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SYNTHESIS, CHARACTERIZATION AND SPECTROSCOPIC STUDY OF NICKEL(II), PALLADIUM(II) AND PLATINUM(IV) COMPLEXES WITH MIXED LIGANDS OF (Z)-N'-(FURAN-3-YLMETHYLENE)ISONICOTINOHYDRAZIDE-CHLOROANILIC ACID

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ABSTRACT. The new complexes of nickel(II), palladium(II) and platinum(IV) with mixed ligands,L^1: (E)-N'-(furan-2-ylmethylene)isonicotinohydrazide and H_2L^2: 2,5-dichloro-3,6-dihydroxycyclohexa-2,5-diene-1,4-dione were synthesized and characterized by FT-IR, EI-MS, NMR and UV-Visible-NIR spectroscopy. The new complexes were prepared in ethanol solutions and in solid state on the 1:1:1 mole ratio of metal ion to the two ligands, L^1 and H_2L^2, respectively. The physicochemical properties; melting point, magnetic susceptibility and molar conductivity were determined. The effect of pH revealed the stability of complexes in solutions. The IR spectra confirmed the bi dentate chelation of chloroanilic acid and HL^1 *via* two –OH sites and nitrogen immine group and oxygen atom of carbonyl moiety, respectively. The observed data form elemental analyses and spectroscopic measurements investigated the octahedral environment around Pt(VI) ion, whereas the geometry of Ni(II) and Pd(II) approved as square-planner.

KEY WORDS: Mixed-ligands of isoniazid, Chloroanilic acid, Spectroscopic properties of (Z)-N'-(furan-3-ylmethylene)isonicotinohydrazide

INTRODUCTION

The mixed ligands of isonicotinic acid hydrazine and its derivatives have attracted great attention in the last decade due to their unique properties [1, 2] and their metal complexes are extensively studied with wide applications of LED industry, trace analyses of toxic metals[3, 4]. A number of functionalized compounds conducted with 2,3-dichloro-5,6-dihydroxycyclohexa-2,5-diene-1,4-dione are readily accessible, so they are promising rich and versatile coordination chemistry and they have heterocyclic π-conjugated systems with good co-ordination abilities to metal centers and own the capability to exhibit luminescence [6-8]. The intra-ligand charge transfer bands located on the free molecules of benzoquinone are mainly wide employed in the applications of LED and photolysis degradation of plastic and many natural pigments [9, 10]. However the incorporation metal ions or mixed ligands beside benzoquinone derivatives might have been developed the emission spectra of the new metal chelates and this may lead to new physical properties in the industrial applications like sensitizer of photochemistry and LED modifications [11, 12].

Material and methods

The starting materials and solvents were purchased from Santa Cruz Bio-technology company used as received without further purification like isoniazid and chloroanilic acid. Melting points were recorded by a Stuart melting point (digital) SMP30 apparatus. FT-IR spectrum was recorded on a Shimadzu 4800 s spectrophotometer in the range (4000-250) cm^{-1}. The electronic spectra of the ligands and their complexes were recorded by Shimadzu UV-Vis 160 ultraviolet spectrophotometer at 25 °C using 1cm quartz cell and examined at the range at (200-1100) nm at

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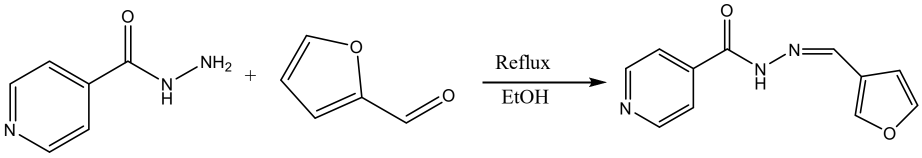
10^{-3} M in methanol. The content of nickel, palladium and platinum in their complexes were estimated by flame atomic absorption spectrometry (FAAS) using a Shimadzu AA680G atomic absorption spectrophotometer at the laboratories of Ibn-Sinaa Company. Elemental analysis for the primary ligand, L^1 and secondary one, HL^2 and their complexes were determined by (CHN) calibration Linear Regression Euro EA elemental analysis. The suggested molecular weight of the two ligands and their complexes were measured by electron ionization mass spectroscopy on GC-MS *via* ES technique. ^{1}H, ^{13}C-NMR spectrum of primary ligand, L^1 and metal complexes were recorded on Bruker DMX-500 spectrophotometer (400 MHz), by using DMSO-d_6. The molar conductance of complexes solutions in DMF solvent were determined at 298°C using an Inolab Muli 740, WTW 82362-Germany. Magnetic susceptibility of prepared complexes was determined at 310 °C by Auto magnetic susceptibility Balance in Mustansiriyah University, College of Science, Chemistry Department.

Synthesis of (E)-N'-(furan-2-ylmethylene)isonicotinohydrazide, L^1

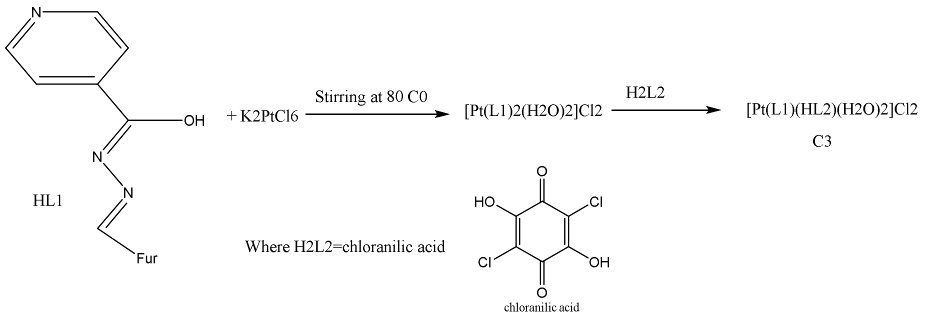
To the solution of isonazid (1.37 g., 10 mmol) in 15 mL of absolute ethanol, 2-formylfuran (0.966 g 10 mmol) in 10 mL ethanol was added. The mixture was refluxed for 2 hours, the reaction was monitored by TLC, and then mixture was allowed to cool down. A solid product was filtered, washed with cold water, dried and re-crystallized from hot ethanol to give compound L1; Scheme 1; M.P. 188-190 °C; color, yellow crystal; Yield 82%; FT-IR υ (cm^{-1}): 3320 (NH_2, asym.), 3100 (NH_2, sym.), 1650 (C=O, amide); R_f (hexane : ethyl acetate; 3:2), ^{1}H NMR (400 MHz, DMSO-d_6), 10.70 (d, 1H, O=C-NH), 8.22 (s, 1H, CH=N-), 7.78 (d, 2H, CH=CH-Py), 7.55 (d, 2H, CH=CH-Py), 6.66 (m, 3H, CH=CH-Furan).



Scheme 1. Synthesis of primary ligand, L^1.

Metals complexes synthesis

To a solution of the metal salts in ethanol, where the salts of ($NiCl_2.2H_2O$, $PdCl_2$ and $PtCl_4$) was added to the primary ligand (2.15 g, 10 mmol) and after stirring the solution on water bath for 15 min, a remarkable change in color turned from green in case of NiL complex to dark olive, the solution of secondary ligand, (2.07 g, 10 mmol) H_2L^2 was added in 10 mL methanol then refluxed the mixture for 2-3 hours. After keeping the mixture for several hours at room temperature, a colored precipitated was form, orange complex of PdL, red complex of NiL and dark yellow C3 complex. The color melting point, yield, metal analysis and solubility of the ligand and it's complexes are given in Table 1.



Scheme 2. Synthesis of metal complexes with mixed ligands.

Table 1. Physical properties and elemental analyses of the prepared compounds

Compound	Color	M.P.	Mwt	Elemental analysis % found (cal.)			Metal M% Found (cal.)	M:L	Suggest formula
				C%	H%	N%			
L^1	Yellow	180-182	215.07	60.99 (61.90)	3.90 (4.22)	18.22 (19.54)	-		$C_{11}H_9N_3O_2$
H_2L^2	Orange	> 300	208.09	36.11 (40.0)	3.00 (3.33)	12.26 (14.0)	-		$C_6H_2Cl_2O_4$
C1	Red	280-282	398.2	29.11 (30.22)	2.07 (1.99)	7.64 (8.50)	19.22 (18.95)	1:2	$C_{13}H_8Cl_2N_3NiO_4$
C2	Orange	290 d	445.97	45.1 (32.4)	3.10 (2.97)	10.72 (18.93)	25.77 (24.39)	1:2	$C_{13}H_8Cl_2N_3NiO_4$
C3	Dark yellow	315 d	641.99	27.76 (26.66)	2.19 (2.44)	15,30 (15.55)	34.02 (33.17)	1:2	$C_{13}H_8Cl_4N_3PtO_6$

RESULTS AND DISCUSSION

The main NMR and MS spectroscopy are the basis for the characterization of the prepared ligands and their mixed metal complexes with nickel(II), palladium(II) and platinum(IV).

^1H NMR spectrum for ligand HL^1

The NMR spectrum of HL^1 in DMSO exhibited weak signal at 12.5 ppm assigned to de shielded –NH proton attached to amide group. As well as the multiple absorption at (6.95-7.5) and (7.92-8.11) ppm are attributed to spin-spin coupling of pyridine and furan rings respectively [13, 14]. Furthermore, the singlet peak at 8.97 ppm is strong evidence for the nuclear spin of immine –CH=N– formed up on condensation of formyl moiety of furan-2-carboxaldehyde and amino group of isoniazid, Figure 1. As well as the data of ^{13}C NMR spectrum of HL^1 was presented in Table 2. The resonance of amide carbon HN-C=O and immine –CH=N– were resonated at 165.25 and 195.50 ppm, respectively, which are good support for condensation reaction of HL^1 [13, 15].

^1H NMR spectrum for $[Pd(L^1)(HL^2)(H_2O)_2]$

The ^1H NMR is main spectroscopic method to show the comparison of chemical shifts between the mono or bi basic chelating ligands with metal chelates, here the shift in the –CH=N- to lowering absorption at 8.18 ppm is assigned to binding of nitrogen atom to the empty orbitals of palladium(II) ion. As well as the disappearance of hydrogen atoms in the enol form of HL^1 ligand in the NMR spectrum of Pd(II) complex revealed the participation of HO-C=N-NH- moiety via de protonation, Figure 2. As well as, the singlet peaks at 9.16 and 8.90 ppm in the spectrum of complex is good evidence for the secondary ligand involving aromatic protons of 1,4-benzoquinine ring [15].

^{1}H NMR spectrum for $[Pt(L^1)(HL^2)(H_2O)_2]$

By the same manner, the chemical shift of –CH=N- in Pt(IV)L complex in DMSO-d_6 solvent showed remarkable lowering absorption to 8.10 ppm is assigned to binding of nitrogen atom to the empty orbitals of platinum(IV) ion. As well as the disappearance of hydrogen atoms in the enol form of HL^1 ligand in the NMR spectrum of Pt(IV) complex revealed the participation of HO-C=N-NH- moiety via de protonation. As well as, the singlet peaks at 9.95 and 10.77 ppm in the spectrum of complex is good evidence for the secondary ligand involving aromatic protons of 1,4-benzoquinine ring [13, 17], Figure 3.

Table 2. NMR data of HL^1 and its C1 complex.

Compound	Chemical shift values in ^{1}H NMR and ^{13}C NMR (ppm) with assignment
HL^1	^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ: 12.50 (s, NH-C=O), δ: 6.95-7.50 (Pyridine-CH=CH), 7.92-8.11 (m, 3H, furan-H), 8.88 (s, 1H, -CH=N-NH), 2.50 -3.321 (s, DMSO solvent). ^{13}C NMR (500 MHz, DMSO-*d*$_6$) δ: 39.32-40.57 (C-DMSO-solvent), 120.40, 129.26 (Py-CH=CH-), 127.10 (furan-r-CH=CH-), 129.87, 139.85, 133.69 (C-O furan), 136.91,165.25 (-C=N-), 195.50 (HN-C=O).
C1	^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ: 11.70 (s, OH-benzoquinone), δ: 6.66-7.43 (Pyridine-CH=CH), 7.90-8.09 (m, 3H, furan-H), 8.50 (s, 1H, -CH=N-NH), 2.50 -3.321 (s, DMSO solvent). ^{13}C NMR (500 MHz, DMSO-*d*$_6$) δ: 31.81 (CH3-NH), 39.32-40.57 (C-DMSO), 124.48 (HC=CH-furan), 125.11-125.04 (2C, C=C=Ar), 127.01 (-C=C-benzoquinone), 125.90 (CH=CH-benzoquinone), 130.66 (C=C-furane), 132.21 (C-O furan), 145.79 (C-N-Pz), 154.12 (-C=N-Pyridine), 190.42 (1-C=O,quinone), 210 (3-C=O, quinone.

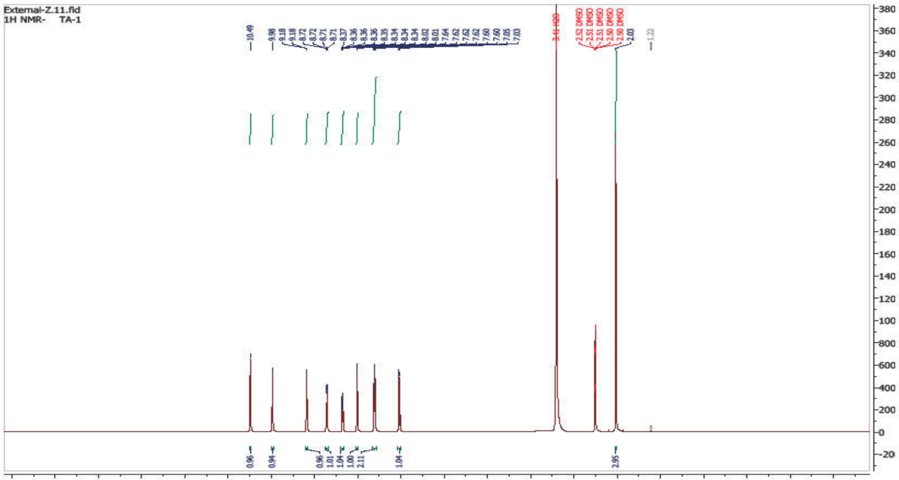


Figure 1. ^{1}H NMR spectrum of L^1 in DMSO-d_6 solvent.

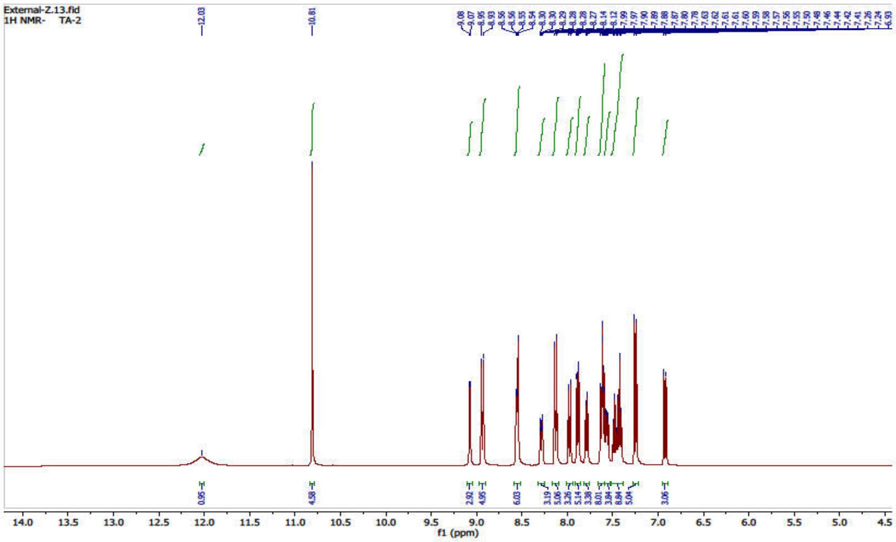


Figure 2. ^{1}H NMR spectrum of PdL complex in DMSO-d_6 solvent.

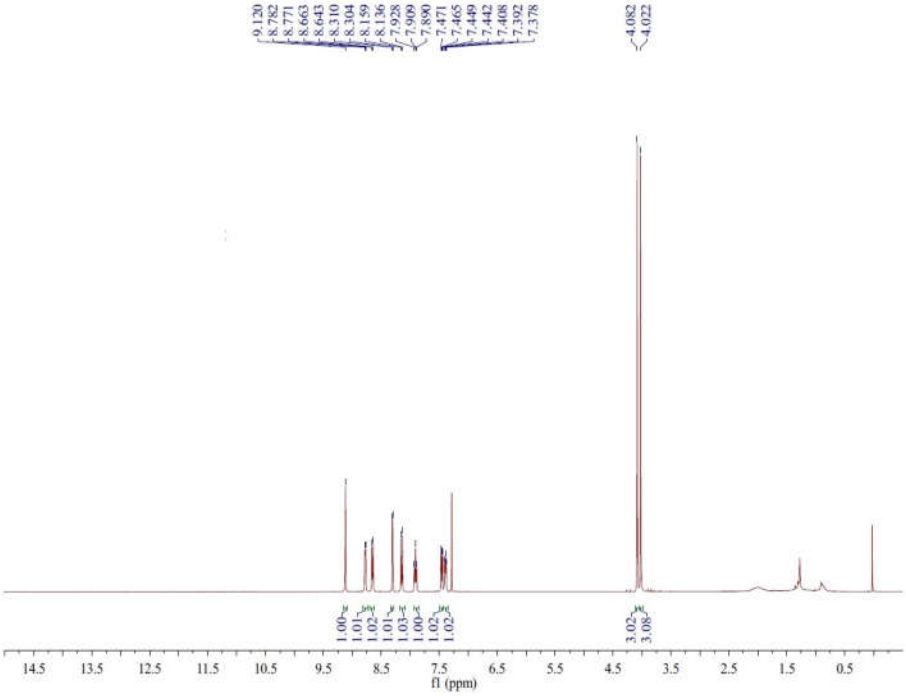


Figure 3. ^{1}H NMR spectrum of PtL complex in DMSO-d_6 solvent.

Mass spectral (MS)

The exact molecular weights of the free primary ligand of isoniazid and its metal complexes formed beside the secondary ligand of benzoquinone, H_2L^2 were depicted through the measurement of electron-spray ionization mass spectroscopy [16]. The MS spectrum of HL^1 exhibited high intensity peak at m/z = 216 assigned to its expected chemical formula. As well as the other variable peaks at 195, 110 and 87 may be attributed to the fragmentation of amino, -OH and furan moieties, respectively. However, the MS spectra of C1, C2 and C3 complexes showed their molecular ions in the mass spectra at 398, 447 and 630, respectively [18, 19]. These base peaks are good evidence for the isotopes of ^{37}Cl and ^{35}Cl isotopes.

FT-IR spectrum of ligand and its complexes

The main bands of FT-IR spectrum of ligand and its complexes are presented in Table 3. The free ligand shows strong bands at 1280 and 1620 cm^{-1} assigning to the vibrations of thioamide –C=O and –C=N– of pyridine and benzoquinone ring. The chelation of nickel(II), palladium(II) and platinum(IV) ions to the active sites –C=O and –N=CH groups have been observed from the lowering in their absorptions to the regions (1670-1580) and (1390-1260) cm^{-1}. As well as the weak to medium bands at around (560-500) and (450-410) cm^{-1} are associated with the coordination bonds of (M-N) and (M-O) respectively [18-20]. As well as the broad bands at (3400-3500) cm^{-1} is assigned to stretching frequency of O-H moiety of coordinated water molecules in C3 complex [15, 18].

Electronic spectra and magnetic susceptibility of complexes

The UV-Vis absorption spectrum of isoniazid Schiff base, HL^1 in methanol exhibited three absorption bands. Two bands at (250 nm, 25000 cm^{-1}) and (290 nm, 35180 cm^{-1}) transitions [19], Figures (4-a,b) and (5-a,b).The solutions of C1, C2 and C3complexes in methanol (10^{-3} M) displayed weak absorptions at (400-1100) nm assigning to d-d spectra and LMCT bands, Figure 4. The square planner complexes of nickel(II) and palladium(II) exhibited weak bands at (510-390) and (350-288) nm which are mainly attributed to $^1A_1g\rightarrow{}^1B_1g$ and $^1A_1g\rightarrow{}^1Eg$ transitions respectively [22, 24]. Furthermore the diamagnetic properties of all complexes is in well-agreement of low-spin state of Pd(II) complex having a d^8 configuration favors the formation of complexes with square planar geometry and d^6 configuration with strong field of the benzoquinone,HL^2 and isoniazid, L^1 ligands, Figure 5.

Molar conductivity of metal complexes

The solutions of metal complexes in DMF solvent (0.001 M) showed electrolytic properties molar conductance values in the region (98-140) $ohm^{-1}.cm^2.mol^{-1}$ which is supporting for the presence of chloride ions in the outer-sphere of complexes structures [25] for C1-C3.

Effect of pH and concentration on Pt (IV)-ligand solutions

The UV-Vis spectra were measured for a set of mixed solutions containing equal quantities of same concentration for Pt^{4+} ion and benzoquinone, HL^2 solutions. The solutions of (10^{-2}-10^{-4}) M were studied while the pH range was between 4.5 and 8.5. Solutions of 10^{-2} M were turbid and out of scale absorbance, while the light color of the 10^{-4} M solutions make them very hard to study and need more time to change solution color, while the solutions sited within the 10^{-3} M concentration obey Lambert-Beer's law and showed a clear intense color [25-27]. A calibration curve was plotted on absorbance against molar concentration for 0.25×10^{-3}, 0.5×10^{-3}, 0.75×10^{-3}, 1×10^{-3} and 1.25×10^{-3} M solution at pH = 6.0 best-fit straight lines were obtained, with correlation factor (r = 0.968).

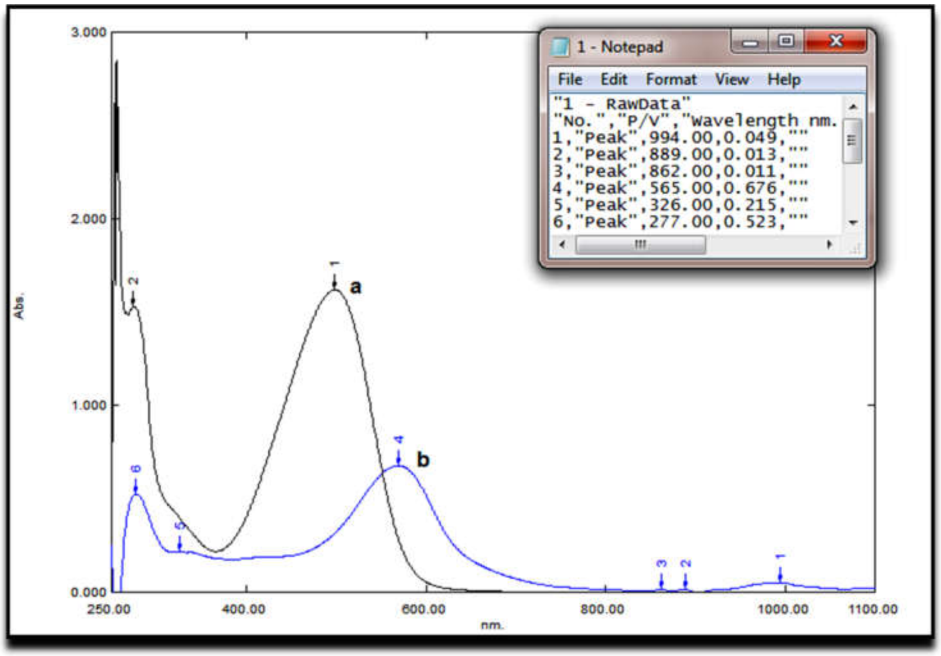


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Figure 4. The UV-Vis spectra of (a) free HL^1 solution in methanol and (b) Pd(II)-L^1-HL^2 solution at pH = 6.5 and concentration = $1x10^{-4}$M.

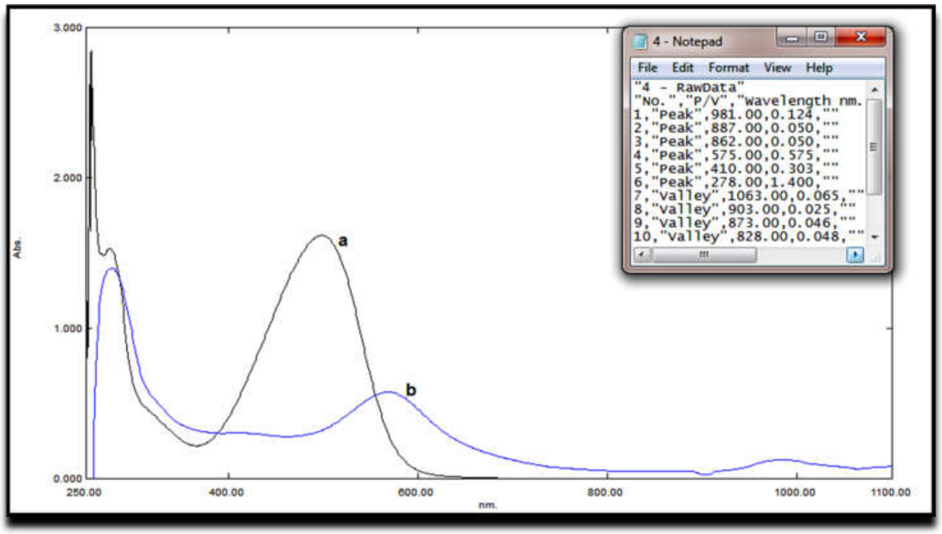

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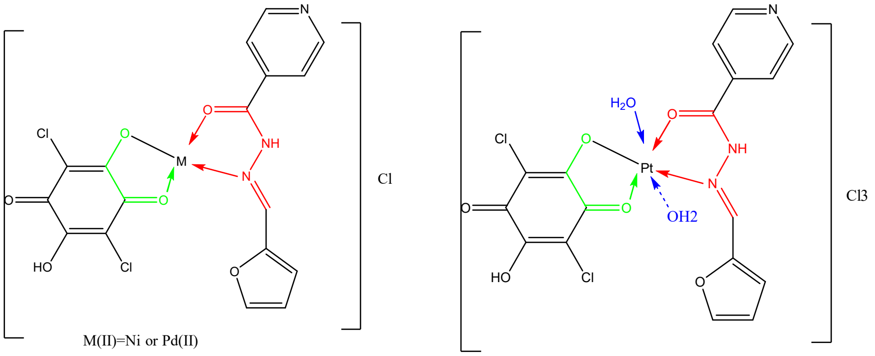
Figure 5. The UV-Vis spectra of (a) free HL^1 solution and (b) Pt(IV)-L^1-HL^2 mixed solution at pH = 6.

CONCLUSION

According to the data observed from elemental analyses and spectroscopic analyses, the square planner geometry around nickel(II) and palladium(II) ions were adopted, Scheme 3. As well as, the intra-ligand charge transfer is the distinct property of 1,4-benzoquinine ligand which is observed in the UV spectra of all prepared metal complexes. As well as the spectroscopic study of platinum(IV) and nickel(II) complexes with mixed ligands of benzoquinone-isonazid showed high stability in solution with respect to pH range (5.5-8.5) which it will be so interested us in the future work to use them in LED and photolysis applications.



Scheme 3. Octahedral structure of C3, and square-planner of C1 and C2 complexes.

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