

Synthesis and characterization of SrO, PbO, NiO nanoparticles and SnO₂ microsphere via a PEG assisted route

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For the first time, nanostar of SrO was prepared by using PEG400 assisted process. Also PbO, NiO nanoparticle and SnO₂ microsphere have been synthesized by this method. In this study PEG400 used as template to control crystal growth, morphology and the sizes of nanoparticles. The products were characterized by SEM, FT-IR, UV-Vis spectrum and nanostar of SrO was characterized by X-ray diffraction (XRD). Results of the XRD analysis of SrO shows the crystal structure. The UV-Vis spectra of SrO, PbO, NiO and SnO₂ illustrate strong absorption peaks at 203, 208, 354 and 382 nm respectively. The SEM photographs of SrO shows nanostar morphology with diameters ranging 412 nm. The SEM photographs of PbO, NiO and SnO₂ show granular morphology with the sizes about 48 nm, 35 nm and 44 μ m, respectively. This article offers a simple, low cost and easy method to synthesis SrO, PbO, NiO and SnO₂. This method can be applied for synthesis of different metal oxides.

Key words: Nanoparticles, PEG400, Nanostar, morphology, Assisted route.

Introduction

Metal oxides play a very important role in many areas of chemistry, physical and materials science [1-6]. The metal oxides are attracting particular attention from scientists due to their easy mode of formation and multifunctional behavior. The research in the area of nano structured metal oxides, gradually gather popularity for their remarkable properties in electronic, magnetic, optical, thermal and mechanical fields [7]. There are many different methods reported for the synthesis of nanometer-sized metal oxides [8, 9]. Among different techniques, template techniques are common to obtain nanostructures. To date, numerous kinds of template molecules have been used to prepare nanocrystals. Of them, polyethylene glycols (PEG) have been used in preparing nanocrystals. It is found that PEG templates can assist in controlling crystal growth, assembly patterns and pore structure [10]. NiO as one of the most important p-type wide band gap transition metal oxide semiconductors, have been attracting attention due to its environmental friendly, high chemical/- thermal stability, and low cost [11]. In recent years, many research efforts have been done for synthesis of NiO nanostructures. For example, Zhang and coworkers reported the synthesis of hollow spheres by a glycerin assisted hydrothermal method [12]. Hu et al obtained NiO nano film through an electrochemical

route [13]. SnO₂ is an n-type semiconductor with a wide band gap and is well known for its applications in gas sensors [14], dye-base solar cells [15], optoelectronic devices [16]. In 2004, Gue and coworkers prepared SnO₂ nano rods with rutile structure via hydrothermal, template method [17]. In 2013, Liang and coworkers prepared SnO₂ nanoparticles via hydrothermal method and the surface properties of nanoparticles were investigated [18]. PbO is an important industrial material due to its unique electronic, mechanical and optical properties [19]. Many methods are applied for synthesis of Lead oxide, including thermal decomposition [20], hydrothermal synthesis [21], and sonochemical [22]. SrO is an important wide band gap metal oxide [23]. It also is a material in production of glass for color television tubes, ferrite magnets, small DC motors, pigments, dryers and paints [24]. There have been many methods for synthesis of SrO such as chemical precipitation, hydrothermal and sol-gel [25].

Herein, a simple and inexpensive method is applied to synthesize SrO nanostar, PbO and NiO nanostructures and SnO₂ microspheres by using PEG as the template.

Experimental

Instruments and reagents

All materials were commercial reagent grade. FT Infrared (FT-IR) spectra were obtained as potassium bromide pellets in the range of 400–4000 cm⁻¹ with a Bruker tensor 27DTGS. UV-VIS spectra were recorded on Lambda 35 spectrophotometer. XRD pattern was recorded by Jeol JDX-8030. Scanning electron microscope (SEM)

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of catalyst was performed on Philips-XL30.

Preparation of nanoparticles

[SrO synthesis]

4 mL SrCl₃.6H₂O (0.1 M) was added to 100 mL beaker containing mixed solution of 40 mL distilled water, 2 mL ammonia and 6 mL PEG(400) and well blended by stirring for 20 min subsequently, the mixed solution was placed for 6 days at room temperature. White crystalline products were collected, washed with distilled water and ethanol several times and dried at 70 °C in a vacuum oven for 7 h. Then calcined for first time at 500 °C for 6 hrs and second time at 650 °C for 36 h to obtain SrO nanostar. Similar procedure is used for preparation of SrO nanoparticles using Sr(NO₃)₂.

[PbO synthesis]

10 mL Pb(OAc)₂.3H₂O (0.1 M), 7 mL NaOH (0.1 mM), 50 mL distilled water and 5 mL PEG400 was added to a conical flask. The mixed solution was placed at room temperature for 30 days. The obtained precipitate was then separated by centrifugation and washed several times with distilled water and ethanol and then calcined at 500 °C for 5 hrs.

[NiO synthesis]

5 mL Ni(OAc)₂.4H₂O (0.5 M) was added to breaker. After that 2 mL ammonia, 60 mL H₂O and 7 mL PEG400 was added to solution. The solution was stirred for 20 min, and then the mixture was placed at room temperature for 14 days. The obtained precipitate was then separated by centrifugation and washed several times with distilled water and ethanol and then calcined at 550 °C for 6 hrs.

[SnO₂ synthesis]

5 mL SnCl₂.2H₂O (0.5M), 8 mL NaOH (5 M), 30 mL distilled water, 1gr PEG (M_w = 6000) were added to a beaker. The solution was stirred for 20 minutes and placed at room temperature for 3 weeks. The obtained precipitate was then separated by centrifugation and washed several times with distilled water and ethanol and then calcined at 600 °C for 4 hrs.

Results and Discussion

The FTIR spectrum of SrO is shown in Fig. 1. In detail, the absorption bands at 3456 cm⁻¹ originates from stretching vibration and deformation vibration of O-H. The strong broad absorption peak at about 1445 cm⁻¹ is assigned to the asymmetric stretching vibration of Sr-O, the sharp absorption bands at 866 cm⁻¹ and 701 cm⁻¹ can be assigned to out of plane bending vibration of Sr-O[26]. The two bands at 1379 and 1469 cm⁻¹ are attributed to the C-O vibrations. [27].

The FTIR spectrum of PbO nanoparticles is shown in Fig. 2. The two peaks at 518, 557 cm⁻¹ are related to stretching vibration of Pb-O [28].

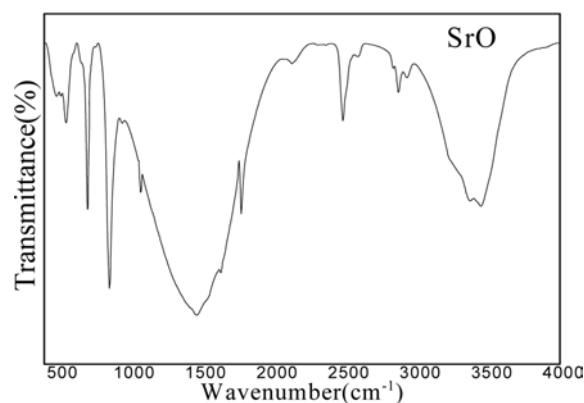


Fig. 1. FT-IR spectra of SrO.

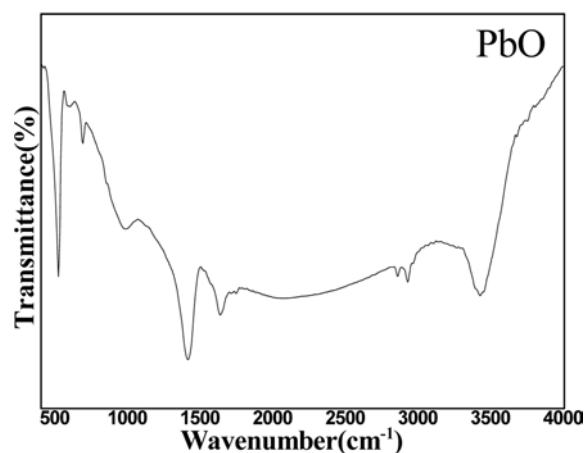


Fig. 2. FT-IR spectra of PbO nanoparticles.

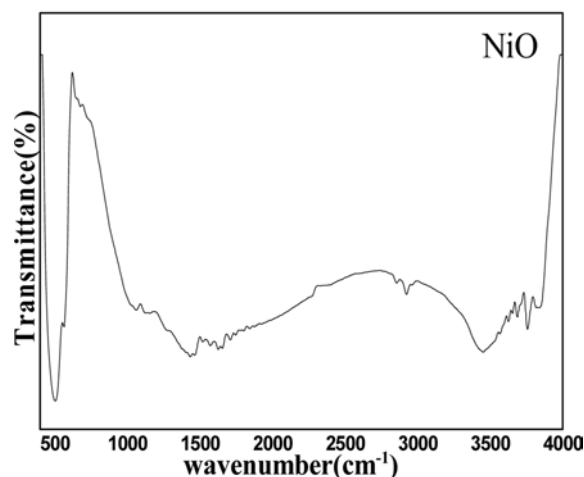


Fig. 3. FT-IR spectra of NiO nanoparticle.

Fig. 3 shows FT-IR spectrum of NiO nanoparticles at room temperature. The strong peak at 493 cm⁻¹ is related to stretching vibration of NiO [29]. The peak at 3415 cm⁻¹ is originated from the symmetric vibration of OH groups.

The FT-IR spectra of SnO₂ nanoparticles is shown in Fig. 4. SnO₂ microstructure has two peaks at 597 cm⁻¹ (Sn-O stretching vibration), 952 cm⁻¹ (Sn-OH stretching

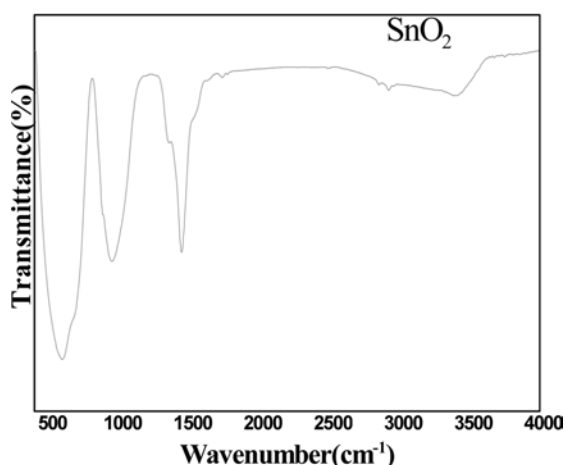


Fig. 4. FT-IR spectra of SnO_2 microsphere.

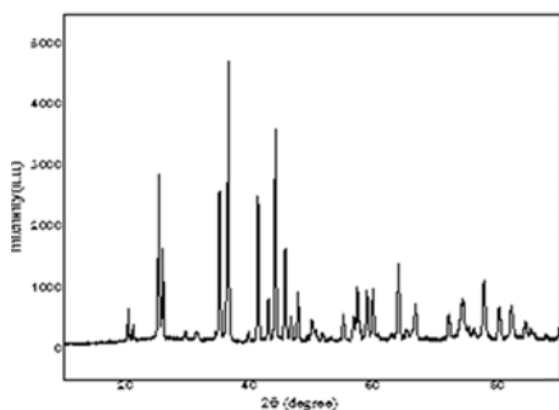


Fig. 5. XRD pattern of SrO nanostar.

vibration) [30].

The XRD pattern of as-obtained SrO nanostar is shown in Fig. 5. The Peak at $2\theta = 29.660$ corresponds to SrO [31]. The peaks at $2\theta = 25.210$, 25.860 are a result of SrCO_3 [18]. Although there are additional weak diffraction peaks (the most intense is at 46.6°) which are related to $\text{Sr}(\text{OH})_2$ [32].

The UV-Vis spectra of SrO, PbO, NiO nanoparticle and SnO_2 microsphere are reported in Fig. 6. Fig. 6(a) shows the strong band around 203 nm which can be attributed to SrO nanostars and nanoparticles [33]. Fig. 6(b) illustrates the band at 208 nm which is probably due to PbO nanoparticles [28]. Fig. 6(c) shows a strong band around 354 nm according to NiO nanoparticles [34]. In Fig. 6(d) the band at 382 nm is related to SnO_2 microsphere [35].

Fig. 7(a) shows SrO nanostars with diameters ranging 412 nm. Fig. 7(b) shows SEM image of SrO nanoparticles, The size of the nanoparticles are around 24 nm. Fig. 8 shows the SEM images of PbO, NiO and SnO_2 . Fig. 8(c) shows the SEM image of PbO with diameters about 48 nm, in Fig. 8(d) the sizes of NiO nanoparticles are about 35 nm and Fig. 8(e) shows the SEM image of SnO_2 microsphere with diameters ranging 44 μm .

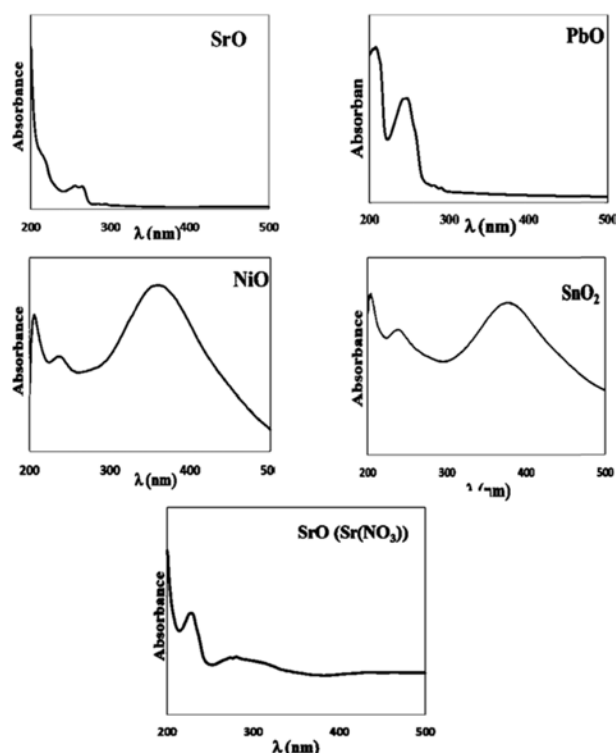


Fig. 6. UV-Vis spectra of SrO, PbO, NiO nanoparticles and SnO_2 microsphere.

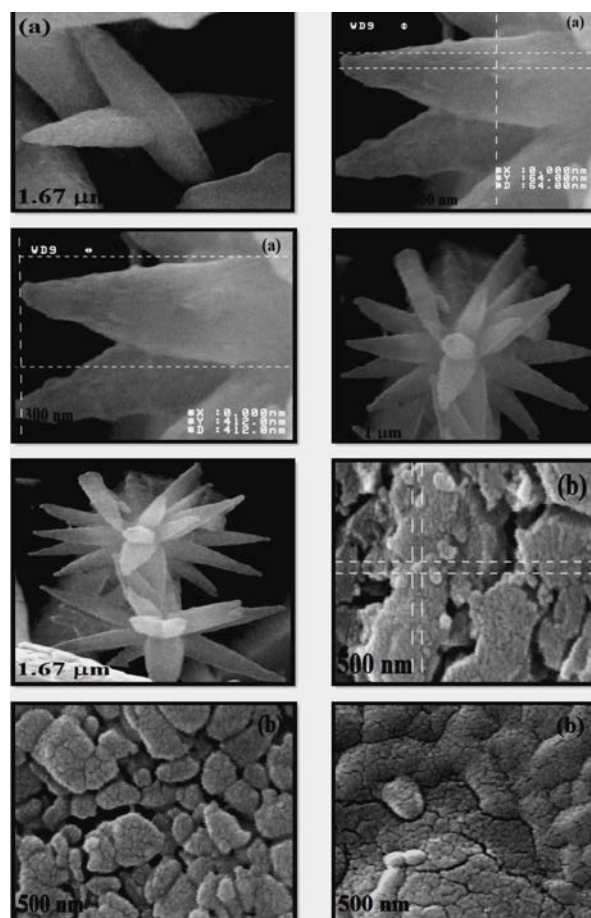


Fig. 7. SEM images of SrO nanostructures (a) SrO nanostar ; (b) SrO nanoparticle.

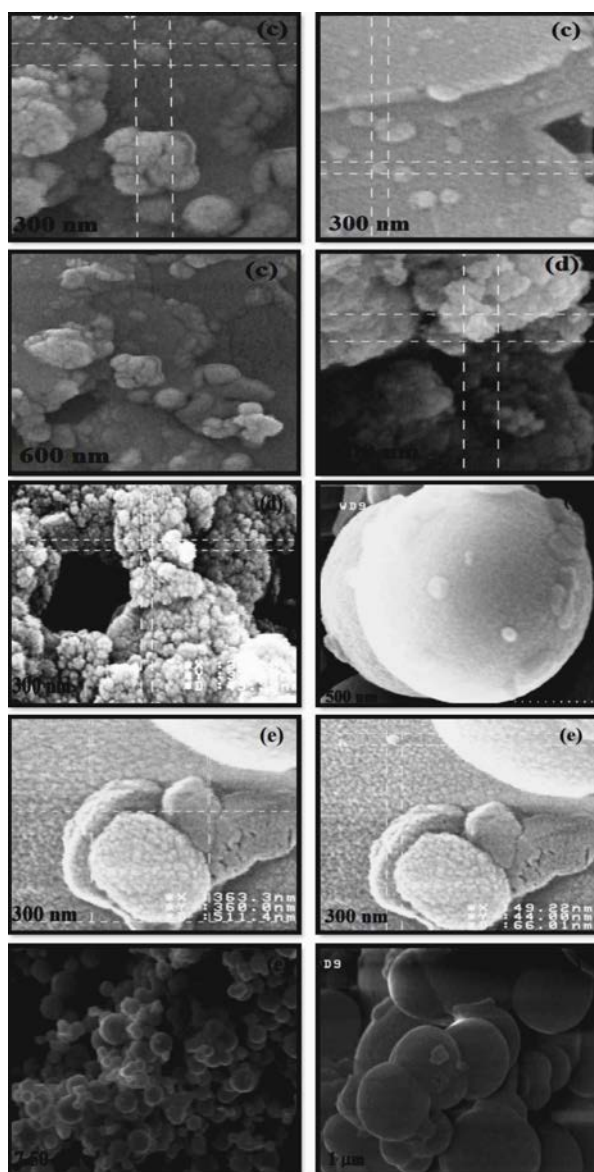


Fig. 8. SEM image of PbO, NiO nanoparticles and SnO₂ microsphere c) PbO nanoparticle; d) NiO nanoparticle; e) SnO₂ microsphere.

Conclusions

This study demonstrates a new method for synthesis of SrO, PbO, NiO and SnO₂ by using a template. PEG400 as a template plays a vital role in the synthesis of nanoparticles. It can assist in controlling crystal growth, assembly patterns and pore structure. In this paper, a new morphology for SrO is identified, nanostar morphology. This method is easy and, low cost, needing no complicated procedures while still applicable for the synthesis of other metal oxides.

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