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Short communication

Synthesis of Ag and Ag–SiO_2 nanoparticles by γ-irradiation and their antibacterial and antifungal efficiency against *Salmonella enterica* serovar Typhimurium and *Botrytis* cinerea

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Abstract

The Ag and Ag–SiO_2 nanoparticles were synthesized by γ-irradiation, and characterized by field-emission transmittance electron microscopy (FE-TEM), field-emission scanning electron microscopy (FE-SEM), and energy dispersive X-ray spectroscopy (EDXS). The electron microscopy images show that well-dispersed Ag nanoparticles of about 7 nm were attached to the surface of SiO_2 nanoparticles of about 350 nm. Antibacterial efficiency of the Ag–SiO_2 nanoparticles was tested against *Salmonella enterica* serovar Typhimurium by measuring the optical density (OD). Without Ag–SiO_2 particles, the *S. enterica* serovar Typhimurium grow gradually, and reach a steady state (fully grow) in about 6 h. At the presence of Ag–SiO_2 particles at 50 ppm, their growth became much slower, reaching a steady state after about 24 h. With Ag–SiO_2 particles at 100 ppm, they did not grow fully even after 58 h. The antifungal efficiency of the Ag–SiO_2 nanoparticles against *Botrytis* cinerea was about 65.0, 99.9, and 99.9% at the concentrations of the particles of 10, 50, and 100 ppm, respectively.

Keywords: γ-Irradiation; Ag nanoparticle; Ag–SiO_2 nanoparticles; Antibacterial efficiency; Antifungal efficiency; *Salmonella enterica* serovar Typhimurium; *Botrytis* cinerea

1. Introduction

Silver compounds have been exploited for their medicinal properties for centuries [1] and it has been known for long time that silver is an effective antimicrobial agent. Gibbard first systematically investigated the antimicrobial activity of silver [2]. He found that if silver was used to be coating cloth or paper, it becomes inactive. Today, silver sulfadiazine is used for topical treatment of burn-wounds [3], and silver nitrate is still used as a prophylaxis in neonatal ophthalmic [4]. Silver nanoparticles have been used as a catalyst for reduction of aromatic nitro compounds [5]. However, little has been reported on the use of silver nanoparticles as an antibacterial or antifungal agent.

Nanometer-sized particles of metals (metallic nanoparticles) and semimetals show interesting characteristics in various properties including optical nonlinearity, specific heat and magnetic properties, which are quantitatively and qualitatively different from those of their respective bulk materials [7,8].

In our previous reports, various nanometer-sized particles have been synthesized by γ-irradiation for use as a catalyst [9-11]. It has been found that hydrated electron (e_{aq}^-), H_3O^+, $H^\bullet$, H_2, (OH, and H_2O_2 are generated in aqueous solution during γ-irradiation. Metallic nanoparticles were produced by reduction of metallic ions by hydrated electrons generated during γ-irradiation. However, the preparation of Ag–SiO_2 nanoparticles by γ-irradiation has not yet been reported.

In this study, Ag and Ag–SiO_2 nanoparticles were prepared by γ-irradiation by reduction of silver ions in SiO_2 colloid solution. The products were characterized by field-emission trans-

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mittance electron microscopy (FE-TEM), field-emission scanning electron microscopy (FE-SEM), energy dispersive X-ray spectroscopy (EDXS) and X-ray diffraction (XRD). In addition, the antibacterial and antifungal efficiency of the Ag–SiO_2 nanoparticles was examined against *Salmonella enterica* serovar Typhimurium and *Botrytis* cinerea, respectively.

2. Experimental

2.1. Chemicals

Silver nitrate ($AgNO_3$) was obtained from Kojima Chemicals Co. Ltd. (Japan). The polyvinylpyrrolidine (PVP, M_w = 10,000), and tetraethylorthosilicate (TEOS) were purchased from Sigma-Aldrich Co. All other chemicals were in reagent grade, and were used without further purification.

2.2. Synthesis of silica nanoparticles by sol–gel method [6]

SiO_2 nanoparticles were synthesized at the molar composition of: 0.3 TEOS/1.0 NH_3/4.4 H_2O/17.0 EtOH by the following procedure. An aqueous solution of NH_4OH (40 mL, 28%) was added to a mixture of deionzed water (80 mL) and ethanol (1000 mL), and then stirred by a mechanical stirrer at 200 rpm. To this clear solution was added 60.0 mL of TEOS while stirring and maintaining pH at 9.5. White SiO_2 nanoparticles were formed after 6 h. The particles were obtained by using centrifuge at 1600 rpm, and washed thoroughly with deionized water and EtOH. The particles were dried at room temperature, and then characterized by energy filtering transmission electron microscope (EF-TEM).

2.3. Synthesis of Ag and Ag–SiO_2 nanoparticles by γ-irradiation

The PVP-stabilized Ag nanoparticles were synthesized by reduction of $AgNO_3$ as described in a previous report [12]. Briefly, $AgNO_3$ (ca. 1.0×10^{-3} M) was dissolved in distilled water in the presence of PVP (M_w = 10,000) as a colloid-stabilizer. Nitrogen gas was bubbled through the solution for 30 min to remove oxygen. Then the solution was irradiated by γ-ray (Co-60 source) under atmospheric pressure and ambient temperature, where the total irradiation dose of 30 kGy (a dose rate = 6.48×10^5 h^{-1}) was used.

Scheme 1 shows the procedure for preparation of Ag–SiO_2 nanoparticles by γ-irradiation. Briefly, the $AgNO_3$ (1.0 M), silica nanoparticles, EtOH (70 mL) and 2-propanol (10 mL) as hydroxyl radical scavenger were mixed and dispersed. Nitrogen gas was bubbled through the solution for 30 min to remove oxygen, and the solution was irradiated by γ-ray at the same condition as that used for the synthesis of Ag-nanoparticles.

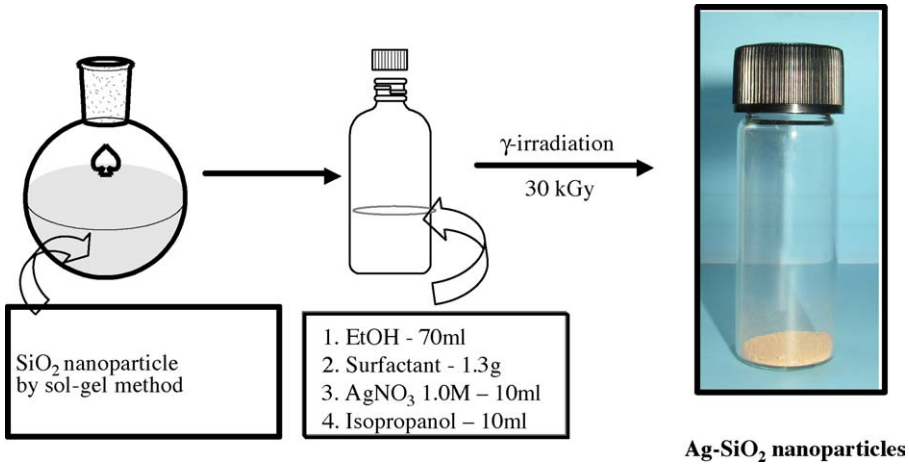
2.4. Antibacterial and antifungal efficiency examination of Ag–SiO_2 nanoparticles against S. enterica serovar Typhimurium and Botrytis cinerea

To examine the antibacterial efficiency of the Ag–SiO_2 nanoparticles, the *S. enterica* serovar Typhimurium was inserted in Luria-Bertani (LB) broth medium of 3.0 mL containing various concentrations of Ag–SiO_2 nanoparticles (6.25, 12.5, 25, 50, and 100 ppm), and cultured by a shaking incubation (37 °C, 200 rpm) for 58 h as described in Table 1 (will be discussed in detail later). The solution was diluted to 1/100 to determine the optical density (OD), which was measured by a spectrophotometer at the wavelength of 600 nm.

To examine the antifungal efficiency of the Ag–SiO_2 nanoparticles, the *Botrytis* cinerea was vaccinated to potato dextrose agar at the presence of Ag–SiO_2 nanoparticles at various concentrations (10, 50, and 100 ppm), and then cultured by incubation for 5 days.

2.5. Characterization

TEM photographs of the PVP-stabilized Ag and Ag–SiO_2 nanoparticles were obtained by a FE-TEM (JEM-2100F, JEOL Co. Ltd., Japan) and a EF-TEM (EM 912 Omega, Carl Zeiss,



Scheme 1. Preparation procedure of Ag–SiO_2 nanoparticles using γ-irradiation.