

One-pot photochemical synthesis of silver nanodisks using a conventional metal-halide lamp



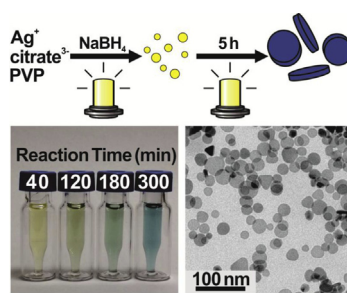
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HIGHLIGHTS

- Monodispersed silver nanodisks are photochemically synthesized using a metal-halide lamp.
- The synthetic conditions of the silver nanodisks are thoroughly investigated.
- The silver nanodisks have surface-affinities with halide ions and are catalytically active.
- Large-scale synthesis of the silver nanodisks is demonstrated.

GRAPHICAL ABSTRACT



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ABSTRACT

One-pot photochemical synthesis of highly monodispersed silver nanodisks (AgNDs) has been developed using a conventional metal-halide lamp. The synthetic conditions, including the roles of each reactant, were thoroughly investigated to disclose what actually determines the quality of the AgNDs. Importantly, to efficiently control the size of the AgNDs under polychromatic conditions at a constant reaction time, we developed a “dilution-control” method by combining the conventional seed-mediated method and our photochemical growth, and we successfully controlled the size of the AgNDs from 15 nm to over 60 nm. We further investigated the chemical properties of the AgND surfaces, which clearly demonstrated their distinctive affinities with halide ions and corresponding optical changes depending on halide species, and their catalytic activity for the reduction of 4-nitrophenol. Most of all, the cost-effective instrumentation, in combination of the large-scale synthesis, possesses a number of advantages over the conventional costly laser-based or multiple LED-based synthesis, which would be useful for the further investigation of the photochemical synthesis of other anisotropic silver nanomaterials.

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1. Introduction

Over the past few decades, non-spherical silver nanostructures [1–5] such as nanocubes [6–10], nanowires [11–13], nanodecahedra [14], nanorods [15–17], and nanoplates [18–21] have been intensively investigated owing to their unique physical and chemical properties. Among these various silver nanostructures,

silver nanoplates have attracted significant interest [22] because of their controllable properties associated with localized surface plasmon resonance (LSPR) [23–25], surface-enhanced Raman spectroscopy (SERS) [26–29], and surface tailorability [30–32]. To date, two main synthetic methods for the planar silver nanostructures have been developed: (1) chemical reduction of Ag^+ using reductive reagents [20,33–35] and (2) photochemical reduction using light illumination [19,36–38]. In particular, photochemical methods have demonstrated how to control the morphologies of the silver nanoplates by changing the wavelength of the light [37,39,40]. In general, this type of morphology control is

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conducted by laser devices that irradiate monochromatic and coherent light to interact with silver nanoparticles and their surface plasmon excitations, resulting in controllable sizes and narrow size-distribution of silver nanoplates [41]. In spite of the advantages of the photochemical synthesis of silver nanoplates, however, the high cost of the laser devices has been a major hurdle for high-throughput production and widespread investigation. Moreover, the limited volume of the reaction mixture in accordance with the focused laser beam has been a major drawback for the large-scale synthesis of the nanoplates, and thus, their extensive applications. To solve these problems, several non-focused and “cost-effective” light sources have been examined for the synthesis of silver nanoplates with comparable or better qualities. For example, a conventional fluorescent lamp emitting white light (350–700 nm) was used as a single light source to synthesize silver nanoplates with different sizes [37]. In spite of the reasonably moderate reaction time (~400 min), however, this method eventually had to regulate the irradiation conditions to specifically control the nanoplate sizes using colored glass filters. Later on, a sodium lamp exhibiting an emission main band in the range of 560–620 nm was used without filters to synthesize silver nanoplates with different shapes by controlling the irradiation time [42]. This method takes advantage of time-dependent control of the morphology of the silver nanoplates; however, this method requires a lengthy reaction time, often over 500 min. Light-emitting diode (LED) lamps with different emission wavelengths (spectral half-width = 30–80 nm) have also been employed to photo-excite citrate anions and accordingly reduce Ag^+ [40,43]. The size and shape of the silver nanoplates were photochemically controlled by controlling the emission wavelengths, but it still required the preparation of multiple lamps emitting lights at the desired wavelengths. Similar to the laser-based photochemical synthesis, this method essentially relies on the nearly monochromatic nature of the irradiation around a specific wavelength of an LED lamp, where the maximum light intensity is obtained. To date, such complicated instrumentation or lengthy synthetic procedures have been the major obstacles for the non-laser-based photochemical synthesis of silver nanomaterials. Therefore, the development of a cost-effective and convenient strategy for the synthesis of size-controlled, large-scale, and monodispersed silver nanoplates would be an important major advance in the field.

Herein, we present the one-pot photochemical synthesis of discoidal silver nanoplates, or silver nanodisks (AgNDs), with controllable sizes using white light from a conventional metal-halide lamp [44]. This method takes advantage of (1) photochemical reduction by citrate anions, (2) shape control by PVP, and (3) size control assisted by the conventional seed-mediated growth method. To elucidate the synthetic mechanism, we thoroughly investigated the experimental conditions, each of which was demonstrated to dramatically affect the morphology of the AgNDs. Importantly, the synthesis eliminated the necessity of expensive and complicated instrumentation, while still providing high-quality monodispersed AgNDs in a large scale.

2. Materials and methods

2.1. Materials

Trisodium citrate dihydrate (cat.#S4641), sodium borohydride (NaBH_4 , cat.# 480886), silver nitrate (AgNO_3 , cat.# 204390), polyvinylpyrrolidone (cat.# PVP40; M.W.40,000, cat.# 81440; M.W. 360,000), a poly(sodium 4-styrenesulfonate) solution (cat.# 434574; M.W. 1,000,000), silver trifluoroacetate (cat.# 482307), poly-L-histidine (cat.#P9386), Triton™ X-100 (cat.#X100), diethylaminoethyl-dextran hydrochloride (cat.#D9885), a poly(acrylic acid, sodium

salt) solution (cat.# 416037), and a poly(styrene-*alt*-maleic acid) sodium salt solution (cat.# 662631) were purchased from Sigma–Aldrich (MO, USA). Ultrapure water was provided by a Direct-Q3® system (Millipore; Billerica, MA). The metal-halide lamp was purchased from Koryo Industry (Seoul, Republic of Korea).

2.2. Synthesis of the AgNDs

Aqueous solutions of PVP (0.015 mL, 0.05 M), AgNO_3 (0.2 mL, 0.005 M), and trisodium citrate (0.5 mL, 0.05 M) were added to 7 mL of pure water in a vial. Then, a NaBH_4 solution (0.08 mL, 0.1 M) was injected into the prepared solution with vigorous stirring; the color of the solution rapidly turned pale yellow. The reduced solution was immediately exposed to a 400 W metal-halide lamp (distance: 50 cm). As the exposure time increased, the pale yellow solution gradually changed first to bright yellow, then to green, and finally to blue. When the color of the solution turned blue, the overall reaction was determined to be over. Finally, the AgNDs were washed by repeated centrifugation at 13,000 rpm for 10 min. The supernatant was removed and the AgNDs were redispersed in a citrate solution (0.05 M). In case of truncated triangular and hexagonal structures, the diameter of truncated triangular and hexagonal structures was obtained from a hypothetical circle that minimally covers their entire area for convenience.

2.3. Dilution-control of the particle size

The size of the AgNDs was controlled by the dilution-control method as follows: The same initial mixture of AgNDs after the injection of NaBH_4 was allowed to react while exposed to the metal-halide lamp for 30 min for the formation of silver nuclei, after which the mixture was diluted with the growth solution (silver nitrate 0.128 mM, sodium citrate 3.207 mM, PVP 10.757 mg/L) for the dilution of the silver nuclei and the corresponding enlargement of the AgNDs; the final mixture was illuminated by the metal-halide lamp for 4.5 h to facilitate full growth of the AgNDs.

2.4. UV–vis spectroscopy

The UV–vis spectra of the AgNDs were acquired at 25 °C from 300 to 900 nm using an Agilent 8453 spectrophotometer.

2.5. Transmission electron microscopy (TEM)

TEM samples were prepared by placing a 2 μL drop of the sample-containing solution onto a carbon-coated Formvar copper grid (400 mesh, Electron Microscopy Sciences). The grids were allowed to dry at room temperature. TEM images were obtained using TECNAI G2 F30ST (FEI) operated at 300 kV or TECNAI 20 operated at 200 kV.

3. Results and discussion

3.1. Standard synthesis and properties of AgNDs

The typical synthesis began with the preparation of the aqueous mixture containing four chemical reagents (silver nitrate 0.128 mM, sodium citrate 3.207 mM, PVP 10.757 mg/L, and sodium borohydride 1.026 mM). At the very beginning of the reaction, the mixture was almost colorless because the initial four chemical reagents are all colorless. After the addition of NaBH_4 , the mixture immediately turned pale yellow and gradually became intense yellow, indicating the formation of silver nanoparticles (diameter < 5 nm) that would subsequently play a role as seeds for further particle growth. As the reaction proceeded under illumination of white light from the

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