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Synthesis and Characterization of Lead Oxide Nanoparticles

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Abstract

Lead oxide nanoparticles were synthesized by sol-gel method using lead acetate and polyvinyl alcohol (PVA) as a precursor. PbO nanopowder were characterised by UV, XRD and FTIR. The thin film of PbO nanopowder by adding PVA were prepared by solution casting method and conductivity of thin film is measured. X-ray diffraction pattern shows the crystalline nature of PbO with grain size 63.00 nm. From UV-spectroscopy the band gap energy is found to be 5.52eV. FTIR spectra confirms the presence of PbO nanoparticles and the dc electrical conductivity is found to be 1.19 x 10^{-5}eV
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Keywords : lead oxide, sol-gel synthesis, XRD, UV-spectroscopy, FTIR, dc electrical conductivity.

1. Introduction :

Nanotechnology involves the study, control and manipulation of materials at the nanoscales, typically having dimensions less than 100nm (K. Mallikarjuna et al., 2012; Natarajan Kannan and Selvaraj Subbalaxmi 2011). In recent years, the synthesis of nanomaterials is an important research in the various scientific and industrial fields (Keating and Natan, 2003; Chen et al., 2005). The properties of such materials are novel and can be engineered by controlling the dimensions of these building blocks and their assembly via physical, chemical or biological methods. (Manoj Singh *et al.,* 2011). Nanostructured materials have unique chemical and physical properties but by their physical application in many fields has stimulated the search for new synthetic method for material.

Lead element has a lot of oxide forms including PbO, Pb_2O_3,PbO_2. Lead oxide (PbO), is an important industrial material due to its unique electronic, mechanical and optical properties and its potential applications in nano devices and functionalized materials (Xi et al., 2004). Due to their unique properties, lead oxides have wide applications such as network-modifiers in luminescent glassy materials, pigments, gas sensors, paints, storage batteries like lead acid, valve regulated lead acid batteries and lithium secondary batteries (karami et al., 2008; Lafronta et al., 2010, Martosetal, 2001) and nanoscale electronic devices. Because of the simplicity of design, low cost of manufacturing, reliability and relative safety there is improve and develop lead oxide characteristics. A variety of physicochemical methods, including thermal decomposition, spray pyrolysis, selected control synthesis, hydrothermal synthesis, sonochemical microwave irradiation synthesis by coordination polymers and pulsed current electrochemical methods have been used to produce nanometer-sized lead oxides. However, sol–gel method which provides a low cost, simple, non-hazardous method for preparing of different nano oxides has not been investigated for nano-sized lead oxides. Sol–gel method shows considerable advantages relatively to the customary methods because it allows controlling the size and morphology of the crystallized particles and lead to production powders with percent of more crystallization phase and high density

In this work, lead oxide nanoparticles were synthesized through the reaction of lead acetate $Pb(CH_3COO)_2$ and polyvinyl alcohol (PVA) by sol-gel methodology. The prepared lead oxide nanopowder were characterized by XRD, UV spectroscopy and FTIR. Thin film of PbO nanoparticles were prepared by adding PVA in proper proportion and the dc electrical conductivity of prepared thin films is recorded.

2. Experimental

2.1 Synthesis of lead oxide nanopowder:

In recent work we use lead acetate $Pb(CH_3COO)_2$ and polyvinyl alcohol (PVA) in 200 ml mixture of lead acetate (4wt %), PVA (16wt %) and water/ethanol (40/60 V/V) was heated at 80 ºC to produce a transparent solution which called "sol". The obtained sol was heated to evaporate major of its solvent and obtain a more viscous gel. The obtained gel was calcinated at 300-500 ºC for 3 hrs. At calcinations process, orange-red type layer nano-structured lead oxide was formed. The prepared lead oxide nanopowder is used to study for XRD, UV spectroscopy, FTIR and the thin film of PbO for dc electrical conductivity.

2.2 Thin Film Sample Preparation :

Thin film of PbO were prepared by dissolving PVA in 10ml distilled water under controlled temp. of 60^0C and continuous stirred for 30 min. Then prepared lead oxide powder form is added into PVA solution at room temperature and continuously stirred for 1 hr. Then the blend solution was divided into petri-dishes. The free standing casting films was formed and cut into several pieces. Prepared thin film of PbO is used to study for dc electrical conductivity.

2.3 Thickness Measurement :

To measure the thickness of the sample we used direct method. In this, the thickness of the sample was measured by the compound-microscope in conjunction with coulometer having a least count of 15.33μm. The thickness of the sample was found out to be 91.98μm.

2.4 X- ray Diffraction :

The X-ray of prepared PbO nanopowder was recorded using Hitachi diffractometer . Applying Scherrer's formula, the crystalline size of PbO sample is calculated.

2.5 UV Spectroscopy :

The UV absorption spectroscopy of the PbO nanopowder was recorded on Perkin Elmer spectrophotometer.

2.6 FTIR Spectroscopy :

Functional groups were analyzed by SHIMADZU – 8400S FT-IR spectrometer. The FTIR spectra were recorded for the range of 400 cm^{-1} to 4000 cm^{-1}.

2.7 Conductivity Measurement :

For measuring conductivity thin film of PbO nanoparticles were prepared in PVA using 1:3 ratio. The films are kept in sample holder. The sample holder assembly was placed in a controlled temperature furnace. The dc electrical conductivity was measured by determining the resistance of a sample within 328-417^{0}K at the rate of 1^0/min. The temperature was recorded by a digital thermometer

3 Result and Discussion :

3.1 X- ray Diffraction :

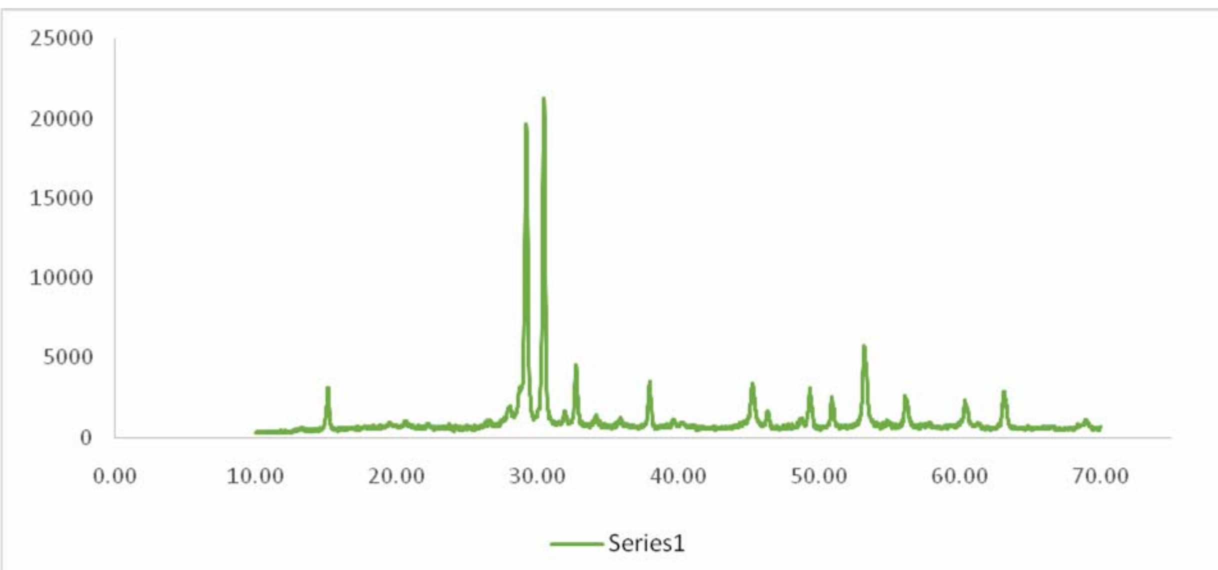

Fig. 1 X- ray diffraction pattern of PbO nanoparticles

Fig. 1 shows X- ray diffraction pattern of PbO nanoparticles. The prominent peaks present in the graph shows the crystalline nature of PbO. The grain size calculated from Debye Scherrer's formula was found to be 63.00 nm. The sharper XRD peaks indicates high crystalline nature of PbO.

3.2 UV absorption Spectroscopy :

Fig. 2 shows UV- absorption spectra of the PbO sample. The band gap energy is calculated by plotting a graph between αhμ and hμ. The band gap energy of the sample is found to be 5.52eV.

3.3. FTIR Spectroscopy :

FTIR spectra for the Lead Oxide Nanoparticles are shown in the Fig.3. The absorption peak at 466.74 cm^{-1} indicates the presence of Pb-O Stretching and also the peak at 557.39 cm^{-1} indicates the presence of oxides. These two peaks are very sharp. It is confirmed that the final product is the presence of lead and oxide.

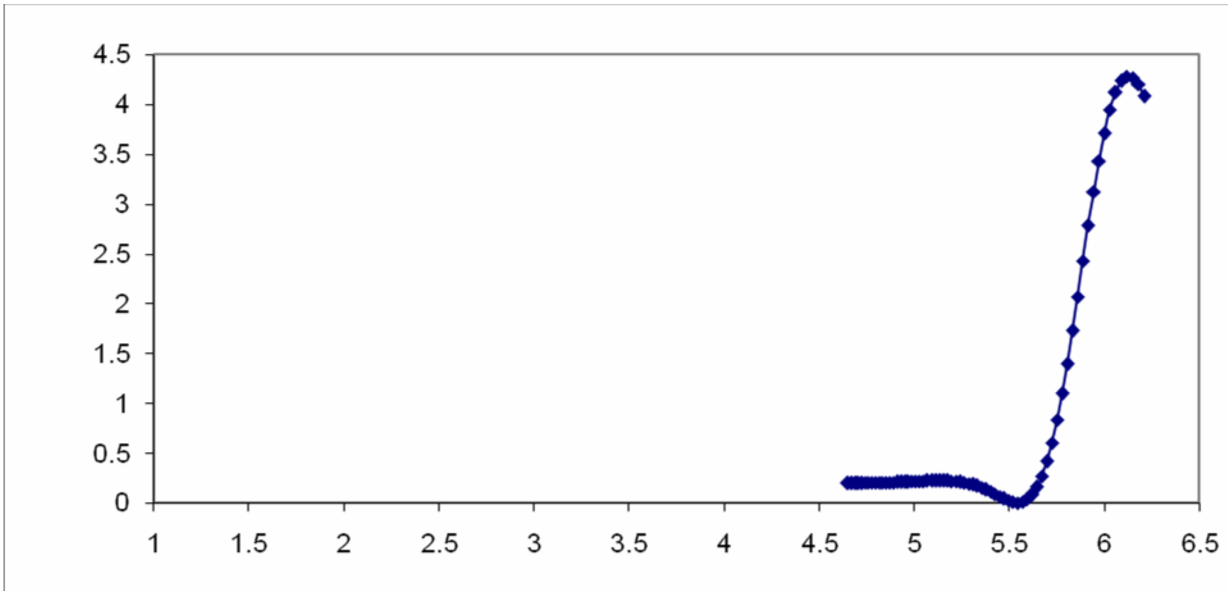

Fig. 2 UV- Absorption Spectroscopy

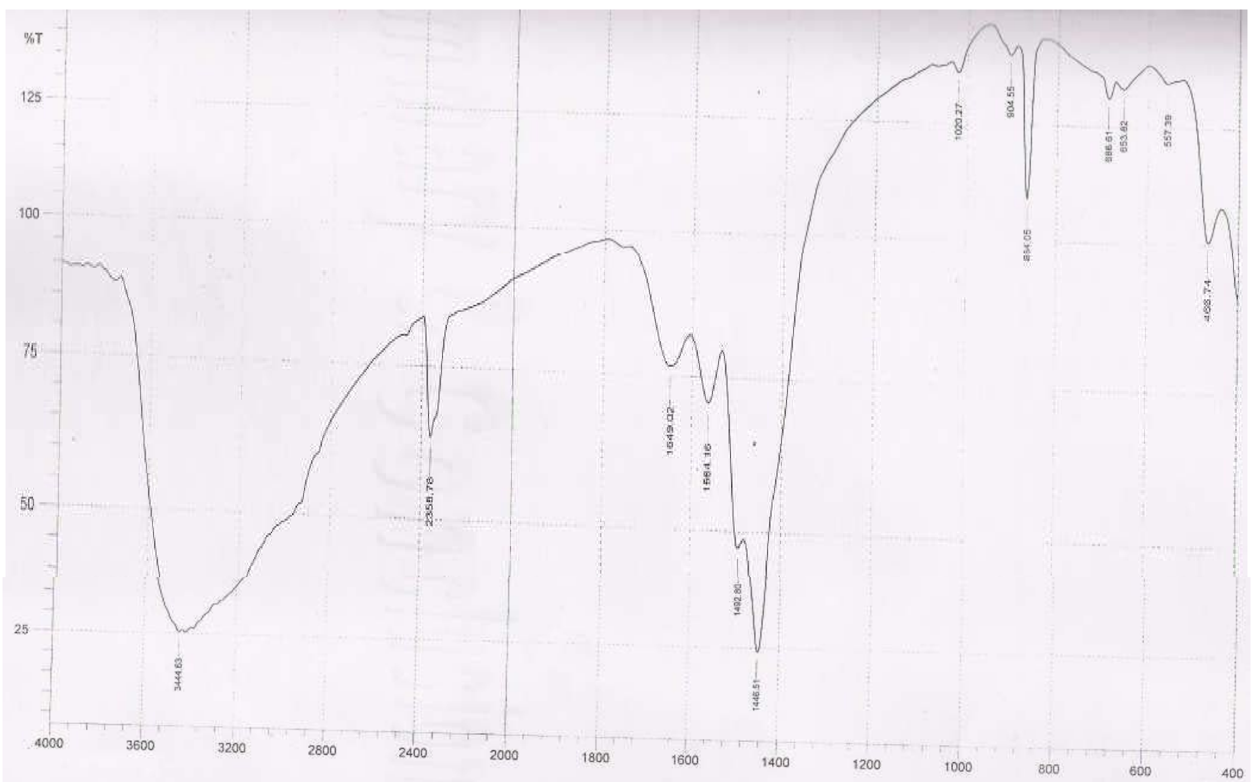

Fig. 3 FTIR of PbO

3.4 DC Electrical Conductivity :

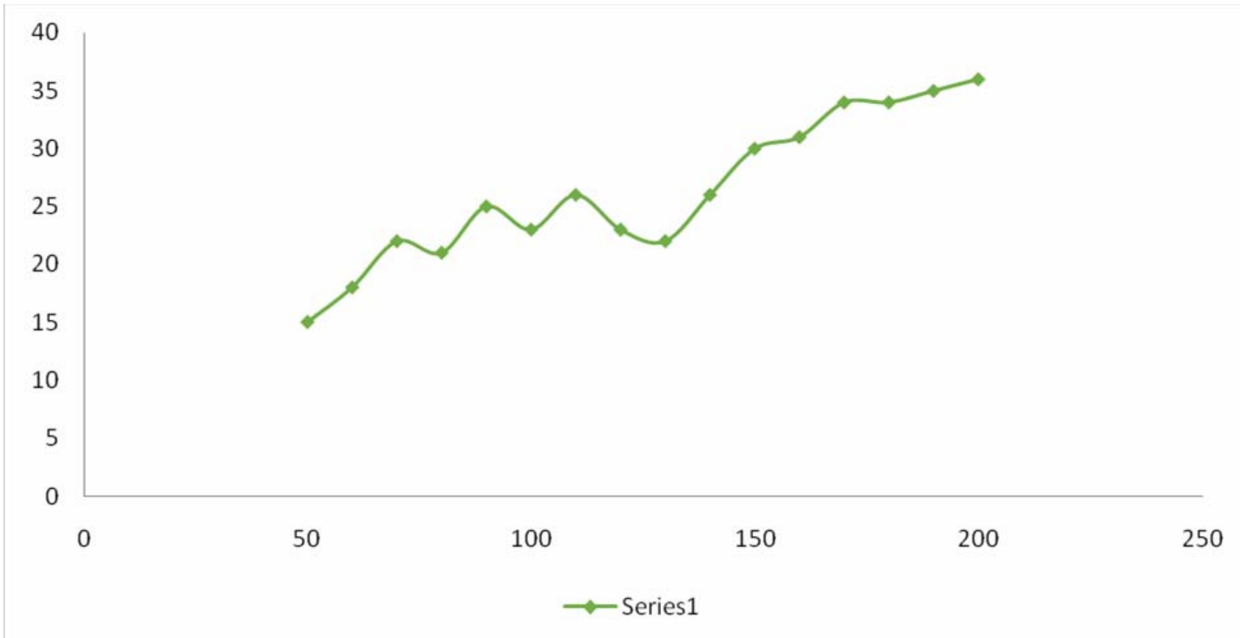

Fig. 4.1 V/I Characteristic of PbO

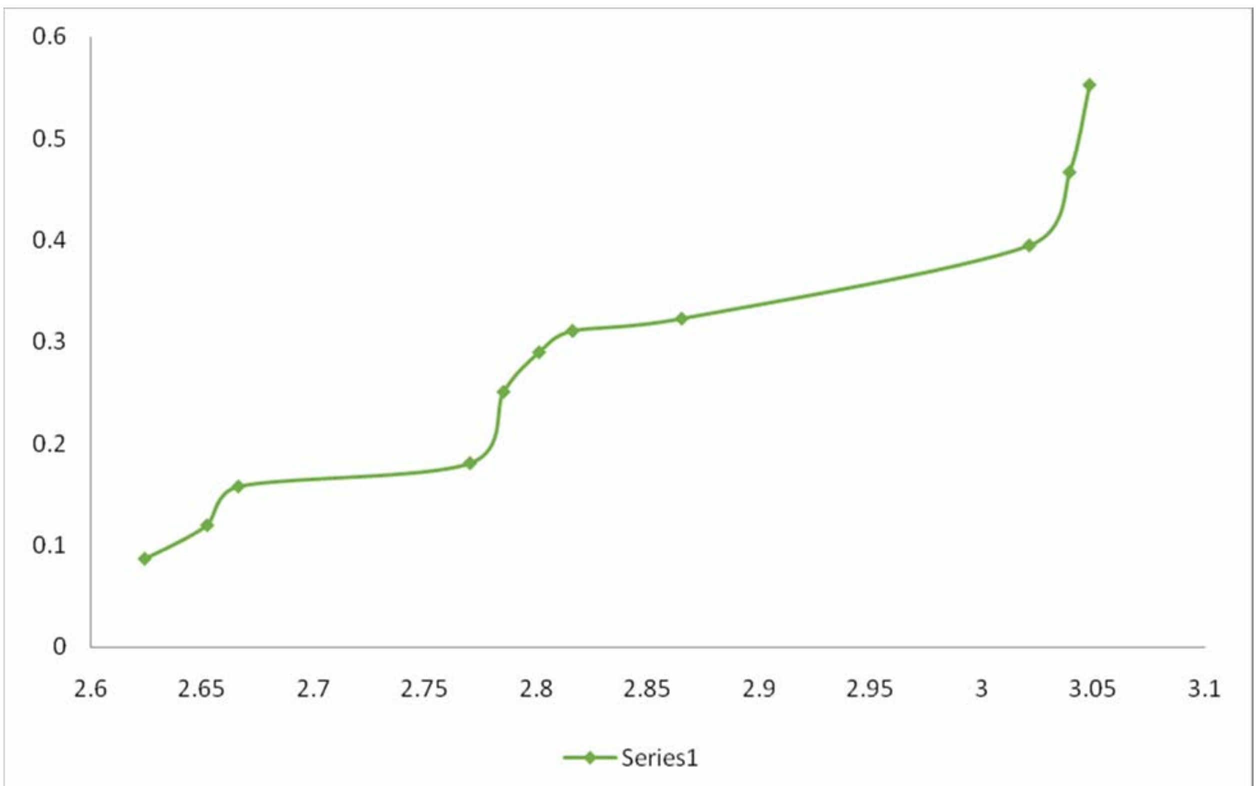

Fig 4.2 D.C Electrical Conductivity of PbO

Fig 4.1 represents the V/I characteristic of PbO filled PVA nanocomposites thin film. The current increase with increasing the voltage drop. Fig 4.2 represent the thermogram i.e logб VS 1/T for PbO filled

PVA nanocomposite thin film. The conductivity increases with increasing temperature. The activation energy is calculated from the graph and is found to be

$E_a = 1.195 \times 10^{-5}$ eV.

Conclusion :

Lead oxide nanoparticle were synthesized successfully by sol-gel method. XRD, UV-Spectroscopy, FTIR-Spectroscopy and DC-electrical conductivity characterization technique confirm the result. The crystalline nature and presence of PbO nanoparticle was confirmed from XRD and FTIR. Band gap energy of PbO nanoparticle was determined by UV-Spectroscopy and it was found to be 5.52eV. The composite of PVA and PbO shows the DC-Electrical conductivity of PbO by two probe method.

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