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Thermal Aqueous Solution Approach for the Synthesis of Triangular and Hexagonal Gold Nanoplates with Three Different Size Ranges

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The synthesis of gold nanoplates was carried out in an aqueous solution by thermal reduction of $HAuCl_4$ with trisodium citrate in the presence of cetyltrimethylammonium bromide (CTAB) surfactant in just 5–40 min. The sizes of the gold nanoplates can be varied from as small as tens of nanometers in width, to several hundreds of nanometers, and even a few microns in width by changing the reagent concentrations, solution temperature, and the reaction time. A [CTAB]/[$HAuCl_4$] ratio of 6 in the reaction solution was found to be favorable for the formation of gold nanoplates. The nanoplates possess well-defined shapes with sharp edges. The small nanoplates exhibit mainly a triangular shape, while larger nanoplates show a mixture of triangular, hexagonal, truncated triangular, and other symmetrical structures. The nanoplates are composed of essentially (111) lattice planes, as revealed by both XRD and TEM results. Nanoplates with widths from several hundreds of nanometers to a few microns absorb light strongly in the near-infrared region. The growth mechanism of these nanoplates was investigated. The ability to synthesize gold nanoplates with these different size ranges in large scale in aqueous solution using simple CTAB capping surfactant should allow more diverse applications of gold nanoplates.

Introduction

The preparation of nanoscale materials with a high degree of size and shape control is important and has been one of the major research efforts in the nanomaterials field, since the physical properties of these materials may be highly dependent on the particle morphology. For example, the surface plasmon resonance absorption spectra between spherical gold nanoparticles and nanorods are different, and the red-shift in the longitudinal mode of the absorption band is dependent on the aspect ratios of the nanorods.[1,2] In addition to the reports on the preparation of spherical and rodlike nanostructures of gold,[3] various other Au particle morphologies have also been synthesized, including nanocubes,[4] nanoplates,[5−17] branched nanocrystals,[18] and hexagonallyshaped nanoparticles.[19] Among these, the systematic

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synthesis of nanoplates with sharp edges and a relatively monodisperse particle size distribution over a wide size range from tens of nanometers to a few microns has not been demonstrated. In fact, unlike the general approach of surfactant capping used for the preparation of gold nanoparticles and nanorods, simple methods of using surfactant molecules such as cetyltrimethylammonium bromide (CTAB) as capping agents for the synthesis of gold nanoplates in pure aqueous solution were not reported until recently.[15] Lee et al. reported the synthesis of gold nanosheets by the preparation of a homogeneous solution of $HAuCl_4$ and poly(ethylene oxide)-poly(propylene oxide)-poly(ethylene oxide) ($PEO_{20}PPO_{70}PEO_{20}$, Pluronic P123) block copolymer in tetrahydrofuran (THF) followed by thermal evaporation of THF at 70 °C.[5] The nanosheets are very large ($\sim$10 μm in width and 100 nm in thickness), but many of them appear to be fractured. Preparation of gold nanoplates with an anionic phospholipid under photoirradiation was delineated by Tominaga and co-workers. The nanoplates are several hundred nanometers in width and many of them have cavities in the middle of the plates.[9] Kirkland et al. prepared triangular gold nanoplates from a boiling aqueous solution of $HAuCl_4$, citric acid monohydrate, and NaOH.[11] The nanoplates are mostly less than 100 nm in width and some large multiply twinned particles are present (tens of nm in diameter). In contrast to the limited methods available for the preparation of gold nanoplates, the growth of silver nanoplates seems to more successful.[20] Thus the ability to synthesize gold nanoplates with well-defined shapes and some degree of particle size control should be very desirable for their greater applications and the demonstration of unique features.

Here, we report the synthesis of gold nanoplates with well-defined triangular and hexagonal shapes. The plates can be prepared to have three different particle size ranges from a few microns to several hundred nanometers and then to several tens of nanometers in width. They are produced by a simple thermal reduction approach in aqueous solution in the presence of CTAB surfactant. The unique optical properties of these plates were characterized, and their possible growth mechanisms were investigated by varying the synthetic conditions.

Experimental Section

Hydrogen tetrachloroaurate(III) trihydrate (Aldrich), cetyltrimethylammonium bromide (Aldrich), and sodium citrate dihydrate (Mallinckrodt, trisodium salt) were used as received. Ultrapure deionized water (18.3 MΩ cm^{-1}) was used for all solution preparations.

The formation of gold nanoplates of different sizes follows basically the same procedure with changes mainly in the reagent concentrations, solution temperature, and reaction time. For the synthesis of large hexagonal and triangular gold nanoplates a few microns in width, 30 mL of an aqueous solution containing 8.33 $\times$ 10^{-4} M trisodium citrate was heated with stirring in a three-neck flask over a heating mantle. To prevent the loss of water, the solution was refluxed by connecting the middle neck of the flask to a condenser. When the temperature of the trisodium citrate solution reached 50 °C, 20 mL of an orange aqueous solution containing 1.25 $\times$ 10^{-3} M $HAuCl_4$ and 7.50 $\times$ 10^{-3} M CTAB, also heated to 50 °C, was injected into the hot trisodium citrate solution. Preheating of the two solutions before mixing reduces the reaction time and can give better control of the distribution of plate sizes. The resulting solution turned from orange to light yellow and then to colorless in 5 min. The loss of solution color is attributed to the reduction of Au^{3+} to Au^{+} by trisodium citrate.[1a] The solution temperature gradually increased to 82 °C after heating for 30 min and stayed at this temperature for 10 more minutes to allow the reaction to proceed to completion. After 40 min, the heat was removed, and the solution had a golden appearance, suggesting the presence of large gold structures dispersed in the solution. The large golden nanoplates slowly precipitated to the bottom of the flask in 1 h. The colorless solution was removed to collect the nanoplates.

Synthesis of medium-sized gold nanoplates (hundreds of nanometers in width) followed the same procedure used to prepare large gold nanoplates except the concentration of trisodium citrate was increased to 1.67 $\times$ 10^{-3} M, which favors the formation of smaller nanoplates. When the two reagent solutions, with the trisodium citrate solution heated to 62 °C and the $HAuCl_4$−CTAB solution at 50 °C, were mixed, the initial orange solution turned colorless, then light blue, and finally brown. The brown color suggests the formation of relatively large gold nanostructures, but it is still not as metallic in appearance as that for the micron-sized gold nanoplates. At this point (usually around 15 min), the solution has reached a temperature of 86 °C, and the flask was separated from the heating mantle. After 5−8 h of cooling, all the top brown solution containing the smaller particles was removed to collect the brown precipitate.

To further reduce the size of the nanoplates to tens of nanometers in width, 15 mL of an aqueous solution containing 1.67 $\times$ 10^{-3} M trisodium citrate was heated to 68 °C. Then 10 mL of an aqueous solution containing 1.25 $\times$ 10^{-3} M $HAuCl_4$ and 7.50 $\times$ 10^{-3} M CTAB heated to 50 °C was injected into the hot trisodium citrate solution. A smaller solution volume was used to rapidly increase the solution temperature. The orange solution turned colorless and then light blue in about 5 min with the final solution temperature reaching 82 °C. Then the flask was separated from the heating mantle and cooled. To collect the nanoplates, the cooled solution was centrifuged at 3000 rpm for 15 min in 5 mL portions (Hermle Z323 centrifuge). The supernatant was removed, and the precipitate was redispersed in deionized water for another round of centrifugation to remove the surfactant.

SEM images were obtained using a JEOL JSM-6330F scanning electron microscope. TEM characterization was performed on a JEOL JEM-1200CXII electron microscope operating at 120 kV. Optical images of large gold nanoplates were obtained with an Olympus BX51 optical microscope fitted to a digital camera. PXRD patterns were acquired using a Rigaku MXP^3 diffractometer with a Cu Kα radiation at $\lambda = 1.5418$ Å. The UV−vis absorption spectra were taken using a JASCO V-570 spectrophotometer.

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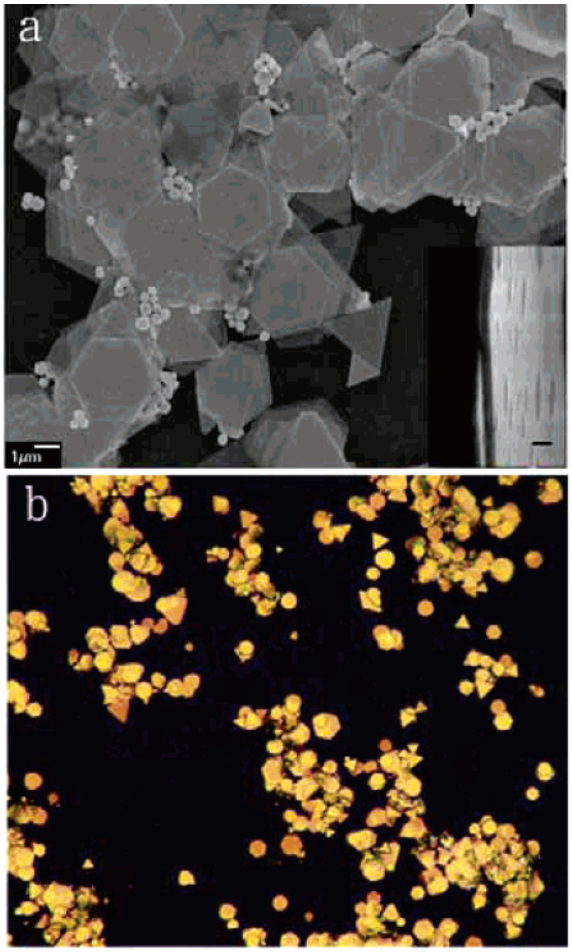

Figure 1. (a) SEM image of the micron-sized gold plates. Inset shows a SEM image of the side view of two overlapping plates. The scale bar in the inset is equal to 100 nm. (b) Optical image of the micron-sized plates showing their golden color. The magnification is ×200.

Results and Discussion

The large gold nanoplates can be conveniently observed using scanning electron microscopy (SEM). Figure 1a shows a typical SEM image of the largest nanoplates that have been synthesized using the described method. The nanoplates have hexagonal, triangular, and truncated triangular (or asymmetric hexagonal) shapes. The combined concentration of hexagonal and truncated triangular nanoplates appears to be higher than that of the purely triangular plates. Purely triangular plates may account for less than one-third of the total nanoplates synthesized, while hexagonal nanoplates represent more than one-third of the total nanoplate structures. Some spherical gold nanoparticles are also present which may be removed by centrifugation. All of the nanoplates also exhibit sharp edges. Even though these plates are considered to be relatively large (3.2 ± 0.4 μm), they are thin enough that the electron beam can penetrate through a stack of overlaying plates and allow the outlines of the underlying plates to be seen. The inset of Figure 1a shows an SEM image of the side view of two overlapping nanoplates for the determination of the plate thickness. The thicknesses of these plates are about 35−55 nm. The plates are large enough that they can be directly observed in an optical microscope. Figure 1b shows an optical image of the large nanoplates (×200). The nanoplates revealed the golden color and metallic shine typical of bulk gold. The image also shows a high percent yield of the gold nanoplates synthesized with relatively few spherical nanoparticles present. Figure 2 gives the size distribution histograms for the three sets of nanoplate samples prepared in this study. A wider plate size distribution can be expected for the larger plates, since slight differences in the growth rate over longer reaction times can produce a wider size range. Recent reports on the synthesis of large gold plates (with sizes of hundreds of nm to tens of μm) also appear to show wide size distributions for the large plates. However, smaller nanoplates exhibit a narrower size distribution (see Figure 2c).

The crystal structure of these nanoplates was characterized with powder X-ray diffraction (XRD) patterns. Figure 3 gives the powder XRD pattern of the large gold plates. The expected diffraction peaks for the face-centered cubic structure of gold are present. However, an exceedingly strong (111) diffraction peak was recorded. In contrast, the (200) diffraction peak is very weak. The (220) diffraction peak is also present, but its intensity is also very weak, less than one-third of that of the (200) peak. Thus the nanoplates are essentially composed of (111) lattice planes, a result consistent with previous reports on the structural characterization of gold nanoplates.[5−7,9−17] The formation of nanoplates with essentially {111} facets may be the result of the lower free energy of the (111) planes relative to those of the (100) and (110) planes.[21]

The medium-sized nanoplates were examined using transmission electron microscopy (TEM). Figure 4a−c show typical TEM images of these nanoplates. Again a mixture of triangular, hexagonal, and truncated triangular nanoplates were observed. The relative distribution of the three shapes in this size range is more random and can vary somewhat from sample to sample, since nanoplates of this size range are produced after a relatively short period of rapid nanoplate growth. The percentage of triangular nanoplates is intermediate between that of the small-sized nanoplate samples and the micron-sized samples. The yield of the medium-sized nanoplates is also high after the removal of the top brown solution, although some highly faceted nanoparticles were observed. The average width of the nanoplates is 486 ± 55 nm, and their thicknesses, as determined from the SEM images (data not shown), are around 10−20 nm. Figure 4d gives the TEM image of a single triangular nanoplate. The plate surface appears to be flat and the edges are sharp. The corresponding selected-area electron diffraction (SAED) pattern of this nanoplate obtained with the electron beam perpendicular to the planar surface of the nanoplate is shown in Figure 4e. The diffraction pattern indicates that the nanoplate is single and crystalline, and its surface is bounded by {111} facets.[5−7,9−17] Similar electron diffraction patterns were observed for the hexagonal nanoplates. The results are consistent with the XRD pattern data for the large gold nanoplates. In addition, nanoplates with unusual structures have also been found. As shown in Figure 4b and 4c, some

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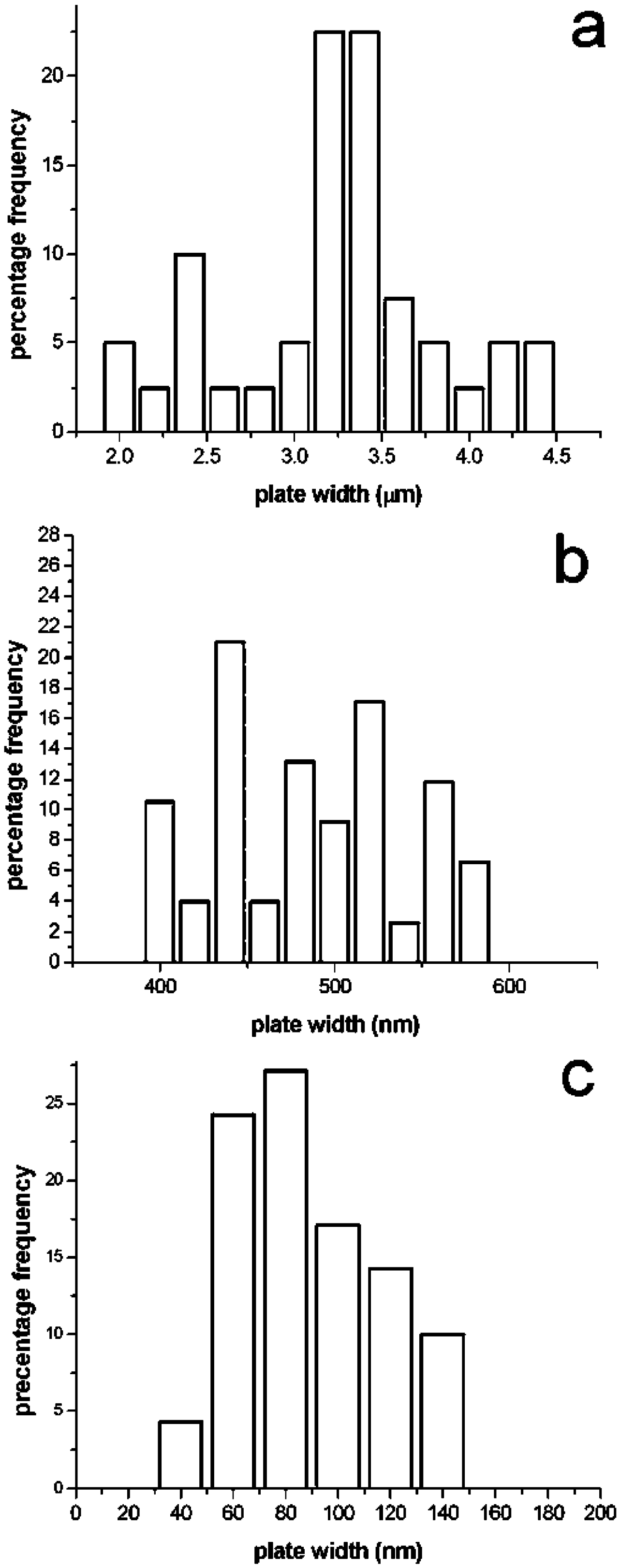

Figure 2. Size distribution histograms of the gold nanoplates. (a) Micron-sized gold plates. (b) Medium-sized nanoplates with widths of hundreds of nm. The size distribution plot reflects the presence of a variety of nanoplate shapes. (c) Small-sized nanoplates. These nanoplates are mainly triangular in shape. The width values for hexagonal and truncated triangular plates were determined by measuring the lengths across opposite edges, and for triangular plates, they are the heights of the triangles.

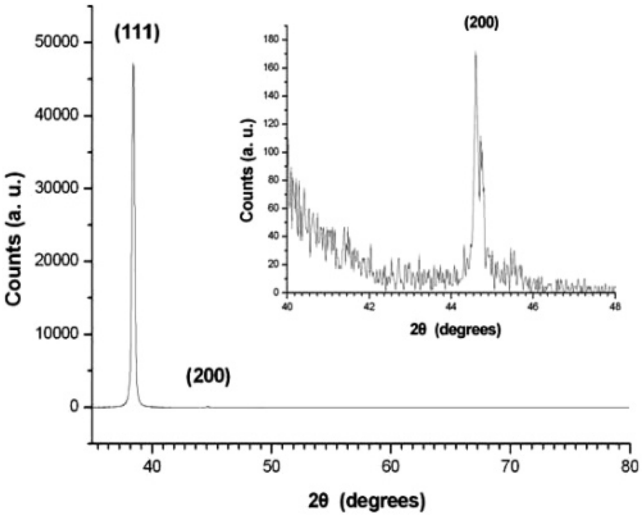

Figure 3. Powder XRD pattern of the micron-sized gold nanoplates. Inset shows the enlarged powder XRD pattern in the (200) peak region.

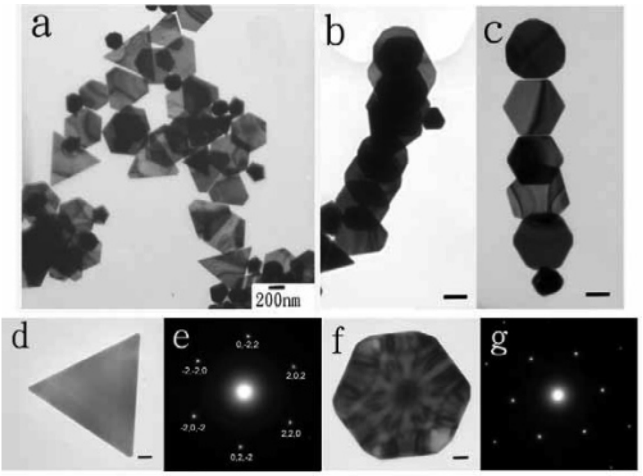

Figure 4. (a) TEM image of medium gold nanoplates. (b and c) TEM images of gold nanoplates arranged into chainlike structures. Scale bar = 200 nm. (d) TEM image of a single triangular gold nanoplate and (e) the corresponding electron diffraction pattern. Scale bar = 50 nm. (f) TEM image of a single quasi-hexagonal gold nanoplate (with 6 long edges and 6 short edges) and (g) the corresponding electron diffraction pattern. Scale bar = 50 nm.

of the hexagonal nanoplates are not quite hexagonal in shape but contain additional edges. One of these quasi-hexagonal nanoplates is shown in Figure 4f. The particle structure is formed by truncating all six corners to form six more edges. The corresponding SAED pattern for this quasi-hexagonal nanoplate is shown in Figure 4g, which is similar to those for the triangular and hexagonal nanoplates with the plate surface bounded by {111} facets.

The smallest gold nanoplates prepared in this study were analyzed with TEM, shown in Figure 5. Most of the nanoplates exhibit triangular structure with sharp edges and flat surfaces. Some highly faceted particles with pentagonal and hexagonal shapes were also observed. However, triangular nanoplates still compose a large fraction of the gold nanostructures formed, as shown by the UV−vis absorption spectrum. The average width of the nanoplates is 85 ± 28 nm, and the plate thicknesses are around 6−17 nm. Some

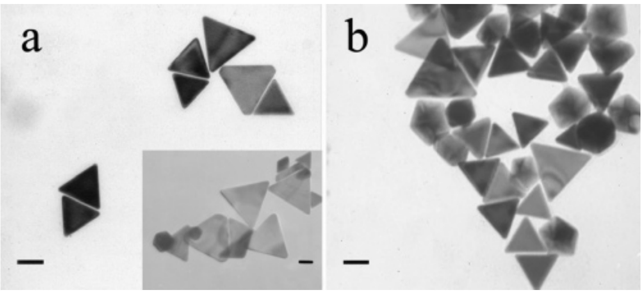

Figure 5. (a and b) TEM images of small gold nanoplates. Scale bar = 50 nm. Plate thickness can be estimated from the inset in (a) with slightly larger nanoplates. Scale bar = 50 nm.

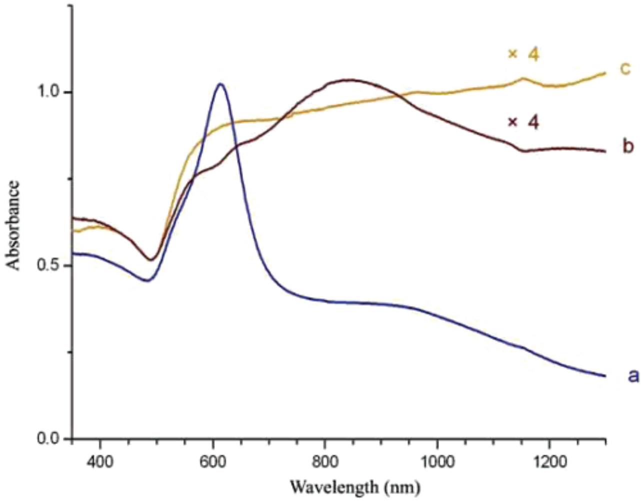

Figure 6. UV−vis absorption spectra for the three different-sized gold nanoplates: (a) small, (b) medium, and (c) micron-sized nanoplates. Absorbance intensities of traces b and c have been multiplied by 4. The spectral discontinuity at ∼1155 nm is the result of a grating/detector change.

triangular plates were also found to arrange edge-to-edge. These nanoplates have the same crystal structure as the larger ones.

It is interesting to note that the small nanoplates are mainly triangular in shape, while the larger plates exhibit hexagonal and truncated triangular structures. It is presumed that triangular nanoplates are dominant when their sizes are still within 150 nm in width, but they may transform into hexagonal, truncated triangular, and other symmetrical structures as the nanoplate size increases. Our preliminary results show that when the reaction for the synthesis of small nanoplates was suddenly quenched with the hot flask placed in an ice bath, some triangular plates with round crystalline protrusions growing from all three angles of a triangle were observed (Supporting Information Figure 1). This structural feature may suggest the mechanism by which triangular plates grow in size or transform into plates of other shapes. Further work is necessary to determine the existence and mechanism of the shape transformation of gold nanoplates.

Because of the different colors exhibited by these nanoplates, optical properties of the nanoplates can be characterized with UV−vis absorption spectroscopy. Figure 6 shows the UV−vis absorption spectra of the three different-sized nanoplate samples. For the small triangular nanoplates, there is a strong surface plasmon resonance band at ∼615 nm and a broad but weak band in the near-infrared (near-IR) region centered at around 950 nm. The band positions are close to those reported for truncated tetrahedral gold nanocrystals.[4c] A shoulder band at 520−530 nm is attributed to the surface plasmon resonance absorption of more spherically shaped particles.[18] The UV−vis absorption spectrum for the medium-sized gold nanoplates shows significant increase in absorption in the near-IR region (Figure 5b). Three bands centered at 576, 642, and 842 nm can be identified. Similar spectral features and the strong absorption in the near-IR region have been observed for triangular nanoprisms with edge lengths of several hundred nanometers.[6] Since our sample contains a mixture of nanoplate shapes over a range of sizes, we cannot unambiguously assign these absorption peaks. The absorption spectrum for the micron-sized gold nanoplates shows a steep increase in the absorption from 500 to 600 nm, and then the absorption increases steadily into the near-IR region. When the spectral features of these different gold nanoplate samples are compared, the medium-sized nanoplate sample shows absorption characteristics resembling those of the large plates. The strong near-IR light absorption of these large gold nanoplates may be useful in biomedical applications.[6,13]

We investigated the factors affecting the formation of gold nanoplates by varying the reaction parameters and comparing the resulting gold nanostructures. A major factor facilitating the formation of gold nanoplates appears to be the reaction temperatures (82−86 °C). At these temperatures, the reduction of $AuCl_4^-$ ions by trisodium citrate proceeds sufficiently fast to supply gold nuclei or gold seed particles. The possibility that a high reaction temperature condition favors the growth of gold nanoplates is also supported by several reports in which the reaction temperature was increased directly or indirectly by heating, photoirradiation, or microwave radiation.[5,9−11,17]

The interactions of CTAB with the gold seed particles under such a high-temperature condition may favor the growth of platelike gold nanostructures; the use of other surfactants such as dodecyltrimethylammonium bromide and sodium dodecyl sulfate under otherwise identical reaction conditions as those used for the preparation of large gold nanoplates produced mainly highly faceted gold nanoparticles and some triangular nanoplates. Of course, the reason for the effectiveness of CTAB over other surfactants tested may be attributed to its substantially lower critical micelle concentration (cmc). The cmc value for CTAB at 60 °C is 1.0 mM.[22] The CTAB concentration in the reaction mixture is 3 mM, thus micelles should have been formed to control the size and shape of the gold nanoparticle seeds. With the help of trisodium citrate to facilitate the formation of nanoparticles with platelike morphology (see discussion below), small nanoplates begin to form.

The relative concentrations of $HAuCl_4$, CTAB, and trisodium citrate were also found to affect the formation and size of gold nanoplates. Under a higher trisodium citrate concentration condition, the reduction of $AuCl_4^-$ ions should

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go faster and form more gold nuclei. This condition may favor a rapid formation of nanoplates with smaller sizes. We found that by doubling the concentration of trisodium citrate from that used for the synthesis of micron-sized gold plates (8.33×10^{-4} M vs 1.67×10^{-3} M), medium-sized nanoplates can be produced in just 15 min. When the reaction time was increased to 40 min, the resulting nanoplates did increase in size but were not as large as the micron-sized plates. On the other hand, by reducing the reaction time used to synthesize the micron-sized plates the nanoplates produced are smaller, but they are still much larger than the medium-sized nanoplates. It should be noted that trisodium citrate may play a role in inducing the formation of the (111) planes, in addition to serving as a reducing agent, and facilitate the growth of nanoplates. The ability of trisodium citrate to affect the crystal growth of ZnO to form platelike ZnO nanostructures under a hydrothermal synthesis condition have been described, in which the growth of ZnO along the ⟨001⟩ orientation was inhibited by the binding of citrate ions.[23,24] In another report, the synthesis of gold nanoplates by aspartate reduction was successful, but not with several other amino acids tested.[7] The presence of multiple carboxylate ions in both aspartate and trisodium citrate molecules may be related to their crystal structure modifying ability. This argument has recently been proven to be reasonable with a report using oxalic acid to prepare hexagonal gold microplates.[17] In addition to the effect of the concentration of trisodium citrate on the plate size control, the relative [CTAB]/[$HAuCl_4$] ratio may also be important for nanoplate formation. A [CTAB]/[$HAuCl_4$] ratio of 6 was used for the synthesis of nanoplates: the use of different reagent ratios may give lower yields of nanoplates. All of these results suggest that the relative concentrations of all reagents need to be controlled to optimize the growth of nanoplates with a particular size range and reduce the presence of highly faceted nanoparticles.

Conclusion

In summary, gold nanoplates with three different ranges of sizes from tens of nanometers to a few microns in width have been prepared using a simple thermal reduction approach with CTAB surfactant in aqueous solution. The nanoplates are formed in relatively short periods of time (that is, 5−40 min), compared to the hours or days of time required in some room-temperature nanoplate preparation procedures.[6,7,20e,20f] A [CTAB]/[$HAuCl_4$] ratio of 6 in the reaction solution was found to be favorable for the formation of gold nanoplates. The nanoplates show triangular, hexagonal, truncated triangular, and other symmetrical shapes. These nanoplates are composed of essentially (111) lattice planes, as revealed by both XRD and TEM results. The nanoplates exhibit different colors depending on their sizes. The ability to synthesize gold nanoplates under such typical preparation conditions as those generally used for the growth of spherical gold nanoparticles should allow greater applications of these nanoplates and demonstration of new ideas. Possible research directions using these triangular and hexagonal nanoplates include creation of novel superstructures through self-assembly or surface modification of the nanoplates and binding of these nanoplates to biological, photosensitive, and electrically responsive molecules for novel property observations. For example, two-dimensional self-assembled superstructures of the small-sized triangular nanoplates may be fabricated on substrates if their sizes are reasonably uniform and the yield is relatively high.

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Supporting Information Available: TEM images of the reaction for the synthesis of small nanoplates which was suddenly quenched by placing the hot flask in an ice bath. This material is available free of charge via the Internet at http://pubs.acs.org.

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