Optik 127 (2016) 8019–8023

Contents lists available at ScienceDirect

Optik

journal homepage: www.elsevier.de/ijleo

Synthesis and characterization of nano spheres decorated silver bromide nanorods using a two step chemical reduction route

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ARTICLE INFO

Article history:
Received 11 April 2016
Accepted 1 June 2016

Keywords:
Nanostructured materials
Chemical synthesis
Transmission electron microscopy
X-ray diffraction

ABSTRACT

We report the preparation of nanospheres protected silver bromide nanorods by direct chemical reduction method with silver nitrate as the metal precursor and sodium borohydride as reducing agent with CTAB doing twin role both as stabilizing agent as well as the source for bromide ion. The peaks in the XRD pattern are in good agreement with the standard values of the face-centered-cubic form of metallic silver bromide and no peaks of other impurity phases were detected. The energy-dispersive spectrum of the nanocolloidal dispersion confirmed the presence of elemental silver and bromide in the synthesized product. The morphology of silver bromide nanoparticles were determined by scanning electron microscopy. TEM observations exhibit astonishing revelation that the colloidal dispersion consist of AgBr nanorods embedded in spherical particles with a particle size distribution in 10–20 nm range.

1. Introduction

Metal nanoparticles demonstrate fascinating optical, electronic, magnetic and chemical properties that differ significantly from their bulk counterparts because of their small size and high surface area to volume ratio [1–3]. To design sophisticated devices in nano regime for various exciting applications, control of the size and morphology of nano crystals is utmost essential. Shape and size control provides an important strategy for designing metallic nanostructures judiciously with novel properties, owing to the strong dependence of physical and chemical properties of metallic nanostructures on their size and shapes. In particular, the formation of nano-sized anisotropic shapes such as rods and wires with tunable optical and electronic properties have been the focus of rigorous research due to their fascinating size and morphology dependent physical and chemical properties and multifaceted application potentials in electronics, sensing, catalysis, and photonics [4–9]. In recent years, silver bromide nanoparticles, hereafter referred to as AgBr NPs, have been found to inhibit bacterial growth in aqueous and solid bromide media because of their remarkable high reactivity due to the large surface to volume ratio [10,11]. Materials containing silver bromide can be thoughtfully employed to get rid of microorganisms on textile fabrics and they can be efficiently used for water treatment [12]. However, the most important application of silver bromide is its use as photographic material and catalysts [13,14]. The aforesaid potential applications make it mandatory that, in developing the routes of synthesis, utmost emphasis ought to be on how to control the size and morphology of silver

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http://dx.doi.org/10.1016/j.ijleo.2016.06.015
0030-4026/

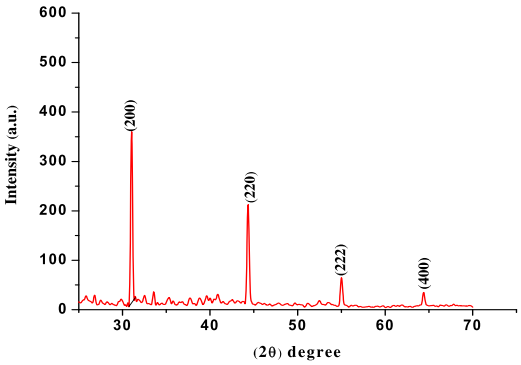

Fig. 1. X-ray diffractogram of silver bromide nanostructures.

bromide nanoparticles. Nevertheless, inspite of several astonishing industrial applications, the AgBr nanoparticles in real time devices were not realized as it is difficult to obtain stable colloidal dispersions in aqueous medium [15–17]. Reports available in the literature indicate that AgBr NPs have been produced using different methods: electrochemical method, thermal decomposition, laser ablation, microwave irradiation, chemical reduction method and sonochemical synthesis. However, the simplest and the most commonly used technique for the production of AgBr NPs is the chemical reduction of metal salts, wherein the reduction of an ionic salt is done in an appropriate medium in the presence of surfactant and reducing agent. Considering its prospective industrial applications, we make an attempt to prepare stable silver bromide nano rods conscientiously by reducing an aqueous solution of $AgNO_3$ with $NaBH_4$ in the presence of cetyl trimethyl ammonium bromide (CTAB). In our experiments, as $NaBH_4$ is a strong reducing agent which reduces Ag^+ to Ag atoms and Ag NPs are highly unstable, Ag atoms could have been converted to Ag^+ in the presence of Br^-. As a result, AgBr NPs are formed. Interestingly, TEM photographs gave a surprising revelation that the colloidal dispersions of AgBr rods were protected by self assembled nano spheres. The synthesized silver bromide nano structures were investigated by various spectroscopic and microscopic techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDS) and transmission electron microscopy (TEM).

2. Experimental

2.1. Material synthesis

All chemicals were used as received without further treatment. In the direct chemical reduction scheme, AgBr NPs were prepared by reducing an aqueous solution of $AgNO_3$ with $NaBH_4$ in the presence of a cationic surfactant, cetyltrimethyl ammonium bromide(CTAB) as a stabilizer. The following procedure has been followed. The silver nitrate (0.02 M, 5 mL) salt solution was vigorously stirred for 15 min using a magnetic stirrer at room temperature. The cetyltrimethyl ammonium bromide (0.03 M, 10 mL) is rapidly injected into this mixture. The color changed from colorless to milky white after another 15 min stirration. Then sodium borohydride (0.03 M, 10 mL) was rapidly injected into this mixture with vigorous stirring using a micro pipette to initiate the reduction of precursor and during this process, the colloidal solution turned deep yellow from milky white due to the formation of small AgBr NPs. After another half an hour stirring, the solution became wine-red color. After aging of one day, 10×2 mL centrifuge tubes were filled with this solution and centrifuged at a speed of 10000 rpm. The CTAB acts both as the source for bromide ion and surfactant. Though the preparation of AgBr NPs by simply mixing CTAB and $AgNO_3$ only involves one step, the present method involves two reactions, initially Ag^+ reduces to Ag atoms by the strong reducing agent $NaBH_4$, and then Ag atoms converted to Ag^+ in the presence of Br^- followed by Ag^+ to AgBr NPs. Moreover, it was noticed in our experiments that the order of chemical mixing affects the resulting colloidal particle's size and size distribution. The procedure that involved first mixing $AgNO_3$ and CTAB and then adding sodium borohydride to the mixed $AgNO_3$—CTAB solution gave the most stable colloidal dispersion and the narrowest particle size distribution as seen in the UV–vis spectrum. The final deposited denser product was washed several times with distilled water and acetone to remove unreacted compounds. The powder was then dried at 65 °C for 15 min in a hot air oven and the final product is used for characterization.

3. Results and discussion

3.1. X-Ray diffraction analysis

The X-Ray diffraction analysis has been recorded by Enraf Nonius CAD4-F diffractometer to confirm the crystallinity and also grain size. The X-Ray diffraction patterns for the various 2θ values (Fig. 1) of the product demonstrate that the silver bromide is crystalline in nature, and the diffraction peaks match those of the cubic silver bromide phase in the Joint