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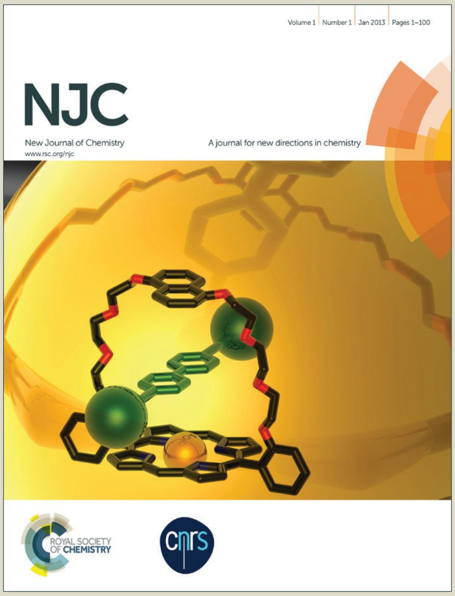

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ARTICLE

Dendritic Gold Nanowires Supported on SiO₂ Nanoparticles Fabricated by Seed Growth Method

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Seed growth methods are useful for the preparation of various shape-controlled nanocrystals, such as rods, cubes, and plates. The seed growth method generally employs seed nanocrystals dispersed in solutions and capping agents with selective adsorption properties. This article describes a novel seed growth method using Au nanoparticles supported on SiO_2 nanoparticles with a diameter of ~160 nm (SiO_2@Au NPs) and a long-chain amidoamine derivative (C18AA) with selective adsorption properties on gold. Selective growth of dendritic Au nanowires (Au DNWs) on the SiO_2@Au NPs took place, but no Au nanocrystals grew using bare SiO_2 NPs. The disadvantages of this method were a long time-consuming preparation and a lower yield of SiO_2@Au DNWs; for this reason, a co-reducing agent was used to increase the formation rate of Au DNWs. We found that the use of ascorbic acid, which has weak reduction powers, brought a considerable improvement to the preparation time and yield of SiO_2@Au DNWs. Furthermore, these SiO_2@Au DNWs showed high morphological and dispersion stabilities compared with unsupported Au DNWs, and it is expected as preparation method of supported shape-controlled nanocrystals with high morphological and dispersion stability.

1. Introduction

Morphological control of metal nanocrystals (NCs) is important for the development of modern material chemistry, because their physical and chemical properties can be easily and extensively tuned by tailoring the particle size and shape.[1-3] Recently, there have been many reports on the fabrication of various shape-controlled metal NCs, such as cubes,[4-8] plates,[9-11] rods,[12-17] and wires.[18-24] These shapes are generally significantly influenced by the reduction rate; a slow reduction rate favors the synthesis of non-spherical NCs such as rods, wires, and plates.[2,23-26] The conventional approach to shape control in metal NCs is the seed-mediated growth method, i.e., seed nanoparticles (NPs) with specific shapes and/or particular crystal facets are grown to obtain the desired nanostructure shape. In the seed growth method, capping agents are typically used to control the growth rate of a specific crystal facet due to selective adsorption on specific crystal facets of metal NPs. For example, Xia *et al.* have successfully synthesized Ag nanowires (NWs) using decahedral Au NPs as seed crystals in the presence of poly(vinylpyrrolidone) (PVP) as a capping agent.[2,27] Here, Ag NWs were prepared by selective growth of the (111) crystal facet on decahedral NPs, because PVP is preferentially adsorbed on the (100) and (110) crystal facets of Ag, which consequently promotes growth of the (111) crystal facet.[2,27-30] Tsuji *et al.* have successfully synthesized cubic, triangular-bipyramidal, and rod-shaped core-shell NCs (Au@Ag) using octahedral, triangular plate, and decahedral seeds, respectively, in the presence of PVP.[31-33] A combination of the seed growth method and appropriate capping agents bearing selective adsorption abilities thus constitutes a workable strategy for preparing NCs with particular shapes. Recently, it is claimed that NCs supported on SiO_2 or carbon nanotubes showed a high stability on their morphology and dispersibility compared with unsupported NCs.[34-36] Therefore, seed growing on SiO_2 materials is very useful for the preparation of the applicative shape-controlled NCs.

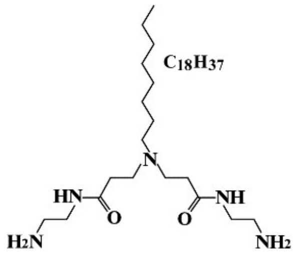

Figure 1. Molecular structure of a long-chain amidoamine derivative (C18AA).

In a previous study,[37] we prepared dendritic Au nanowires (Au DNWs) with diameters of 100–200 nm by utilizing the selective adsorption properties of a long-chain amidoamine derivative (C18AA, Figure 1). We also demonstrated that Au DNWs grew selectively from both ends of straight Au NWs.[37-40] This article describes the preparation of SiO_2-cored Au DNWs (SiO_2@Au DNWs), which were grown from Au NP seeds adsorbed on SiO_2 NPs using C18AA as reducing and capping agent (Figure 2). The SiO_2@Au DNWs showed a high dispersibility in water, and their morphological stability was

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Electronic Supplementary Information (ESI) available: [TEM, TEM-EDX, and QCM measurement]. See DOI: 10.1039/x0xx00000x superior to unsupported Au DNWs regardless of pH conditions (Figure 2).

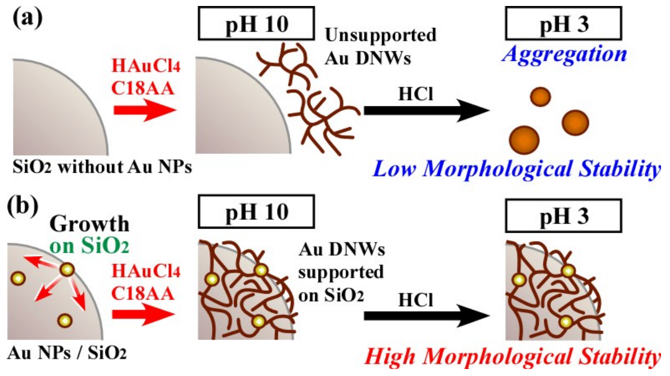

Figure 2. Schematic illustration of growth of (a) unsupported Au DNWs and (b) Au DNWs supported on SiO_2.

2. Experimental

2.1. Materials

3N tetraethoxysilane (TEOS), 1M sodium hydroxide, and ethanol were obtained from Kanto Chemicals. Hydrogen tetrachloroaurate tetrahydrate ($HAuCl_4$•$4H_2O$) was obtained from Nacalai Tesque. Aqueous ammonia (28 wt%) was obtained from Wako Pure Chemical Industries. They were used as received without purification. C18AA was synthesized according to a method from a previous paper.[41-43]

2.2. Preparation

2.2.1. SiO_2 nanoparticles

SiO_2 NPs were prepared by the Stöber method.[44] TEOS (3 g) and ethanol (25 g) were added to a mixture of 28 wt% aqueous NH_3 (4 g), water (3 g), and ethanol (25 g), and the resultant solution was stirred for 24 hours at room temperature. The average diameter of the SiO_2 NPs was 160 nm. To prepare a water dispersion of SiO_2 NPs, the as-prepared dispersion (5 g) of SiO_2 NPs was centrifuged at 5000 rpm for 30 min. The precipitated SiO_2 NPs were collected and washed with 5 mL water for three cycles, and the rinsed SiO_2 NPs was dispersed in 5 mL water (Figures 3a and S1a).

2.2.2. Supported Au nanoparticles

An aqueous dispersion of SiO_2 NPs (0.2 mL) and a 24 mM aqueous solution of $HAuCl_4$ (0.2 mL) were added to a 0.5 wt% aqueous solution of C18AA (1.0 mL). The mixture was heated to 55°C for 8 h, upon which the colour changed to purple-red, indicating the formation of Au NPs. To separate SiO_2@Au NPs from non-adsorbed Au NPs, the reddish dispersion was centrifuged at 5000 rpm for 30 min, and a re-dispersion (1.4 mL) of Au NPs deposited onto SiO_2 NPs (SiO_2@Au NPs) was used as a seed solution (Figures 3b and S1b).

2.2.3. Supported dendritic Au nanowires

SiO_2@Au NPs (0.05 mL) and 24 mM $HAuCl_4$ aqueous solution (0.2 mL) were added to 2 wt% C18AA aqueous solution (1.0 mL). The mixture was left for 3 days at room temperature without stirring (Figures 4b and c).

2.2.4. Improving preparation method of supported dendritic Au nanowires

After SiO_2@Au NPs (0.05 mL) and 24 mM $HAuCl_4$ aqueous solution (0.2 mL) were added to 2 wt% C18AA aqueous solution (1.0 mL), 0.48 mL of ascorbic acid aqueous solution (2, 10, or 100 mM) was quickly added and the mixture was left at room temperature without stirring (Figures 7 and S6). The amounts of ascorbic acid in the 2, 10, and 100 mM aqueous solutions (0.48 mL) were 0.96, 4.8, and 48 μmol, respectively.

2.3. Morphological and dispersion stability by pH change

1M HCl or 1M NaOH aqueous solution were added to unsupported Au DNWs (Figure 5) or SiO_2@Au DNWs (Figure 7e) dispersion prepared using 10mM aqueous solution of ascorbic acid (ESI).

2.4. Characterization

Transmission electron microscopy (TEM) observation was carried out using a JEOL JEM-1011 operating 100 kV. High-resolution TEM (HR-TEM) was performed using a JEOL 2100 instrument equipped with an energy-dispersive X-ray spectrometer (EDX) operated at 200 kV. UV-vis spectroscopy (JASCO, V-570) was conducted after centrifuging the dispersion at 5000 rpm for 10 min and re-dispersion in water.

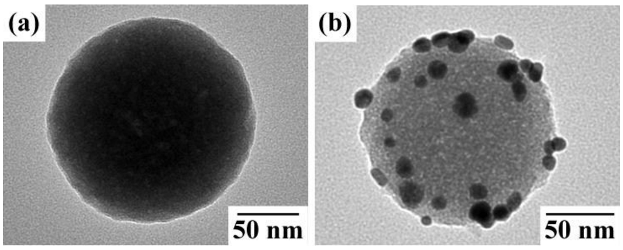

Figure 3. TEM images of (a) as-prepared SiO_2 NPs and (b) SiO_2@Au NPs after centrifugation.

3. Results and discussion

3.1. Preparation of SiO_2@Au DNWs

The TEM images in Figure 3 show that spherical Au NPs on the cored SiO_2 (SiO_2@Au NPs, Figures 3b and S1b) were obtained when $HAuCl_4$ and C18AA were added to the SiO_2 NPs (Figures 3a and S1a) dispersion. Here, C18AA acts not only as a capping agent but also as a reducing agent.[37] We conducted preparation of Au DNWs from seeded-Au sites on SiO_2@Au NPs by adding $HAuCl_4$ in the presence of [C18AA] = 2.0 wt%. First, we evaluated the effect of $HAuCl_4$ used on the growth of Au NPs on SiO_2 and examined the suitable amount of $HAuCl_4$ and C18AA (ESI). The spherical morphology of the Au NPs did not change significantly using 0.1 and 0.4 mL of aq. $HAuCl_4$, but their average diameter became larger (from 11 nm to 14 and 19 nm, respectively), indicating that the Au NPs served as the seed (Figures 4a and d, ESI). Similar products were formed in [C18AA] of 0.5 and 4.0 wt% at a constant volume of 0.2 mL aq.

$HAuCl_4$ (ESI, Figure S2). At [C18AA] = 2.0 wt% and with 0.2 mL aq. $HAuCl_4$, the morphology of some Au NPs changed from spherical structure to dendritic structure (Figure 4b), while almost Au NPs retained a spherical structure but with the average diameter increasing to 16 nm (Figure 4c).

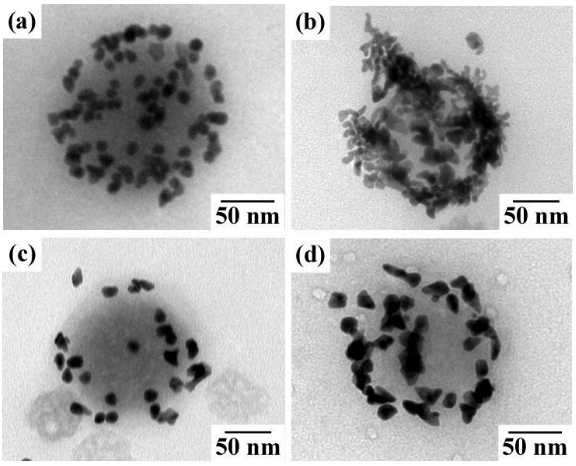

Figure 4. TEM images of SiO_2@Au NCs prepared by adding (a) 0.1 mL, (b, c) 0.2 mL, and (d) 0.4 mL $HAuCl_4$.: Only SiO_2@Au NPs were prepared using (a) 0.1 mL and (d) 0.4 mL $HAuCl_4$. SiO_2@Au NCs containing DNWs and NPs were prepared using (b,c) 0.2 mL $HAuCl_4$.

As a blank test, we conducted Au growth experiments using bare SiO_2 NPs without Au NPs under the same conditions used to obtain dendritic Au NWs from Au seeds of SiO_2@Au NPs. TEM images (Figure 5, ESI) showed that there were no Au NCs on the SiO_2 NPs, but Au DNWs formed in the bulk solution, indicating that the use of Au NPs as seeds is essential for the production of Au DNWs on SiO_2 NPs.

In the preparation of non-spherical Au NCs, control of the reduction rate is crucial.[2,23-26] Since the amine groups in C18AA are weakly reducing,[9,37] Au growth from the Au NP seeds on SiO_2 was expected to progress under slow reduction conditions. We confirmed the slow reduction progress by TEM observation at various reaction times (ESI, Figure S3). Only spherical NPs (no Au DNWs) were observed at 4 and 24 hours (Figure S3), but at 3 days SiO_2@Au DNWs as well as SiO_2@Au NPs were observed in the dispersion (Figures 4b and c). When the reaction temperature was increased from room temperature to 55°C to increase the rate of reduction, we were unable to obtain Au DNWs, only Au NPs. Furthermore, not Au DNWs only spherical Au NPs was obtained using $NaBH_4$ with strong reducing power as reducing agent (ESI, Figure S4). These results indicate that the formation of SiO_2@Au DNWs requires slow reduction conditions.

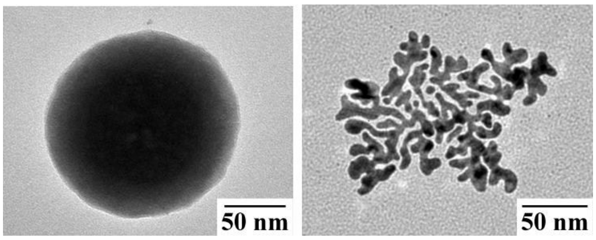

Figure 5. TEM images of SiO_2 NPs and Au DNWs prepared using SiO_2 NPs as seeds.

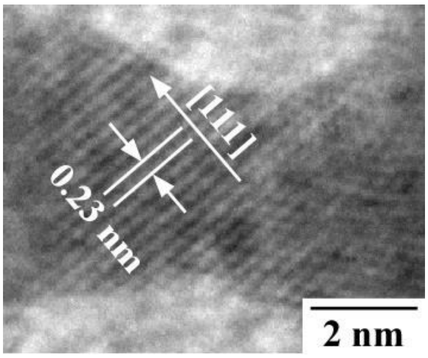

Figure 6. High-resolution TEM (HR-TEM) image of gold in SiO_2@Au DNWs.

TEM-EDX spectra showed that SiO_2@Au DNWs were composed of gold and silica (Figure S5). High-resolution TEM (HR-TEM) images (Figure 6) showed that gold in SiO_2@Au DNWs grew in the [111] direction, because a periodic fringe of 0.23 nm, which corresponds to (111) lattice spacing, was observed along the long axis of the Au DNWs (Figure 6).[18-20,37-40] According to a previous report,[37,41] C18AA is preferentially adsorbed on (100) and (110) facets rather than the (111) facet; thus, the growth of these facets should be favourable. Consequently, branches of Au DNWs grow in the [111] direction.

3.2. Improving preparation method of SiO_2@Au DNWs

The selective adsorption properties and weak reduction power of C18AA were very useful for the formation of Au DNWs on SiO_2 NPs; however, the disadvantages of this method were a long time-consuming preparation and a lower yield of SiO_2@Au DNWs. To overcome this obvious disadvantage, we used a co-reducing agent of ascorbic acid with weak reducing power to fulfil the requirement for slow reduction conditions. The co-reducing agent system was expected to shorten the preparation time and further increase the yield of SiO_2@Au DNWs. Note that ascorbic acid is one of famous reducing agent having a slow reduction power and is used for preparing Au nanorods.[13,14] Figures 7 and S6 shows representative TEM images of Au NCs covering SiO_2 in the presence of 0.96, 4.8, and 48 μmol of ascorbic acid at reaction times of 1 and 3 days. Au DNWs covering SiO_2 were successfully prepared within 1 day, and the yield of Au DNWs on SiO_2 was also greatly improved, as shown in Figures 7. Figure 7 and S6 showed that a low dose (0.96 μmol) of ascorbic acid affected the growth of Au seeds only slightly, with growth progress that was very similar to that of the system without ascorbic acid. However, large doses (4.8 and 48 μmol) considerably promoted seed growth of Au on SiO_2, and comparison of TEM images taken after 1 day and 3 days indicated that growth was completed within 1 day. Interestingly, the use of 4.8 μmol of ascorbic acid resulted in the formation of crystals with completely different morphology from those obtained using 48 μmol; the former were a mophead-like shape, while the latter adopted a gnarled shape (Figures 7e and f).

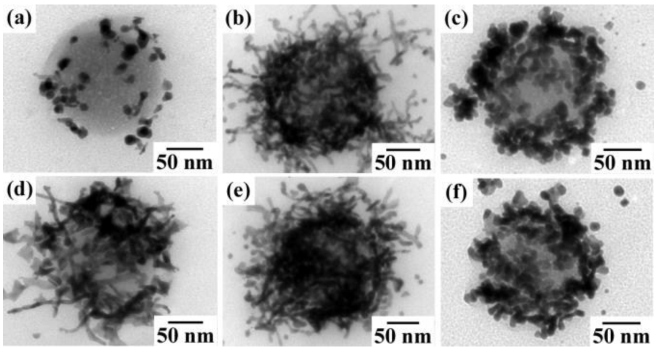

Figure 7. TEM images of SiO_2@Au NCs prepared using (a) 0.96, (b) 4.8, and (c) 48 μmol ascorbic acid after 1 day, and TEM images of SiO_2@Au NCs prepared using (d) 0.96, (e) 4.8, and (f) 48 μmol ascorbic acid after 3 days. The pH value was 7.0.

UV-vis spectroscopy was very useful for examining morphological changes in the Au NCs, because the surface plasmon (SP) band of Au NCs is highly sensitive to shape.[2,45,46] Figure 8 shows UV-vis spectra of as-prepared SiO_2 NPs (Figure 3a), SiO_2@Au NPs as seed NPs (Figure 3b), SiO_2@Au NCs containing DNWs and NPs prepared without ascorbic acid (Figures 4b and c), and SiO_2@Au DNWs prepared using 4.8 μmol of ascorbic acid (Figures 7e). The SP band of the Au NPs on SiO_2 appeared at ~530 nm (Figure 8b), which corresponds to the standard spherical Au NPs.[41,47-49] In contrast, the dispersions containing SiO_2@Au DNWs and SiO_2@Au NPs (Figures 4b and c) showed a SP band at ~530 nm and a broad peak at ~800 nm (Figure 8c).[50,51] The band at ~800 nm is derived from non-spherical Au NPs.[50,51] Furthermore, when ascorbic acid was used, the SP band at ~530 nm disappeared, while the broad peak at ~800 nm remained (Figure 8d). These results are in good agreement with the TEM images of the system using ascorbic acid, indicating the presence of SiO_2@Au DNWs but the absence of SiO_2@Au NPs.

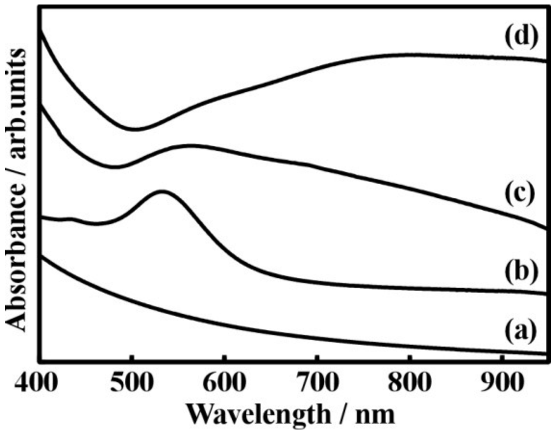

Figure 8. UV-vis spectrum of (a) as-prepared SiO_2 NPs, (b) SiO_2@Au NPs as seeds, (c) SiO_2@Au NCs containing DNWs and NPs, and (d) SiO_2@Au DNWs.

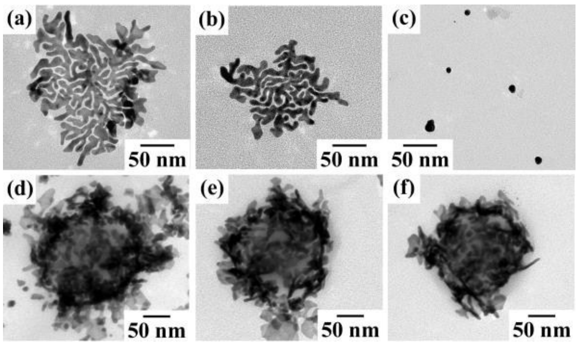

Figure 9. TEM images of unsupported Au DNWs at pH value of (a) 10.0, (b) 5.0, and (c) 3.0. TEM images of SiO_2@Au DNWs at pH value of (d) 10.0, (e) 5.0, and (f) 3.0.

3.3. Morphological and dispersion stability

In general, dispersion and morphological stability of NCs were improved by attaching NCs on support such as SiO_2 and carbon nanotubes.[34-36] In the case of the unsupported Au DNWs, dendritic morphology was remained at pH 5.0, and 10.0 (Figures 9a and b), though it changed to spherical structure at pH 3.0 (Figure 9c). Finally, aggregated nanoparticles precipitated in the vials at acidic condition. Previously we reported that ultrathin Au NWs were deformed at acidic condition of pH < 4 [39], due to the protonation of ternary amine of C18AA. QCM measurements (ESI, Figure S7) showed the adsorption amount of C18AA onto the Au surface decreased from 12.3 ng/mm^2 (pH 7.0) to 9.2 ng/mm^2 (pH 3.0). It seemed that Au DNWs was also deformed due to desorption of C18AA by pH changes. On the contrary, in the case of SiO_2-supported Au DNWs, the morphology was not changed by pH change (Figures 9d-f). Namely, morphological and dispersion stability of Au DNWs was improved by *supporting* on SiO_2. In a previous work [39], improvement of morphological stability of the Au NWs was shown by the *coating* with SiO_2, which required the several steps to introduce SiO_2 layer after the preparation of NWs. This seed-growth on SiO_2 was novel and efficient method to prepare shape-controlled NCs and to support the NCs with high morphological and dispersion stability.

4. Conclusions

We have described novel seed-mediated growth from spherical Au nanoparticles on SiO_2 nanoparticles using long-chain amine derivative (C18AA) as a capping and reducing agent. Using SiO_2-supported Au nanoparticles as seeds, SiO_2-supported dendritic Au nanowires—dendritic Au nanowires grown from Au nanoparticles on SiO_2—were obtained under slow reduction conditions. Furthermore, a co-reducing-agent system using long-chain amine derivative (C18AA) and ascorbic acid, retaining the slow reduction conditions, improved the preparation time and yield of SiO_2-supported dendritic Au nanowires compared with the system using only long-chain amine derivative (C18AA). The morphological and dispersion stability of SiO_2-supported dendritic Au nanowires improved compared with unsupported dendritic Au nanowires. This novel seed growth method using Au nanoparticles on SiO_2 was very useful to prepare shape-controlled nanocrystals with high morphological and dispersion stability, for applying as nanocatalysts.

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Graphical Abstract

SiO_2 supported Au dendritic nanowires were prepared by novel seed growth method, and showed high morphological and dispersion stabilities.