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Structural characterization, electrochemical, photoluminescence and thermal properties of potassium ion-mediated coordination polymer

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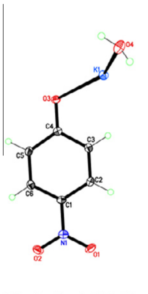
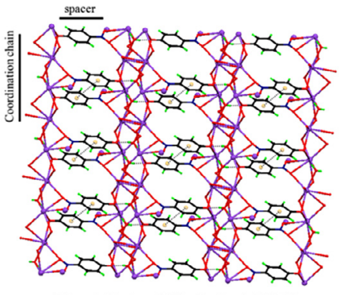
HIGHLIGHTS

- A novel polymeric potassium complex of *p*-nitrophenol is reported.
- Molecular structure of the complex was determined by X-ray diffraction technique.
- Electrochemical and thermal properties of the complex were investigated.

GRAPHICAL ABSTRACT



$[K(p\text{-nitrophenolate})(H_2O)]_n$



Polymeric Structure of $[K(p\text{-nitrophenolate})(H_2O)]_n$

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ABSTRACT

A polymeric potassium complex of *p*-nitrophenol was synthesized and characterized by analytical and spectroscopic techniques. Molecular structure of the complex was determined by single crystal X-ray diffraction study. X-ray structural data show that crystals contain polymeric K^+ complex of *p*-nitrophenol. Asymmetric unit consists of one *p*-nitrophenolate, one K^+ ion and one water molecule. All bond lengths and angles in the phenyl rings have normal C*sp*2–C*sp*2 values and are in the expected ranges. The *p*-nitrophenolate is close to planar with small distortions by some atoms. Each potassium ion in the polymeric structure is identical and eight-coordinate, bonded to four nitro, two phenolate oxygen atoms from five *p*-nitrophenolate ligands and two oxygen atoms from two water molecules. Electronic, electrochemical, photoluminescence and thermal properties of the complex were also investigated.

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Introduction

p-Nitrophenol is a product of the enzymatic cleavage of several substrates such as *p*-nitrophenyl acetate (used as a substrate for carbonic anhydrase) and *p*-nitrophenyl-β-D-glucopyranoside and other sugar derivatives which are used to assay various glycosidase enzymes. Amounts of *p*-nitrophenol produced by a particular enzyme (alkaline phosphatase) in the presence of its corresponding substrate can be measured with a spectrophotometer at around 405 nm and used as a proxy measurement for the amount of the enzyme activity in the sample. *p*-Nitrophenol shows two polymorphs in the crystalline state. The alpha-form is colorless pillars and unstable at room temperature. The beta-form is yellow pillars and stable at room temperature, and gradually turns red upon irradiation of sunlight. Usually, *p*-nitrophenol exists as a mixture of these two forms [1]. Potassium complexes of *p*-nitrophenyl compounds have been reported in literature [2–6]. Molecular structure of potassium complex of *p*-nitrophenyl sulfate (PNPS) was structurally characterized by X-ray diffraction study. The crystal structure of the compound [K(PNPS)] contains

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p-nitrophenylsulphate anions and potassium cations. Each K^+ ions is 8-coordinate with approximate square antiprism geometry [2].

In this study, polymeric potassium complex of *p*-nitrophenol (KNP) was prepared and characterized by spectroscopic and analytical methods. Molecular structure of the complex was determined by single crystal X-ray diffraction study. Electronic and electrochemical properties of the complex were also investigated.

Experimental

General methods

All starting materials and organic solvents were purchased from commercial sources and used as received, unless noted otherwise. IR spectrum was performed on a Perkin Elmer, Spectrum 400 (resolution; 0.5–4 cm^{-1}). CHN analysis was performed using a CE-440 Elemental analyzer. The ^1H- and ^{13}C NMR were performed using a BrukerAvance 400. Mass spectrum was recorded on a Thermo Fisher Exactive Orbitrap mass spectrometer coupled to an Advion TriVersa Nanomate injection system.

X-ray structures solution and refinement for the polymeric complex

X-ray diffraction data for the complex was collected at 100(2) K on a Bruker ApexII CCD diffractometer using Mo-$K\alpha$ radiation (λ = 0.71073 Å). Data reduction was performed using Bruker SAINT. SHELXTL was used to solve and refine the structures [7]. The structures were solved by direct methods and refined on F^2 using all the reflections [8]. All the non-hydrogen atoms were refined using anisotropic atomic displacement parameters and hydrogen atoms bonded to carbon were inserted at calculated positions using a riding model. Hydrogen atoms bonded to water oxygen atom (O4) were located from difference maps and refined with temperature factors riding on the carrier atom. Details of the crystal data and refinement are given Table 1. Hydrogen bond parameters are given in Table 2 and bond lengths and angles are given in Table 3.

Preparation of the polymeric complex (KNP)

p-Nitrophenol (1 mmol, 0.139 g) in acetone (10 ml) and K_2CO_3 (0.5 mmol, 0.069 g) in acetone (5 ml) were mixed and refluxed for 4 h at 358 K. An intense light brown coloration appeared immediately. After one week, well-formed light brown crystals grew on the walls. A single crystal of dimensions 0.33 × 0.23 × 0.16 mm^3 was selected from mother liquor and mounted for X-ray diffraction study at low temperature.

Yield: 88%, color: light brown, M.p.: 127 °C. Anal. (%) Found (Calculated for $C_6H_6KNO_4$): C 36.95(36.92), H 3.10(3.07), N 7.18(7.20). ^1H NMR (δ, ppm): 6.25–7.55 (4H, Ar–H). ^{13}C NMR (δ, ppm): 115.20–149.10 (Ar–C). Mass Spectrum (LC/MS APCI): m/z 196 $[M+1]^+$ (40%), 195 $[M]^+$ (100%). FTIR (KBr, cm^{-1}): 2960 $\nu(C\text{–}H)_{aromatic}$, 1455 $\nu(NO_2)$, 1345 ν(phenolic C–O). UV–vis (λ_{max} (ε_{max}), nm). EtOH; 344(0.42 × 10^3), 459(0.36 × 10^3), MeOH; 319(1.19 × 10^3), 449(0.61 × 10^3), DMF, 316(0.67 × 10^3), 448(0.35 × 10^3), Ethylacetate; 349(0.19 × 10^3), 454(0.13 × 10^3).

Table 1
Crystallographic data.

Identification code	K1
Empirical formula	$C_6H_6KNO_4$
Formula weight	195.22
T/K	100(2)
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell	
a (Å)	10.4828 (2)
b (Å)	7.2887 (1)
c (Å)	11.2186 (2)
α (°)	90
β (°)	117.682 (1)
γ (°)	90
Volume (Å^3)	759.06 (2)
Z	4
Calculated density (g/cm^3)	1.708
Crystal size (mm^3)	0.33 × 0.23 × 0.16
Abs. coeff. (mm^{-1})	0.670
Refl. collected	6878
Ind. Refl. [R_{int}]	1894 [0.0204]
R_1, wR_2 [$I > 2\sigma(I)$]	0.0245, 0.0637
R_1, wR_2 (all data)	0.0274, 0.0655
Goodness-of-fit on F^2	1.064
CCDC number	987883

Table 2
Hydrogen-bond geometry for the complex (Å, °).

D–H···A	D–H	H···A	D···A	D–H···A
O4–H4A···O3*	0.79 (2)	2.00 (2)	2.7863 (13)	172.7 (18)
O4–H4B···O3**	0.84 (2)	1.97 (2)	2.7781 (13)	160.1 (17)

Symmetry codes.
* $-x, -y, -z$.
** $x, y-1, z$.

Table 3
Bond lengths and angles for the complex (Å, °).

K1–O4	2.6652 (10)	O2–K1^{vii}	2.8493 (9)
K1–O3	2.7683 (9)	O2–K1^{iv}	2.8972 (10)
K1–O4^{i}	2.7819 (10)	O2–N1	1.2484 (14)
K1–O1^{ii}	2.8308 (9)	O1–N1	1.2430 (14)
K1–O2^{iii}	2.8493 (9)	O3–C4	1.2891 (14)
K1–O3^{iv}	2.8704 (9)	N1–C1	1.4187 (15)
K1–O2^{v}	2.8972 (10)	C1–C6	1.3983 (17)
K1–O1^{v}	2.9813 (10)	C1–C2	1.4006 (16)
O1–K1^{ii}	2.8309 (9)	C2–C3	1.3735 (17)
O1–K1^{iv}	2.9813 (10)	C3–C4	1.4293 (16)
O3–K1^{i}	2.8705 (9)	C4–C5	1.4292 (16)
O4–K1^{iv}	2.7819 (10)	C5–C6	1.3693 (17)
O4–K1–O3	146.88 (3)	O3^{iv}–K1–O1^{v}	72.43 (3)
O4–K1–O4^{i}	135.11 (3)	O2^{v}–K1–O1^{v}	43.26 (3)
O3–K1–O4^{i}	77.05 (3)	N1–O1–K1^{ii}	161.62 (8)
O4–K1–O1^{ii}	73.16 (3)	N1–O1–K1^{iv}	93.89 (7)
O3–K1–O1^{ii}	126.34 (3)	K1^{ii}–O1–K1^{vi}	88.22 (2)
O4^{i}–K1–O1^{ii}	85.21 (3)	N1–O2–K1^{vii}	149.50 (8)
O4–K1–O2^{iii}	74.97 (3)	N1–O2–K1^{iv}	97.82 (7)
O3–K1–O2^{iii}	76.12 (3)	K1^{vii}–O2–K1^{iv}	94.89 (3)
O4^{i}–K1–O2^{iii}	147.90 (3)	C4–O3–K1	106.54 (7)
O1^{ii}–K1–O2^{iii}	96.75 (3)	C4–O3–K1^{i}	122.55 (7)
O4–K1–O3^{iv}	77.20 (3)	K1–O3–K1^{i}	91.71 (2)
O3–K1–O3^{iv}	130.91 (3)	K1–O4–K1^{iv}	95.94 (3)
O4^{i}–K1–O3^{iv}	58.85 (3)	O1–N1–O2	120.96 (10)
O1^{ii}–K1–O3^{iv}	73.39 (3)	O1–N1–C1	119.66 (10)
O2^{iii}–K1–O3^{iv}	152.11 (3)	O2–N1–C1	119.36 (10)
O4–K1–O2^{v}	72.63 (3)	C6–C1–C2	120.99 (11)
O3–K1–O2^{v}	89.17 (3)	C6–C1–N1	119.41 (10)
O4^{i}–K1–O2^{v}	111.82 (3)	C2–C1–N1	119.56 (11)
O1^{ii}–K1–O2^{v}	143.95 (3)	C3–C2–C1	119.46 (11)
O2^{iii}–K1–O2^{v}	85.11 (3)	C2–C3–C4	121.43 (11)
O3^{iv}–K1–O2^{v}	88.25 (3)	O3–C4–C5	121.71 (11)
O4–K1–O1^{v}	107.77 (3)	O3–C4–C3	121.31 (10)
O3–K1–O1^{v}	72.57 (3)	C5–C4–C3	116.98 (10)
O4^{i}–K1–O1^{v}	69.23 (3)	C6–C5–C4	121.54 (11)
O1^{ii}–K1–O1^{v}	144.549 (14)	C5–C6–C1	119.60 (11)
O2^{iii}–K1–O1^{v}	118.04 (3)		

Symmetry codes.
^{i} $-x, y+1/2, -z+1/2$.
^{ii} $-x+1, -y, -z+1$.
^{iii} $-x+1, y-1/2, -z+1/2$.
^{iv} $-x, y-1/2, -z+1/2$.
^{v} $x-1, -y+1/2, z-1/2$.
^{vi} $x+1, -y+1/2, z+1/2$.
^{vii} $-x+1, y+1/2, -z+1/2$.

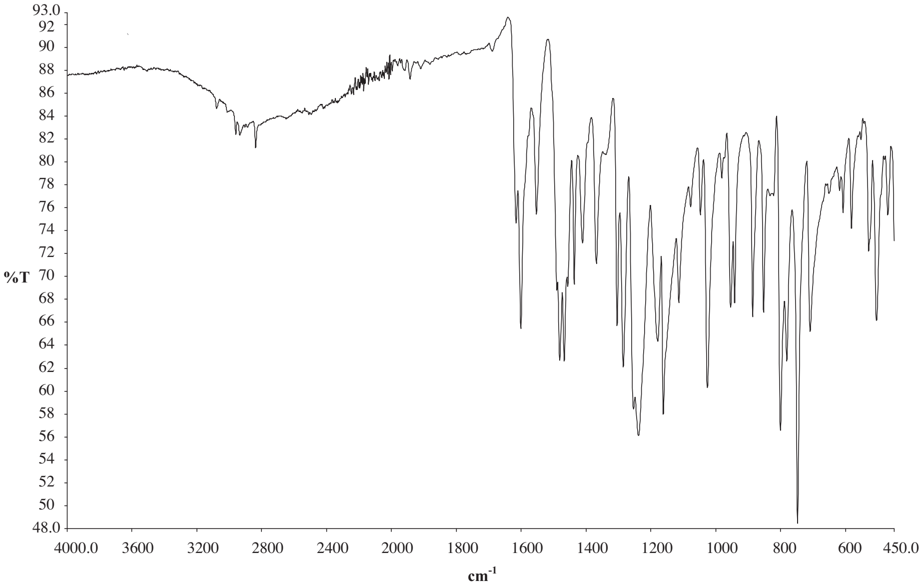

Fig. 1. FT-IR spectrum of the K^+ complex.

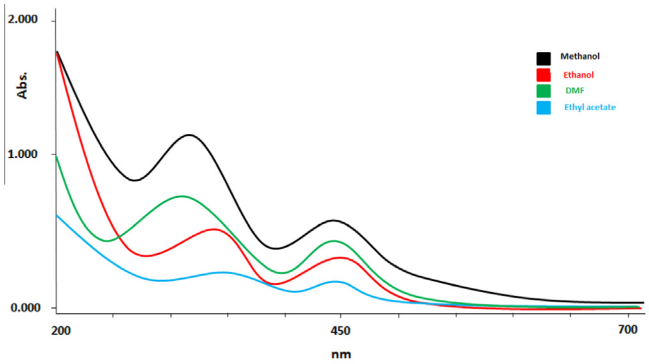

Fig. 2. Electronic absorption spectra of the coordination polymer complex (KNP) in the different solvents.

Results and discussion

A novel polymeric potassium complex (KNP) was synthesized by the reaction of *p*-nitrophenol and K_2CO_3 in acetone in high yield and purity. The complex is stable at room temperature and soluble in common organic solvents such as DMSO and DMF, EtOH, MeOH, CH_3CN, CH_3Cl and CH_2Cl_2. Elemental analysis data are given in the experimental section and are in well agreement with the theoretical values. In the ^1H NMR spectrum of the complex KNP, the multiplets in the 6.25–7.55 ppm range can be attributed to the aromatic ring protons. In the ^{13}C NMR spectrum, the aromatic ring carbon atoms were observed in the range of 115.20–149.10 ppm. In the mass spectrum of the complex KNP, the molecular ion peak was observed at m/e 195. In the FT-IR spectrum of the complex, the vibrations at 2960, 1455 and 1345 cm^{-1} can be assigned to the aromatic υ(C–H), nitro υ(NO_2) and phenolic υ(C–O) groups vibrations, respectively Fig. 1. In order to investigate the solvent effect, the electronic properties of the complex were studied in the different solvents such as EtOH, MeOH, DMF and ethylacetate. The electronic spectra of the coordination complex are given in Fig. 2. While the complex has maximum absorption band in the MeOH, it has lowest band in the ethylacetate solvent. In all solvents, two absorption bands were absorbed in the 459–316 nm range. These absorption bands can be attributed to the n–π and π–π* transitions [9]. In ethanol, the absorption bands shifted to longer wavelength. This situation shows that the polarity of the solvents effected the transitions.

Electrochemical properties of the coordination polymer complex (KNP) were studied in DMF – 0.1 M NBu_4BF_4 as supporting electrolyte at 293 K. In order to study the effects of the solvent and the scan rates on electrochemical behaviors, two different solvents (DMF and acetonitrile) and different scan rates (100, 150, 200, 250, 500, 750, 1000 mV/s) were used against an internal ferrocence–ferrocenium standard. The obtained data are given in Table 4. The electrochemical curves of the complex KNP are shown

Table 4
The electrochemical data of the polymeric potassium complex (KNP).

Compound	Solvent	Concentration (M)	Scan rate (mV/s)	E_{pa} (V)	E_{pc} (V)	I_{pa}/I_{pc}	$E_{1/2}$ (V)	ΔE_p (V)
KNP	DMF	1×10^{-3}	100	−0.18, 0.41, 0.89	0.49, −1,61	0.83	–	0.40
			250	−0.25, 0.42, 0.87	0.47, −1.61	0.89	–	0.40
			500	−0.32, 0.36, 0.81	0.45, −1.60	0.80	–	0.36
			750	−0.35, 0.32, 0.76	0.41, −1.61	0.78	–	0.35
			1000	−0.41, 0.28, 0.72	0.37, −1.60	0.75	–	0.35
	ACN		100	−1.25, −0.61, 0.20	0.75, −0.62	1.00	0.61	0.60
			250	−1,24, −0.62, 0.21	0,74, −0.62	1.00	0.62	0.62
			500	−1,23, −0.65, 0.20	0.73, −0. 65	1.00	0.65	0.58
			750	−1,22, −0.67, .020	0.72, −0.67	1.00	0.67	0.52
			1000	−1.21, −0.70, 0.23	0.70, −0.70	1.00	0.70	0.51

All the potentials are referenced to Ag+/AgCl; where E_{pa} and E_{pc} are anodic and cathodic potentials, respectively. $\Delta E_p = E_{pa} - E_{pc}$. $E_{1/2} = 0.5 \times (E_{pa} + E_{pc})$.

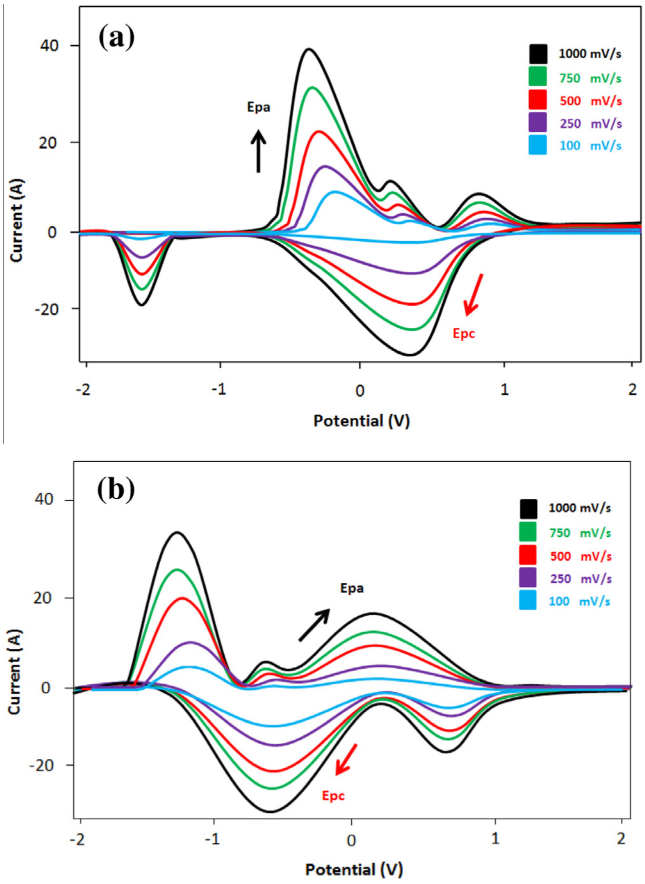

Fig. 3. (a and b) Cyclic voltammograms of the polymeric potassium complex (KNP) in the presence of 0.1 M NBu_4BF_4 in the (a) DMF and (b) CH_3CN solutions (1×10^{-3} M).

in Fig. 3a and b. In DMF and CH_3CN solvents, the complex shows three anodic and two cathodic peak potentials at all scan rates. In DMF solution, the complex shows the quasi-reversible redox process. On the other hand, in CH_3CN solution, the complex has five reversible redox process and, in this process, the nitro group of the *p*-nitrophenol compound reduces to the primary amine group and again oxidizes to the nitro group.

DSC study of the polymer complex was performed in the 20–500 °C temperature range. The DSC curve of the complex KNP is shown in Fig. 4. The melting point of the polymer complex is about 127 °C. The melting point is shown as a strong endothermic peak. The peaks at about 245 and 280 °C temperatures are crystallization peaks of the complex. The coordination polymer complex decomposed to the metal oxide (K_2O) at the higher temperatures.

X-ray structure of the complex

X-ray structural data shows that crystals contains polymeric K^+ complex of *p*-nitrophenol. Asymmetric unit consists of a *p*-nitrophenolate, a K^+ ion and a water molecule (Fig. S1). All bond lengths and angles in the phenyl rings have normal C*sp*2–C*sp*2 values and are in the expected ranges (Table 3). The ligand (*p*-nitrophenolate) is close to planar with small distortions by some atoms. Each potassium ion in the polymeric structure is identical and eight-coordinate, bonded to four nitro, two phenolate oxygen atoms from five *p*-nitrophenolate ligands and two oxygen atoms from two water molecules (Fig. 5). The same coordination contacts are extended between the other symmetry-related molecules in their respective planes to form a 2D coordination layers. Each oxygen atoms of the ligand and water molecules bridge two potassium ions in the structure. K–O distances range between 2.6652(10) and 2.9813(10) Å. The K–O4 (water) distance is the shortest distance at 2.6652(10) Å.

The ligand *p*-nitrophenol is deprotonated and each phenolate oxygen atom is involved in hydrogen bonding with water molecule (O3···HO4) strengthening the polymeric structure (Figs. 6, S2 and Table 2). Each water molecule bridge two potassium ions and involved in two hydrogen bonding (as donor) with two phenolate oxygen atoms (as acceptor) with distorted tetrahedral geometry.

Each potassium ions in the polymeric structure is linked benzene rings (spacer) (Fig. 6). The shortest K–K distance in the chain is 4.047 Å (Fig. S2). There are π–π edge to edge stacking interac-

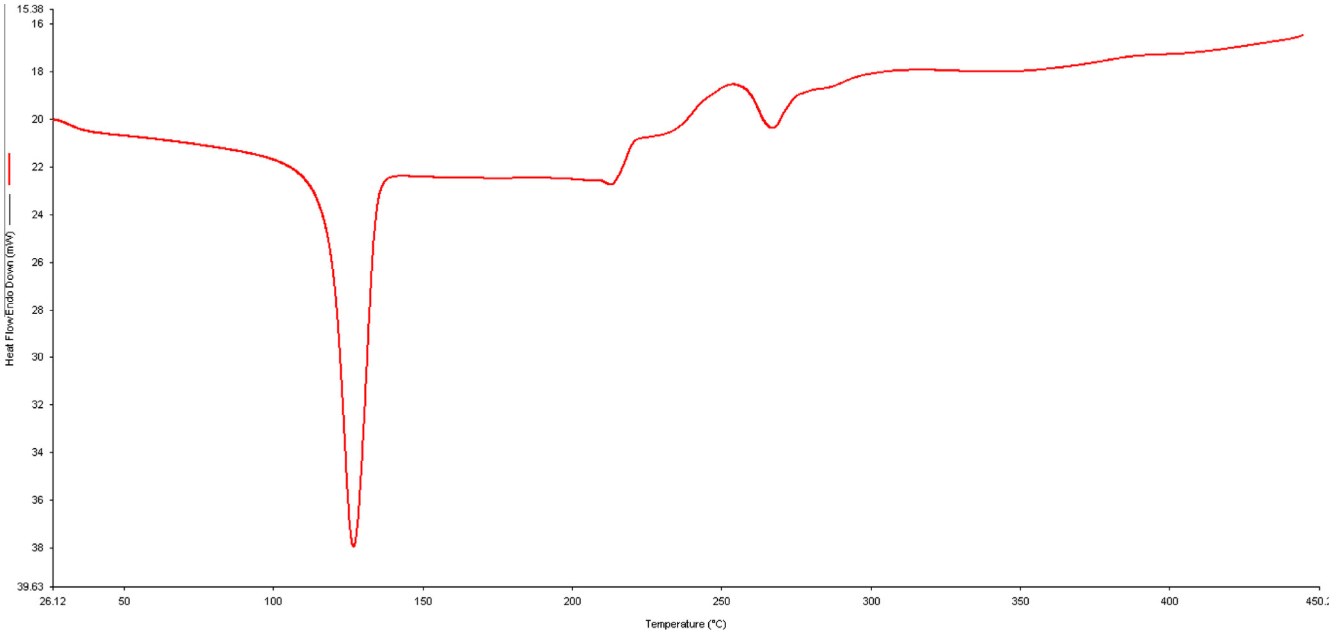

Fig. 4. DSC curve of the polymeric potassium complex under nitrogen atmosphere in the 20–500 °C temperature range.

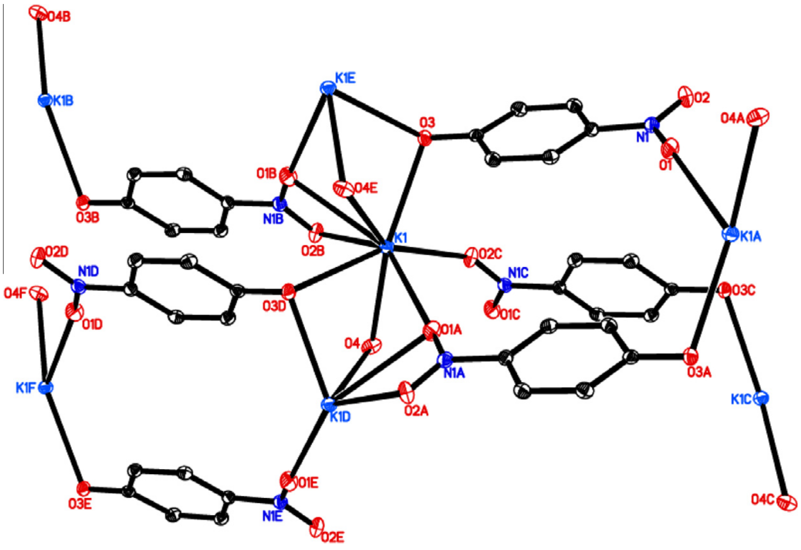

Fig. 5. Molecular structure of the complex showing coordination around potassium ions, hydrogen atom are omitted for clarity.

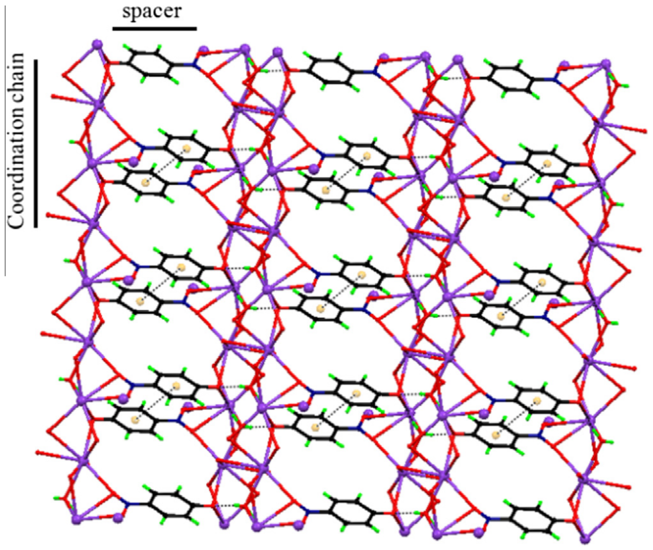

Fig. 6. Polymeric chains showing hydrogen bonding within the chains and π–π interactions within the spacers.

tions observed within the structure. The N1–C3 edge of the nitrophenolate ring is stacked with the same section of an adjacent molecule under symmetry of operation of $1 - x, 1 - y, 1 - z$; C3 and N1 are separated by a distance of 3.314 Å and centroid–centroid distance is 3.881 Å.

Table 5
The obtained absorption data from different concentrations of the DMF solution KNP complex.

Concentration (M)	KNP			
	Excitation		Emission	
	λ (nm)	A (abs)	λ (nm)	A (abs)
1×10^{-3}	557.61	1000.00	613.23	497.45
1×10^{-4}	556.42	832.66	622.46	260.57
1×10^{-5}	555.31	536.61	625.19	132.23
1×10^{-6}	554.21	372.20	634.72	61.21
1×10^{-7}	547.06	330.10	637.61	39.52

Molecular structures of Na^+ and Mg^{2+} complexes of *p*-nitrophenol have been reported [10–11]. It is informative to compare molecular structure of the polymeric K^+ complex of *p*-nitrophenol with Na^+ and Mg^{2+} analogous. The structure of Na^+ complex of *p*-nitrophenol contains *p*-nitrophenol molecules, *p*-nitrophenolate anions and water molecules surrounding the sodium ion. One *p*-nitrophenol molecule deprotonates for each sodium ion to balance the charge. Each Na^+ is six-coordinate bonding to two nitro group oxygens of *p*-nitrophenol and *p*-nitrophenolate and four water oxygen atoms (Fig. S3). Water molecules each serve to bridge two Na^+ ions resulting in a polymeric chain along the two fold axis cations. The infinite polymeric chains $[Na(p\text{-nitrophenol})(p\text{-nitrophenolate})(H_2O)_2]_n$ are linked *via* hydrogen bonding between the phenolate anions and phenol molecules. The structure of Mg^{2+} complex of *p*-nitrophenol $[Mg(p\text{-nitrophenolate})_2(H_2O)_2]_n$ is isotypic with that of the Na^+ complex. In the structure of both Na and Mg complexes of *p*-nitrophenol, phenolic oxygens do not involve in coordination with metal ions but involves in hydrogen bonding however in the structure of K^+ complex phenolate oxygen atoms coordinate to the K^+ ions.

The structure of the K^+ complex with the analogous complex of the related ligand 4-(4-nitrophenyl)phenol was reported by Haase et al. [12]. In general terms, the published structure of the polymeric complex $[K(4\text{-}(p\text{-nitrophenyl})\text{phenolate})(H_2O)]_n$ (Fig. S4) is very similar to that of the complex $[K(p\text{-nitrophenolate})(H_2O)]_n$; in both cases the K^+ ion is 8-coordinate, both have similar K–O bond distances. Both complexes show similar hydrogen bonding and π–π interactions.

The effect of different concentration on the photoluminescence properties of the metal complex

The effect of different concentration on the photoluminescence properties of the polymeric potassium complex (KNP) was investigated in the 1.0×10^{-3}–1.0×10^{-7} M range in the DMF solution. Obtained data are given in Table 5. At room temperature, the polymeric potassium complex exhibits similar emission spectra in the UV–vis region. The emission and excitation spectra of the polymeric potassium complex (KNP) in the DMF are shown in Fig. 7a and b. In the 1.0×10^{-3} M concentration, the polymeric potassium complex have highest emission peaks in the 628–666 nm range. In the 1.0×10^{-7} M concentration, the emission peaks of the polymeric potassium complex have both lowest intensity and also shift

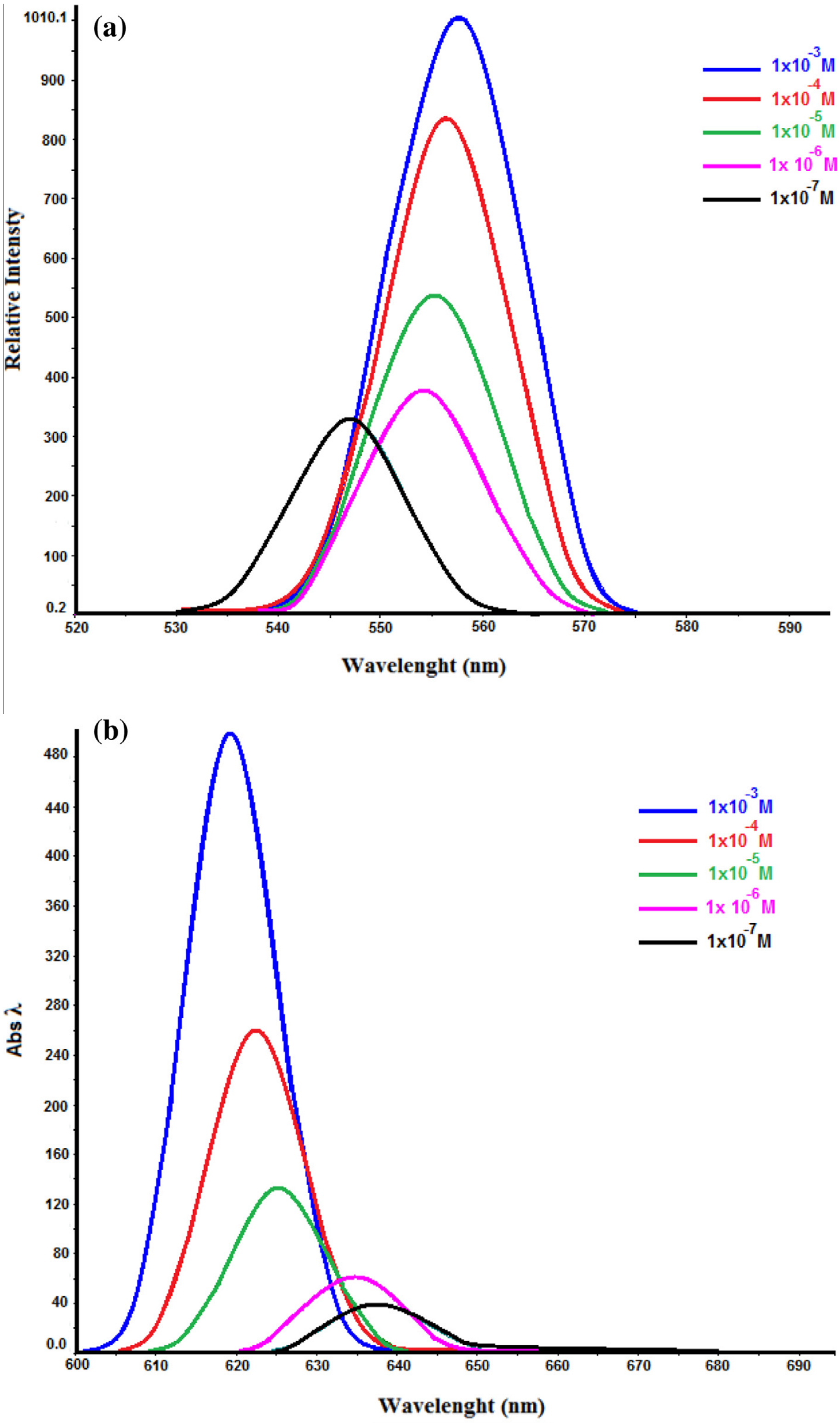

Fig. 7. (a and b) Photoluminescence of the polymeric potassium complex (KNP) (a) excitation, (b) emission in the DMF solution ($1 \times 10^{-}3$–1×10^{-7} M).

to the lower regions (628 nm). The quantity of the polymeric potassium complex have the effect on the emission peaks. Similar properties were shown also for excitation peaks of the polymeric potassium complex. In the 1.0×10^{-3} M concentration, the ligands have highest excitation peaks in the 557–547 nm range. In the 1.0×10^{-7} M concentration, the excitation peaks of the ligands have both lowest intensity and also shift to the lower regions (547 nm).

Conclusions

In conclusion, a novel polymeric complex was prepared from *p*-nitrophenol and K_2CO_3. Molecular structure of the complex was successfully characterized by X-ray diffraction study. Each potassium ion in the complex is eight-coordinate, coordinated to four nitro, two phenolate oxygen atoms from five *p*-nitrophenolate ligands and two oxygen atoms from two water molecules. The complex has an interesting thermal, electrochemical and electronic properties. Thermal study shows that crystallization peaks of the complex were found to be 245 and 280 °C. The redox processes in the CH_3CN solution were found to be reversible at all potentials.

Acknowledgement

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Appendix A. Supplementary data

CCDC 987883 contains the supplementary crystallographic data for the complex. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44)1223–336-033; or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.saa.2015.01.114.

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