



Crystal structure and luminescence properties of a new nanostructure lead(II) complex: a precursor for preparation of pure phase nanosized PbO

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ABSTRACT

The reaction of 1,3-diphenylpropane-1,3-dione (HL) ligand with lead(II) nitrate under hydrothermal conditions led to the formation of a novel lead complex with singular structural features. The characterization of title complex was performed by spectroscopy methods such as ¹H NMR, UV, and IR and elemental analyses (CHN) and crystal structure of prepared lead (II) complex was determined by single-crystal X-ray diffraction. The facile and productive sonochemical method was used to prepare nano-size particles of the title complex at room temperature. The prepared nano-size-complex was characterized by elemental analysis, scanning electron microscopy (SEM), IR spectroscopy and X-ray powder diffraction (XRD). The nano-size lead oxides that prepared by calcination of the nano-size complex and crystalline bulk complex showed the initial particle size of the precursor is influential on the particle size of the derived PbO nanoparticles. Optical property investigation of the PbO nanoparticles at room temperature showed that the size of PbO nanoparticles has an important role on their optical behavior.

1. Introduction

During the last two decades, pronounced interest has been devoted to the preparation of novel metal coordination compounds with multi-dimensional networks, which involves a self-assembly of organic multitopic ligands with suitable functional groups and metallic centers. Concomitantly, this interest also is due to the unique structural topologies and potential applications in many fields, such as lasers, telecommunication technology, transistors and optical sensors for very particular probes and in bioanalysis [1-5]. Investigation of the molecular structures of a large number of coordination compounds of main and transition metal ions exhibited that the selection of an appropriate ligand and the knowledge of the nature and number of the binding modes and binding geometry of it is one of the most important tasks to improve the molecular recognition ability of coordination compounds. A change in length, symmetry and flexibility of organic ligands provides serviceable

conditions for making new classes of compounds with distinctive arrangement and applications [6-10]. It seems potential utilization of nano-metal-organic coordination compounds is necessary for nano-technological applications. Nevertheless, preparation and investigation of their properties have not yet been comprehensively scrutinized [11-15]. The chemical and physical properties of materials strongly rely on their size and shape. Nano-materials completely have different properties and behavior than their bulk particles form because of their small size and large surface-to-volume ratios [16-19]. Therefore, developing nano-metal-organic coordination polymers in any mold with remarkable properties should be very important and beneficial for the future success of science and technology in the nanoscale field. For the first time, Wang reported preparation of non-coordination polymers and by this time, several distinctive synthetic methods have been suggested for the preparation of nanosized materials [20-27]. However, present synthetic techniques are costly and time-consuming and

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introducing alternative methods for the preparation of novel nano-materials with uncommon properties are essential for further advances in nanotechnology.

In the present work, sonochemical route as a facile and productive method was used for the preparation of a uniform nano-size lead(II) coordination polymer. Sonochemistry as a favorable synthesis method can resolve problems encountered with the routine preparation methods by saving time and energy and diminishing the expense of final products. In this work, a new lead complex and its nano-size counterpart with 1,3-diphenylpropane-1,3-dione (HL) as a ligand have been synthesized under hydrothermal conditions and sonochemical method, respectively. Moreover, the solid-state transformation method as an alternative synthetic method has been used for the preparation of nano-size lead(II) oxide from nano-size complex and bulk crystalline complex as a precursor for understanding the effect of the initial particle size on the size of final derived PbO nanoparticles.

2. Results and Discussion

2.1. Synthesis

A new lead(II) coordination polymer as light yellow needle-like crystals was successfully synthesized with 1,3-diphenylpropane-1,3-dione (HL) ligand under hydrothermal conditions. Then nanosize of the same compound was prepared by the sonochemical method. Calcination of both compound at 600 °C resulted in the formation of nano-size lead oxide particles with different sizes.

The results of preparation of nano-size lead(II) complex using the sonochemical method as a facile and cost effective route shows this approach can be an alternative and inexpensive route for the preparation of nano-size coordination polymers. All spectroscopic data exhibit that both nano-size and bulk lead(II) complex have the same structures with a formula of $\text{Pb}_2(\text{C}_{15}\text{H}_{11}\text{O}_2)_4$.

2.2. Characterization of title lead complex

The structure of the prepared lead(II) compound was verified by elemental analyses (CHN) and single-crystal X-ray diffraction analyses. The distinct difference between the IR spectrum of title complex and free ligands and also the presence of the stretching vibration bands of Pb–O at 510 cm^{-1} indicate unambiguously coordination of 1,3-diphenylpropane-1,3-dione ligand to Pb(II) ion. Relatively weak stretching vibration bands of the aromatic and aliphatic C–H bonds are observed in the range of $3000\text{--}3050\text{ cm}^{-1}$ and $2930\text{--}3000\text{ cm}^{-1}$, respectively, in the IR spectrum of complex. The Pb–O stretching vibration band appeared at a frequency similar to those discerned in other lead(II) complexes [28]. The IR results of the prepared nano-size

complex are consistent with that of the bulk complex which exhibits; they have the same structure.

In order to provision a valuable information about metal-ligand binding and determination of additional structural property details of the prepared complex in the solution, the ^1H NMR spectrum of the lead(II) complex was recorded. The ^1H NMR spectrum of complex exhibits the anticipated aliphatic and aromatic peaks with the exact integration which somewhat shifted to the low field relative to those of free ligands. The reason of this shift can be related to the electron density transfer from the ligands to the lead atom as an acceptor. To show the nano-size and bulk complex have the same structure, the powder X-ray diffraction pattern of nano-size complex was recorded and compared to the simulated XRD pattern of the single-crystal data. As can be seen in Fig 1, there is good correlation with a small distinction in 2θ values between the simulated XRD pattern of crystal data, and the XRD pattern of nano-size complex prepared by the sonochemical method. Since the experimental data are in good agreement with the simulated powder XRD pattern based on single-crystal data, therefore, it can be concluded the sonochemically prepared compound is a single-phase material.

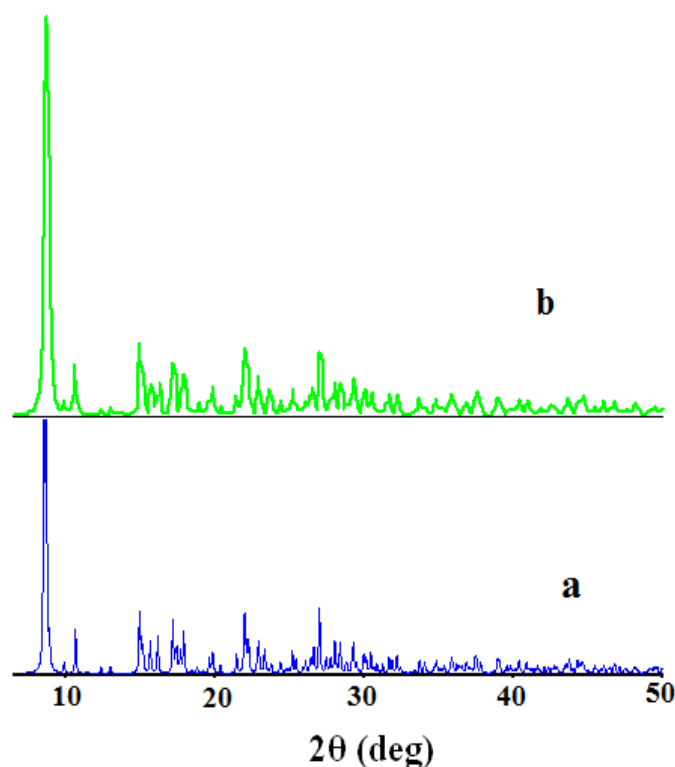


Fig. 1. The XRD pattern of prepared crystalline bulk complex (a) and nano-size complex (b)

The scanning electron microscopy (SEM) technique was exploited to determine the size and morphology of prepared nano-size and crystalline bulk complex. As the SEM images in Fig. 2 shows, both crystalline bulk and nano-size complex have sphere-like morphology, and the average diameters of particles are about 190 and 70 nm, respectively.

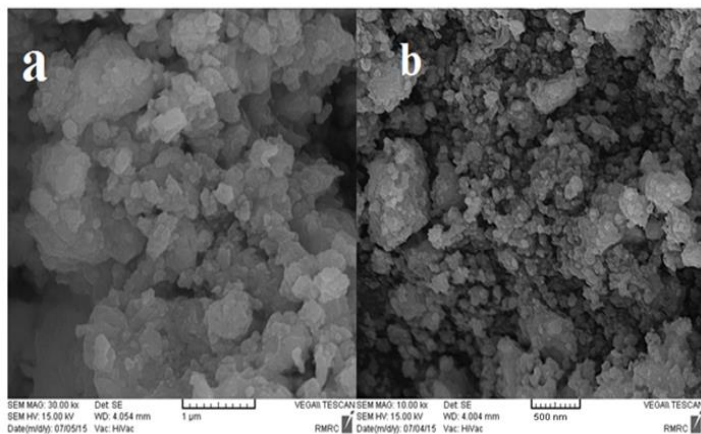


Fig. 2. SEM of prepared crystalline bulk complex (a) and nano-size complex (b)

2.3. Description of crystal structure of lead complex

Crystal structure analysis of prepared compound displays the attendance of a dinuclear, centrosymmetric dimeric, complex that crystallizes in the monoclinic system with the space group of $P2_1/c$. Crystal data and refinement parameters are provided in Table 1. Selected angles and bond lengths are listed in Table 2.

Table 1 Crystal data and refinement details for lead(II) complex

Chemical formula	$\text{Pb}_2(\text{C}_{15}\text{H}_{11}\text{O}_2)_4$
M_r	1307.33
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, b, c (Å)	10.3873 (5), 20.6144 (9), 12.2359 (6)
β (°)	107.952 (5)
V (Å ³)	2492.5 (2)
Z	2
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	6.80
Data collection	
Diffractometer	Rigaku Oxford Diffraction SuperNova diffractometer
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	21707, 5622, 4538
R_{int}	0.041
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.035, 0.075, 1.06
No. of reflections	5622
No. of parameters	316
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	1.51, -0.94

Table 2 Selected bond lengths (Å) and bond angles (°) for lead complex.

Bond	Dist.	Bond	Dist.
Pb(1)-O(1)	2.296(3)	Pb(1)-O(2)	2.356(3)
Pb(1)-O(3)	2.309(3)	Pb(1)-O(4)	2.470(3)
Angle	(°)	Angle	(°)
O(1)-Pb(1)-O(2)	75.30(12)	O(1)-Pb(1)-O(3)	83.09(12)
O(1)-Pb(1)-O(4)	74.64(12)	O(2)-Pb(1)-O(3)	79.08(11)
O(2)-Pb(1)-O(4)	141.94(12)		

The coordination environment of the Pb1 ion with coordination number of five may be described as a distorted square pyramid (Fig. 3). Crystal structure of complex **1** can be considered as a dimer unit that was made of a building block of Pb_2O_2 with a center of inversion. The coordination sphere of Pb(II) atom resulting from the arrangement of the donor oxygen atoms indicates obviously an apparent vacancy. This vacant space displays that the electron lone pair of pb atom is stereochemically active in the structure of prepared lead(II) complex. The bond lengths of the Pb-O bonds on the edge of the Pb1 ion which are opposite to the putative lone pair in comparison with the Pb-O bonds adjacent to them confirm this property. As shown in scheme 1, the 1,3-diphenylpropane-1,3-dione anions adopt two different coordination modes, labeled as **a** and **b**. In mode **a**, the L^- anion is coordinated to one Pb atom in the chelating mode. In mode **b**, the L^- anion with tridentate bridging mode coordinated to two Pb atoms. The distance of $\text{Pb1}\dots\text{Pb1}$ separated by the L^- anion is 4.145 Å. The length of Pb—O bond is in the range between 2.296 (3)–2.718(3) Å. As it was analyzed by CSD 2015, these values are in accordance with those reported for other Pb(II)—O donor complexes. In the structure of prepared complex, the attendances of weak inter- and intramolecular interactions are the principal controlling agent in the managing of packing. The junctions between the binuclear units by the hydrogen bonding interactions and C-H... π (C17–H17...Cg(C25–C30) and C19–H19...Cg(C1–C6)), Pb... π interactions (Fig. 4 and 5) lead to growth of the structure of complex to a 3D supramolecular network.

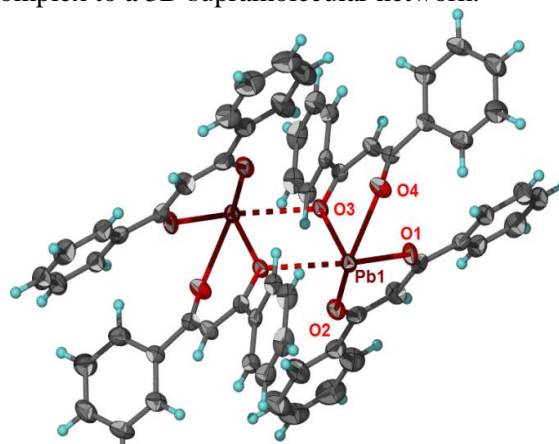
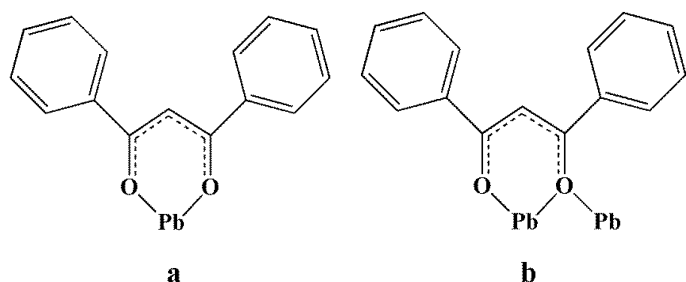


Fig. 3. An ORTEP view of prepared lead(II) complex.



Scheme 1. Coordination modes of 1,3-diphenylpropane-1,3-dione anion.

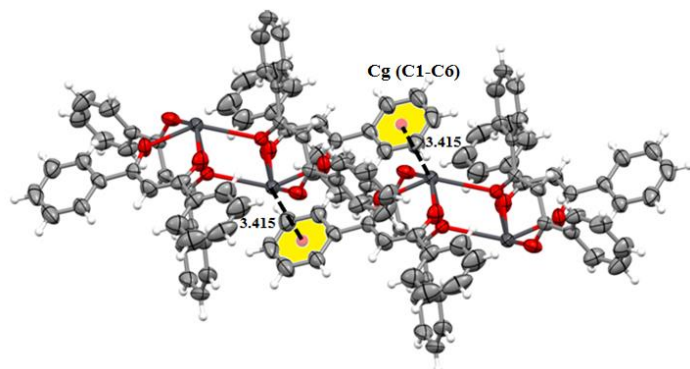


Fig. 4. A perspective view of Pb... π interactions.

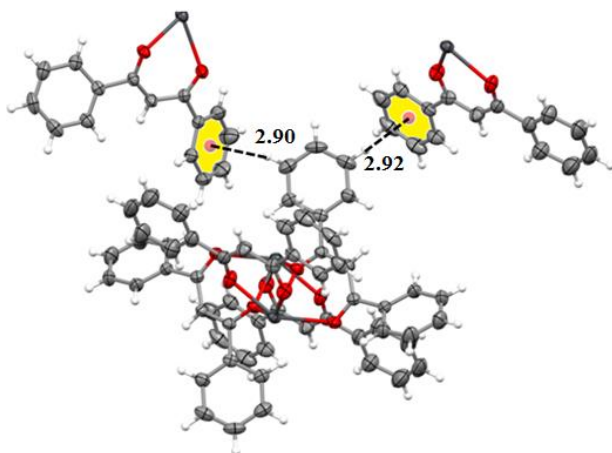


Fig. 5. A perspective view of C-H... π interactions.

2.4. Preparation of lead(II) oxide from lead complex (1)

Recently, considerable efforts have been devoted to learning preparation methods and applications of lead oxide particles. Nonetheless, the novel challenges of scientists are the discovery of alternative and efficient methods for the preparation of pure nano-size lead oxide particles, and also understanding and controlling the size of lead oxide particles during formation [29-31]. In this context, we used the prepared nano-size and crystalline bulk complex as precursors for the preparation of nano-size lead(II) oxide particles and studied the effect of initial particle size of precursor on the particle size of the PbO nanoparticles prepared from them. The results

showed that coordination compounds can be appropriate precursors for the preparation of nano-size metal oxides particles such as nano-size lead oxide particles.

As shown in Fig. 6, the XRD pattern of nano-size lead(II) oxide particles that obtained from the calcination of the bulk complex at 600 °C is in good correlation with the JCPDS files (No. 38-1477). The prepared lead(II) oxide has an orthorhombic structure with lattice parameters of $a = 5.49$, $b = 5.89$ and $c = 4.75$ Å and $Z = 4$. The phase purity of the prepared nano-size lead oxide was confirmed by assigning all reflections and absence of extra reflections in XRD pattern.

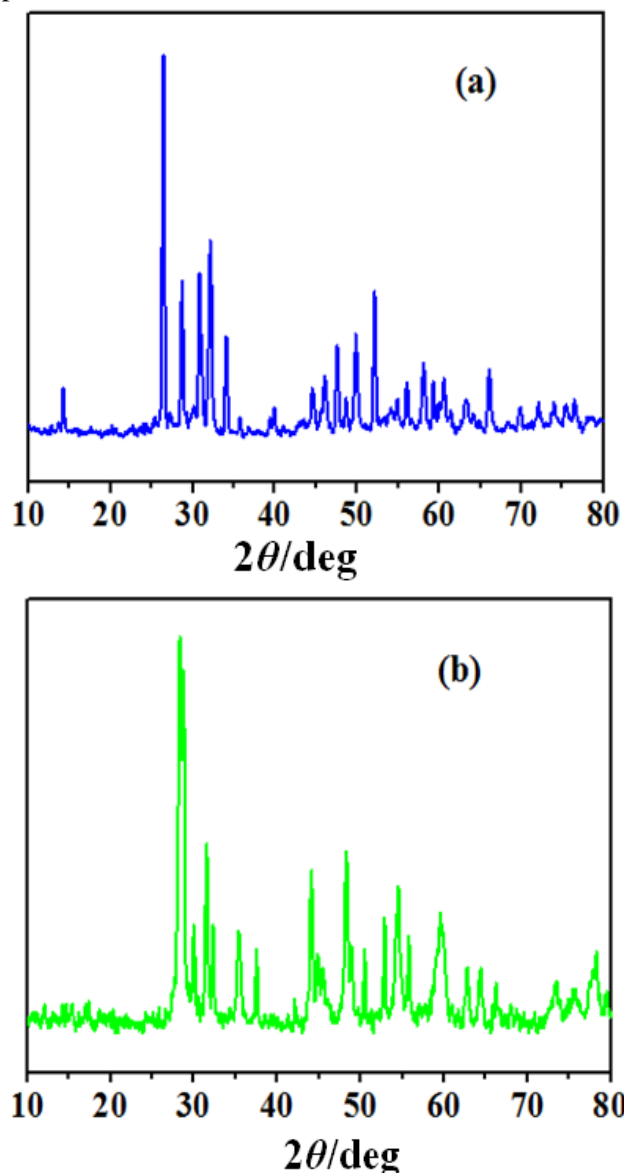


Fig. 6. XRD patterns of lead oxide obtained from the crystalline bulk complex (a) and lead oxide obtained from nano-size complex (b).

The investigation of size and morphology of the prepared nano-size lead(II) oxide from crystalline bulk complex showed particles have a good size distribution and a fair particle segregation with an average diameter at about 70 nm (Fig. 7). Calcination product of the bulk complex showed a coordination compound could be

used as a precursor for the preparation of nano-size metal oxides.

To investigate the effect of initial size of coordination compound on the particle size of final metal-oxides, prepared nano-size complex was calcined in the same condition of bulk complex. As seen in Fig. 6, the XRD pattern of nano-size lead oxide obtained from the calcination of nano-size complex is similar to the XRD pattern of the lead oxide obtained from the bulk complex. This similarity reveals that both lead oxides have the same structure with the same lattice parameter

The SEM image of nano-size lead oxide obtained from the nano-size complex exhibits an average diameter of about 30 nm with a good size distribution (Fig. 7). These results reveal that the particle size of coordination compounds used as precursors has a significant effect on the size of final metal oxide particles. It is noticeable that lead(II) oxide particles prepared by calcination of nano-size complex have smaller size than that of prepared from bulk complex.

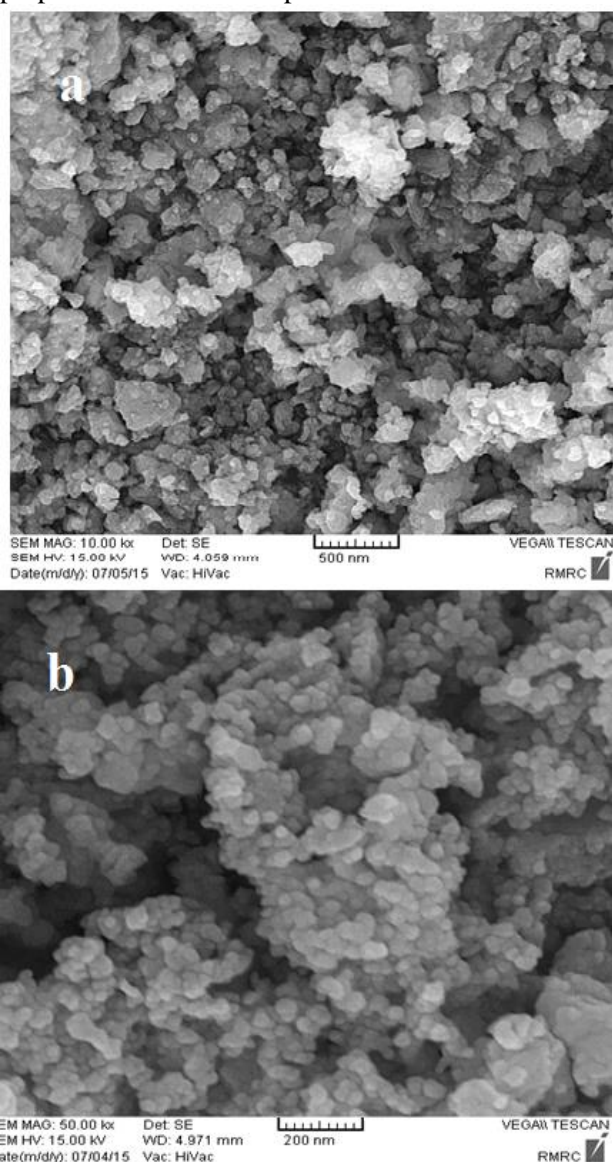


Fig. 7. SEM images of lead oxide obtained from the crystalline bulk complex (a) lead oxide obtained from nano-size complex (b).

2.5. Optical properties of nano-size lead(II) oxide particles

All physical and chemical properties of semiconductor materials such as lead(II) oxide depend sensitively on their size. Nano-materials with the spacious number of surface molecules show new and unique properties that cannot be observed in their bulk analogs. To verify that the decreasing particle size of materials leads to a blue-shift in their absorption edge, absorption spectra of nano-size lead oxides prepared from the calcination of the nano-size complex and crystalline bulk complex were recorded and the results are illustrated in Fig. 8. The values of 300 and 390 nm can be seen for the absorption onset of lead(II) oxide particles prepared from nano-size and crystalline bulk complex, respectively, in the Fig. 6.

The band gap computation of lead(II) oxides prepared from the nano-size and crystalline bulk complex showed the values of 4.13 and 3.17 eV, respectively, [32]. Those results show that the nano-size lead(II) oxide derived from the nano-size complex have a larger band gap relative to that of prepared from the crystalline bulk complex.

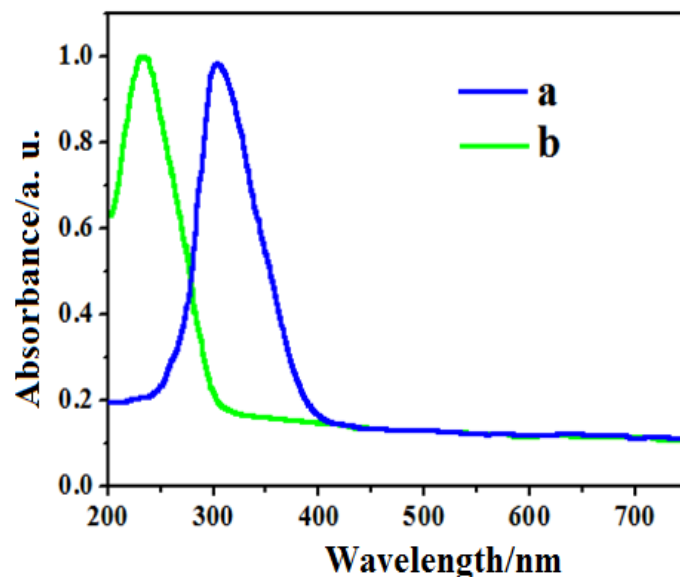


Fig.8. UV-Vis absorption spectra of lead oxide obtained from the crystalline bulk complex (a) lead oxide obtained from nano-size complex (b).

The fluorescence spectra of lead oxides particles are illustrated in Fig. 9. As can be seen, the maximum emission of lead oxide prepared from the nano-size complex, similar to the absorption spectra, shows a blue-shift relative to that of the lead oxide prepared from the crystalline bulk complex. The results of optical properties investigation of prepared lead(II) oxide particles show that the physical and chemical properties of materials can be controlled by changing their size.

3. Conclusion

A novel lead(II) complex and its nano-size particles were synthesized and fully characterized. A new and

simple method namely sonochemical method has been used for the preparation of nano-size complex.

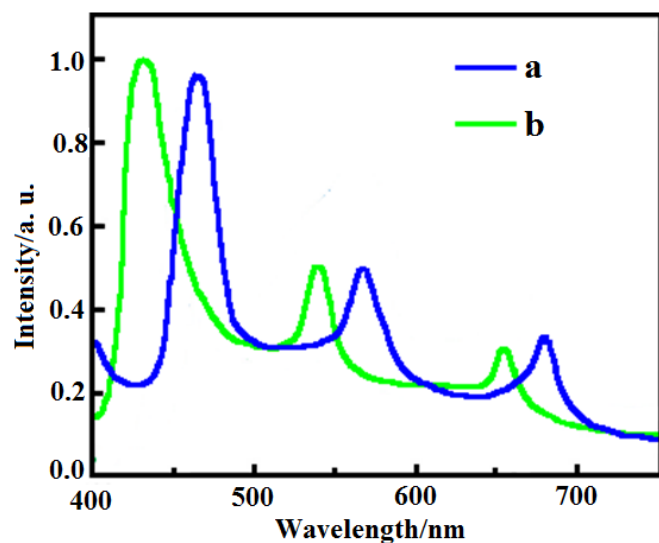


Fig. 9. Photoluminescence spectra of lead oxide obtained from the crystalline bulk complex (a) lead oxide obtained from nano-size complex (b).

The results showed that sonochemical method as a facile and cost effective route can be used for preparation of nano-size coordination polymers. Furthermore, calcination of prepared nano-size and crystalline complex led to formation of nano-size lead(II) oxide particles, which characterized by XRD and SEM. Since, lead oxide prepared from nano-size complex had a smaller size in comparison with lead oxide prepared from crystalline bulk complex, it can be concluded that the initial particle size of complex has an important role on the size of the final derived lead oxide particles. Finally, investigation of optical properties of prepared lead oxides showed that the altering of particle size of materials can be used to tune their photophysical properties in a broad range.

4. Experimental

4.1. Materials and physical techniques

Lead(II) nitrate and 1,3-diphenylpropane-1,3-dione (HL) were purchased from Sigma-Aldrich and used without further purification. All solvents before applying were dried according to standard procedures [33]. Elemental analyses of compounds have been evaluated by Thermo Finnigan Flash-1112EA microanalyzer. For recording of infrared spectra, a Bomem MB-series FT-IR instrument has been utilized. NMR spectra were inscribed on a Bruker AVANCE 300 operating at 300.3 MHz. The luminescence and absorption spectra of compounds were obtained on USB2000 and Shimadzu 2,100 spectrometers, respectively. A Philips XL-30 scanning electron microscope (SEM) with gold coating was used for recording of the morphology of particles.

Crystallographic measurements were carried out by utilizing a STOE IPDS-II diffractometer supplied with a graphite monochromated Mo-K α radiation at 100K. Accurate unit cell parameters and orientation matrices were achieved from least-squares refinements. The structure was solved by direct method and refined by full-matrix least-squares procedure on F^2 . The SHELXL-2014/6 crystallographic software package was used for all refinements.

4.2. Synthesis of complexes

4.2.1. Preparation of single crystals (1)

Pb(NO₃)₂ (0.5 mmol, 0.16 g) was added to a 1:2 methanol/DMF (15.0 mL) solution of 1,3-diphenylpropane-1,3-dione (0.22 g, 1 mmol) in a 20 mL glass tube. The solution was transferred to a glass vessel and heated hydrothermally from room temperature to 80 °C at 2 °C/min, kept at 80 °C for 72 h and then allowed to cool to room temperature during 18 h. After filtration, the yellow needle-like crystals were obtained (yield 73%, m.p. > 270 °C). Anal. Calc (%) for C₆₀H₄₄O₈Pb₂: C, 55.12; H, 3.39; N, 0.0. Found (%): C, 55.10; H, 3.38; N, 0.0. IR (KBr, cm⁻¹): 3035 (w), 2082 (w), 1585 (s), 1440 (s), 1148 (m), 742 (m). ¹H NMR (CDCl₃, ppm): 7.28 (Ph-H), 7.19 (Ph-H), 7.12 (Ph-H), 5.95 (s, =CH-). Eventually, the calcination of title complex at 600 °C for 4 h have been made in a furnace and in a static atmosphere in order to achieve nano-size lead oxide particles.

4.2.2. Preparation of nano-size complex (1) by sonochemical method

For preparation of nano-size complex, a solution of Pb(NO₃)₂ (0.5 mmol, 0.16 g) in 10 mL methanol was placed in a high-density ultrasonic probe. To this solution, a solution of 1,3-diphenylpropane-1,3-dione (0.22 g, 1 mmol) in 10 mL methanol was added dropwise. The acquired precipitates were subsequently washed with ether, filtered and dried in air (yield 84%, m.p. > 270 °C). Anal. Calc (%) for C₆₀H₄₄O₈Pb₂: C, 55.12; H, 3.39; N, 0.0. Found (%): C, 55.10; H, 3.38; N, 0.0. IR (KBr, cm⁻¹): 3035 (w), 2082 (w), 1585 (s), 1440 (s), 1148 (m), 742 (m). Finally, calcination of prepared nano-size complex in a furnace at 600 °C for 4 h led to formation of nano-size lead(II) oxide particles.

5. Supplementary material

CCDC 1539030 includes the crystallographic data for complex reported in the article.

Copies of the data can be achieved 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.

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