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Synthesis and Characterization of Barium Lead Tungstate

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Abstract:

The use of metal oxide nanoparticles have been studied due to their novel optical, electronic, magnetic, thermal and potential applications as catalysts, gas sensors, photo-electronic devices, ..etc. In this research work, we report a simple, soft chemical route for synthesizing $BaPbWO_4$ and $SrPbWO_4$ nanoparticles using cheap chemicals such as Barium nitrate (precursor salt), Strontium Nitrate by Sol-Gel method. The final product was dried at room temperature over night and calcined at 700^OC for 2h to get phase-pure product. The prepared nanoparticles (as prepared and heat-treated samples) were characterized by Scanning electron microscopy (SEM).

Keywords: Synthesis, Nanoparticles, Characterization, SEM, $BaPbWO_4$, $SrPbWO_4$,

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A. INTRODUCTION:

Nanomaterials research has witnessed an exponential growth during the decade. In the nanometric range, metallic and small band gap semiconducting materials exhibit fascinating quantum phenomena. Large band gap materials such as oxides stabilize in their high temperature phases and exhibit enhanced surface phenomena like catalysis and reduced reaction barriers for solid-state reactions[1-2]. Indeed, in the nanometric range, materials may be expected to behave quite differently from both molecular and bulk states since the ratio of the number of surface atoms to the number of bulk atoms is quite high. As a consequence of this increased ratio the number of valence unsatiated atoms in the nanoparticle becomes significant. There is thus a curiosity to understand the behaviour of materials in the nanolength scale and if possible to exploit the new properties exhibited by materials purely as a consequence of the smallness of size. Nano oxide materials have found wide range applications particularly as catalysts and as starting materials for making advanced structural ceramics. During sintering and shaping of oxidic materials for practical

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applications, use of nano sized particle as starting materials can be of great advantage because of the availability of large surface areas of the nanoparticles.

Nanostructures of metal oxides have shown their revival of interest in the fabrication of energy saving and harvesting devices such as Lithium ion batteries[1-5], fuel cells[6-7], solar cells[8],transistors/FETs[9], Light emitting devices (LEDs)[10], hydrogen production by water photolysis and its storage[11], water and air purification by degradation and adsorption of organic/inorganic pollutants and toxic gases [12], environmental monitoring by their applications in the fabrication of gas, humidity and temperature sensors[13], UV-screening[14] and photo detectors[15]. Instead of these they have also fabulous applications in biological and medical sciences such as drug delivery, cancer treatments, fluorescent imaging, bio labelling and bio tagging etc [16]. Oxides of transition metals have strong ferromagnetism with high Curie temperature and are used as magnetic read, write heads and data storage devices [17]. Transition metal doped active oxides such as ZnO, CuO, TiO_2, Al_2O_3 ..etc are called diluted magnetic semi conductors (DMS) and are applicable in the fabrication of spin based electronic devices i.e., spintronics. Similarly rare earth elements such as Eu, Nd, Sm, Tb doped metal oxides are usually used as phosphor materials for fabrication of LEDs, displays and laser materials.

Metal oxides are expected replacement and alternative of silicon and metal nitrate based expensive electronic devices and ICs. Recently, oxide based nanomaterial such as ZnO, TiO_2, CuO_2 and so on have revolutionized the nano materials research because of the availability or possibility of soft chemical synthesis besides tremendous application potential. One of the salient features of these oxide nano materials is the biocompatibility which opens avenue for interdisciplinary research to have better bridge up between physicist and biotechnologist. Binary semiconducting oxides, such as ZnO, TiO_2, CuO_2/Cu_2O, SnO_2, In_2O_3 and CdO, have distinctive properties and are now widely used as transparent conducting oxide materials [18] and sensors SnO_2 nano materials are regarded as one of the most important sensor materials for detecting leakage of several inflammable gases owing to their high sensitivity to low gas concentrations[19-20]. Indium–doped tinoxide (In: SnO_2, ITO) film is an ideal material for flat panel displays because of its high electrical conductivity and high optical transparency[21-22] and ZnO is regarded as an ideal alternative material for ITO because of its lower cost and easier etc hability. This chapter deals with methods of synthesis, characterisation of barium strontium lead tungstate.

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Recently, a variety of methods have been used for the synthesis of nanoparticles such as aerosol, pyrolysis, calcination and surfactant based colloidal synthesis. Sol gel process is one of the important methods in the preparation of nanoparticle. Most of the nanoparticles prepared by sol gel processing are amorphous in nature and it requires further heat treatment process for the production of crystalline particles. The nanoparticles synthesised by sol gel process are characterised by SEM (scanning electron microscope). The nano crystalline nature gives these nanoparticles unique properties that have consequently lead to their usefulness in photocatalysis.

B. Materials and Methods:

The sol-gel process, improves the evolution of inorganic networks through the formation of a colloidal suspension (sol) and gelation of the sol to form a network in a continuous liquid phase (gel). The precursors by synthesizing these colloids consist usually of a metal or metalloid element surrounded by various reactive ligands. The starting material is processed to form a dispersible oxide and forms a sol in contact with water or dilute acid. Removal of the liquid from the sol yields the gel, and the sol-gel transition controls the particle size and shape. Calcination of the gel produces the oxide.

Sol-gel processing refers to the hydrolysis and condensation of alkoxide-based precursors such as $Si(OEt)_4$ (tetraethyl orthosilicate or TEOS). The reactions involved in the sol-gel chemistry based on the hydrolysis and condensation of metal alkoxide $M(OR)_2$ can be described as follows:

$$MOR+H_2O \longrightarrow MOH + ROH \text{ (hydrolysis)}$$

$$MOH+ROM \longrightarrow M\text{-}O\text{-}M + ROH \text{ (Condensation)}$$

Sol-gel method of synthesizing nanomaterials is very popular amongst chemists and is widely employed to prepare oxide materials. The sol-gel process can be characterized by a series of distinct steps.

Objectives:

i. Synthesis of metal tungstate nanoparticles by Sol-Gel method

ii. Characterize the nanoparticles using Scanning Electron Microscopy (SEM) technique.

C. EXPERIMENTAL:

Reagents required: Sodium tungstate, Lead nitrate, Barium tungstate, Strontium tungstate, Sucrose, PVA EDTA, diethylamine ..etc were of expected AR grade of purity.

Step1:

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Synthesis metal tungstate particles: 25ml of equimolar(0.5) concentration of Sodium tungstate and lead nitrate solutions were prepared. Barium nitrate was added drop by drop to the round bottom flask with constant stirring with a magnetic stirrer. Barium lead tungstate particle is obtained.

Step 2:

Synthesis of Barium lead tungstate nanoparticle: To the synthesised Barium lead tungstate particle, equimolar (0.2M) mixture of EDTA (25mL) and diethylamine(25mL) was added. White gel was formed; to this add 0.02M PVA (15mL) and 0.02M sucrose (10mL). Heat the mixture at 90ºC for 1 hour to obtain the gel. This gel was taken to the high temperature(700ºC) in the furnace to obtain the required nanoparticle(Scheme-1).

Step 1:

Synthesis metal tungstate particles: 25ml of equimolar(0.5) concentration of Sodium tungstate and lead nitrate solutions were prepared. Strontium tungstate was added drop by drop to the round bottom flask with constant stirring with a magnetic stirrer. Strontium lead tungstate particle is obtained.

Step 2:

Synthesis of Strontium lead tungstate nanoparticle: To the synthesised Strontium lead tungstate particle, equimolar (0.2M) mixture of EDTA (25mL) and diethylamine(25mL) was added. White gel was formed; to this add 0.02M PVA (15mL) and 0.02M sucrose (10mL). Heat the mixture at 90ºC for 1 hour to obtain the gel. This gel was taken to the high temperature (700ºC) in the furnace to obtain the required nanoparticle (Scheme-2).

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D. a. Synthesis of Barium Lead tungstate nanoparticle flow chart:

$Na_2WO_4.2H_2O$(0.5M of 25ml) + $Pb(NO_3)_2$(0.5M of 25ml)

Add $Ba(N0_3)_2$(0.5M of 25ml)

Drop wise with continuous stirring for 2 hrs

↓

$BaPbWO_4$ precipitate

Add 0.2M, 25mL of (EDTA+ diethyl amine)

↓

White gel

Add 0.02M, 25mL (PVA+Sucrose)

↓

Subjected to high temperature

Heat

↓

Fine powder of $BaPbWO_4$ nanoparticle

Scheme-1

b. Synthesis of Strontium Lead tungstate nanoparticle flow chart:

Na_2WO_4. $2H_2O$(0.5M of 25ml) + $Pb(NO_3)_2$(0.5M of 25ml

Add $Sr(N0_3)_2$(0.5M of 25ml) Drop wise with continuous stirring for 2 hrs

↓

$SrPbWO_4$ precipitate

Add 0.2M, 25mL of (EDTA+ diethylamine)

↓

White gel

Add 0.02M, 25mL(PVA + Sucrose)

↓

Subjected to high temperature

Heat

↓

Fine powder of $SrPbWO_4$ nanoparticle

Scheme-2

E. Characterization of nanoparticles:

In nanotechnology, nanoparticles synthesized either biologically or chemically must be characterized in order to understand their intrinsic properties such as size, monodispersity, aqueous stability, the net charge, adsorption to biomolecules, aggregation and flocculation in various media. This provides vital information in terms of application of this nanoparticle. For instance, it provides answers to know whether a particular nanoparticle can be used in biological applications, or else to improve their synthetic processes, and/or chemical functionalization.

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A variety of characterization techniques are currently available some which precede the advent of nanoscience and technology and mostly drawn from material science. The development of new and integrated methods suited to probe nanomaterials is however, a continuous process. The common techniques used in the characterisation of nanoparticles are, ultraviolet-visible (UV) spectroscopy, Fourier transform infrared spectroscopy(FTIR), inductively coupled atomic/optical emission spectroscopy(ICP AES/OES), fluorescence spectroscopy(FS), X-ray photoelectron spectroscopy(XPS), scanning/transmission electron microscopy (SEM/TEM), dynamic light scattering(DLS), atomic force microscopy(AFM) and energy dispersion and analysis of X-rays(EDAX).

Scanning electron microscopy (SEM): In SEM, high resolution images are generated by focusing a high-energy beam of electrons on the surface of the specimen in a raster scanning fashion. These electrons interact with specimen to produce signals that provides information about the sample such as the surface morphology, elemental or chemical composition, crystal structure and positions of atoms or materials that makes up the sample. A high-energy beam of electrons are thermoionically emitted from an electron gun fitted with a tungsten or lanthanum hexaboride (LaB_6) cathode filament towards an anode; another source of electrons can be through field emission (FE). The most common filament is tungsten because of its affordability and high melting temperature compared to other metals.

A typical electron beam with energy within the 0-50keV is focused by two consecutive condenser lenses into a very fine spot of approximately 5mm. As the 1º electron beam strikes and interacts with the sample surface, the energy of the electron is dissipated due to continuous random scattering and absorption and effectively spreads into a tear drop shaped volume of the sample (interaction volume) extending about 1-5 µm into the surface. Several secondary signals are thus generated which are picked up by specialized detectors depending on the type of instrumentation.

F. RESULT AND DISCUSSION:

The shape, size and microstructure of the prepared metal oxides were examined using Leica-440 Cambridge Stereo scan, scanning electron microscope image. The SEM was operated at 220Kv.

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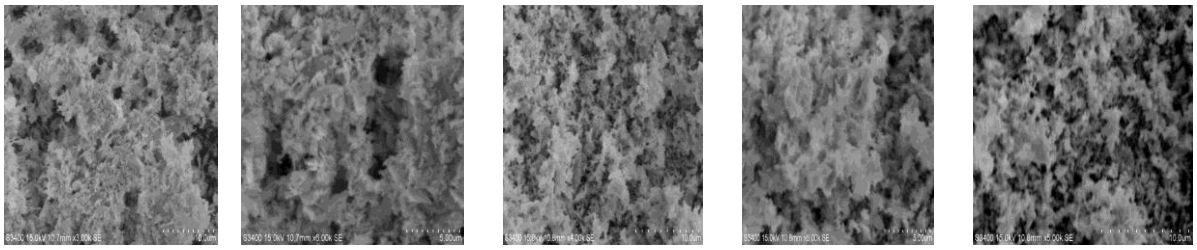
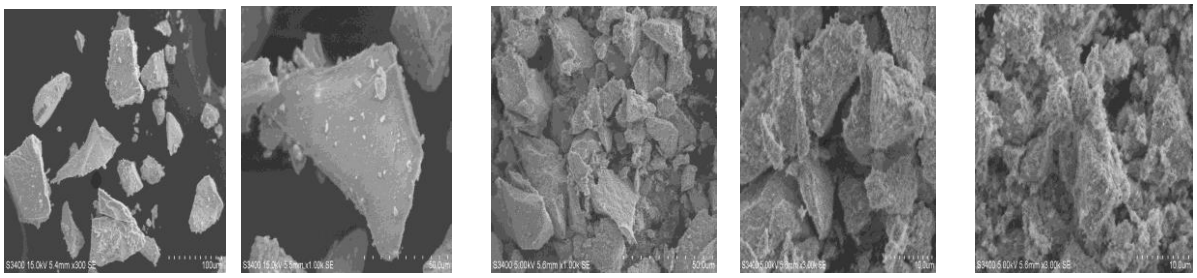


SEM Images of Synthesized $BaPbWO_4$ Nanoparticles



SEM Images of Synthesized $SrPbWO_4$ Nanoparticles

G. CONCLUSION:

From the above observations of SEM images, it may be concluded that all the ceramic materials showed nanosizes, forming different shapes and sizes of agglomerates starting from nanosphere to nanorods.

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