

Synthesis and Characterization of Selenium Nanoparticles and their Photocatalytic Activity

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Abstract

The objective of present study demonstrates a simple soft chemical method to prepare selenium (Se) nanoparticles. Various characterization techniques such as X-ray diffraction (XRD), scanning electron microscope (SEM), and Ultraviolet-Visible (UV-Vis) spectroscopy have been used to characterize synthesized nanoparticles. The XRD studies synthesized indicated the formation zinc selenide (ZnSe) along with Se nanoparticles. The SEM showed spherical shape Se nanoparticles of average size about 525 nm. The synthesized nanoparticles were also studied for their photocatalytic activity for degradation of methylene blue (MB) under UV light irradiation. The study showed 99% degradation efficiency in 90 min. It has been observed that the presence of Se in ZnSe reduces the photo-corrosion under light irradiation and improved photocatalytic stability.

Keywords

Photocatalysis, Selenium, Nanoparticles, Methylene blue, Degradation

Introduction

Industrial development has an important role in economic's growth of countries. However, the rapid growth of industries is leaving adverse impact on human life in term of polluting water and air [1]. Water pollution is one of the biggest challenges worldwide. Many industries like textile, pharmaceutical industries, etc. that release hazardous effluents such as toxic chemicals and organic dye pollutants into water which imposes serious health problems for both aquatic and terrestrial ecosystem [2, 3]. Therefore, it is necessary to remove such type of pollutants from water. To address these issues, various techniques such as ion exchange, adsorption, membrane reverse osmosis, and membrane filtration has been used for treatment of contaminated water. [4-6]. Photocatalysis is the most successful technique for removal of organic pollutant from water. In this process, photo-induced electron and hole stimulate the redox reaction with the pollutants. Several types of semiconductors photocatalyst have been synthesized with band gap from UV to visible for removal of dye pollutants [7-10]. Among them, anatase titanium dioxide and zinc oxide (ZnO) are most promising materials for solar cell, hydrogen production, and degradation of pollutants from water [11-13]. The problem associated with these materials exhibiting large band gaps and photo-corrosion stability. Researchers across the world had reported various types of zinc, copper, cadmium, vanadium, chromium, etc. based metal oxide photocatalysts for degradation of dye [14-16]. Huey-Yee Chai et al. has prepared ZnO through hydrothermal method and reported photocatalytic activity of ZnO was 75% in 90 min under UV light [17]. Shasha Fu and his co-worker reported hollow perovskite-type LaCoO₃ nanoparticles prepared via surface-ion adsorption method [18]. They reported 87% degradation of MB over LaCoO₃,

in 100 min under UV light. However, most of them have low photocatalytic efficiency due to high electron-hole pair recombination, photo corrosion, and low absorption of light which make them unstable catalyst under light irradiation. Here, we synthesized Se nanoparticle along with ZnSe by simple co-precipitation method. These nanoparticles show excellent photocatalytic activity under UV light irradiation. The Se nanoparticle acts as a co-catalyst which minimize electron hole pair and improves photocatalytic activity

Materials and Methods

Materials: Zinc acetate dihydrate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$), selenium dioxide powder (SeO_2), diethylene glycol, and ethylene diamine (high purity, >99%) were purchased from Merck. The sodium acetate anhydrous (CH_3COONa), thiourea (NH_2CSNH_2), and MB were purchased from Loba chemie Pvt. with purity 99.0%. The De-ionized water was used in the experiment to prepare the solution.

Synthesis procedure

Se nanoparticles was prepared by simple soft chemical approach followed by co-precipitation method. Briefly, Zinc acetate dihydrate and thiourea in 1:2 molar ratio were dissolved 100 mL diethylene glycol solution under 15 min magnetic stirrer and 10 min for sonication to make clear solution. We prepared another solution in which selenium oxide powder and sodium acetate in 1:1 ratio was dispersed in 100 mL distilled water along 20 mL ethylenediamine. Both the solution mixture were mixed together and kept under continuous stirring for 15 min. Afterward, solution mixtures is refluxed at 150 °C for 2 h under magnetic stirrer. The obtained precipitate was washed several times with distilled water to remove by-product. The dark yellowish green color precipitate was dried in oven overnight at 60 °C to obtain powder sample.

Characterization techniques

Various instrumental techniques have been used to characterize the synthesized Se nanoparticles. The crystal structure has been analyzed by powder XRD patterns using RIGAKU X-ray Diffractometer (model: SMART LAB) with Cu-K α radiation ($\lambda = 0.1518$ nm) at room temperature. UV-Vis absorption spectra was measured by Shimadzu UV-2600 spectrophotometer consisting of double beam photometric system. SEM was used to observe the surface morphology of sample.

Photocatalytic experiment

The photocatalytic experiment was performed under UV light using MB as a model dye. The experiment was conducted by adding 24 mg amount of catalyst in 80 mL dye solution (10 ppm) and kept under stirring for 15 min to obtain the equilibrium of the reaction. Initially, 2 mL solution were extracted in Eppendorf and then solution mixture was exposed under UV light with continuous stirring for 90 min. Every 15 min interval, 2 mL solution was extracted and then absorbance peak of MB at 665 nm was recorded using UV-vis absorption spectrophotometer. The photocatalytic efficiency was calculated as follow:

$$\text{Photodegradation efficiency (\%)} = (1 - C/C_0) \times 100$$

Where, C_0 is the concentration of dye at time "0" and C is the concentration of dye at time "t".

Results and Discussion

Structural studies

XRD pattern as shown in Figure 1 reveals the formation of hexagonal structure of Se nanoparticles with JCPDS Card No. 00-042-1425. Apart from Se nanoparticles, the other additional peaks observed at $2\theta = 270^\circ$ and 450° correspond to (111) and (220) crystal plane of ZnSe which is well matched with the JCPDF Card No. 00-005-0522. The presence of sharp and intense peaks confirmed the highly crystalline Se nanoparticles with average crystallite size of 86 nm.

The surface morphology of synthesized nanoparticles were examined by SEM analysis. SEM images as shown in Figure 2 clearly shows the nanoparticles were spherical as well

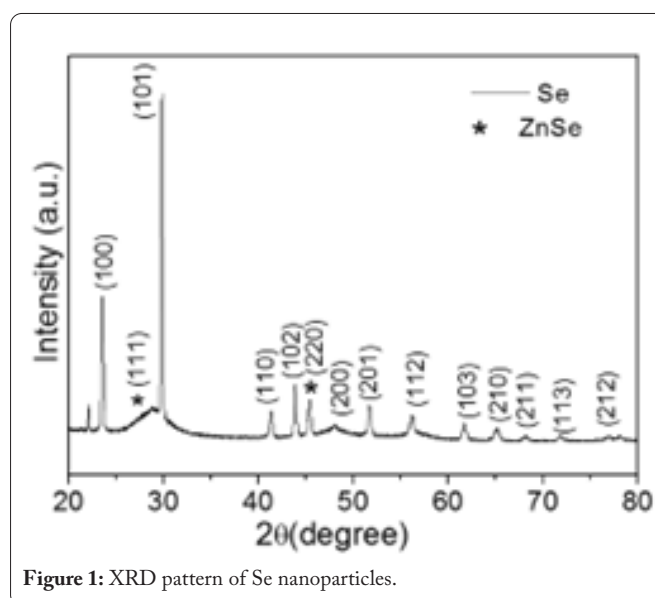


Figure 1: XRD pattern of Se nanoparticles.



Figure 2: SEM micrograph of Se nanoparticles.

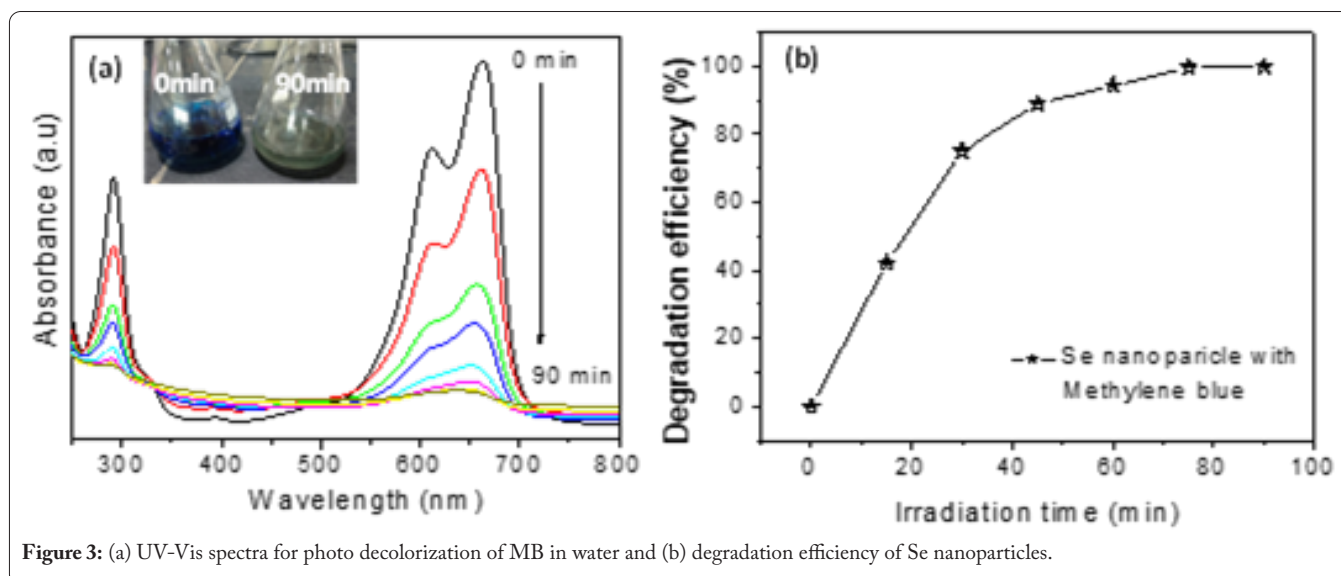


Figure 3: (a) UV-Vis spectra for photo decolorization of MB in water and (b) degradation efficiency of Se nanoparticles.

as hemispherical in shape. The average size of a Se nanoparticles is around 520 nm.

Photocatalytic Activity

To study the catalytic activity of Se nanoparticles, MB was chosen as model dye. The photocatalytic activity were studied under UV light irradiation to degrade the MB. When the sample is irradiated with UV light of energy either equal or greater than the band gap energy of sample, electrons from valence band (V.B) are excited to conduction band (C.B) and leaving holes in V.B. Thus, electrons in the C.B are responsible for reduction of aqueous solution to produce superoxide radical ions and holes in V.B of sample is accountable for oxidized water molecules to generate hydroxyl radicals. These generated radical species are responsible for degradation of MB. Figure 3 shows photo-decolorization of MB under UV light irradiation. The time dependent absorption spectra of MB over Se nanoparticles (Figure 3a) reveals that absorbance peak of measured at 665 nm decreases with time and decolorized dye into 90 min under UV light irradiation as shown in inset of Figure 3a. It was observed that 99% of dye molecules were degraded after 90 min under UV light irradiation (Figure 3b). Thus, Se nanoparticles exhibits much higher catalytic performance by minimizing the electron hole pair recombination rate.

Conclusions

A simple soft chemical approach was adopted for successful synthesis of Se nanoparticles. The XRD technique confirmed the formation of Se nanoparticles along with ZnSe. SEM image of as-synthesized nanoparticle confirms the spherical shape particle of size 520 nm. The nanoparticle was investigated for decolorization of MB dye under UV light irradiation. The photodegradation efficiency about 99% achieved in 90 min.

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