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Facile Synthesis and Characterization of Au-Urchin@Ag Core-Shell Nanoparticles

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ABSTRACT

Core-shell structures of numerous metal nanoparticles have captured significant research attention; however, a proper control of their synthetic approach, morphologies and optical properties still remains challenging. Herein, an anisotropic coating of silver onto sea urchin-like gold nanoparticles so called Au-urchin@Ag core-shell nanoparticles were facilely prepared based on the seed mediated growth approach at room temperature. Ascorbic acid was used as green reductant for the coating of Ag shell onto pre-synthesized urchin gold seeds in the presence of polyvinyl pyrrolidone as capping agents. The Au-urchin@Ag core-shell nanohybrids exhibit a highly anisotropic morphology with tunable shell thickness and optical properties in the range 650–800 nm. Controlling the reaction rate by modulating the reaction pH plays a decisive role to form core-shell structure. Furthermore, the Au-urchin@Ag nanohybrids display a gradual blue-shift in the spectral response upon increasing the amount of $AgNO_3$ precursor or reducing the dosage of Au-urchin seeds. The synergistic effect derived from the coupling of anisotropic silver shell and spiky shaped gold cores with multiple generated hot-spots suggests their great potential for plasmonic sensing, catalysis and biological applications.

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1. Introduction

Noble metal nanoparticles especially gold and silver nanoparticles have captured tremendous research attention due to their intriguing structure dependent optical and electronic properties [1]-[5]. To date, various structures of gold and silver nanoparticles including spheres, rods, cubes, spikes, prism, and spikes etc. have been successfully prepared and demonstrated a great potential in numerous applications such as plasmonics, thermaltherapy, and bio-imaging [1], [6]-[9]. Among them, urchin-like which are also referred as spiky, thorny, or branched nanoparticles are of particular interest due to the existence of multiple hot-spots from sharp edges and corners on their surface [10]-[14]. These hot-spots can concentrate and induce a huge electromagnetic field enhancement when their intrinsic plasmon absorbance peak matches the excitation wavelength [15]. Therefore, the synthesis of metallic nanoparticles with anisotropic morphologies and controllable optical properties is of great scientific and technological importance [16], [17].

Furthermore, it is well accepted that the optical properties of metallic nanoparticles have a strong correlation to their morphology and compositions [18], [19]. Although gold is preferred for plasmonic sensing and biological applications due to its broader optical tunability, high chemical stability and bio-compatibility, silver generally exhibits better plasmonic sensitivity and electromagnetic field enhancement attributed to its greater scattering intensity [20], [21]. However, the aqueous synthesis of silver nanoparticles with complex morphologies and tunable optical response in the Vis/NIR region remains challenging due to the fast diffusion rate of Ag atoms [19]. Consequently, many studies have exploited the coupling of silver and gold to form bimetallic nanoparticles with tunable optical properties, improved sensitivity and stability [20], [22]-[25]. While the uniform deposition of silver on various gold nanostructures have been successfully demonstrated, [26]-[29], recent studies have focused on developing silver coating on urchin-like gold nanoparticles with few examples have been reported [19], [21], [30], [31]. For example, the researchers successfully prepared a uniform silver layer of 2–10 nm deposited onto urchin gold nanoparticles by polyol process at 150 °C in which pentanediol was used as reducing agent in the presence of polyvinyl pyrrolidone as capping agent [19]. Besides, hexadecyltrimethylammonium bromide (CTAB) was also widely exploited as stabilizing agent in the synthesis of AuAg bimetallic nanoparticles, though it can cause difficulty for further surface modification [21], [30]. Furthermore, the effect of different factors on the growth of silver shell with controllable thickness and optical properties still requires more elucidation [18], [20].

Herein, we reported on a facile and controllable coating of anisotropic silver shell onto sea-urchin-like gold nanoparticles, so-called Au-urchin@Ag core-shell nanoparticles via seed mediated growth approach at room temperature. The urchin-like gold nanoparticles were pre-synthesized based on co-reduction of $HAuCl_4$ and $AgNO_3$ by ascorbic acid and in the presence of gold nanospheres. These urchin-like gold nanoparticles were subsequently used as templates for the further growth of silver shell with controllable thickness and optical properties. The effects of different reaction parameters including the initial pH and the dosages of Au-urchin seeds and $AgNO_3$ precursors on the morphology and corresponding optical properties of the resulting nanoparticles were also examined.

2. Materials and Methods

2.1. Materials

Gold (III) chloride hydrate ($HAuCl_4 . xH_2O$, ~52% basis), silver nitrate ($AgNO_3$, 99%), polyvinyl pyrrolidone (PVP, 360000 Da), and L-Ascorbic acid (99%) were purchased from Sigma Aldrich. Hydrochloric acid (HCl, 36%), sodium hydroxide (NaOH) and concentrated nitric acid (HNO_3) were purchased from Xilong Scientific Co., Ltd. (China). All chemicals were used as received without purification. All glassware were washed intensively with aqua regia solution ($HCl:HNO_3$, 3:1 v/v) and double distilled water prior to use.

2.2. Methods

Synthesis of gold nanospheres: the spherical gold nanoparticles were synthesized based on the Turkevich method [32]. Briefly, 1.525 mL $HAuCl_4$ (10 mM) was added into a 125 mL erlen containing 55.475 mL double distilled water under magnetic stirring. The solution was heated to boiling within a silicon bath. Afterward, 3 mL trisodium citrate (10 mg/mL) was quickly added. The color gradually changed from colorless to light pink and red wine within 25 minutes of vigorous stirring as the sign for the formation of gold nanospheres. Subsequently, the heater was removed whereas the dispersion was kept gently stirring until it cooled to room temperature. Finally, double distilled water was added to adjust the concentration of gold nanoparticles at 0.5 mM correlated to the amount of Au^{3+} and the dispersion was kept in refrigerator at 4 °C for further use.

Synthesis of sea urchin like gold nanoparticles (Au-urchin): the seed mediated growth approach was exploited to prepare sea urchin-like nanoparticles as previously reported with modification [9], [21]. In a typical synthesis, 1.2 mL spherical gold nanoparticles, 1.080 mL $HAuCl_4$ (10 mM), 0.3 mL $AgNO_3$ (10 mM), and 0.18 mL HCl (1.0 M) were added into a beaker containing 17.4 mL double distilled water under magnetic stirring for 5 minutes. Afterward, 0.9 mL of freshly-prepared ascorbic acid (10 mg/mL) was rapidly added under vigorous stirring. The color immediately changed to greenish blue which was attributed to the formation of sea urchin-like Au nanoparticles. After 2 minutes of stirring, 9.025 mL of PVP solution (10 mg/mL) was drop-wise added into the dispersion under gently stirring for 30 minutes to stabilize the as-formed nanoparticles. The stabilized urchin-like nanoparticles were finally kept in refrigerator at 4 °C for further use.

Synthesis of silver coating of sea urchin-like gold nanoparticles (so-called Au-urchin@Ag core-shell nanoparticles): Au-urchin@Ag core-shell nanoparticles were synthesized based on the seed mediated growth approach. Typically, an aliquot (1.0 mL) of as-synthesized Au-urchin nanoparticles, 0.34 mL PVP solution (10 mg/mL), and 0.05 mL of $AgNO_3$ solution (10 mM) were added into a vial containing 1.67 mL of double distilled water at various pH conditions and subsequently sonicating for 20 seconds to ensure complete dispersion. The initial pH was adjusted by adding different volumes of NaOH (10 mM) from a stock solution. After 5 minutes, 0.15 mL of ascorbic acid (10 mg/mL) was rapidly added and the above mixture was gently magnetic stirred for 1 hour. Finally, the as-prepared core-shell nanohybrids were collected by centrifugation and rinsed with double distilled water. This purification step was repeated twice to remove all unreacted agents.

2.3. Characterization

The morphologies of the as-synthesized nanoparticles were analyzed by TEM (JEM-1010). Their optical properties were recorded by UV-Vis (UH5300, Hitachi). The crystallography was performed by X-rays diffraction spectroscopy (XRD, Bruker D8 Advance) operated with Cu Kα radiation ($\lambda = 1.5406$ Å) at 40 kV and 30 mA and scan rate of 3 deg/min.

3. Results and Discussion

Herein, gold nanospheres synthesized by the citrate method exhibit spherical morphologies with a narrow size distribution of about 25 nm in diameter, Figure 1a. Consequently, their UV-Vis spectrum shows a sharp peak at ~525 nm which is a typical plasmon peak of gold nanospheres, Figure 1c. The resultant sea urchin-like gold nanoparticles (Au-urchin) developed from these gold nanospheres display an urchin-like shape with multiple spikes protruding from a spherical core. Their corresponding optical spectra displays a broader peak at ~787 nm which is the result of the plasmon hybridization of core particle and multiple tips, Figure 1b-c [33]. Interestingly, the shoulder peak observerd at ~525 nm is attributed to the solid spherical cores. Those urchin-like gold nanoparticles were utilized as templates for the growth of core-shell structures, so-called Au-urchin@Ag nanoparticles.

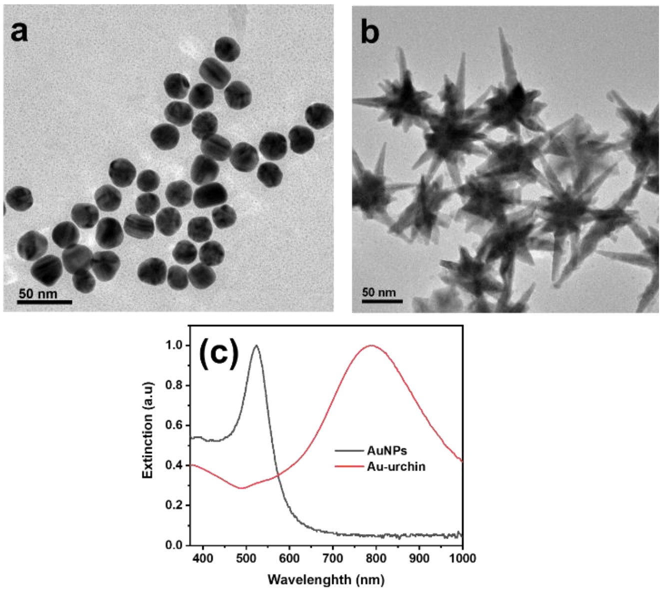

Figure 1. *TEM images of (a) spherical gold nanoparticles, (b) urchin-like gold nanoparticles (Au-urchin); and (c) the UV-Vis spectra of spherical gold nanoparticles and urchin-like gold nanoparticles, respectively. All the UV-Vis spectra were normalized to 0–1 for visualization.*

To elucidate the growth of silver coating of the Au-uchin core particles, we have investigated the effects of different parameters including the initial pH and the dosages of urchin-like seeds and $AgNO_3$ precussor. Herein, ascorbic acid, a mild reducing agent which is also known as vitamin C, was used to trigger the reduction of Ag^+ ions in the presence of Au-urchin nanoparticles. Recently, ascorbic acid is widely used as reductant in the green synthesis of various metallic nanoparticles [34]. The reducing ability of ascorbic acid is derived from ascorbate ions which are their favorable form in alkaline condition [35]. To understand the effect of pH on the reducing power of ascorbic acid, we have prepared Au-urchin@Ag nanoparticles at various pH conditions. The reaction was extended to 2 days in which the LSPR spectra of as-synthesized nanoparticles was collected and monitored after each 1-day interval. We found that with pH lower than 10, ascorbic acid was unable to reduce Ag^+ ions since there is no obvious change in the spectral response of Au-urchin was observed. At pH 10, the freshly prepared Au-urchin@Ag hybrids displays a LSPR peak at 692 nm. However, this peak shifted back to 680 nm after 1-day storage, Figure 2a-b, possibly due to the slow reaction rate at this pH condition. At pH 11, the freshly prepared Au-urchin@Ag bimetallic nanoparticles exhibit a LSPR peak at ~680 nm, Figur 2a-c. This peak was kept constant during 2-day observation, suggesting the quick completion of reaction at this pH condition. A same trend was also observed for the Au-urchin@Ag hybrids synthesized at pH 12 in which the freshly prepared bimetallic nanoparticles display a consistent absorption peak at ~685 nm shortly after being synthesized, Figure 2a-d. These results clearly indicate that the reaction rate is accelerated upon increasing the pH value which are also in good agreement with what was previously reported [36]. The reducing ability of ascorbic acid is attributed to the ascorbate ion; therefore, at higher pH values, the reducing capacity of ascorbic acid increases, which in turn accelerates the reaction rate. Nothworthy, at pH 12, we observed the appearance of a negligible peak located at ~410 nm in their optical spectrum which is possibly due to the co-existence of silver nanospheres. To verify that, the reaction was also conducted at pH larger than 12. The color of the dispersion was immediately transferred to yellowish brown with a sharp and narrow absorbance peak at ~410 nm which is the typical peak of silver nanospheres and a secondary broad peak at ~680 nm of Au@Ag hybrids, Figure 2a. The accelerated reaction rate at this extreme alkaline condition possibly enables the fast coalescence of abundant freshly-reduced Ag^0 atoms in the growth solution to dominantly produce silver nanoparticles instead of depositing onto the surface of Au-urchin seeds. Therefore, controlling the reaction rate via reaction pH plays a critical role in regulating the silver deposition process and thus the formation of core-shell nanostructures.

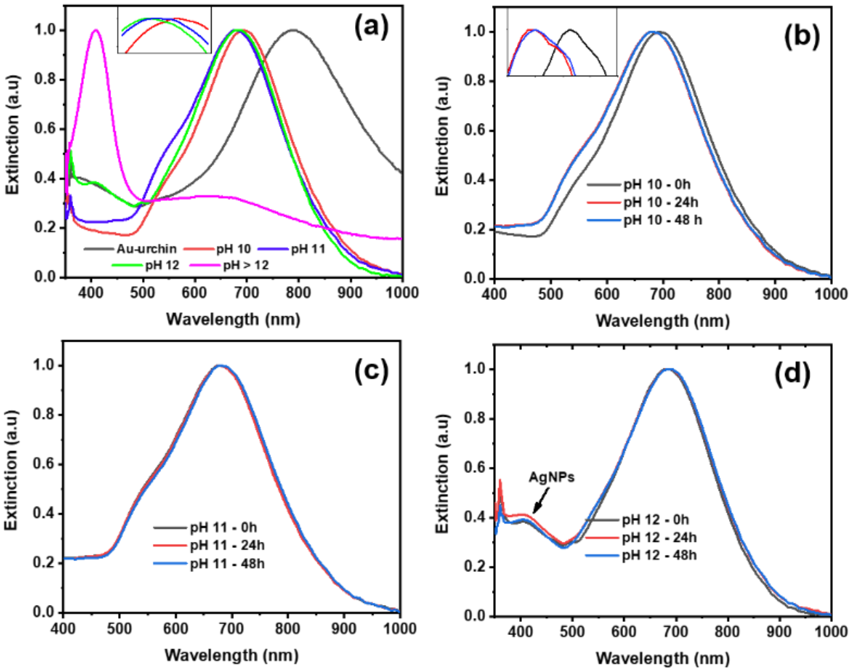

Figure 2. *UV-Vis spectra of silver coated urchin-like gold nanoparticles (so-called Au-urchin@Ag core-shell nanoparticles) synthesized at different pH conditions (a); UV–Vis spectra of Au-urchin@Ag core-shell nanoparticles monitored over 2 days at pH 10 (b), pH 11 (c), and pH 12 (d). All the UV-Vis spectra were normalized to 0–1 for visualization.*

We also investigated the impact of AgNO$_3$ precussor on the formation of core-shell structures by altering the volume of AgNO$_3$ (10 mM) added from a stock solution. An increase in the added amount of AgNO$_3$ leads to a progressive blue-shift of the LSPR maximum position from 787 down toward 650 nm, Figure 3a. Correspondingly, Au-urchin@Ag hybrids with heterogeneous boundary and thicker silver shell were also observed as the amount of AgNO$_3$ increased, Figure 4. The silver coating is likely to develop from the core and expand outward upon AgNO$_3$ addition. The blue-shift in the spectral response was also reported for silver coating of gold nanoparticles with other morphologies [26], [27]. Besides, an increase in silver shell thickness causes a reduction in the tip length and tip sharpness of Au-urchin and thus resulting in the blue-shift of obtained plasmon spectral maximum toward that of quasi-spherical silver nanoparticles where Au-urchin particles are fully covered. These results clearly suggest the crucial role of controlling the amount of $AgNO_3$ in order to tailoring the tip sharpness and thus optical properties of the obtained Au-urchin@Ag core-shell nanoparticles.

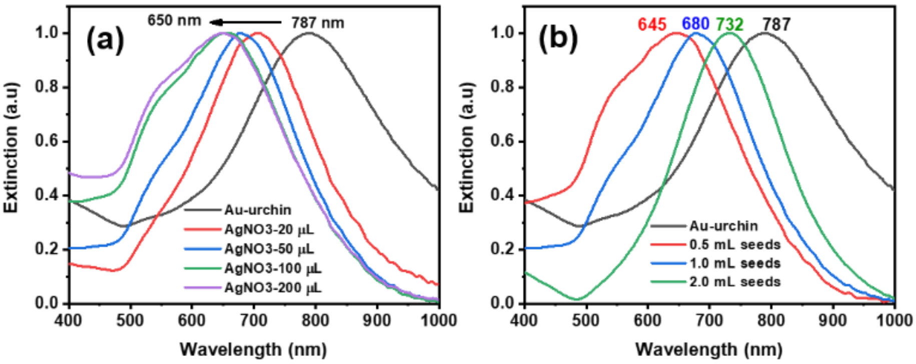

Figure 3. *The UV-Vis spectra of silver coated urchin-like gold nanoparticles (so-called Au-urchin@Ag core-shell nanoparticles) synthesized at different added volumes of $AgNO_3$ (10 mM) (a) and at different amount of Au-urchin seed particles (b). All the UV-Vis spectra were normalized to 0–1 for visualization.*

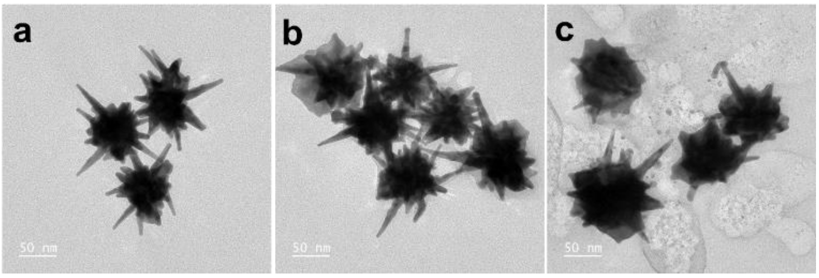

Figure 4. *(a) the TEM image of urchin-like gold nanoparticles (so-called Au-urchin); (b, c) the TEM images of silver coated urchin-like gold nanoparticles (so-called Au-rchin@Ag core-shell nanoparticles) synthesized with the addition of 50 µL and 100 µL $AgNO_3$ (10 mM), respectively.*

To understand the effect of the amount of Au-urchin seeds on the growth of Au-urchin@Ag core-shell nanoparticles, we have also conducted a series of experiments by altering the added amount of Au-urchin seed particles whereas fixing other parameters. The resultant LSPR peak shifts to shorter wavelengths upon decreasing the added dosage of urchin seed particles, Figure 3b. It is attributed to the increase in silver coating thickness produced at low amount of added Au-urchin templates which also agrees well with what was observed for $AgNO_3$ addition. These results suggest an alternative way to a better control over the optical properties of Au-urchin@Ag core-shell nanohybrids based on modulating the amount of seeds added.

4. Conclusion

In this study, silver coated sea urchin-like gold nanoparticles were successfully synthesized based on a seed growth method at room temperature. Ascorbic acid was used as green reductant to reduce Ag^+ ions on the surface of pre-synthesized gold urchin nanoparticles in the presence of PVP as capping agent. These core-shell nanoparticles exhibited tunable optical properties with anisotropic silver coating. We found that controlling the reaction rate via regulating the reaction pH plays critical role in the formation of Au-urchin@Ag core-shell structures and thus their optical properties. Besides, their shell thickness and corresponding optical properties are also facilely controlled by altering the amounts of Au-urchin seeds and $AgNO_3$ precursor in the growth solution. This proposed green synthetic route herein can be extended to the controllable fabrication of core-shell structures of various metallic nanoparticles. Importantly, the highly anisotropic morphology combined with the synergistic effect from silver and gold coupling are believed to offer these Au-urchin@Ag hybrids remarkably improved activities which are beneficial for plasmonic, catalytic, and biomedical applications.

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Conflict of Interest

The authors declare no conflicts of interest.

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