

Sonochemical Synthesis of Novel Nano Flower Lead(II) Metal-organic Coordination Polymer: A New Precursor to Produce Nano-sized PbO

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The new nano flower Pb(II) 1D metal-organic chain $\{[\text{Pb}(\text{phen})(\mu\text{-CH}_3\text{COO})]_n(\text{BF}_4)\}_n(1)$ (phen = 1,10-phenanthroline) was synthesized by a sonochemical irradiation assisted method. The compound was characterized by scanning electron microscopy (SEM), elemental analysis, FT-IR spectroscopy, X-ray powder diffraction (XRPD) and single crystal X-ray analysis. The X-ray analysis reveals a 1D chain metal-organic coordination polymer structure for 1 that is further extended into a 3D supramolecular structure by π - π and other labile interactions. The coordination number of the lead(II) ions is determined as about eight, $\text{PbF}_2\text{N}_2\text{O}_4$. The lead oxide nanoparticles were prepared at 180 °C by the thermolysis of 1. The average diameter of the nanoparticles to be 27 nm. The size and morphology of the prepared PbO samples were further observed using SEM.

Keywords: Nanometal-organic coordination polymer, Ultrasonic irradiation, 1D chain, Nano PbO

INTRODUCTION

Metal-organic coordination compounds are one of the most important classes of organic-inorganic hybrid materials which have been advanced in recent years. These materials have been studied in the past two decades. Metal-organic coordination polymers are a class of organic-inorganic materials consists of metal ions linked together through multi-dentate organic ligands, to form a polymeric chain. These materials have received a great deal of attention in a wide range of different areas such as catalysis, sensing, luminescence, separation and storage used [1].

In total ligand molecules structure and dimensions, type of one-dimensional, two-dimensional or three-dimensional framework determines the final. As a result, the final choice of ligands suitable for the intended application framework is very important. So Crystal engineering of metal-organic coordination polymer to build new features and new

applications, has attracted much attention [2-17].

Among the various methods of synthesis of these materials, methods and simple sonochemical one of the common methods used. The benefits of this approach are cost-effective, easily controlled, to synthesize these materials at the nanoscale. Among linkers, organic aromatic ligands are considered among the most important ligand. Reducing the size of coordination polymer at the nanoscale using ultrasound done. The effect and power of ultrasonic waves on the shape and size of the pores of the particles affect [3,29,30].

We focus our attention on design and synthesis lead(II) coordination compounds, and recently we have reported some nano lead(II) coordination polymer [4,21]. In this paper we report the new asymmetrical lead(II) metal-organic coordination polymer in the presence of 1,10-phenanthroline ligand with tetrafluoroborate and acetate anions. The work presented here, describes a simple sonochemical synthetic method for preparation of nano-structures of this metal-organic coordination polymer and its use in the preparation of lead oxide nanoparticles.

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EXPERIMENTAL

Materials and Physical Measurements

The chemicals were obtained from Sigma-Aldrich and were used as-received without further purification. Elemental analyses of the samples were carried out by a Vario Microanalyzer. The IR spectra were recorded on a Bruker Vector 22 FT-IR spectrometer by using KBr disks in the 4000-400 cm^{-1} range. X-ray powder diffraction (XRD) measurements were done using an X'pert diffractometer (Panalytical) with monochromatized $\text{Cu-K}\alpha$ radiation. Using mercury simulated XRD powder patterns based on single crystal data were obtained [22]. The morphology of the nanostructured compound was determined via scanning electron microscopy (SEM) (S-4200, Hitachi, Japan) and transmission electron microscopy (JEM-2200FS, JEOL Ltd., Japan). In order to ultrasonic irradiation, a multiwave ultrasonic generator (Sonicator_3000, Misonix Inc., Farmingdale, NY, USA), equipped with a converter/transducer and titanium oscillator (horn), 12.5 mm in diameter, operating at 20 kHz was used with a maximum power output of 600 W at room temperature for 1 h.

Preparation of Single Crystal and Nanostructure of $\{[\text{Pb}(\text{phen})(\mu\text{-CH}_3\text{COO})] \cdot (\text{BF}_4)\}_n(1)$

By addition of 30 ml of a 0.1 M solution of the ligand "phen" and 30 ml of a 0.1 M solution of NaBF_4 into 30 ml of a 0.1 M solution of $\text{Pb}(\text{CH}_3\text{COO})_2$ in water under sonication using a high density ultrasonic probe, operating at 20 kHz and 600 W, nanostructures of $\{[\text{Pb}(\text{phen})(\mu\text{-CH}_3\text{COO})] \cdot (\text{BF}_4)\}_n(1)$ were prepared. The obtained precipitates were filtered off, washed with water, and then dried in air.

Product 1: d.p. = 356 °C. Analysis: Found; C 37.00, H 2.50, N 6.00%. Calculated for $\text{C}_{14}\text{H}_{12}\text{BF}_4\text{N}_2\text{O}_2\text{Pb}$: C 36.45, H 2.62, N 6.07%. FT-IR (selected bands, in cm^{-1}): 635m, 722s, 851s, 1054vs, 1103vs, 1150s, 1343s, 1419s, 1428s, 1542m, 1573s, 3065w.

For preparation and isolate single crystals of $\{[\text{Pb}(\text{phen})(\mu\text{-CH}_3\text{COO})] \cdot (\text{BF}_4)\}_n(1)$ suitable for an X-ray structure determination, 1 mmol of "phen" and NaBF_4 was placed in one arm of a branched tube, and 1 mmol of $\text{Pb}(\text{CH}_3\text{COO})_2$ (0.07 g) was placed in the other. To fill both

arms, methanol was then carefully added and the tube was sealed, and the ligand-containing arm was immersed in an oil bath at 60 °C. The other arm was left at ambient temperature. The yellow crystals (d.p. = 358 °C) had deposited in the arm at ambient temperature after 3 days. They were then filtered off, washed with acetone and ether, and air-dried (yield: 80%).

Found; C 36.45, H 2.62, N 6.07%. Calculated for $\text{C}_{14}\text{H}_{12}\text{BF}_4\text{N}_2\text{O}_2\text{Pb}$: C 37.00, H 2.50, N 6.00%. FT-IR (selected bands, in cm^{-1}): 639m, 721s, 851s, 1055vs, 1072vs, 1150s, 1343s, 1415s, 1428s, 1542m, 1573s, 3066w.

Synthesis of Lead(II) Oxide Nanoparticles

The light yellow solution was formed after dissolving of 0.1 mmol of $\{[\text{Pb}(\text{phen})(\mu\text{-CH}_3\text{COO})] \cdot (\text{BF}_4)\}_n(1)$ in 10 ml of oleic acid. This solution was degassed for 45 min and then heated to 180 °C for 3 h. Finally a black precipitate was formed at the end of reaction. Ethanol at large excess and toluene in small amount were added to the solution, and PbO nanoparticles were separated by centrifugation. The final solids were washed with ethanol and dried in ambient air (yield: 54%).

Crystallography

A crystal of the compound (yellow, Parallelepiped, size $0.65 \times 0.12 \times 0.07$ mm) was mounted on a glass fiber with epoxy adhesive. Data collection was performed using an X-ray diffractometer (SMART APEX II, Bruker) with graphite-monochromated $\text{Mo K}\alpha$ radiation ($\lambda = 0.71073$ Å), operating at 50 kV and 30 mA over 2θ ranges of 7.34 to 52.00°. No significant decay was observed during the data collection.

The processing of data was done by the Bruker AXS Crystal Structure Analysis Package [23]. The following modules of the AXS package were used. Data collection: APEX2 (Bruker, 2010); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT (Bruker, 2009); absorption correction: SADABS (Bruker, 2008); structure solution: XPREP (Bruker, 2008), and SHELXS-97 (Sheldrick, 2008); structure refinement: SHELXL-97 (Sheldrick, 2008); molecular graphics and publication materials: SHELXTL (Sheldrick, 2008). The scattering factors of neutral atoms were taken from Waber and Cromer [24]. At a low temperature the sample underwent phase transition. Thus; the

Table 1. Crystal Data and Structure Refinement for $\{[\text{Pb}(\text{phen})(\mu\text{-CH}_3\text{COO})] \cdot (\text{BF}_4)\}_n(1)$

Empirical formula	$\text{C}_{14}\text{H}_{12}\text{BF}_4\text{N}_2\text{O}_2\text{Pb}$
Formula weight	534.27
Temperature (K)	293(2)
Crystal system	triclinic
Space group	$P\bar{1}$
a (Å)	7.082
b (Å)	10.589
c (Å)	10.876
α (°)	72.08
β (°)	87.92
γ (°)	85.83
Volume (Å ³)	773.9
Z	2
ρ_{calc} (mm ³)	5.080
μ (mm ⁻¹)	16.598
F(000)	1134.0
Crystal size (mm ³)	$0.65 \times 0.12 \times 0.07$
2 θ range for data collection	8.52-59.02°
Index ranges	$-8 \leq h \leq 9, -14 \leq k \leq 14, -13 \leq l \leq 14$
Reflections collected	6196
Independent reflections	3753[R(int) = 0.0574]
Data/restraints/parameters	3753/0/217
Goodness-of-fit on F ²	1.105
Final R indexes [$I \geq 2\sigma(I)$]	$R_1 = 0.0512, wR_2 = 0.1420$
Final R indexes [all data]	$R_1 = 0.0571, wR_2 = 0.1539$
Largest diff. peak/hole (e Å ⁻³)	4.79/-2.59

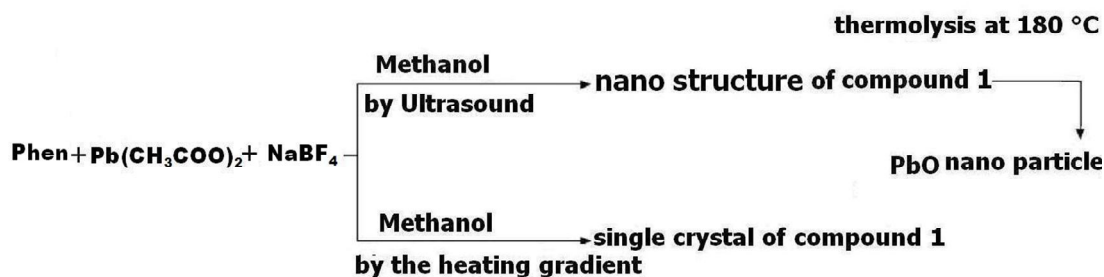
data was collected at room temperature. The crystal is monoclinic space group $P\bar{1}$, based on the systematic absences, E statistics, and successful refinement of the

structure. The structure was then solved by direct methods. Full-matrix least-square refinements minimizing the function $\sum w(F_o^2 - F_c^2)^2$ were applied to the data acquired

Table 2. Selected Bond Lengths (Å) and Angles (°) for {[Pb(phen)(μ-CH₃COO)]·(BF₄)_n} (1)

Pb1-N9	2.549(8)	Pb1-N8	2.480(8)
Pb1-O100	2.506(6)	Pb1-O100 ¹	2.733(7)
Pb1-O200 ²	2.494(6)	Pb1-O200	2.737(7)
O100-Pb1 ¹	2.733(7)	O200-Pb1 ¹	2.737(7)
O200-Pb1 ³	2.494(6)	Pb1-F1	2.930(4)
Pb1-F2	2.895(4)	N9-Pb1-O100 ¹	141.0(3)
N9-Pb1-O200 ¹	143.2(2)	N8-Pb1-N9	66.1(3)
N8-Pb1-O100 ¹	83.9(3)	N8-Pb1-O100	79.4(3)
N8-Pb1-O200 ²	80.1(3)	N8-Pb1-O200 ¹	83.7(3)
O100-Pb1-N9	82.2(3)	O100-Pb1-O100 ¹	67.8(2)
O100-Pb1-O200 ¹	113.5(2)	O100 ¹ -Pb1-O200 ¹	46.64(18)
O200 ² -Pb1-N9	86.1(2)	O200 ² -Pb1-O100 ¹	113.7(2)
O200 ² -Pb1-O100	159.1(3)	O200 ² -Pb1-O200 ¹	67.8(2)
F1-Pb1-F2	43.01(3)	F1-Pb1-O100	106.37(2)
F1-Pb1-O200	125.28(3)	F2-Pb1-O200	106.03
F2-Pb1-O100	125.17(2)		

¹1-X, 1-Y, 1-Z; ²1+X, +Y, +Z; ³-1+X, +Y, +Z.



Scheme 1. Materials produced and synthetic methods

form the compound. The refining of non-hydrogen atoms done anisotropically. All H atoms were placed in geometrically calculated positions, with C-H = 0.95 (aromatic), and 0.98 (CH₃) Å, and refined as riding atoms, with Uiso (H) = 1.5 UeqC (CH₃) or 1.2 UeqC (other C). Crystallographic data, bond lengths, and angles are given in Tables 1 and 2.

RESULTS AND DISCUSSION

The reaction between the ligand with Pb(CH₃COO)₂ and NaBF₄ led to the formation of the new lead(II) metal-organic polymer {[Pb(phen)(μ-CH₃COO)]·(BF₄)_n} (1). Nanostructures of compound 1 were obtained by ultrasonication in a methanolic solution, and the single

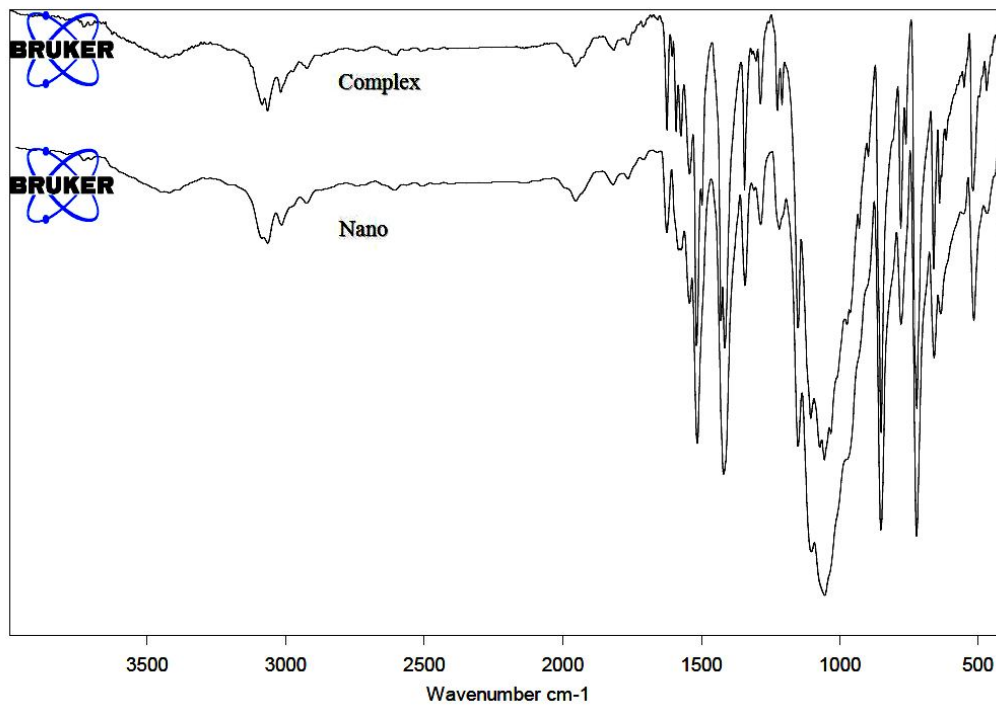


Fig. 1. FT-IR spectra of crystal and nanostructure of $\{[\text{Pb}(\text{phen})(\mu\text{-CH}_3\text{COO})] \cdot (\text{BF}_4)\}_n$ (1) produced by branched-tube method and sonochemical method.

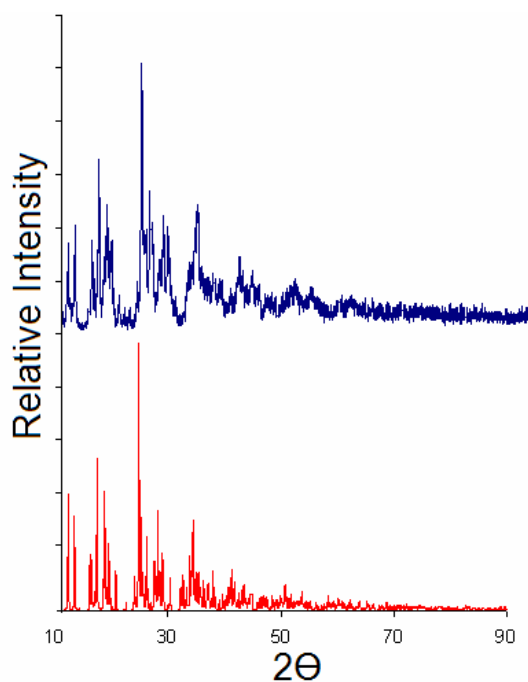


Fig. 2. XRPD patterns (a) computed from single crystal X-ray data of compound 1, (b) nano-structure of compound 1.

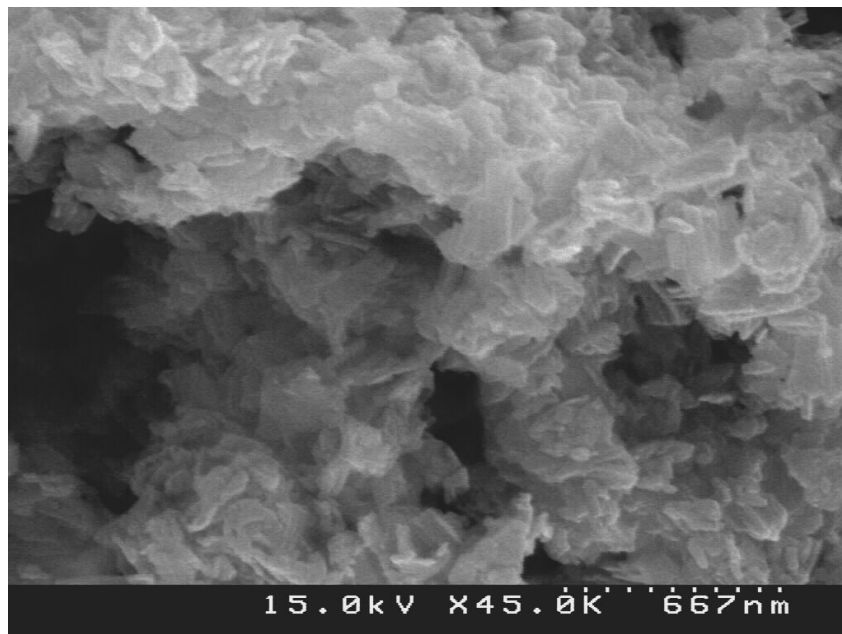


Fig. 3. SEM photographs of $\{[\text{Pb}(\text{phen})(\mu\text{-CH}_3\text{COO})]\cdot(\text{BF}_4)\}_n$ (1) nano structures.

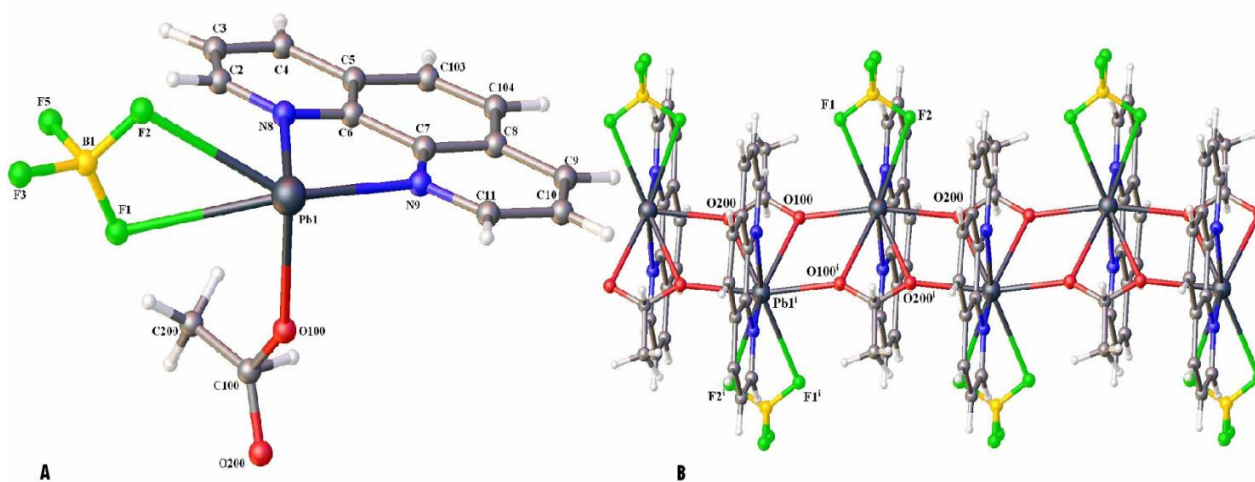


Fig. 4. A) Molecular asymmetrical unit of $\{[\text{Pb}(\text{phen})(\mu\text{-CH}_3\text{COO})]\cdot(\text{BF}_4)\}_n$ (1) [the coordination sphere of Pb1 center is not completed]. B) Fragment of the coordination polymer showing the 1D metal-organic polymer.

crystalline sample was obtained using a heat gradient applied to a solution of the reagents (the branched tube method). Scheme 1 gives an overview of the methods used for the synthesis of $[\text{Pb}(p\text{-2-einh})\text{NO}_2]_n$ (1) using the two different routes.

The IR spectrum and elemental analysis of the single crystalline material and the nanostructure are identical (Fig. 1). The FT-IR spectra of the single crystalline materials and the nanostructures show the characteristic absorption bands of the “phen” ligand. The relatively weak band at around

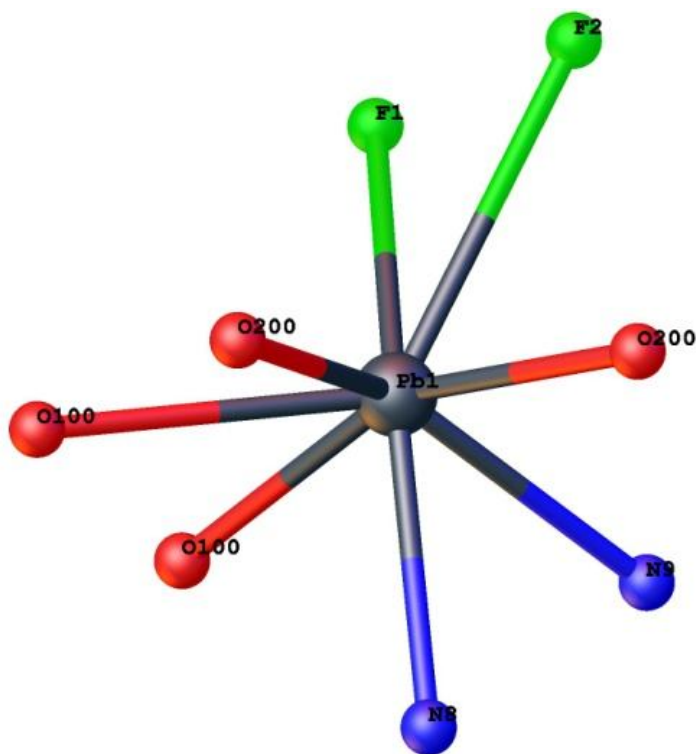


Fig. 5. Schematic representation of lead(II) environment.

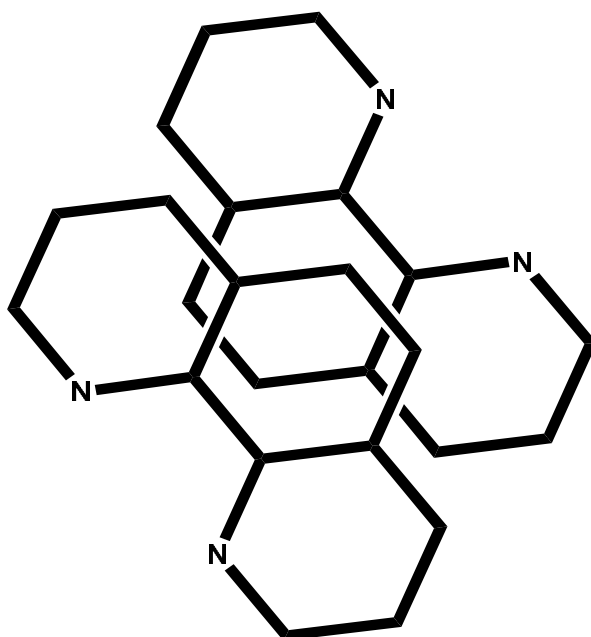


Fig. 6. Projection of the nearest neighbor pairs π - π stacks of heteroaromatic bases in (1).

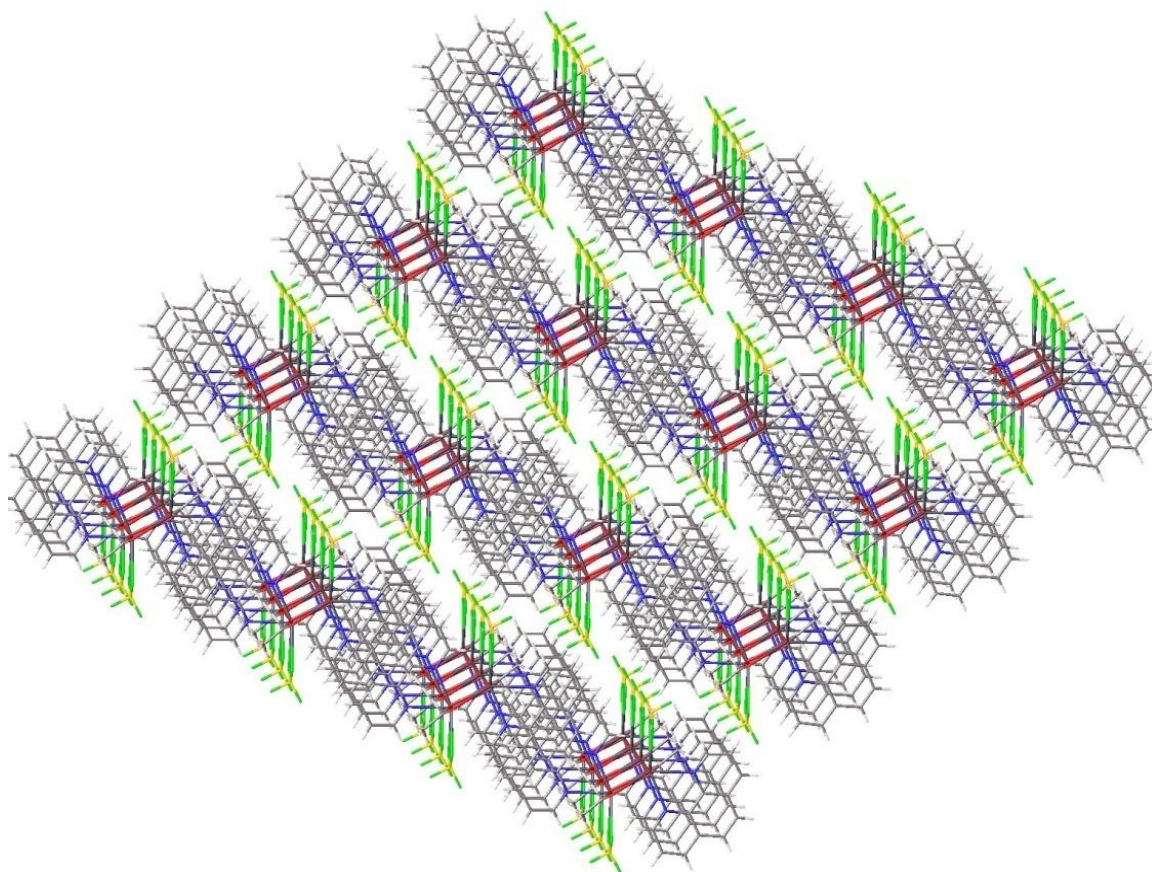


Fig. 7. From discrete architecture to supramolecular 3D zigzag metal-organic polymer.

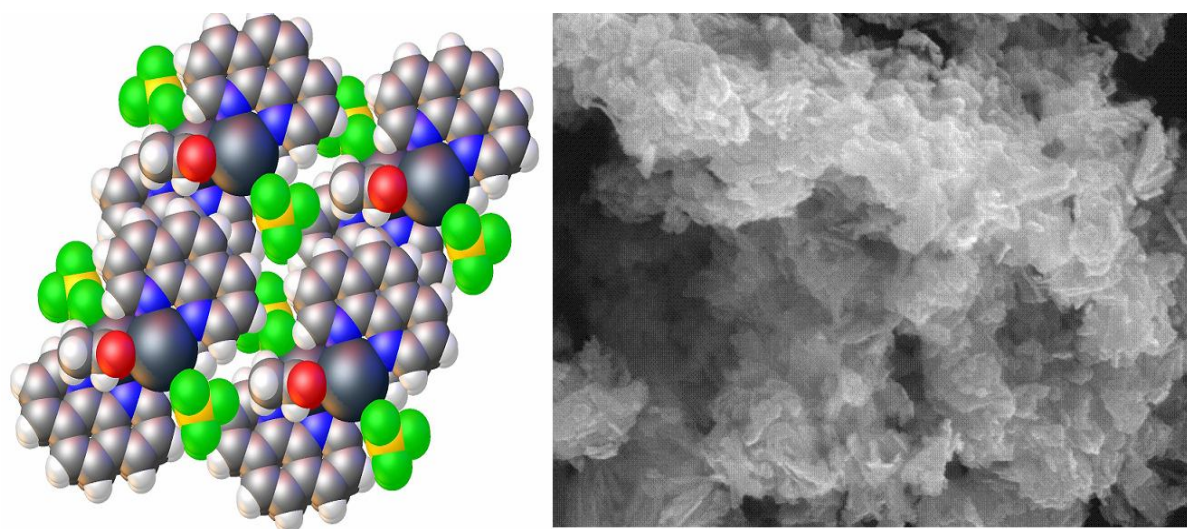


Fig. 8. Left: packing of the structure on a molecular level, Right: morphology of the nanostructure.

Table 3. Selected Non-covalent Contacts in the Crystal Structure of 1

C-H...F	H...F (Å)	C...F (Å)	<F...H-C (°)
C4-H4... F3	2.531	3.460	160.27
C10-H10... F3	2.613	3.219	126.11
C20-H20... F5	2.701	3.067	103.30

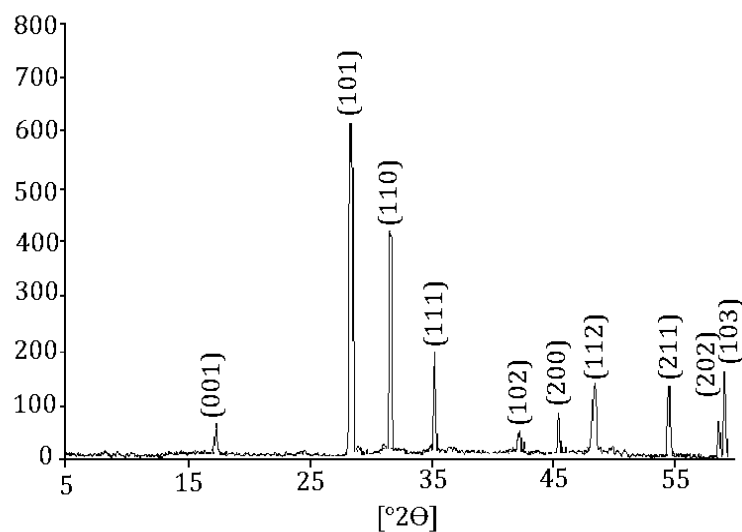


Fig. 9. XRD pattern PbO prepared after thermolyses of compound $\{[\text{Pb}(\text{phen})(\mu\text{-CH}_3\text{COO})] \cdot (\text{BF}_4)_n\}_n$ (1).

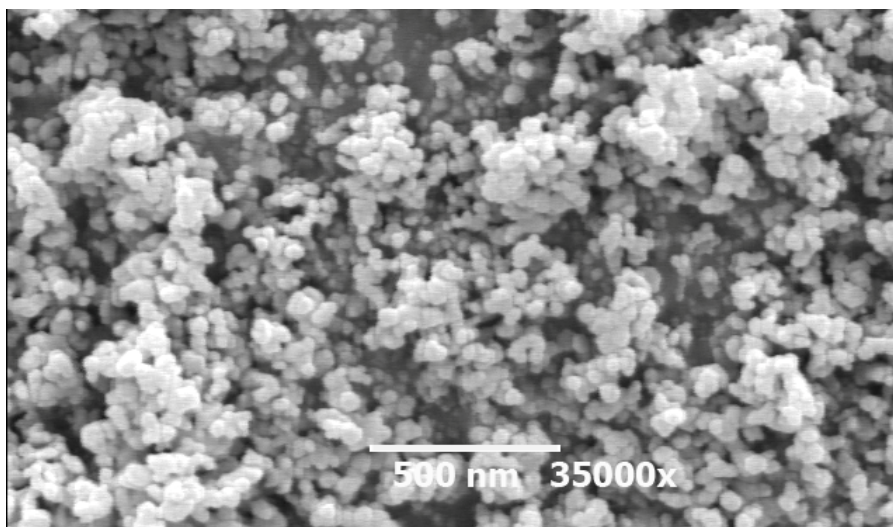


Fig. 10. SEM photograph of PbO nano-structures (produced by thermolyses of nano structures).

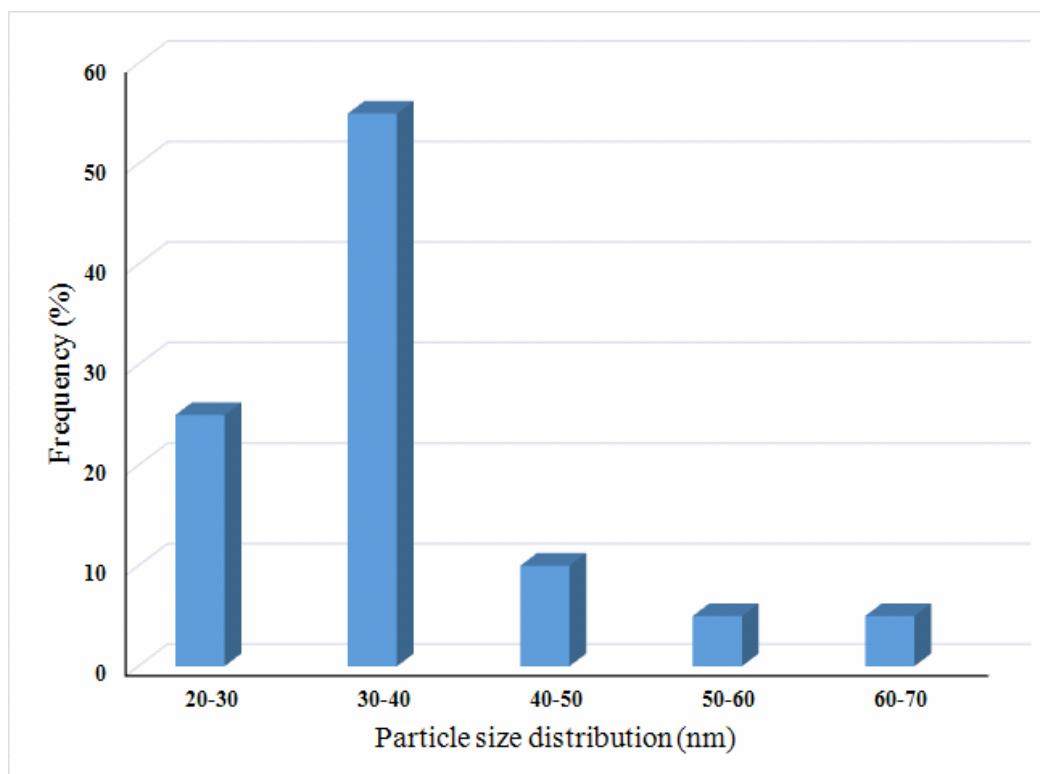


Fig. 11. Size distribution of PbO nanoparticles.

3066 cm^{-1} is attributed to the absorption of the aromatic CH hydrogen atoms. The vibrations aromatic rings of “phen” ligand were seen in the frequency range of $1400\text{-}1600\text{ cm}^{-1}$. The band at 1150 cm^{-1} is due to the BF_4^- stretching vibration.

The XRD pattern of compound 1 simulated from single crystal X-ray data is shown at Fig. 2a. Figure 2b shows the experimental XRD pattern of compound 1, prepared by using the sonochemical process. Between the experimental and simulated powder X-ray diffraction patterns, slight differences in 2θ , were observed (Fig. 2b). This shows that the compound obtained by the sonochemical route is to a large extent identical to the single crystalline sample. By the Scherrer formula ($D = 0.891\lambda/\beta\cos\theta$, where D is the average grain size, λ is the X-ray wavelength (0.15405 nm), and θ and β are the diffraction angle and full-width at half-maximum of an observed peak) the estimated value of average grain size is 23 nm .

Figure 3 shows the nanostructures observed by scanning electron microscopy. Interestingly, the morphology of

compound 1 prepared by the sonochemical method (Fig. 3) is nano flower with thickness of $23\text{-}25\text{ nm}$. Further investigation required for explanation of the mechanism of formation of these structures. However, the supramolecular structure of the compound may influence. The morphology of nanostructured compound can be affected by the packing of the structure on a molecular level [25-27] (Fig. 9). Based on single crystal X-ray crystallography (Table 1), the structure of $\{[\text{Pb}(\text{phen})(\mu\text{-CH}_3\text{COO})] \cdot (\text{BF}_4)\}_n$ crystallizes in the triclinic system with space group $P\bar{1}$, taking the form of a one dimensional metal-organic polymer in the solid state (Fig. 4b). Figure 4a indicates a view of the asymmetrical unit structure of 1, together with a selected atom numbering scheme.

Figure 5 shows that, the lead(II) atom is coordinated by three nitrogen atoms of two “phen” ligands with Pb-N distances of $2.549(8)$, $2.480(8)\text{ \AA}$, and four oxygen atoms of tri acetate anion with Pb-O distance of $2.494(6)$, $2.737(7)$, $2.733(7)$ and $2.560(7)\text{ \AA}$ and two Fluorine atoms of one tetrafluoroborate anion with Pb-F distances of $2.930(8)$ and

2.895 (8) Å in a three-fashion with a PbF₂N₂O₄ donor set. Therefore, the coordination number of the lead(II) atom is eight with symmetrical geometry, therefor the coordination sphere of lead(II) centers are *holodirected* geometry.

The F...H-C interactions are usually classified as weak non-covalent bonding between adjacent atoms. However, the observed distance values for these interactions (Table 3) propose relatively strong interaction between these atoms. (Table 4)

As shown in Fig. 6, In the structure π - π stacking interactions observed between the parallel aromatic rings belonging to adjacent chains. The interplanar distance of the aromatic rings are 3.544 Å, that are noticeably shorter than the normal π - π stacking [28]. As a consequence, the labile interactions also allow the the 1D zigzag molecular architecture to interact with neighboring chains and to extend the structure to 3D supramolecular architecture. Thereby, the coordination sphere of lead(II) ions in this complex may be controlled by the labile and π - π stacking interactions.

Nanoparticles of PbO

By the decomposition of precursor 1 in oleic acid as a surfactant at 180 °C, under an air atmosphere PbO nanoparticles were prepared. The XRD pattern of PbO nanoparticles is seen in Fig. 9. All the peaks in Fig. 14 can be attributed to a tetragonal structure with a lattice parameter of $a = 3.947$ and $c = 4.988$ Å, consistent with the standard data file (JCPDS, No: 85-0711).

The size and morphology of the as-obtained PbO samples were further evaluated using scanning electron microscopy (SEM). This process resulted in the regular shape of lead(II) oxide nanoparticles (Fig. 10). The size distribution of PbO particles was found to be in the range of 18-70 nm, respectively.

CONCLUSIONS

In this study, a novel nano flower lead(II) 1D metal-organic coordination polymer containing 1,10-phenanthroline ligand, tetrafluoroborate and acetate anions via sonochemical route is described for first time. The morphology of 1 prepared sonochemically is composed of nano flower with thickness of 17-70 nm. The compound

takes the form of a one dimensional metal-organic coordination polymer in the solid state and through several labile interactions interacting with neighbouring chains, the 1D structure extends to a 3D supramolecular coordination polymer. Uniform PbO nanoparticles were obtained by thermolysis of compound 1.

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