

Crystal Structure, Fluorescence Spectroscopy, and Electrochemical Property of Two Neodymium Coordination Polymers with Phenoxy Acids¹

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Abstract—Two nine-coordination coordination polymers of neodymium, $[\text{Nd}_2(p\text{-C}_8\text{H}_4\text{O}_4)(o\text{-C}_8\text{H}_4\text{O}_4)_2 \cdot 4\text{H}_2\text{O}]_n$ (**I**), $[\text{Nd}_2(\text{C}_{10}\text{H}_4\text{O}_8)(\text{C}_{10}\text{H}_2\text{O}_8) \cdot 2\text{H}_2\text{O}]_n$ (**II**), have been prepared by hydrothermal reaction of $\text{Nd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ with terephthalic acid and phthalic acid, or benzenetetracarboxylic anhydride and determined by means of IR, UV, fluorescence, TG-DTA, cyclic voltammetry (CV) and X-ray single-crystal diffraction methods (CIF files CCDC nos. 1006206 (**I**), 979309 (**II**)). Yellow-green luminescence could be observed at 391 nm ($\lambda_{\text{ex}} = 305$ nm) for complex **I** and 370 nm ($\lambda_{\text{ex}} = 331$ nm) for the complex **II**. The emission of complexes **I** and **II** may be due to the $\pi^* \rightarrow n$ transition, which may be assigned to the ligand-to-metal charge-transfer bands. Compared with complex **II**, the complex **I** exhibits a stronger fluorescence intensity for the different coordinated environment. Cyclic voltammetric measurement of the two compounds reveal that the compounds both have a couple of irreversible redox peak, indicating that the two polymers were both corresponded to the unusual $\text{Nd}(\text{III})/\text{Nd}(\text{V})$.

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INTRODUCTION

Rare earth elements was known as “the material of the 21st century” [1, 2], it has a unique physical and chemical properties. China has a large amount of rare earth which has a profound impact on economic and social development [3, 4]. Since the 20th century, research of rare earth for people has been more and more widely, especially in the field of coordination chemistry [5–7]. Neodymium as a member of the rare earth elements has some incomparable spectroscopic properties and it’s complexes with organic ligands in probe light-emitting materials [8], superconducting materials, structure, molecular sieve, catalytic and biological activity, and other fields have caught people’s widely attention [9–12].

Phenoxy acid is a very important kind of carboxylic acid. The carboxylic acid has rich way of coordination, most of the complexes of rare earth and carboxylic acid are stable, so it occupies an important position in coordination chemistry. The carboxyl group can not

only bonded to the metal ion in a variety of ways with the ligand, but also form a secondary structural elements of the multinuclear metal ions, and then constructed with a variety of polymer network structure [13–15]. What’s more, the substitution position of the carboxyl group more variable can be assembled into a plurality of metal ions and formed to a new stabilized topologies. Carboxylic acid coordination polymer has been widely studied in the field of light, catalysis and materials [16, 17]. A great number of the compounds of $\text{Nd}(\text{III})$ and carboxylic acid were synthesized and characterized, for example, the compound of $\text{Nd}(\text{III})$ and 2,3-pyrazinedicarboxylic acid was synthesized by SHE Jiangboin 2011 [18]. Renata Łyszczek had also synthesized the compound of $\text{Nd}(\text{III})$ and biphenyl-4,4'-dicarboxylate [19]. Along the pace of their predecessors, we have synthesized two new polymers of $\text{Nd}(\text{III})$ $[\text{Nd}_2(p\text{-C}_8\text{H}_4\text{O}_4)(o\text{-C}_8\text{H}_4\text{O}_4)_2 \cdot 4\text{H}_2\text{O}]_n$ (**I**), $[\text{Nd}_2(\text{C}_{10}\text{H}_4\text{O}_8)(\text{C}_{10}\text{H}_2\text{O}_8) \cdot 2\text{H}_2\text{O}]_n$ (**II**), and phenoxy acids. The crystal structures, spectroscopy and electrochemistry behavior of the obtained compounds are studied.

¹ The article is published in the original.

EXPERIMENTAL

Materials and physical measurement. All chemicals were analytical grade and deionized water used as solvent without purification. Elemental analysis (C, H) was performed on a Perkin-Elmer 2400 elemental analyzer. The infrared spectrum was recorded as a KBr pellet on a Nicolet 170SXFTIR spectrometer in the range 400–4000 cm⁻¹. The UV spectrum was obtained on a Lambda 35 spectrophotometer in the range 200–800 nm. Fluorescence data was collected on a LS-55 fluorescence spectrophotometer. Thermal gravimetric analysis was tested under nitrogen with a heating rate of 20°C/min using U.S. PERKIN ELMER Company Diamond TG–DTA thermal analysis system. Cyclic voltammetry (CV) were obtain on a model CH1660C electrochemical analyzer controlled by a personal computer at room temperature.

Synthesis of I. A mixture of phthalic acid (0.3328 g, 2.0 mmol), Nd(NO₃)₃ · 6H₂O (0.2193 g, 0.5 mmol), terephthalic acid (0.3318 g, 2.0 mmol) and H₂O (15 mL) was stirred for half an hour in air, and pH value of the solution was adjusted to 2.5 by the addition of hydrochloric acid. The mixture was then transferred to a Teflon-lined stainless steel autoclave (25 mL) and kept at 170°C for 5 days. After the autoclave had cooled to room temperature, purple crystals were filtered off, washed with distilled water, and air-dried to give a yield of 30% based on Nd.

For (C₂₄H₂₀O₁₆Nd₂)_n (I)

anal. calcd., %:	C, 33.1;	H, 2.8.
Found, %:	C, 33.5;	H, 2.3.

Synthesis of II. A mixture of benzenetetracarboxylic anhydride (0.1125 g, 0.5 mmol), Nd(NO₃)₃ · 6H₂O (0.2210 g, 0.5 mmol) and H₂O (15 mL) was stirred for half an hour in air, and pH value of the solution was adjusted to 2.0 by the addition of hydrochloric acid. The mixture was then transferred to a Teflon-lined stainless steel autoclave (25 mL) and kept at 170°C for 2 days. When the solution was cooled to room temperature, yellow crystals were filtered off, washed with distilled water, and air-dried to give a yield of 40% based on Nd.

For (C₂₀H₁₀O₁₈Nd₂)_n (II)

anal. calcd., %:	C, 28.4;	H, 1.5.
Found, %:	C, 28.6;	H, 1.2.

X-ray structure determination. Single crystals were performed by X-ray crystallographic analyses at 296(2) K on a Bruker SMART APEX CCD fine-focus sealed tube diffractometer with graphite monochro-

mated MoK_α ($\lambda = 0.71073 \text{ \AA}$) radiation. Data collection, indexing, and initial cell refinements were carried out using SMART software. Frame integration and final cell refinements were carried out using SAINT software. Absorption corrections for each data set were applied using SADABS program. Structure solution, refinement, and generation of publication materials were solved by methods with SHELXTL [20, 21]. The crystal parameters, data collection, and refinement results are summarized in Table 1. Selected bond lengths and angles are listed in Table 2.

Crystallographic data for structures I and II have been deposited with Cambridge Crystallographic Data Center (nos. 1006206 (I), 979309 (II); deposit@ccdc.cam.ac.uk or <http://www.ccdc.cam.ac.uk>).

RESULTS AND DISCUSSION

In structure I, phthalic acid acts as bridge to link four neodymium atoms, the two oxygen atoms of the carboxylic group coordinate to the same neodymium atom, in which one of them adopt a bidentate chelating mode connecting another neodymium atom. The two oxygen atoms of the other carboxylic group coordinate to two different neodymium atom (Fig. 1a).

Terephthalic acid also acts as bridge to link two neodymium atoms, in which each deprotonated carboxylic group coordinates to one neodymium. Thus, the Nd atom is coordinated by nine oxygen atoms, five from phthalic acids, two from terephthalic acids and two from two coordinated water molecules. The C–O distances of the carboxylic groups are typical, ranging from 1.231(8) to 1.274(10) Å. The O(1) and O(5) respectively connect two different neodymium atoms, since one oxygen atom can provide two pairs of lone pair electrons. Due to the steric hindrance, the Nd(2)–O(5) (2.909 Å) is much longer than others, ranging from 0.2349(5) to 0.2588(6) nm, while the Nd(1)–O(1) (2.648 Å) is also longer. Along y axis, a one-dimensional chain structure is formed by the carboxylic group of phthalic acid (Fig. 1b), every two adjacent chains is linked by the carboxylic group in terephthalic acid to generate a two-dimensional mesh structure (Fig. 1c). The shortest distance of complex I between the two neighboring benzene rings is 3.5587 Å (as shown in Fig. 1d). The extensive offset face-to-edge $\pi \cdots \pi$ stacking interactions increases the stability of the structure for the coordination polymers.

Complex II is a three-dimensional framework containing nine-coordinate neodymium centers. There are two kinds of carboxylic groups, protonated carboxylic groups and deprotonated in the structure (Fig. 2a). The pyromellitic acid which contains protonated carboxylic groups acts as bridge to link six

Table 1. Crystallographic data and refinement parameters for coordination polymers **I** and **II**

Parameter	Value	
	I	II
Formula weight	852.88	826.76
Crystal size, mm	0.10 × 0.08 × 0.07	0.12 × 0.11 × 0.08
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
Volume, Å ³	1311.43(13)	534.9(2)
<i>a</i> , Å	6.2078(4)	6.3947(17)
<i>b</i> , Å	8.8862(5)	9.356(3)
<i>c</i> , Å	25.2194(14)	9.544(3)
α, deg	88.605(2)	88.476(3)
β, deg	87.090(2)	74.413(3)
γ, deg	70.719(2)	76.715(3)
<i>Z</i>	2	2
ρ _{calcd} , g m ⁻³	2.160	2.567
Absorption coefficient, mm ⁻¹	3.997	4.902
<i>F</i> (000)	824	394
θ Range for data collection, deg	2.93–25.25	2.22–26.00
Index ranges	$-7 \leq h \leq 7, -10 \leq k \leq 8$ $-30 \leq l \leq 26$	$-7 \leq h \leq 7, -11 \leq k \leq 11,$ $-11 \leq l \leq 11$
Reflections collected/unique (<i>R</i> _{int})	10341/4662 (0.0581)	4594/2086 (0.0338)
Reflections with <i>I</i> ≥ 2σ(<i>I</i>)	4662	2086
Goodness-of-fit on <i>F</i> ²	1.035	0.992
Number of refinement parameters	379	185
<i>R</i> ₁ , <i>wR</i> ₂ (<i>I</i> ≥ 2σ(<i>I</i>))*	0.0771, 0.0680	0.0270, 0.0679
<i>R</i> ₁ , <i>wR</i> ₂ (all data)*	0.0400, 0.0788	0.0263, 0.0685
Δρ _{max} /Δρ _{min} , e Å ⁻³	0.0963/–1.195	1.419/–1.687

* **I.** $R_1 = \sum |F_{\text{o}}| - |F_{\text{c}}| / \sum |F_{\text{o}}|$; $wR_2 = \sum [w(F_{\text{o}}^2 - F_{\text{c}}^2)^2] / \sum [w(F_{\text{o}}^2)^2]^{1/2}$; $w^{-1} = [\sigma^2(F_{\text{o}})^2 + (0.0259P)^2 + 1.0007P]$, $P = (F_{\text{o}}^2 + 2F_{\text{c}}^2)/3$. **II.** $R_1 = \sum |F_{\text{o}}| - |F_{\text{c}}| / \sum |F_{\text{o}}|$; $wR_2 = \sum [w(F_{\text{o}}^2 - F_{\text{c}}^2)^2] / \sum [w(F_{\text{o}}^2)^2]^{1/2}$; $w^{-1} = [\sigma^2(F_{\text{o}})^2 + (0.0430P)^2 + 1.1566P]$, $P = (F_{\text{o}}^2 + 2F_{\text{c}}^2)/3$.

Table 2. Selected bond distances (Å) and angles (deg) for **I** and **II**

Bond	<i>d</i> , Å	Bond	<i>d</i> , Å
I			
Nd(1)–O(5)	2.420(5)	Nd(2)–O(3)	2.349(5)
Nd(1)–O(13)	2.434(5)	Nd(2)–O(8)	2.474(5)
Nd(1)–O(10)	2.501(5)	Nd(2)–O(5)	2.909(5)
Nd(1)–O(14)	2.502(6)	Nd(2)–O(6)	2.510(5)
Nd(1)–O(9)	2.541(5)	Nd(2)–O(11)	2.507(5)
Nd(1)–O(2)	2.588(6)	Nd(2)–O(1)	2.500(5)
Nd(1)–O(1)	2.648(5)	Nd(2)–O(15)	2.482(5)
Nd(1)–O(7)	2.478(5)	Nd(2)–O(12)	2.462(6)
Nd(1)–O(4)	2.478(5)	Nd(2)–O(16)	2.459(6)
C(7)–O(2)	1.244(9)	C(7)–O(1)	1.274(10)
C(8)–O(4)	1.231(8)	C(8)–O(3)	1.255(9)
C(15)–O(5)	1.236(9)	C(15)–O(6)	1.273(9)
C(16)–O(7)	1.257(8)	C(16)–O(8)	1.265(9)
C(20)–O(10)	1.258(9)	C(20)–O(9)	1.259(9)
C(24)–O(11)	1.267(10)	C(24)–O(12)	1.242(9)
II			
Nd(1)–O(1)	2.510(3)	C(4)–O(1)	1.254(5)
Nd(1)–O(2)	2.512(3)	C(4)–O(2)	1.261(5)
Nd(1)–O(2)	2.608(3)	C(5)–O(3)	1.259(5)
Nd(1)–O(3)	2.565(3)	C(5)–O(4)	1.264(5)
Nd(1)–O(4)	2.535(3)	C(9)–O(5)	1.247(5)
Nd(1)–O(5)	2.433(3)	C(9)–O(6)	1.255(5)
Nd(1)–O(6)	2.381(3)	C(10)–O(7)	1.222(5)
Nd(1)–O(7)	2.539(3)	C(10)–O(8)	1.310(6)
Nd(1)–O(9)	2.453(3)		
Angle	ω , deg	Angle	ω , deg
I			
O(5)Nd(1)O(2)	118.53(18)	O(4)Nd(1)O(2)	73.23(18)
O(10)Nd(1)O(1)	144.17(18)	O(9)Nd(1)O(1)	149.60(17)
O(2)Nd(1)O(1)	49.54(17)	O(3)Nd(2)O(1)	73.92(18)
O(8)Nd(2)O(1)	80.22(18)	O(3)Nd(2)O(8)	136.55(17)
O(1)Nd(2)O(6)	114.33(16)	O(3)Nd(2)O(5)	67.12(17)
O(8)Nd(2)O(5)	70.94(16)	O(1)Nd(2)O(5)	67.57(15)
O(6)Nd(2)O(5)	46.85(16)	Nd(2)O(1)Nd(1)	111.15(19)
II			
O(6)Nd(1)O(1)	80.87(11)	O(6)Nd(1)O(5)	76.84(11)
O(9)Nd(1)O(1)	102.06(12)	O(5)Nd(1)O(1)	72.3(1)
O(5)Nd(1)O(2)	136.68(10)	O(6)Nd(1)O(2)	145.48(11)
O(1)Nd(1)O(2)	113.21(9)	O(5)Nd(1)O(9)	66.27(12)
O(5)Nd(1)O(4)	142.10(11)	O(6)Nd(1)O(4)	72.47(11)
O(1)Nd(1)O(4)	122.74(10)	O(9)Nd(1)O(4)	130.83(12)
O(6)Nd(1)O(7)	88.80(12)	O(2)Nd(1)O(4)	73.57(10)
O(5)Nd(1)O(3)	137.13(10)	O(5)Nd(1)O(7)	87.02(11)
O(5)Nd(1)O(2)	101.71(10)	O(1)Nd(1)O(7)	158.48(11)
O(1)Nd(1)O(2)	50.76(9)	O(1)Nd(1)O(3)	73.19(10)
O(2)Nd(1)O(2)	63.38(11)		

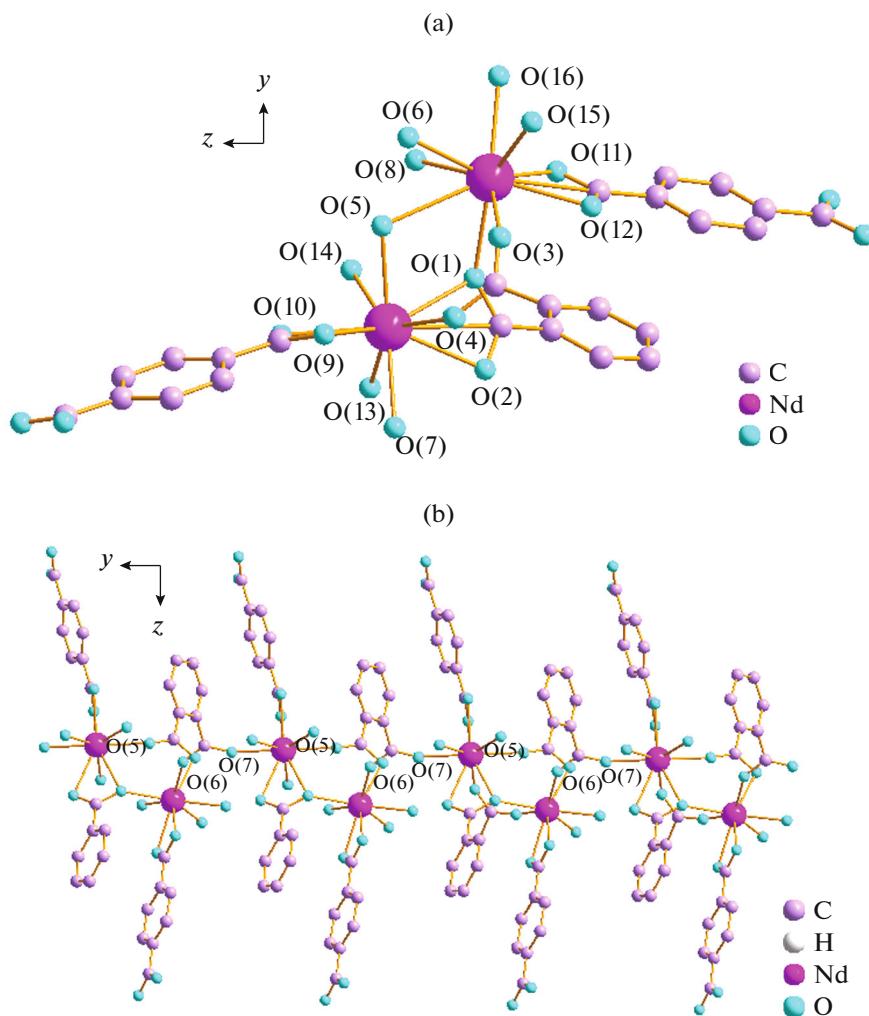


Fig. 1. Molecular structure of complex I (all hydrogen atoms are omitted for clarity (a); the 1D structure of complex I (b); the 2D structure of complex I (c); the distance of two neighboring aromatic rings of complex I (d)).

neodymium atoms, each protonated carboxylic group coordinates to one neodymium atom and each deprotonated carboxylic group connects to two neodymium atoms. The pyromellitic acid that have no protonated carboxylic groups also acts as bridge to link six neodymium atoms, the two *para*-carboxylate groups adopt a bidentate chelating mode coordinating to the same neodymium atom while the other two *para*-carboxylate groups adopt a bidentate chelating-bridging mode connecting two neodymium atoms. Thus, the environment around Nd is coordinated by nine oxygen atoms which one comes from protonated carboxylic group, seven come from deprotonated carboxylic groups and one comes from a coordinated water molecule. The Nd–O distances range from 2.381 to 2.608(3) Å. The C–O distances of the deprotonated carboxylic groups range from 0.1247(5) to 1.264(5) Å and a clear difference of C–O distances within the protonated carboxylic group is observed: C(10)–O(8)

(1.310(6) Å) is much longer than C(10)–O(7) (1.222(5) Å). The two adjacent Nd(1) atoms and O(2) atoms form a stable rhomboid since the larger radius of neodymium atom can make the tension become smaller to form a ring. Along *z* axis, a one-dimensional chain structure is formed by the O(5)–C(9)–O(6) in the deprotonated carboxylic group (Fig. 2b), the two chains are linked by the carbonyl oxygen of the protonated carboxylic group to form a two-dimensional mesh structure. The bridging carboxylate groups in ligands connect each other to produce a three-dimensional structure (Fig. 2c). The shortest distance of complex II between the two neighboring benzene rings is 3.5378 Å (Fig. 2d). Similar to complex I, the extensive π – π stacking increases the stability of the structure.

The IR spectrum of complex I exhibits a strong absorption at 1543 cm⁻¹ and complex II also shows

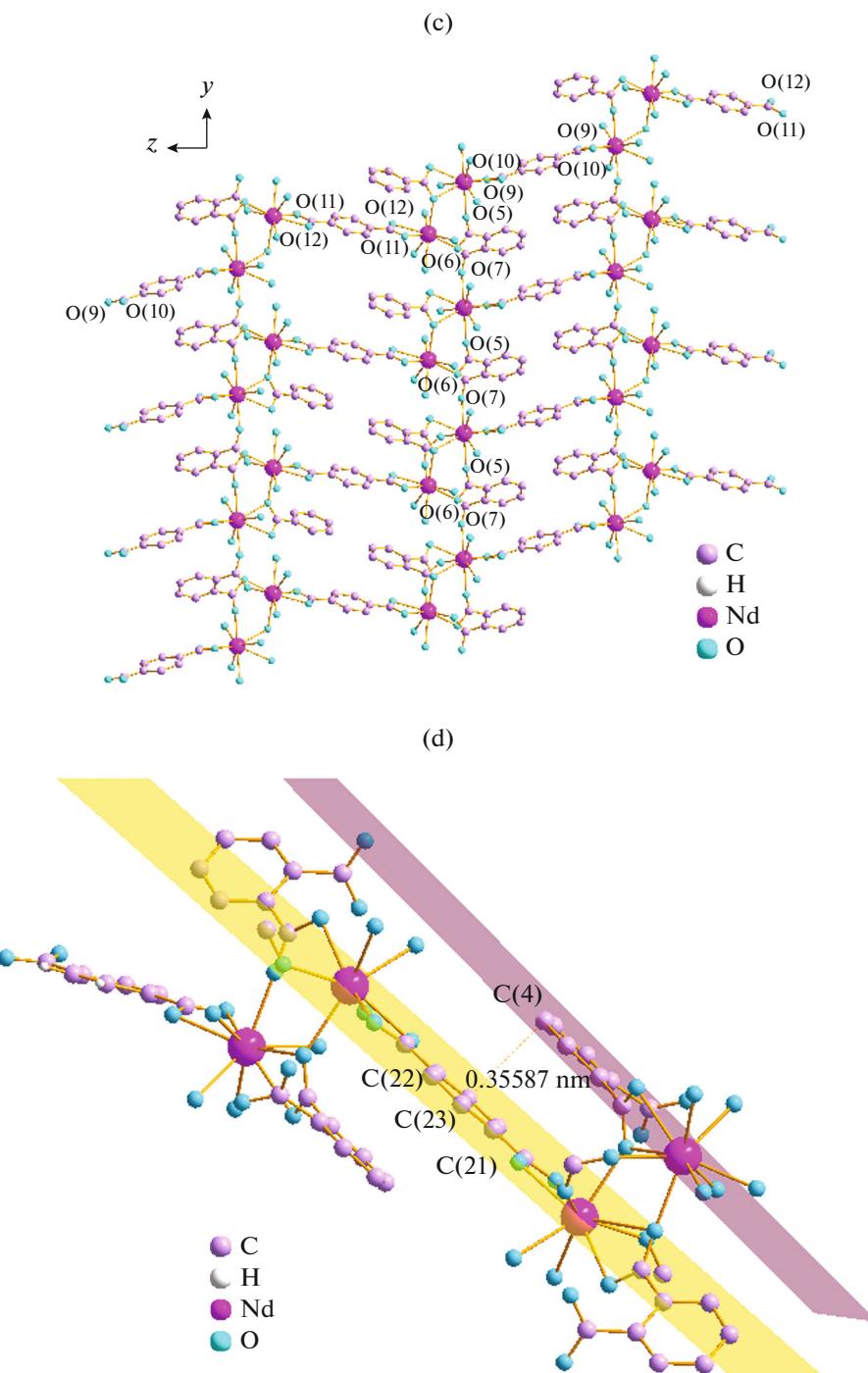


Fig. 1. (Contd.)

several obvious peaks at 1450–1600 cm^{-1} due to the stretching vibration of C=C on the benzene [22]. The observation of an absorption peak at 1680 cm^{-1} for $-\text{COOH}$ indicates the presence of carboxylate groups in complex **I** and the carboxylate groups are at 1667 cm^{-1} in complex **II**. This is also in agreement

with the results of the single crystal X-ray diffraction analysis.

The UV spectrum of coordination polymers were measured in H_2SO_4 solution within the wavelength range of 200–500 nm. Two absorption bands at 230 and 276 nm were observed for complex **I**, and

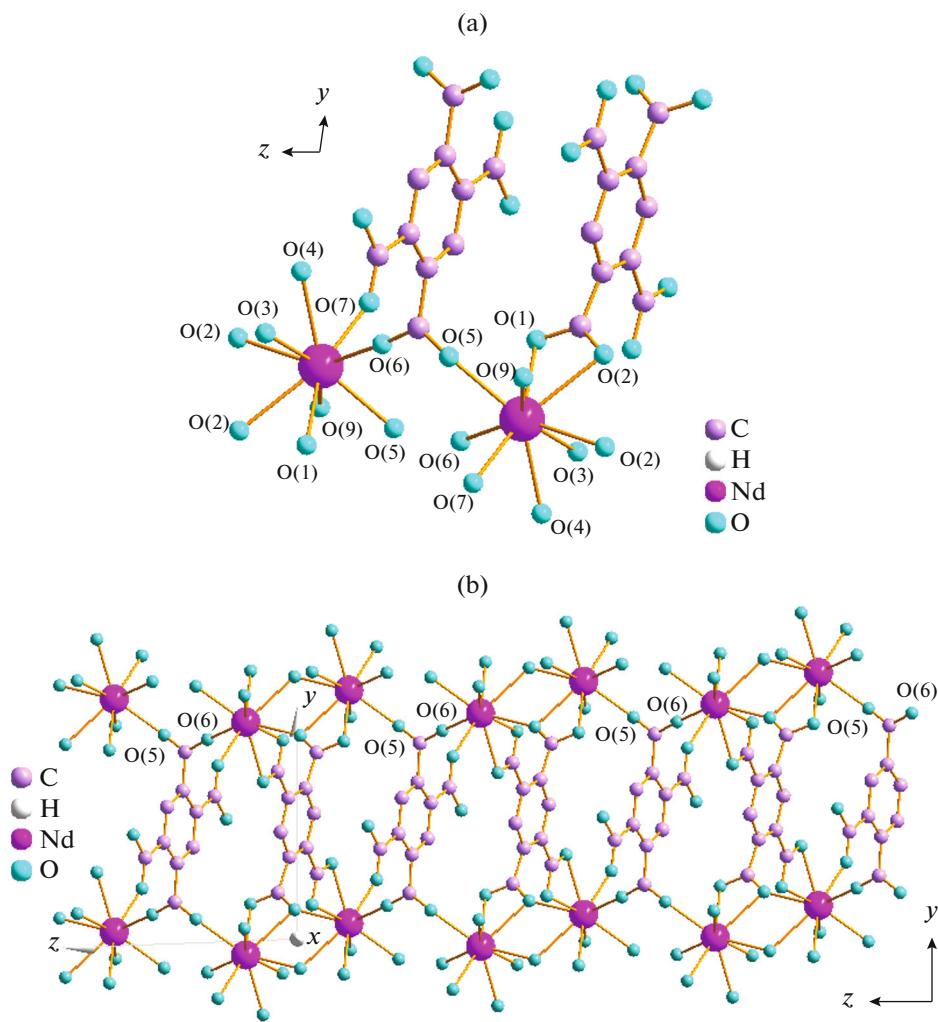


Fig. 2. Molecular structure of complex **II** (all hydrogen atoms are omitted for clarity) (a); the 1D structure of complex **II** (b); the 2D structure of complex **II** (c); the distance of two neighboring aromatic rings of complex **II** (d).

complex **II** also showed two bands at 225 and 280 nm, which can be respectively attributed to the $\pi-\pi^*$ transition of the phenyl ring [23, 24]. Compared with the ligand, these bonds of the complexes show slightly shifts which can be attributed to the binding of the ligands to the central metal ions [25].

It has been shown that the low molar absorptivity of the neodymium ions do not favor a strong emission when excited directly, the emission of neodymium ions can be observed by employing the ligands that can absorb and transfer the energy to the central neodymium ions. The ligand is excited to the singlet state, from where part of the energy is transferred to the triplet state through inter system crossing. When the energy levels are favorable, the triplet excited state can transfer the energy to the metal centers, resulting in metal centered luminescence. The success of this transfer of energy is reflected in the suppressing of the

intra-ligand emission in the luminescence spectra [26, 27]. The emission spectrum of complexes **I** and **II** in H_2SO_4 are shown in Fig. 3. The phthalic acid show a emission bands with the maximum at 361 nm and the terephthalic acid are at 385 nm while complex **I** exhibits intense broad emission bands with the maximum at 391 nm upon excitation at 305 nm, which the bank is slightly red-shift after forming complex **I**. It can be proved that the phthalic acid and the terephthalic acid are involved in coordination. However, complex **II** also exhibits a broad emission band with the maximum at 370 nm upon excitation at 331 nm while the benzenetetracarboxylic anhydride show no fluorescence intensity, which is the evidence that benzenetetracarboxylic anhydride is also involved in coordination. The emission of complex **I** and **II** may be due to the $\pi^* \rightarrow n$ transition, which may be assigned to the ligand-to-metal charge-transfer bands [28, 29]. Com-

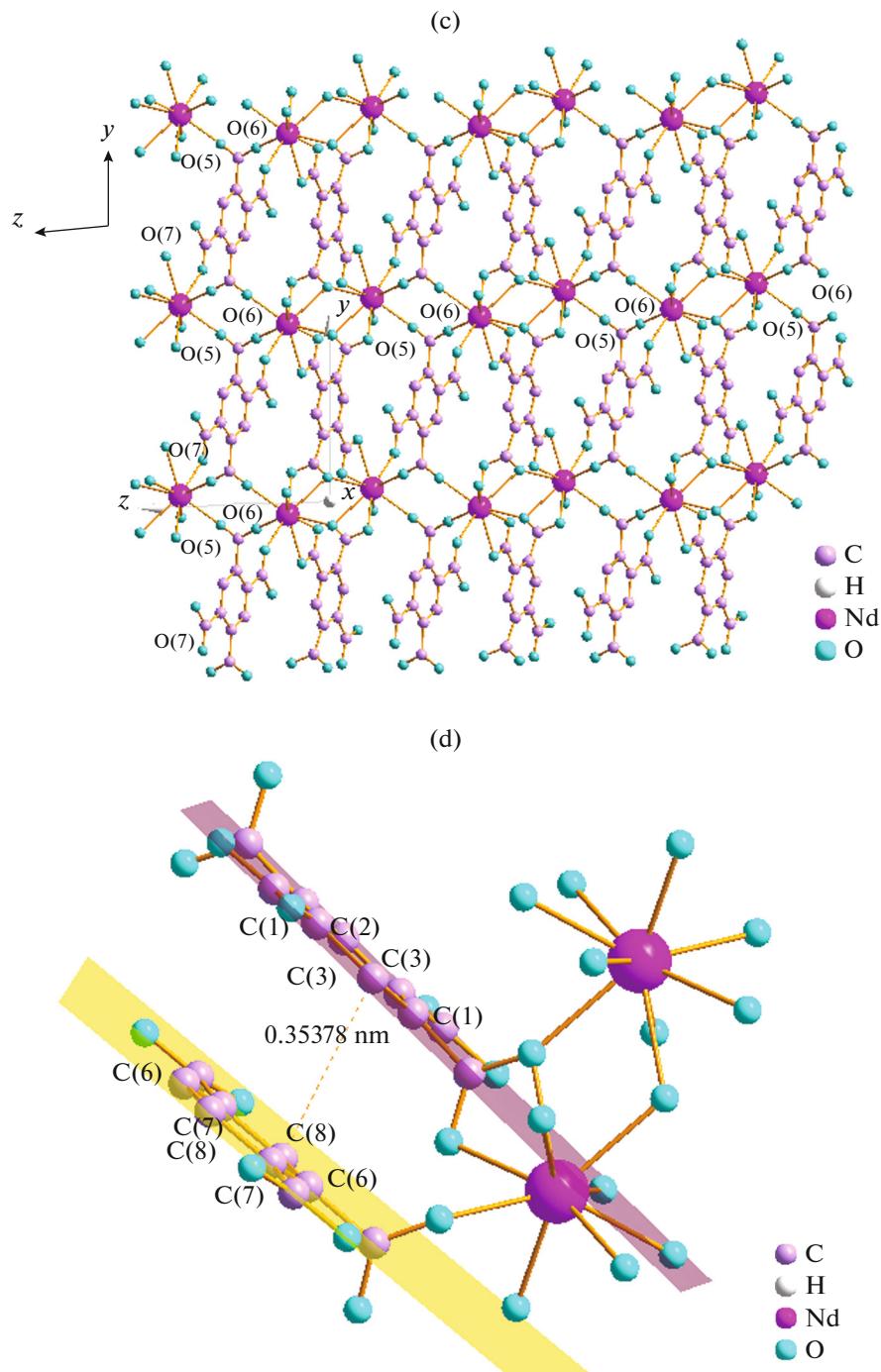


Fig. 2. (Contd.)

pared with complex **II**, complex **I** exhibits a stronger fluorescence intensity for the different coordinated environment.

CV measurement of the complexes were carried out in H_2SO_4 solution at 25°C using carbon electrodes as

the working electrode and the reference electrode at a scan rate 10 mV/s. As show in Fig. 4, for complex **I**, a couple of reduction and oxidation was recorded in the range -0.25 – 0.4 V. The oxidation peak $E_{\text{pa}1} = 0.129$ V, and the reduction peak $E_{\text{pc}1} = 0.047$ V, $\Delta E_{\text{p}1} = 82$ mV.

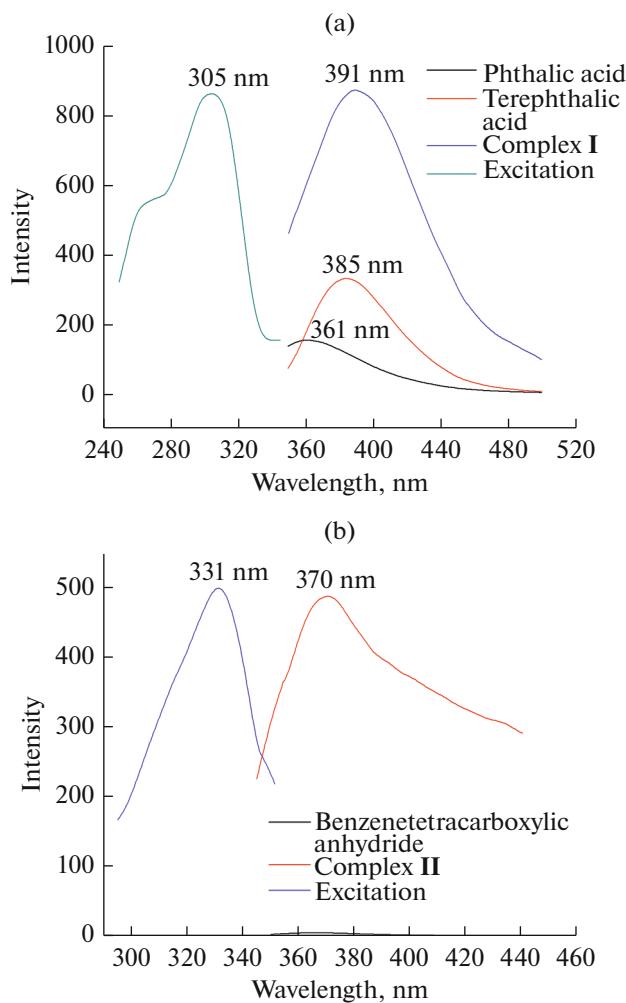


Fig. 3. Fluorescence spectra of complexes I (a) and II (b) in H_2SO_4 .

$E_{\text{pa}1/2} = 0.093$ V and $E_{\text{pc}1/2} = -0.069$ V, $\Delta E_{\text{p}1/2} = 0.024$ V. The result that the transition was irreversible could be concluded, because the $\Delta E_{\text{p}1}$ is not equal to 59 mV [30]. According to $|E_{\text{p}} - E_{\text{p}1/2}| = 47.7/\alpha n$ ($\alpha = 0.5$), the electron transfer number of complex I involved in the processes [31] could be calculated to be two.

For complex II, a couple of reduction and oxidation was recorded in the range -0.3 – 0.3 V. $E_{\text{pa}2} = 0.083$ V, $E_{\text{pc}2} = -0.051$ V, $\Delta E_{\text{p}2} = 134$ mV, $E_{\text{pa}2/2} = 0.056$ V, $E_{\text{pc}2/2} = -0.032$ V and $\Delta E_{\text{p}2/2} = 0.088$ V. The electron transfer number of complex could be also calculated to be two. The result correspond to the fact that Nd(III) change to Nd(V) for complex I and complex II.

The TG measure show a three-step weight loss both complexes I and II (Fig. 5). For complex I, the

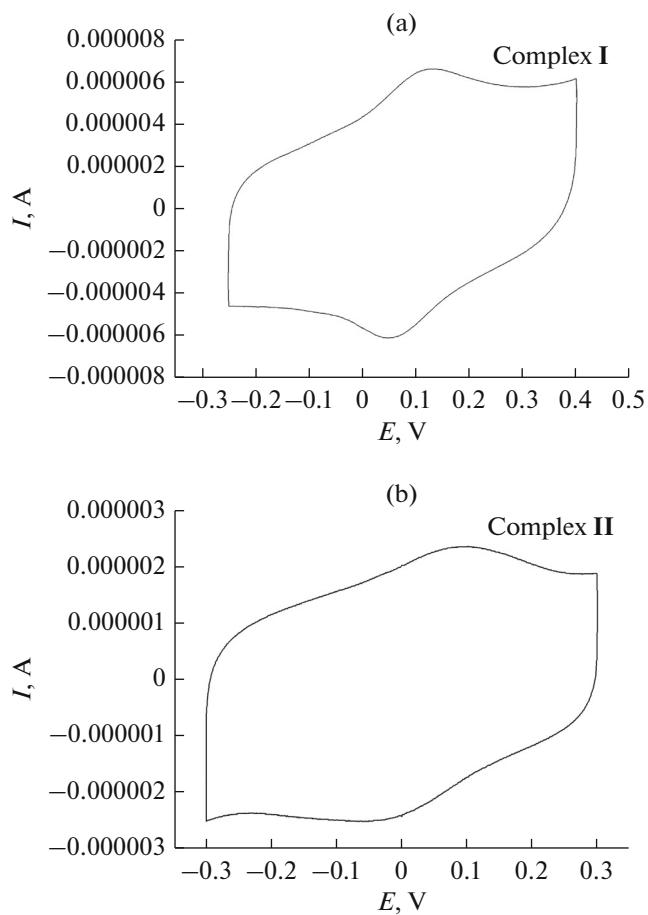


Fig. 4. CV of complexes I (a) and II (b) in H_2SO_4 solution; scan rate: 5 mV/s.

first loss of 12.85% was four coordinated water molecules and a carboxy group on the terephthalic acid between 150 and 260°C (calcd. 13.60%), accompanied with an obvious peak at 194°C. The second loss (13.23%) from 260 to 350°C corresponds to the loss of the rest of the terephthalic acid (calcd. 14.07%) with a peak at 300°C. The final loss was the decomposition of the phthalic acid, which started at 350°C until the Nd_2O_3 (40.71%) had been the last remaining (calcd. 39.40%), accompanied with a peak at 375°C. For complex II, the loss (3.77%) of the two coordinated water molecules ended at 360°C [32] (cacl. 4.35%) with a obvious peak at 350°C. The next was the loss of three carboxy groups (15.58%) on the benzenetetracarboxylic anhydride between 360 and 520°C which contains protonated carboxylic groups (calcd. 15.97%). The final loss occurred in the range of 520 to 770°C, which was the loss of the rest of the benzenetetracarboxylic anhydride that contains protonated carboxylic groups and the benzenetetracarboxylic

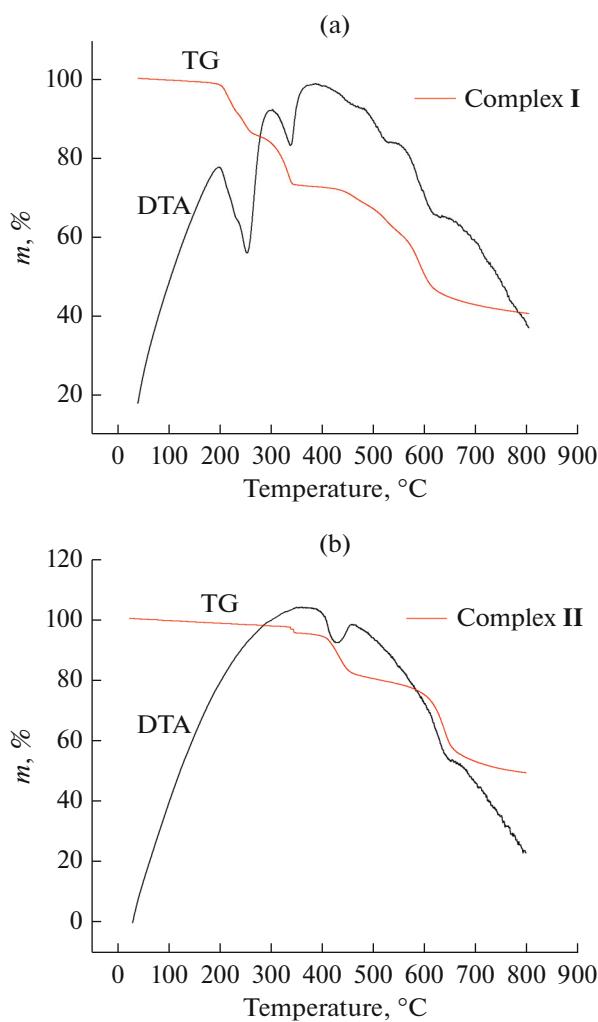


Fig. 5. TG—DTA curve of complexes I (a) and II (b).

anhydride that contains deprotonated carboxylic groups started to decompose. Above these, the results of the TG show that complex **II** has higher thermal stability than complex **I**.

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