



Synthesis of selenium particles with various morphologies



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ABSTRACT

Uniform selenium spherical particles were prepared by reducing selenous acid with hydroquinone in the presence of Daxad 11G. The red colored colloidal dispersions displayed a distinct plasmon band at ~612 nm and were stable for extended time due to the negative surface potential of the particles. Structural analyses indicated that the Se spheres were aggregates of nanosize subunits crystallized in the hexagonal system. Selenium wires and rods were obtained by changing the pH and the composition of the precipitated dispersions and incubating them for extended time at moderate temperatures. The addition of a co-solvent played a major role in the re-crystallization of selenium spheres into anisotropic structures.

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1. Introduction

The excellent photoconductivity and enhanced piezoelectric, thermoelectric, and non-linear responses of selenium are routinely exploited in many applications including solar cells, rectifiers, photographic exposure meters, photocells, pressure sensors, and xerography [1,2]. The range of applications can be further broadened when the element is available in dispersed form [3]. For example, selenium nanoparticles paired with suitable bio-degradable stabilizers are often used in the bio-medical field [4]. For these reasons, the study of selenium particles has gained lately considerable importance and significant efforts are directed toward their synthesis, characterization, and modification. Dispersed selenium with various crystalline structures has been obtained in a wide variety and shapes including nanowires [5], nanoribbons [6], nanoplates [7], nanotubes [8], and spheres [2]. Gates et al. [9] were the first to synthesize trigonal Se nanowires using a solution precipitation process. Johnson et al. [1] synthesized both amorphous and monoclinic selenium in reverse micelles. Amorphous selenium (a-Se) with a mean particle size of 2 nm was also prepared in aqueous solution by Dimitrijevic and Kamat [10]. Such dispersions, however, were subject to strong photo-degradation and thus unstable. Quintana et al. [11] used pulsed laser ablation to prepare amorphous selenium particles and deposited them on various substrates (Au, Si, glass). Trigonal selenium (t-Se) particles have also been produced by adsorption through vapor phase diffusion [12], confinement in zeolite pores or cancrinite nanochannels [13], and crystallization of amorphous selenium melt [14]. The majority of these processes yield stable selenium dispersions only at low

concentrations and are not suitable for large-scale manufacturing. For this reason, the development of many practical applications involving dispersed selenium particles has been slow or put on hold. This paper describes a simple, volumetrically efficient, and easily scalable precipitation method for producing monodisperse selenium particles in either dried form or as stable dispersions. The process consists of reducing selenous acid with hydroquinone in the presence of a dispersing agent (Daxad 11G) and includes several novel elements. First, we show for the first time that hydroquinone can be used effectively for the preparation of dispersed elemental selenium. Secondly, the method generates stable dispersions of uniform selenium particles at significantly higher concentrations than previously demonstrated. Finally, it is being shown that the precipitated uniform selenium spheres can be subsequently converted into anisotropic particles.

2. Experimental

2.1. Materials

Selenous acid (H_2SeO_3 , 97%) and hydroquinone ($\text{C}_6\text{H}_6\text{O}_2$, 99%) were obtained from Alfa Aesar (Massachusetts, USA). Daxad 11G (sodium salt of naphthalene sulfonate formaldehyde condensate) was purchased from Fluka (Missouri, USA). All reagents were used without further purification. All glassware was thoroughly cleaned before the experiments. Deionized water (DI) was used in the preparation of all solutions.

2.2. Preparation of selenium spheres

The reacting solutions of selenous acid (100 cm^3 , 1.4 mol dm^{-3}) and hydroquinone (50 cm^3 , 2.2 mol dm^{-3}) were prepared by

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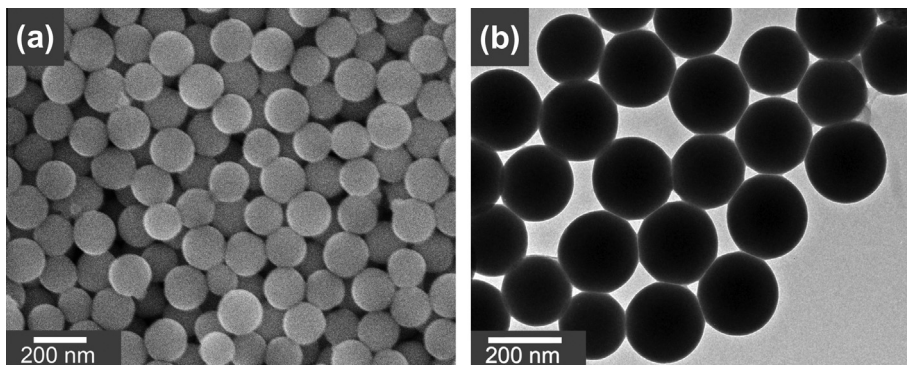


Fig. 1. FESEM (a) and TEM (b) images of selenium particles formed in the experimental conditions given in Section 2.2.

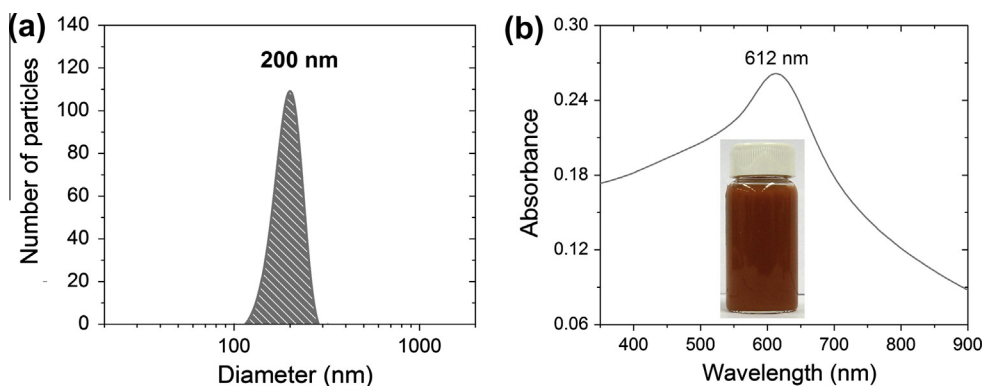


Fig. 2. (a) Particle size distribution of Se particles shown in Fig. 1 and (b) UV-Vis spectrum and photo (inset) of the selenium dispersion prepared in the conditions given in Section 2.2.

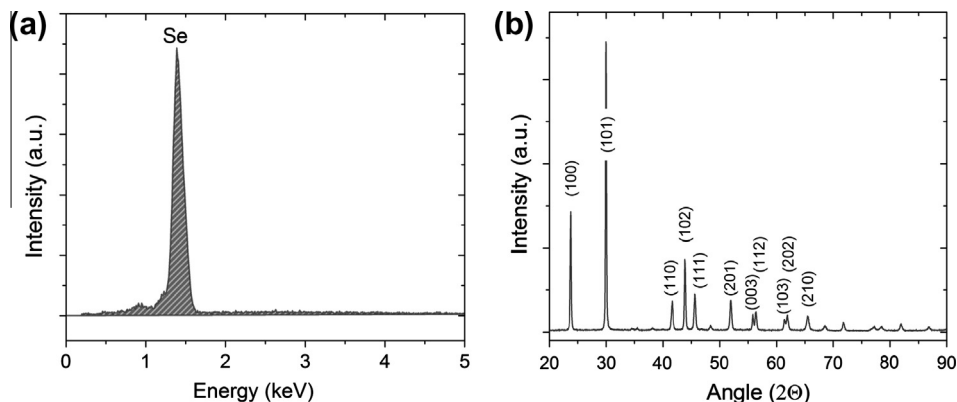


Fig. 3. (a) EDX and (b) X-ray diffraction patterns of selenium particles shown in Fig. 1.

dissolving the respective compounds in deionized water. The dispersant (2.2 g Daxad 11G) was dissolved in the latter. After maintaining the solutions at 65 °C for 10 min, the selenous acid was added quickly to the stirred hydroquinone/Daxad 11G solution. While holding the temperature at 65 °C, the stirring was continued for 30 more minutes to ensure the complete reduction of selenium and the formation of a stable colloidal dispersion. For structural characterization, the particles were separated by centrifugation, washed three times with DI water and dried for 4 h at 40 °C.

2.3. Characterization

The size and morphology of selenium particles were assessed by transmission electron microscopy (TEM) and field emission

scanning electron microscopy (FESEM). The size distribution of the particles was determined from the electron micrographs using ImageJ software. Their degree of dispersion was assessed by comparing the SEM size and the average particle size determined by dynamic light scattering (DLS) analysis. The optical properties of selenium particles were monitored as a function of time with a Perkin–Elmer Lambda 35 UV–Vis Spectrophotometer. Small dispersion aliquots (1.0 cm³) removed from the reaction vessel were first diluted in a 50 cm³ volumetric flask and then transferred in the 10-mm-optical-path-length quartz cuvette. The scanning range used was from 400 nm to 900 nm and the scanning speed was 960 nm/min. The surface charge of Se particles was measured with a ZetaPals instrument (Brookhaven Instruments). The structure of particles was investigated by X-ray diffraction (XRD) using a

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