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Au–Ag core–shell nanoparticles with controllable shell thicknesses for the detection of adenosine by surface enhanced Raman scattering

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ABSTRACT

In this study, we report a feasible aqueous chemical method to synthesize Au core–Ag shell nanoparticles (NPs) by seeding growth approach. The concentration of Au NPs plays an important role in growing the different thicknesses of Ag shells. The design of signaling aptamer utilizes the target-induced switching between an aptamer/DNA duplex and an aptamer/target complex. The surface enhanced Raman scattering (SERS) signal was enhanced upon the hybridization of the Raman reporter-labeled DNA released from the target-induced displacement with the captured DNA immobilized on the silicon substrate with Au–Ag core–shell NPs. This substrate with 12.4 nm Ag shell shows high reproducibility, strong enhancement, and a low detection limit (1 nM). The enhancement in DNA-based adenosine detection properties shall be advantageous in applications for other aptamer sensing systems.

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1. Introduction

Bimetallic core–shell nanoparticles (NPs) have attracted extensive interest due to the addition of a second metal shell, providing a method to control chemical or physical properties of the NPs [1–4]. Core–shell NPs have played an important role in changing the surface plasmon band, enhancing the stability and dispersion of the colloid, and regulating the magnetic, optical, or catalytic properties [1–8]. These properties strongly depend on the composition, shapes, and sizes of core–shell NPs [9–14]. Bimetallic core–shell NPs have been synthesized through various methods, such as electroless plating [15], surface reaction [16], seeding-mediated synthesis [1,14], and self-assembly [8]. Among them, seeding-mediated synthesis is a typical and easy approach in preparing bimetallic core–shell NPs [1,14]. The seeding-mediated synthesis strategies based on the temporal separation of nucleation and growth processes are considered to be very efficient method for controlling the sizes and shapes of bimetal core–shell NPs [14]. For the seeding-mediated growth method, low temperature, and solution-phase synthesis have the potential for low cost and industrial-scale fabrication.

Nobel metallic NPs composed of free-electrons such as Au and Ag are known to provide strong resonance optical responses to irradiation by light, which result in amplification of light-induced processes undergone by molecules localized on their surfaces, such as Raman scattering, giving rise to surface-enhanced Raman scattering (SERS) [17,18]. SERS is a highly sensitive and selective technique that allows for detection of chemical or biological molecules in very low concentration and provides rich structural information [19]. The enormous enhancement in SERS can be attributed to electromagnetic and chemical enhancement mechanisms [20]. Electromagnetic enhancement arises due to the localized surface plasmon resonances (LSPR) mode, which can focus light into nanosized volumes, drastically increasing the electrical field intensity near the nanoparticles. Chemical enhancement arises from interactions between the molecule and the nanoparticles as a result of changes to the molecular electronic states. The electromagnetic mechanism is typically thought to contribute most of the enhancement (10^5–10^8), and the chemical enhancement contributes rather less (10–10^3) [18]. The size, morphology, and composition of the surface plasmon band should aid in controlling the intensity of SERS [19,21]. Comparison of the calculated and measured surface plasmon extinction spectra were repeatedly employed as one of the criteria in distinguishing between the alloy and core–shell structure of the bimetallic Au–Ag NPs [21]. A strategy for extracting optical constants of the Au–Ag core–shell NPs from their measured surface plasmon extinction spectra has been recently reported [22]. Furthermore, it was shown that Au–Ag core–shell NPs can be employed as SERS-active surfaces. SERS enhancement as a function of the core–shell Au–Ag NPs

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composition and the state of their aggregation were investigated using pyridine and other types of probe adsorbates [23,24]. However, understanding the effect of shell thickness on SERS signals and the synthesis of a thicker Ag shell without aggregation is still unclear.

The present study has successfully synthesized the different shell thicknesses of Au–Ag core–shell NPs by the seeding-mediated method. The appropriate concentration of Au NPs and sodium citrate are important for growing the different thicknesses of Ag shells in an aqueous solution. The silicon substrate with self-assembled Au–Ag core–shell NPs can be used to detect adenosine by a structures-switch aptamer. The fabrication of DNA-based adenosine sensors is facile, high reproducibility, stability, and low detection limit (1 nM), which shall be advantageous in applications for other aptamer sensing systems.

2. Experimental

2.1. Chemical reagents

Hydrogen tetrachloroaurate(III) trihydride, ethanol, hydrogen peroxide, adenosine, and phosphate buffered saline (PBS) were purchased from SIGMA. Silver nitrate, trisodium citrate dihydride, and sulfuric acid were purchased from J.T. Baker. 3-Aminopropyltrimethoxysilane (APTMS) and 3-mercapto-1-hexanol (MCH) were purchased from Alfa Aesar. The three types of deoxyribonucleic acid (DNA) were synthesized by MDBio, Inc. All chemicals were of analytical grade and used as received. De-ionized water (>18 MΩ/cm) was used throughout the experiments.

2.2. Apparatus

The morphology of nanostructures was examined with a field emission scanning electron microscope (FESEM) using a JEOL JSM-6700F SEM operating at 10 kV accelerating voltage. A JEOL-2010 transmission electron microscope (TEM) operating at 200 kV was used to examine the microstructures. An electron dispersive spectrometer (EDS) attached to the TEM was used to determine the composition of NPs. A Hitachi U-2900 UV–vis spectroscopy was employed to characterize the optical properties of NPs. The zeta potential was measured by Delsa^{TM} Nano (Brookhaven, USA). The Raman spectra were performed by Confocal Raman Microscope (HORIBA, LabRAM HR) at room temperature in the backscattering configuration. The source light was He–Ne laser emitting at a wavelength of 632.8 nm.

2.3. Preparation of Au–Ag core–shell NPs

The Au NPs were synthesized by adding 1 mL of 38.8 mM trisodium citrate to 10 mL of a boiling aqueous solution of 1 mM hydrogen tetrachloroaurate (III) trihydride under vigorous stirring. After reaching a deep red color, boiling and stirring were continued under refluxing for 10 min and then cooled at room temperature. Next, different volumes of prepared Au NPs were diluted with 8 mL of de-ionized water and reheated to a boil, followed by adding 1 mL of 100 mM trisodium citrate and vigorous stirring. Finally, 2 mL of 5 mM silver nitrate was added, and the boiling continued for 10 min. The Au–Ag core–shell NPs solution was cooled and stored at room temperature. In the second step, the double core–shell structure was converted from Au NPs to Au–Ag core–shell NPs.

2.4. Preparation of substrate

Si (1 0 0) wafers were cleaned in a boiling piranha solution (H_2O_2:H_2SO_4, 3:7, v/v) for 10 min, and then rinsed with de-ionized water and ethanol. The substrates were immersed into an ethanol solution containing 5 mM APTMS, and then heated under reflux for 2 h. The substrates were rinsed with ethanol and de-ionized water, and then dried under a N_2 purge. The Au NPs or Au–Ag core–shell NPs were deposited onto the amino-terminated bonding by using an immersion method. After immersing the substrates in a solution of Au NPs or Au–Ag core–shell NPs for an appropriate time, the substrates were immediately rinsed with de-ionized water and dried under a N_2 purge.

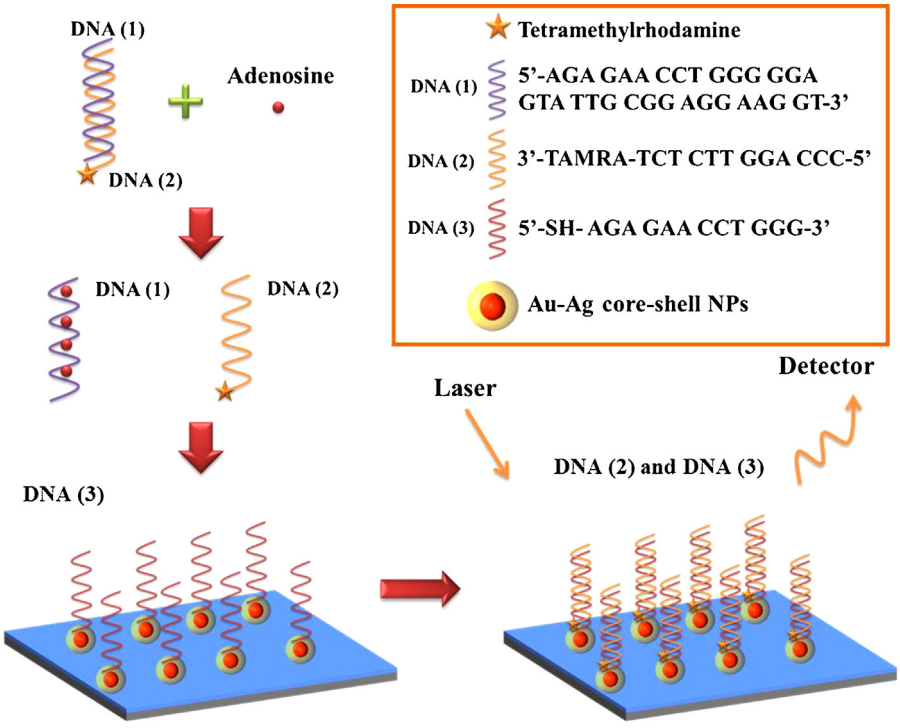

Fig. 1. Schematic diagram describes the preparation of sensing system for adenosine.

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