ISSN : 0974 - 746X Volume 9 Issue 2

Inorganic CHEMISTRY

An Indian Journal

Full Paper

ICAIJ, 9(2), 2014 [37-39]

Synthesis and characterization of nano lead (II) oxide sulfate by coordination polymer as precursor

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ABSTRACT

A lead (II) coordination polymer $[Pb(bpy)(SCN)_2]_n$ (bpy = 2,2'-bipyridine) has been synthesized and characterized by elemental analysis and IR spectroscopy. The nano lead (II) oxide sulfate $Pb_2(SO_4)O$ was synthesized by calcination of Pb (II) coordination polymer at 450 °C which was characterized by x-ray powder diffraction (XRD) and scanning electron microscopy (SEM).

KEYWORDS

Nano;
Lead (II);
Coordination polymer;
Calcination;
$Pb_2(SO_4)O$.

INTRODUCTION

Metal coordination polymers have been studied widely as they represent an important interface between synthetic chemistry and materials science with specific structures, properties, reactivities that are not found in mononuclear compounds[1-8]. Controlling the growth of materials at the submicrometer scale is of central importance in the emerging field of nanotechnology. Several methods can be applied to synthesize PbO nano structures with well-defined shapes,but to the best our knowledge less use of lead (II) coordination polymers to produce $Pb_2(SO_4)O$ nanoparticles has been reported. This article focuses on the synthesis of lead (II) coordination polymer $[Pb(bpy)(SCN)_2]_n$ as well as its use in preparing $Pb_2(SO_4)O$ nano structures.

EXPERIMENTAL

General

All reagents and solvents for the synthesis and analysis were commercially available and were used as received. IR spectra were recorded using PERKIN-Elmer 597 and Nicolet 510P spectophotometers. Microanalyses were carrid out using a Heraeus CHN-O-Rapid analyzer. The nano particles of lead (II) oxide sulfatewere characterized with a scanning electron microscope with gold coating.

Preparation of $[Pb(bpy)(NCS)_2]_n$

Compound 1 was prepared using reported method[9]. A solution of 2,2'-bipyridine (0.088 g, 0.5 mmol) in hot ethanol (10 ml) and hot aqueous solution of lead (II) nitrate (0.16 g, 0.5 mmol) and KSCN (0.049 g, 0.5 mmol) were mixed. The resulting colorless solution was heated, stirred and left to evaporate at room temperature. After a few days, colorless crystal was isolated which was washed with acetone and air dried.

(a) Compound 1

Yield: 0.239 g, 50% m.p. 280ºC (Found: C, 30.63; H, 1.80; N, 11.50. calcd. For $C_{12}H_8N_4PbS_2$ C, 30.03; H, 1.67; N, 11.69). IR (cm^{-1} Selected bands): 740(s), 1010(s), 1110(vs), 1530(s), 1610(s), 2040(vs), 3050(w).

Synthesis of $Pb_2(O_4)O$ nanoparticles

Nanoparticles of $Pb_2(SO_4)O$ have been genetated by thermal decomposition of single crystals of compound 1 at 500 ºC for 3h respectively. The morphology and size of the as-prepared nanoparticles $Pb_2(SO_4)O$ sample were further investigated using Scanning Electron Microscopy (SEM) and X-ray powder diffraction (XRD).

RESULTS AND DISCUSSION

Reaction between a felexible organic nitrogen donor ligand, 2,2'-bipyridine,and mixtures of lead (II) nitrate and potassium thiocyanate yielded crystalline material formulated $[Pb(bpy)(NCS)_2]_n$. The IR spectera display characteristic absorbtion bands for the ligand and the thiocyanate anion. The relatively weak absorbtion bands at 3078 cm^{-1} are due to the C–H modes involving the aromatic ring hydrogen atoms. The C–H modes involving the aliphatic hydrogen atoms of ligand 2,2'-bipyridine in compound $[Pb(bpy)(NCS)_2]_n$ reveal around 3015-3020 $cm^{–1}$. The absorption bands with variable intensity in the frequency range 1400-1580 $cm^{–1}$ correspond to ring vibrations of the py moiety of the ligand. The characteristic bands of the thiocyanate anion appear at *ca* 2040 cm^{-1}. The structure of compound $[Pb(bpy)(NCS)_2]_n$ (1) was previously analyzed and reported. The single-crystal X-ray structural analysis shows that the structure of compound is a unique one-dimensional supramolecular framework in which the lead atoms are eight-coordinated, $Pb_4N_4S_4$. Two of the the coordinated sites are occupied by the nitrogen atoms of bidentate bipyridine, the four nitrogen atoms are clustered about one pole of the symmetry axis with the with the sulfur atoms about its opposite and rather long (Pb-S=3.25-3.46). Compound 1 crystallizes in the monoclinic crystal system with space group C2/c. Nanoparticles of$Pb_2(SO_4)O$ were generated by thermal decomposition of compound 1. The final product upon calcination of compound 1 at 400 ºC is $Pb_2(SO_4)O$ based on XRD pattern and SEM images.

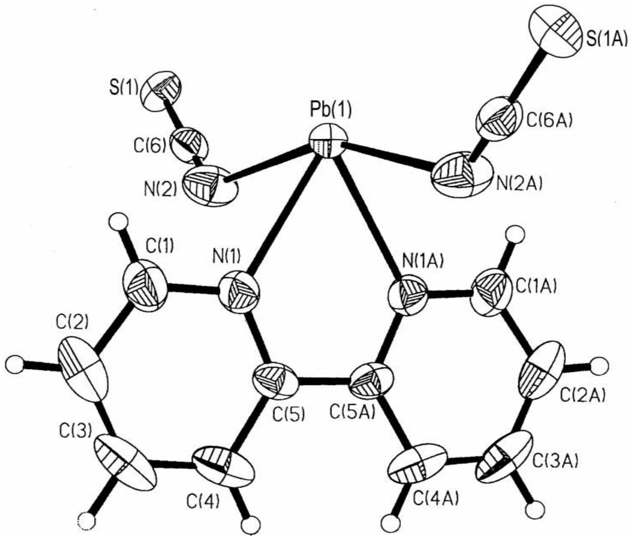

Figure 1 : ORTEP diagram of the $[Pb(bpy)(NCS)_2]_n$ compound

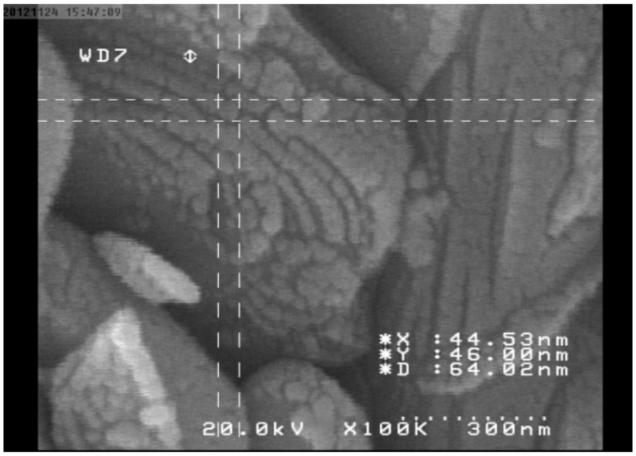

Figure 2 : SEM of nano-particle of $Pb_2(SO_4)O$ produced by calcinations of $[Pb(bpy)(NCS)_2]_n$

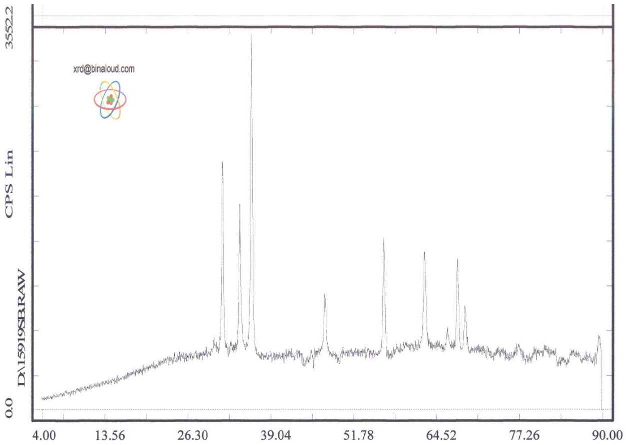

Figure 3 : XRD patern of $Pb_2(SO_4)O$ produced by calcinations of $[Pb(bpy)(NCS)_2]_n$

CONCLUSION

One lead (II) coordination polymer wassynthesized and characterized by elemental analysis and IR spectroscopy. Nanoparticles of $Pb_2(SO_4)O$ was synthesized by calcinations of compound 1. The nanomaterial was characterized by SEM, XRD and IR spectroscopy. This study demonstrates the coordination polymers may be suitable precursors for the preparation of nanoscale materials.

ACKNOWLEDGEMENTS

This work was supported by the Islamic Azad University of Firoozabad.

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