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## A simple sonochemical approach for synthesis of selenium nanostructures and investigation of its light harvesting application

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## ABSTRACT

Selenium (Se) nanostructures were synthesized by a sonochemical method using  $\text{SeCl}_4$  as a new precursor for Se nanostructures. Moreover, hydrazine, potassium borohydride, and thioglycolic acid were used as reducing reagents in aqueous solution. Ultrasonic power, irradiation time, reducing agent, solvent, HCl, NaOH, and the surfactant were changed in order to investigate the effect of preparation parameters on the morphology and particle size of selenium. The obtained Se with different morphologies and sizes was characterized by XRD, SEM, TEM, EDS, and DRS. The selenium nanostructures exhibited enhanced photocatalytic activity in the degradation of methylene blue (MB) under visible light irradiation. Furthermore, to examine the solar cell application of as-synthesized selenium nanostructure, FTO/TiO<sub>2</sub>/Se/Pt-FTO and FTO/Se/CdS/Pt-FTO structures were created by deposited selenium film on top of the TiO<sub>2</sub> layer and FTO glass prepared by Doctor's blade method, respectively.

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### 1. Introduction

In the past few decades, nanomaterials have been extensively studied for the degradation of organic pollutants as well as solar cell application [1–3]. Elemental selenium (Se) is a narrow band gap (~1.7 eV) semiconductor which has been extensively used in solar cells, photocatalyst, xerography, and rectifiers [4–6]. It is also a photoconductor with a relatively low melting point (~217 °C), high refractive index and high reactivity leading to a wealth of functional materials such as Ag<sub>2</sub>Se [7–9], CdSe [10], ZnSe [11,12], PbSe [13,14], and NiSe [15–19]. There are some methods for selenium nanostructures preparation which involve solution-phase [20], Green Synthesis [21,22], microwave-polyol method [23] and ultrasonic irradiation approach [24,25]. Among various methods for the preparation of nanostructures, ultrasonic method is more promising in terms of low cost, simply control the shape and particle size, low processing temperature, simplicity and potential for large-scale production. Recently, the ultrasonic process as a fast, convenient, and economical method has been widely used to generate novel nanostructure materials under ambient conditions [24–29]. The chemical effects of ultrasound arise from

acoustic cavitation, which is the formation, growth, and implosive collapse of bubbles in a liquid. The growth of the bubble occurs through the diffusion of solute vapor into the volume of the bubble, while the collapse of the bubble occurs when the bubble size reaches its maximum value. When solutions are exposed to ultrasound irradiation, the bubbles are implosively collapsed by acoustic fields in the solution. According to hot spot theory, very high temperatures (>5000 K) are obtained upon the collapse of a bubble. Since this collapse occurs in less than a nanosecond, very high cooling rates (>10<sup>10</sup> K/s) are also obtained [30,31]. These extreme conditions can drive a variety of chemical reactions to fabricate nano-sized materials. It is widely accepted that the properties of nanomaterials have a close relationship with their morphology, size, size distribution, crystallinity, [32,33] and control over the morphology and size of inorganic materials at micro- and nano-scale level is an important goal. Therefore, in this research we investigate the effect of preparation parameters such as ultrasonic power, irradiation time, reducing agent, solvent, HCl, NaOH, and surfactant on the morphology and particle size of selenium which was synthesized from  $\text{SeCl}_4$  as selenium precursor by a sonochemical method. To our knowledge, it is the first time that  $\text{SeCl}_4$  is used as the selenium source for the synthesis of Se nanostructures. Thioglycolic acid was used as both the solvent and the capping agent. It is noteworthy that, in suitable conditions, thioglycolic acid can

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form a complex with  $\text{Se}^{4+}$  ions that turn to Se clusters; furthermore, it can reduce  $\text{SeCl}_4$  to Se, which means it can work as a reducing agent under ultrasonic irradiation. Moreover, the as-synthesized Se nanostructures were utilized as the photocatalyst for the degradation of methylene blue (MB) and as the photo-anode material for fabrication of FTO/ $\text{TiO}_2$ /Se/Pt-FTO and FTO/Se/CdS/Pt-FTO solar cell structures to examine their solar cell application.

## 2. Experimental

### 2.1. Materials and physical measurements

All the chemicals used in this method were of analytical grade and used as-received without any further purification. Ultrasonic irradiation was accomplished using a high-intensity ultrasonic probe (Sonicator 3000; Bandeline, MS 72, Germany, Tihorn, 20 kHz, 80 W  $\text{cm}^{-2}$ ) immersed directly in the reaction solution. X-ray diffraction (XRD) patterns were recorded by a Philips-X'PertPro, X-ray diffractometer using Ni-filtered  $\text{Cu K}\alpha$  radiation at scan range of  $10 < 2\theta < 80$ . Scanning electron microscopy (SEM) images were obtained on LEO-1455VP equipped with an energy dispersive X-ray spectroscopy. Transmission electron microscope (TEM) images were obtained on a Philips EM208S transmission electron microscope with an accelerating voltage of 100 kV. The energy dispersive spectrometry (EDS) analysis was studied by XL30, Philips microscope. The diffused reflectance UV-visible spectrum (DRS) of the sample was recorded by an Ava Spec-2048TEC spectrometer. Photocurrent density–voltage ( $J$ – $V$ ) curve was measured by using computerized digital multimeters (Ivium-n-Stat Multichannel potentiostat) and a variable load. A 300 W metal xenon lamp (Luzchem) served as assimilated sun light source, and its light intensity (or radiant power) was adjusted to simulated AM 1.5 radiation at 100  $\text{mW}/\text{cm}^2$  with a filter for this purpose.

### 2.2. Synthesis of selenium nanostructures

At first, 0.05 g (0.23 mmol) of  $\text{SeCl}_4$  as selenium precursor was dissolved in 30 ml of distilled water, TGA/water, and TGA under magnetic stirring. In the second step, different solutions were made by dissolving 0.14 mmol of different surfactants such as CTAB, SDS, TGA, PEG-600, TGA/PEG, TGA/SDS, and TGA/CTAB in 8 ml of distilled water and were added subsequently to the above solution; then, 2 ml of hydrazine (1 M), potassium borohydride (1 M) as reducing agent were added drop-wise. During these processes no sample was observed. Finally, the colorless mixtures were loaded into a beaker where the reaction was performed in an ultrasonic digestion system at room temperature. After 10 min, the colorless solutions turned red, which indicates the presence of  $\text{Se}^{2+}$ . The color of the solution changed to black when the reaction time was prolonged to 20 min. After irradiation, the system was allowed to cool to room temperature by itself, and the obtained precipitate was collected by filtration, and washed with absolute ethanol and distilled water several times. Finally, the products were dried in vacuum at 70 °C for 5 h. The reaction conditions are listed in Table 1.

### 2.3. Photocatalysis experiments

In order to test the photocatalytic activity of the Se nanostructures, the decolorization of methylene blue (MB) in suspension of Se nanostructures under high-pressure mercury lamp illumination was studied. A 250 W high-pressure mercury lamp (GYZ-250) was fixed at a distance of 30 cm above the surface solution. The

measured luminous intensity was 0.5  $\text{kW m}^{-2}$ . Then, 15 mg/L of photocatalyst nanoparticles was added into 150 ml of 25  $\text{g l}^{-1}$  methylene blue (MB) aqueous solution. Subsequently, the aqueous suspension was magnetically stirred for 30 min in dark to achieve the adsorption–desorption equilibration to discount the adsorption of the substrate on the catalyst. Afterwards, under ambient conditions and stirring, the beakers were exposed to UV light for 30, 45, 60, 75, 90, 105, and 120 min, respectively. Then, 15 ml of sample was taken out after a determined period of time and centrifuged at 5250 rpm for 5 min. Finally, the absorbance spectra of the methylene blue (MB) solution were recorded by a UV–vis spectrophotometer (Shimadzu UV–vis spectrometer) and the decolorization rate was calculated according to the absorbance change. The decolorization efficiency (%) is calculated as decolorization efficiency (1).

$$(\%) = \frac{C_0 - C}{C_0} \times 100 \quad (1)$$

where  $C_0$  is the initial concentration of dye and  $C$  is the concentration of dye after photo irradiation.

### 2.4. Fabrication of FTO/Se/CdS/Pt-FTO cell

A selenium thin film for the solar cell was prepared by the Doctor's blade technique. First, a suspension was provided by dispersing 0.05 g of synthesized Se powder into a solution containing 5 ml of ethanol, 0.05 g of ethyl cellulose, and 0.05 ml of Triton X100. Then, this suspension was coated on washed Fluorine-tin-oxide (FTO)-coated glass substrate (1  $\text{cm} \times 1 \text{cm}$ ). Subsequently, the glass substrate was kept at 350 °C for 30 min to eliminate the organic compounds. Afterwards, a CdS layer was deposited on a Se thin film with chemical bath deposition (CBD). The CBD process was performed with a solution consisting of 0.01 M of  $\text{Cd}(\text{NO}_3)_2$  and 0.08 M of thiourea. The bath was heated to 80 °C and then the sample was immersed. A counter-electrode was made from deposition of a Pt solution on FTO glass. Later, this electrode was placed over CdS/Se electrode. The redox electrolyte consisting of 0.05 M of  $\text{LiI}$ , 0.05 M of  $\text{I}_2$  and 0.5 M of 4-tert-butylpyridine at acetonitrile as a solvent was introduced into the cell through one of the two small holes drilled in the counter electrode. Finally, these two holes were sealed by a small square of sealing sheet and characterized by  $I$ – $V$  test.

### 2.5. Fabrication of FTO/ $\text{TiO}_2$ /Se/Pt-FTO cell

Electrophoresis deposition (EPD) was utilized to the prepare  $\text{TiO}_2$  films. During EPD, the cleaned FTO glass remained at a positive potential (anode) while a pure steel mesh was used as the counter (cathode) electrode. The linear distance between the two electrodes was about 3 cm. Power was supplied by a Megatek Pro-programmable DC Power Supply (MP-3005D). The applied voltage was 10 V. The deposition cycle was repeated 4 times, each time 5 s, and the temperature of the electrolyte solution was kept constant at 25 °C. The coated substrates were air dried. The apparent area of the film was 1  $\times$  1  $\text{cm}^2$ . The resulted layer was annealed under an air flow at 500 °C for 30 min. Electrolyte solution consisted of 120 mg/l of  $\text{I}_2$ , 48 ml/l of acetone, and 20 ml/l of water. For deposition of Se powder on the FTO glass substrate, a paste of Se was initially prepared. The slurry was produced by mixing and grinding 1.0 g of the nanometer sized Se with ethanol and water in several steps. Afterwards, the ground slurry was sonicated with ultrasonic horn (Sonicator 3000; Bandeline, MS 72, Germany) and then mixed with terpeneol and ethyl cellulose as binders. After removing the ethanol and water with a rotary-evaporator, the final paste was prepared. The prepared Se paste was coated on  $\text{TiO}_2$  film by a Doctor blade technique. After that the electrode was gradually

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