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Synthesis and characterization of oxo-peroxo vanadium complexes derived from 2-hydroxy-1-naphthaldehyde isonicotinoylhydrazone

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ABSTRACT

This paper describes the preparation, characterization and structural assessment of oxo-peroxo vanadium complexes derived from 2-hydroxy-1-naphthaldehyde isonicotinoylhydrazone (H_2npih). After presenting in brief the rationale for the present work, preparation and characterization of the complexes is described. The molecular peroxo compound (**1**) was isolated from reaction of V_2O_5 dissolved in 30% H_2O_2 with hydrazone in ethanol. Further, alkali metal oxo-peroxo vanadium complexes (**2**) to (**4**) were prepared from reaction of V_2O_5 dissolved in 30% H_2O_2 with hydrazone in ethanol medium raising the pH of the reaction medium by alkali metal and ammonium hydroxide to about 6.5. Similarly, pyridinium and substituted pyridinium oxo-peroxo complexes (**5**) to (**7**) were obtained by carrying out reactions in the presence of pyridine or 3-picoline or 4-picoline instead of alkali metal and ammonium hydroxides. These complexes have zero magnetic moment values indicating their diamagnetic nature. Further, the compounds are ESR inactive. The analytical data and Infrared spectroscopic studies of these complexes are suggested to have a seven coordinate polyhedra around the metal centre and their tentative structures have been assigned.

Keywords: *hydrazone, isonicotinoylhydrazine, oxo-peroxo, polyhedra, tridentate*

1. INTRODUCTION

Vanadium occurs as an "essential trace" element in diverse living organisms and has wide spread involvement in enzymatic and physiological activities [1-2]. The coordination chemistry of vanadium complexes are of immense interest due to their significance in various biochemical, pharmacological and catalytic activities [3-4]. Vanadium possesses the ability to assume various oxidation states ranges from -1, 0, +1, +2, +3, +4, +5 [5]. Under physiological conditions, *in vivo*, vanadium complexes are usually stable in their +4 and +5 oxidation states. Among various transition metal complexes, the importance of vanadium chemistry is currently receiving considerable recognition owing to its diverse applications in biology, pharmacology and their catalytic activity.

Peroxovanadium(V) species have attracted the attention of pharmacologists with respect to their insulin mimetic functions [6,7], namely, vanadium(V) exerts insulin mimetic activities in a synergic manner with peroxide [8]. Peroxovanadium(V) species formed in vivo have been considered to be relevant to insulin mimesis [9]. It has been demonstrated that discrete peroxovanadium(V) complexes are also effective insulin mimics [10-12]. However, it is anticipated that the chemistry of the insulin mimetic activities of peroxovanadium(V) complexes is also very much complicated.

Hydrazones derived from isonicotinic acid hydrazide and their metal complexes have been shown to have biological activity specially, the enhanced antitubercular activity [13]. Further, the related hydrazones formed from pyridoxal and isonicotinic acid hydrazide (pyridoxal isonicotinoyl hydrazone, pyrinhH_2), (pyrinhH_2) has recently been shown to be an effective chelator of iron(III) and thus has potential to be used to mobilize iron from tissues that are overloaded with iron and may find applications in the treatment of thalassemia [14, 15]. The potential of pyrinhH_2 has been examined and confirmed in a variety of biological and biochemical assays [16–18]. Recent studies have shown that 2-hydroxy 1-naphthaldehyde isonicotinoyl hydrazone (H_2npih) have appreciable activity in hepatocyte assay. The presence of naphthyl group in H_2npih introduces bulky fragment in the hydrazone. The hydrazones having bulky fragments in their molecular skeleton give rise to complexes having discrete molecular arity. In view of such properties associated with the H_2npih, we became interested in the study of reactions of V_2O_5 with H_2npih in the presence of H_2O_2 under variety of experimental conditions and isolation of the resulting species and their characterization. We wanted to know whether the present hydrazone leads to the formation of oxo-peroxo complexes having discrete molecular arity. Further, we were interested to know whether the present hydrazone coordinates to the metal centre in keto form or enol form. Accordingly, the present paper describes the synthesis of oxo-peroxo vanadium complexes of the title hydrazone and their characterization by various physico - chemical techniques. An effort has been made to establish their structure by IR spectroscopic study.

2. Experimental

2.1. Materials and methods

V_2O_5 of E. Merck, grade was employed. Organic chemicals such as isonicotinoylhydrazine ($C_5H_4NCONHNH_2$) and 2-hydroxy-1-naphthaldehyde [$C_{10}H_6$(OH)(CHO)] were E. Merck reagents. The organic solvents viz. Ethanol, methanol, chloroform, benzene, diethyl ether, acetone, methylene chloride, acetonitrile, dimethyl sulphoxide and dimethyl formamide were purified by standard literature procedures. C, H and N were determined by Heraeus Carbo Erba 1108. Room temperature magnetic susceptibility measurements were made on a Sherwood Scientific Magnetic Susceptibility Balance. The molar conductance of the complexes at 10^{-3} M dilution in DMSO were measured on a Direct Reading Conductivity meter-303 with a dip type conductivity cell at room temperature. IR spectra were recorded on a Perkin-Elmer Spectrophotometer in the range 4000-450cm^{-1} in KBr discs. The ESR spectra of the compounds in powdered form at room temperature and liquid temperature were recorded at X-band frequency on a Variance E-112X/2 band spectrometer, DPPH was used as an internal field marker.

2.2. Synthesis

2.2.1. Synthesis of ligand (H_2npih)

To prepare hydrazone, ethanol solutions (100mL, 100mmol) of isonicotinoylhydrazine in hot conditions were allowed to interact with a slightly larger amount of 2-hydroxy-1-naphthaldehyde (105mL, 100 mmol) than required for a 1:1 molar ratio. The reaction mixture was refluxed for about 1h. The precipitate thus obtained on cooling the solutions to room temperature were filtered by suction, washed with ethyl alcohol and recrystallized from ethanol [19] and dried in an electric oven maintained at ca 70^{O}C. Yield 75%. Anal. Calcd. for $C_{17}H_{13}N_3O_2$: C, 70.10; H, 4.47; N, 14.43. Found: C, 69.67; H, 4.50; N, 13.96.

2.2.2. Synthesis of complexes

2.2.2.1. Synthesis of [VO(O_2) (Hnpih) (H_2O)] (1)

V_2O_5 (0.32g, 1.76 mmol) was suspended in ethanol (20 mL) and stirred for about 10 minutes to make it homogeneous. To this suspension, 10 mL 30% H_2O_2 was added accompanied by gentle stirring. This mixture was stirred for another 10 minutes. A vigorous reaction occurred. The reaction mixture was filtered to remove any undissolved material. This resulted a clear dark red solution. H_2npih (1.0g, 4.15 mmol) was dissolved in ethanol (120 mL) by boiling. The hot H_2npih solution was added slowly to V_2O_5 solution accompanied by slow stirring. This gave a yellowish coloured solution. Stirring was continued for 15 - 20 minutes accompanied by cooling to room temperature. This precipitated a brown compound after 1hr which was filtered, washed with ethanol and dried at ambient temperature over anhydrous $CaCl_2$.

2.2.2.2. Synthesis of $A_2[V_2O_2(O_2)_3(Hnpih)_2].2H_2O$. [A=K(2), Na(3) and NH_4(4)]

In order to prepare K_2 [V_2O_2 (O_2)$_3$ (Hnpih)$_2$].2H_2O (**2**), V_2O_5 (0.32g, 1.76 mmol) was suspended in ethanol (20 mL) and stirred for about 10 minutes. 10 mL 30% H_2O_2 was added to this suspension accompanied by stirring. This mixture was stirred for about 10 minutes and a vigorous reaction occurred. The reaction mixture was filtered to remove any undissolved material. H_2npih (1.0g, 3.44 mmol) was dissolved in 120 mL ethanol by boiling. The resulting hot solution was added slowly to V_2O_5 solution accompanied by gentle stirring. Stirring was continued for 15 - 20 minutes accompanied by cooling to room temperature. This gave an yellowish coloured solution. To this solution, 10% KOH in water was added drop by drop bringing pH to 6.5. The reaction mixture was stirred for another 30 minutes. This precipitated a greenish coloured compound which was then filtered, washed with ethanol and dried over anhydrous calcium chloride.

The compounds $Na_2[V_2O_2(O_2)_3(Hnpih)_2].2H_2O$ (**3**) and $(NH_4)_2$ [$V_2O_2(O_2)_3(Hnpih)_2$].2H_2O (**4**) were prepared by essentially the above procedure by using NaOH and NH_4OH instead of KOH.

2.2.2.3. Synthesis of $(BH)_2$ [V_2O_2 (O_2)$_3$ (Hnpih)$_2$], [BH = pyH (5), 3-picH (6) and 4 - picH (7)]

In order to prepare (pyH)$_2$ [V_2O_2 (O_2)$_3$ (Hnpih)$_2$] (**5**), V_2O_5 (0.32g, 1.76 mmol) was suspended in 20 mL ethanol and stirred for about 10 minutes. 10 mL 30% H_2O_2 was added to this suspension accompanied by gentle stirring. This mixture was stirred for about 10 minutes. Any undissolved material was removed by

filtration. H_2npih (1.0g, 3.44 mmol) was dissolved in ethanol (120 mL) by boiling. The resulting hot solution of H_2npih was added slowly to V_2O_5 solution accompanied by gentle stirring. The solution was cooled to room temperature followed by addition of pyridine (1.8 mL) accompanied by stirring for one hour. The colour of the solution changed to brown and then precipitated a brown-coloured compound. The precipitate was filtered and washed with ethanol and isolated in the usual way.

The compounds (3-picH)$_2$[$V_2O_2(O_2)_3$(Hnpih)$_2$] (**6**) and (4-picH)$_2$ [$V_2O_2(O_2)_3$(Hnpih)$_2$] (**7**) were also prepared by essentially the above procedure by using 2.2 mL each of 3- picoline (3- picH) and 4 - picoline (4- picH) respectively instead of pyridine (pyH).

3. Result and discussion

3.1. Analytical data and the physical properties

The analytical data reveal the formation of oxo-peroxo compounds of vanadium of the following compositions.

[VO (O2) (Hnpih) (H2O)] (**1**)

A_2[V2O2(O2)3 (Hnpih)2].2H2O [A = K (**2**), Na (**3**), NH_4 (**4**)]

(BH)$_2$[V2O2(O2)3(Hnpih)$_2$] [BH = pyH (**5**), 3-picH (**6**) and 4- picH (**7**)]

The complexes (**2**) to (**4**) are yellow while the complexes (**1**) and (**5**) are brown and the complexes (**6**) and (**7**) are dark red. The alkali metal and ammonium peroxo complexes are freely soluble in water, DMSO, DMF and partially in methanol and ethanol. On the other hand, pyridinium and picolinium oxoperoxo vanadium(V) are soluble in DMSO and DMF only. All of the complexes are insoluble in common organic solvents such as acetone, dichloromethane, diethyl ether, acetonitrile and benzene.

The complexes along with their colour, analytical data and molar conductance have been set out in the **Table 1**.

The weight loss experiments for complexes (**1**) to (**7**) were carried out in the temperature range 110^0 - 180^0C. When these complexes are heated at 110^{o}C, they lose active oxygen molecules. The complex (**1**) loses weight corresponding to half oxygen molecule only while complexes (**2**) to (**4**) lose weight corresponding to one and half oxygen molecule and two water molecules. On the other hand, the weight loss in the complexes (**5**) to (**7**) corresponds to one and half oxygen molecules and no water molecules are lost. These weight loss studies showed that the complexes (**1**) and (**5**) to (**7**) do not have any water molecule in their lattice structure. While the complexes (**2**) to (**4**) contains two water molecules in their lattice structure. The complexes (**2**) to (**7**) showed no weight loss when heated at 180^{o}C indicating that the complexes do not have water molecules in the first coordination sphere around the metal centre. On the other hand, the complex (**1**) shows weight loss corresponding to one water molecule at 180^{o}C which suggests one water molecule is bonded to the metal centre. The compounds possess zero magnetic moment and are ESR silent suggesting that vanadium is present in +5 oxidation state in them. The analytical results suggested that vanadium and peroxo groups are in 1:1 molar ratio in the complexes (**1**), while in 1:1.5 molar ratio in the remaining complexes.

3.2. Molar Conductance

The molar conductance values for the complex (**1**) and (**5**) to (**7**) have been determined in DMSO while those of complexes (**2**) to (**4**) in aqueous medium due to solubility reasons. The molar conductance values for the complexes (**1**) and (**5**) to (**7**) lie in the region 11.7 - 26.6 ohm $^{-1}$ cm ^{2} mol $^{-1}$. The molar conductance values for

the complexes (**5**) to (**7**) also fall below the value reported for 1:1 electrolyte indicating their non - electrolytic nature [20]. The higher molar conductance values of the complexes (**5**) to (**7**) might be attributed to ionic nature of the complexes, most probably, 2:1 electrolyte. However, the values are still less than those of 2:1 electrolyte, probably, due to low ionic mobility of anionic and cationic coordination sphere because of their larger size. It appears that the compounds exist as ion-pairs.

On the other hand, the molar conductance values for the complexes (**2**) to (**4**) fall in the region 403.8 - 496.0 ohm^{-1} cm^2 mol^{-1}. The molar conductance of these compounds in water is higher than that expected for 2:1 electrolyte. It has, generally, been observed [21, 22] that owing to their instability, the molar conductance of many peroxo metal complexes cannot be measured.

3.3. Magnetic and ESR spectral studies

These complexes possess zero magnetic moment. This suggests their diamagnetic nature consistent with the presence of vanadium in these complexes in +5 oxidation state. This is also confirmed from ESR spectral studies of the complex (**1**), recorded as representative sample, which is ESR silent.

Table 1: Analytical data and the physical properties of the oxoperoxo vanadium(V) complexes derived from 2-hydroxy-1-naphthaldehyde isonicotinoylhydrazone

Sl. No.	Complex	Colour	Analysis Found					Molar conductance $ohm^{-1}cm^2mol^{-1}$
			V	O_2^{2-}	C	H	N	
4.1	$[VO(O_2)(Hnpih)(H_2O)]$	Dark yellow	12.13 (12.53)	8.12 (7.86)	50.61 (50.12)	3.38 (3.43)	10.09 (10.31)	11.7
4.2	$K_2[V_2O_2(O_2)_3(Hnpih)_2].2H_2O$	Yellow	11.35 (11.03)	10.02 (10.39)	44.61 (44.15)	3.05 (3.03)	8.81 (9.09)	496.0
4.3	$Na_2[V_2O_2(O_2)_3(Hnpih)_2].2H_2O$	Yellow	11.13 (11.43)	11.00 (10.76)	45.29 (45.73)	3.09 (3.14)	9.72 (9.41)	447.4
4.4	$(NH_4)2[V_2O_2(O_2)_3(Hnpih)_2].2H_2O$	Yellow	12.00 (11.56)	10.59 (10.88)	45.76 (46.25)	3.22 (3.17)	10.00 (9.52)	403.8
4.5	$(pyH)_2[V_2O_2(O_2)_3(Hnpih)_2]$	Brown	11.13 (11.51)	9.68 (9.90)	54.90 (54.43)	3.76 (3.71)	11.92 (11.55)	25.20
4.6	$(3\text{-picH})_2[V_2O_2(O_2)_3(Hnpih)_2]$	Redish	10.61 (10.22)	9.39 (9.62)	55.79 (55.31)	3.98 (4.01)	11.39 (11.22)	24.7
4.7	$(4\text{-picH})_2[V_2O_2(O_2)_3(Hnpih)_2]$	Redish	9.93 (10.22)	10.00 (9.62)	54.93 (55.31)	4.05 (4.01)	10.95 (11.22)	26.6

3.4. IR Spectra

Structurally significant IR bands for these complexes have been set out in **Table 2**. The IR spectra for hydrazone and the complexes (**1**) to (**5**) have been shown in Fig. 4.1 to 4.7

The IR spectrum of the hydrazone, H_2npih shows strong bands at 3220 and 3040 cm^{-1}. These bands are asigned to have composite character due to bands arising from νOH and νNH vibrations of naphtholic -OH and secondary -NH groups. The position of these bands suggests the presence of strong intramolecular hydrogen bonding between secondary amine hydrogen, naphtholic - OH hydrogen, the >C=O and > NH groups.

The IR spectra of the complexes (**1**) to (**7**) show either a very strong broad band in the region 2900 - 3550 cm^{-1} in the complexes (**2**) and (**3**) or three to four bands in the region 3000- 3500 cm-1 in the complexes (**1**) and (**4**) to (**7**). The IR spectra of complexes are complicated in this region because of occurrence of bands due to stretching vibrations of secondary -NH groups, vibrations of lattice and coordinated water molecules in the complexes (**1**) to (**4**). In the ammonium oxo-peroxo vanadate (**4**), the bands due to stretching vibrations of the NH_4^+ group also appear in this region. Further, complicacy is added as the spectra of the complexes are recorded in KBr pellets which might absorb water molecules from atmosphere during the preparation of the complexes. The bands observed in the region 3049 - 3450 cm^{-1} in the complexes (**5**) to (**7**) are attributed to arise from -NH stretching vibrations of secondary -NH group and -NH group of protonated pyridyl N-atom of pyridinium or substituted pyridinium ion.

The uncoordinated hydrazone shows a strong band at 1679 cm-1. This band is assigned to ν(C = O) stretching vibration. This band remains almost unperturbed in position in the complexes (**1**) to (**7**) ruling out the possibility of coordination of carbonyl oxygen atom to the metal centre. The band due to bending C–O vibration at 1279 cm^{-1} in H_2npih remains either almost unshifted in position as in complex (**2**) or shifts to higher frequency by 1-5 cm^{-1} as in the remaining complexes. Such a feature associated with this band indicates weak bonding between naphtholate oxygen atoms and metals centres [23]. The amide II + ν(C–O) (naphtholic) band appearing at ca. 1551 cm^{-1} shifts to lower frequency by 1-18 cm^{-1} in the complexes. This indicates bonding of ligand through naphtholate oxygen atom [24].

The absorption band due to azomethine group appears at 1623 cm^{-1} as a medium intensity band. This band splits into two bands in all of the complexes which appears in the region 1619 - 1626 cm^{-1} and 1597 - 1604 cm^{-1}. The average position of this band shifts to lower frequency by 3 to 23 cm^{-1} in the complexes. The lower shift of ν(C=N) band in the complexes derived from H_2npih may be attributed to drainage of electron density from azomethine nitrogen atom to the metal centre.

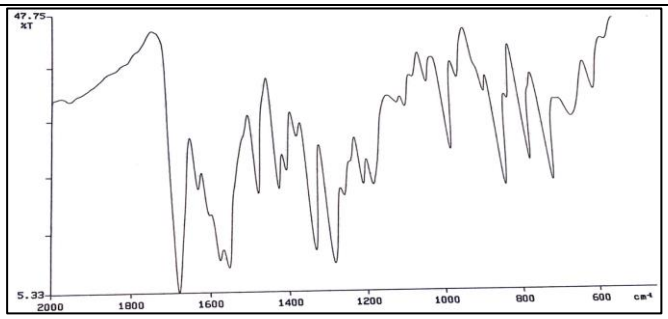
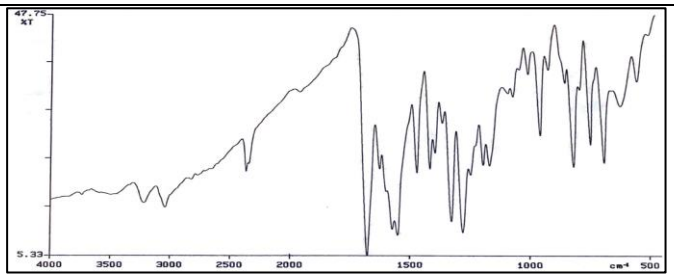
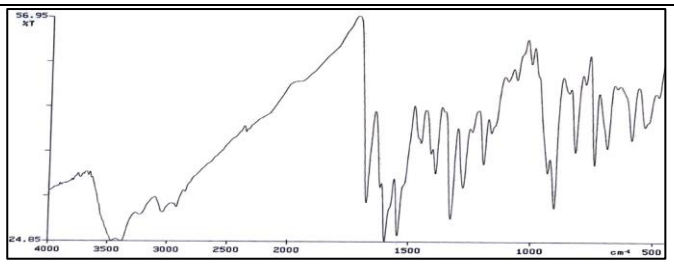
The free hydrazone shows a weak band at 997 cm^{-1} which is assigned to arise due to pyridyl ring breathing vibrations. This band shifts to higher frequency by 4-33 cm^{-1} indicating coordination through pyridyl nitrogen atom.

The IR spectra of all of the complexes (peroxo) show a single strong band in the region 952 - 958 cm^{-1}. This band is assigned to ν(V=O) arising from the terminally bonded V=O group [25-27]. This band masks the medium intensity band at 952 cm^{-1} in the IR spectrum of the uncoordinated hydrazones.

The IR spectra of all of the complexes except the complex (**1**) shows two bands in the regions 835 - 848 cm^{-1} and 815 - 816 cm^{-1}respectively. The position and intensity of these bands indicate that they originate from the presence of peroxide ligand in the complexes. The band in the region 835 - 848 cm^{-1} owes its origin to the ν_1 mode of a triangularly bonded, bidentate, chelating O_2^{2-} (C_{2v}) group [28]. The IR spectrum of the ligand shows another strong band in the region 815 - 816 am^{-1}. This band is assigned to arise due to the ν(O - O) mode of bridging (σ : σ) peroxide [29]. Although ν(O - O) mode of bridging (σ : σ) peroxide group is IR forbidden, yet the forbidden vibrations may become allowed as a result of particular symmetry properties of overall ligand field in the complexes. The symmetric (ν_s) and asymmetric (ν_{as}) modes associated with V-O_2 stretches have been located in the regions 582 - 610 cm^{-1} and 534 - 559 cm^{-1} respectively.

In the compound (**4**), shows an additional sharp band appears at 1410 cm^{-1} which is clearly due to the N-H deformation mode of NH_4^+ ion [30]. The ν(N-H) modes arising from NH_4^+ could not be identified clearly owing to their overlap with the ν(O-H) mode originating from the lattice water and ν(N-H) mode originating from the secondary NH group of coordinated hydrazone and ν(N-H) of protonated pyridine/ 3-picoline/ 4-picoline.

Table 2: Infrared spectra of ligand (H_2npih) and complexes (**1-7**)

Probable name/formula	Infrared spectrum
2-Hydroxy-1-naphthaldeyhyde isonicotinoylhydrazone (H_2npih)	
[$VO(O_2)(Hnpih)(H_2O)$] (**1**)	
K_2 [$V_2O_2(O_2)_3(Hnpih)_2$].2H_2O (**2**)	

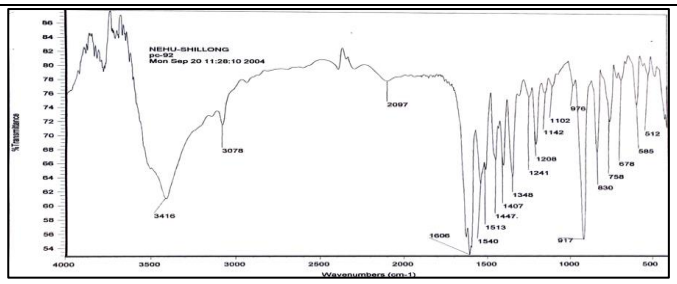
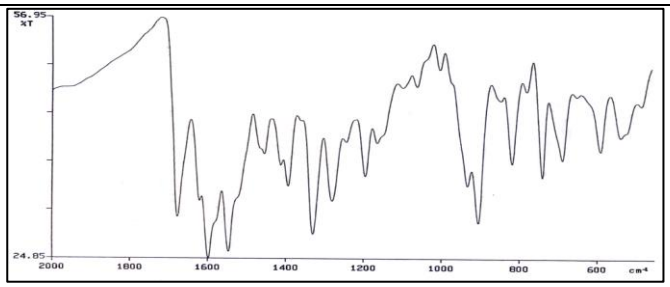
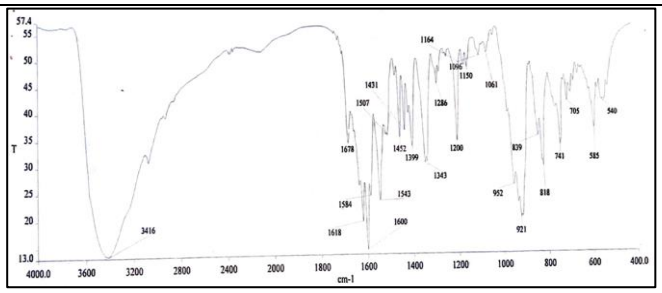
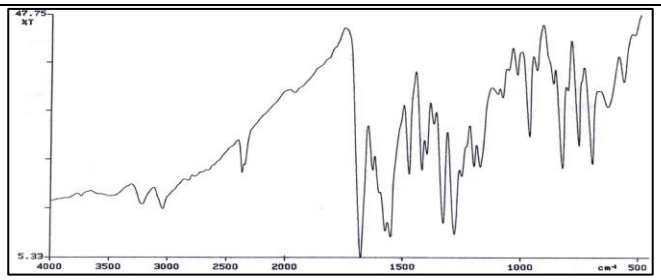
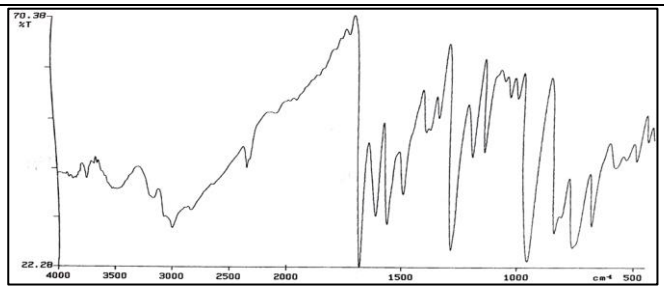
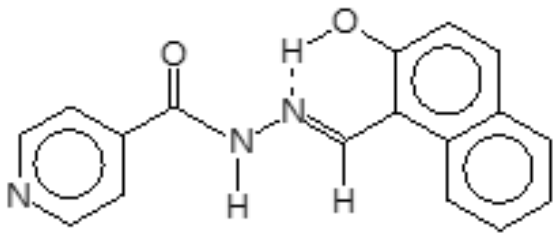
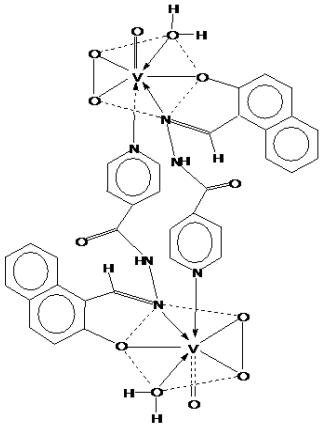
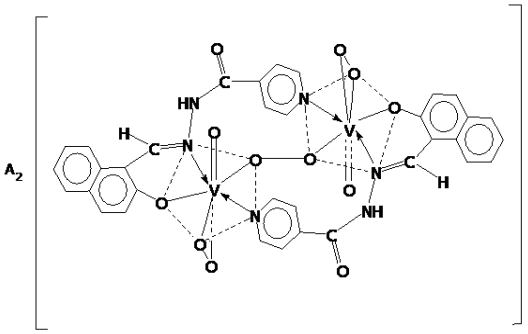
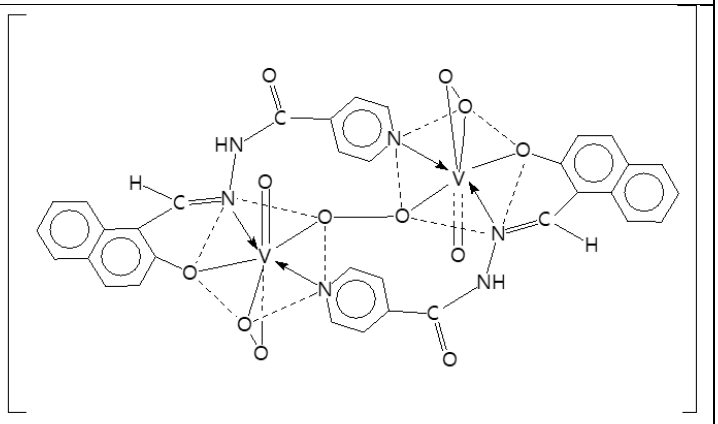
Na_2 [$V_2O_2(O_2)_3(Hnpih)_2$].$2H_2O$ (**3**)	
$(NH4)2$ [$V_2O_2(O_2)_3(Hnpih)_2$].$2H_2O$ (**4**)	
$(pyH)_2$ [$V_2O_2(O_2)_3(Hnpih)_2$] (**5**)	
$(3\text{-picH})_2$ [$V_2O_2(O_2)_3(Hnpih)_2$] (**6**)	
$(4\text{-picH})_2$ [$V_2O_2(O_2)_3(Hnpih)_2$] (**7**)	

Table 3: Suggested structure for 2-Hydroxy-1-naphthaldeyhyde isonicotinoylhydrazone (H_2npih), [$VO(O_2)(Hnpih)(H_2O)$] (**1**), $A_2[V_2O_2(O_2)_3(Hnpih)_2].2H_2O$ [A = K(**2**), Na(**3**) and NH_4(**4**)] and $(BH)_2[V_2O_2(O_2)_3(Hnpih)_2$] [BH = pyH (**5**), 3-picH (**6**) and 4-picH (**7**)]

Probable name/formula	Suggested structure
2-Hydroxy-1-naphthaldeyhyde isonicotinoylhydrazone (H_2npih)	
[$VO(O_2)(Hnpih)(H_2O)$] (**1**)	
A_2 [$V_2O_2(O_2)_3(Hnpih)_2$].$2H_2O$ [A = K(**2**), Na(**3**) and NH_4(**4**)]	 $2H_2O$
$(BH)_2$ [$V_2O_2(O_2)_3(Hnpih)_2$] [BH = pyH (**5**), 3-picH (**6**) and 4-picH (**7**)]	

4. Conclusion

It is evident from the experimental evidences presented and discussed above that H_2npih functions as a monobasic tridentate ligand in all of the complexes. The hydrazone coordinates to the metal centre through carbonyl oxygen atom, azomethine nitrogen atoms and pyridyl nitrogen atom. The peroxo group is bonded to the metal centres as a bidentate chelating ligand in the complex (**1**) while in the remaining complexes, one peroxo group is bonded to the each metal centre as a bidentate chelating ligand and other one as a bridging bidentate ligand.

Alkali metal ions and pyridinium and picolinium ions are present outside the coordination sphere. In all of the peroxo complexes, the vanadium atom is present as VO^{3+} group. All of the peroxo complexes are suggested to have a seven coordinate pentagonal bipyramidal structure. Considering the presence of chelated and bridging peroxo groups, a terminal oxo group and monobasic tridentate hydrazone ligand and the presence of one coordinated water molecule in the complex (**1**) and all of the complexes may be represented by seven coordinate polyhedra (**Table 3**).

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