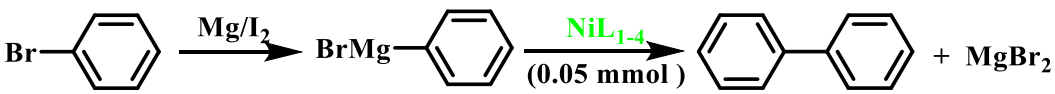
Fig. 4. ^{13}C NMR spectrum of the [NiL$_1$] Schiff base complex.

nickel complexes in PBS without nicotine, no irreversible oxidation peaks were seen. However, after injecting 0.08 mM nicotine into PBS, nickel complexes showed an oxidation peak at + 0.948 V and a peak current of 0.0764 mA/cm^2. This proved that nickel complexes with a high overpotential are the sites of nicotine oxidation. Nickel complexes were one of the best for nicotine oxidation at

Table 4
Catalytic efficiency of Nickel(II) Schiff base complexes.

Complex	Oxidation of alcohols				Aryl-aryl coupling	
	Substrate	Product	Yield[a]	TON[b]	Yield (g)	Yield (%)
$[NiL_1]$			31	31	0.67	70
			37	37		
$[NiL_2]$			29	31	0.71	73
			32	33		
$[NiL_3]$			39	41	0.69	72
			44	44		
$[NiL_4]$			28	28	0.73	75
			34	36		

[a] Yield was calculated using GC and compared to the results of real sample analysis.
[b] The proportion of product moles obtained to catalyst moles utilized.



Scheme 3. C_6H_5–H_5C_6 Coupling reactions of the nickel(II) Schiff base complexes.

lower potential (+0.9 V) and higher current density (0.57 mA/cm^2). These characteristics suggested that a nickel complexes hybrid film might be employed to minimize the overpotential for nicotine electro-catalytic oxidation. The phenyl moiety of nicotine and the abundant electron density of nickel complexes interact *via* π-stacking, which improves the electrocatalytic effect. Throughout electrocatalysis, the nicotine molecule's pyrrolidine group is electro-oxidized by n-methyl hydroxylation (NMH) [37–40].

4. Conclusions

In this work, we have described the new nickel(II) complexes encompassing Schiff base ligands such as acetoacetotoludide/actetoacetanilide with *o*-aminothiophenol/o-aminophenol in 1 : 2 stoichiometric ratio. The formation of new nickel(II) complexes were established by analytical and various spectroscopic (NMR, IR and electronic) methods. The geometry of complexes NiL_1-NiL_4 revealed a square-planar arrangement around the metal ion *via* tetradentate fashion. Furthermore, the well-defined nickel(II) complexes exploited as a highly active catalyst for the oxidation of variety alcohols to obtained assorted appropriate oxidized products using molecular oxygen as a greener oxidant, which is eco-friendly method. Likewise, nickel(II) complexes further utilized for the synthesis of biphenyl product by aryl-aryl coupling reaction *via* Suzuki-Mayura method. Moreover, the synthesized nickel(II) Schiff base complexes were exploited sensing behavior towards the detection of nicotine at low concentrations by electrochemically. Starting

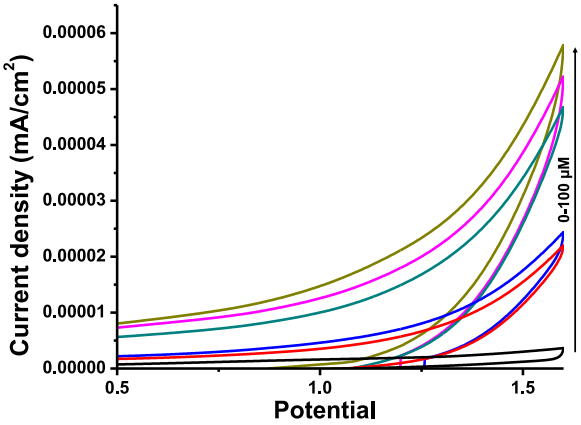


Fig. 5. Electrochemical behavior of NiL_1 complex in PBS buffer solution with increasing concentrations of nicotine (0–100 μM).

off, it can be said that even at low concentrations, every nickel complex found in this study demonstrates noticeably high selective catalytic/sensing abilities. Consequently, the creation of a new catalyst would undoubtedly take this class of coordination chemicals into consideration.

Data availability statement

The article and/or the supplemental material provide all the information required to assess the conclusions drawn in the work.

CRediT authorship contribution statement

Madaselvi Rathinam: Writing – review & editing, Writing – original draft, Validation, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Paranthaman Vijayan:** Writing – review & editing, Writing – original draft, Validation, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Selvakumar Balasubramanian:** Writing – original draft, Validation, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Sasikumar Ponnusamy:** Writing – review & editing, Writing – original draft, Validation, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Arunachalam SaravanaVadivu:** Writing – review & editing, Writing – original draft, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Mohamed Abbas:** Elhamid Abbas, Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Brindha Banu Balakrishnan:** Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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