MethodsX 1 (2014) 118–123

Contents lists available at ScienceDirect

MethodsX

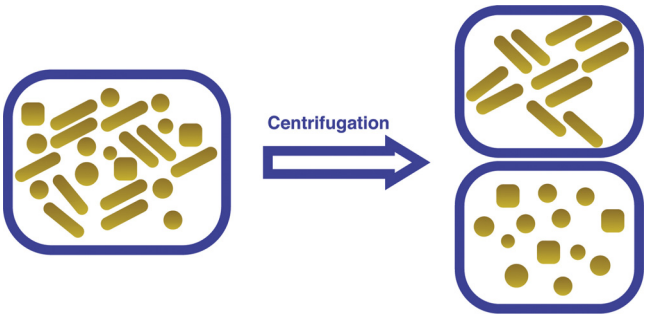
journal homepage: www.elsevier.com/locate/mex

Rapid purification of gold nanorods for biomedical applications

Federica Scaletti [a,b], Chang Soo Kim [b], Luigi Messori [a,**], Vincent M. Rotello [b,*]

[a] Department of Chemistry "Ugo Schiff", University of Florence, Via della Lastruccia 3-13, 50019 Sesto Fiorentino, Florence, Italy
[b] Department of Chemistry, University of Massachusetts, 710 North Pleasant Street, Amherst, MA 01003, USA

GRAPHICAL ABSTRACT



ABSTRACT

Small gold nanorods (GNRs) with longitudinal plasmon absorption in the near-infrared window (700–900 nm) are of great interest for *in vivo* optical applications (*e.g.*, photothermal therapy) and for their high-payload-to-carrier ratio for drug delivery. Common synthetic strategies for GNR production afford spherical and cubical nanoparticles in addition to the desired GNRs. Thus, several methods have been proposed for the selective separation of GNRs from the reaction by-products. For example, centrifugation has been used to separate the high aspect ratio (AR) GNRs (AR > 4). However, it is difficult to separate small sized GNRs with low AR (AR ≤ 4) that are particularly promising for biomedical applications. Here, we describe a simple and fast procedure for the separation of small GNRs with AR of 4, and length of 28 nm from reaction by-products.

* Corresponding author. Tel.: +1 413 545 2058.
** Corresponding author.
E-mail addresses: luigi.messori@unifi.it (L. Messori), rotello@chem.umass.edu (V.M. Rotello).

http://dx.doi.org/10.1016/j.mex.2014.07.007

The shape separation is achieved through centrifugation according to the following steps:

- Isolation of all gold products of the reaction from the excess of cetyltrimethylammonium bromide through a first cycle of centrifugation.
- Optimization of the speed and the time of centrifugation for the separation of GNRs from the reaction by-products.
- Shape separation of GNRs through a second cycle of centrifugation.

The effectiveness of this procedure is documented.

ARTICLE INFO
Method name: Selective separation of gold nanorods from by-products
Keywords: Gold nanorods, Centrifugation, Nanoparticle, Shape separation, Purification, Surface plasmon resonance
Article history: Received 23 May 2014; Accepted 30 July 2014; Available online 10 August 2014

Method

The final reaction mixture for GNRs contains the desired GNRs, a variety of gold nanoparticles (NPs) of different shapes (spherical, cubic, *etc.*). To obtain an homogeneous population of GNRs, two cycles of centrifugation were carried out. After the synthesis, all reaction products (spheres, cubes, and rods) were first isolated and purified from the excess of cetyltrimethylammonium bromide (CTAB) through centrifugation at high rpm. CTAB needs to be removed as much as possible for many applications owing to its cytotoxicity [1]. Then, shape separation was afforded through a second cycle of centrifugation adapted from the method reported by Sharma et al. [2] and optimized for the size of interest.

Due to the large shape-dependent differences in the respective sedimentation velocities, in the course of centrifugation, spheres and cubes were sedimented at the bottom of the centrifuge tube while most GNRs remained in the supernatant solution. Therefore, an efficient shape separation can be achieved by tuning the centrifugation times and speeds.

Method details

Step 1: GNR synthesis

Materials

Sodium borohydride (>98%), cetyltrimethylammonium bromide (>99%), and silver nitrate (99.5%) were purchased from Acros Organics. Chloroauric acid (99.9%) was purchased from Strem Chemical, and L-(+)-ascorbic acid was purchased from Alfa Aesar. All chemicals were used as received. All the solutions were prepared with deionized water produced by a Millipore System.

Procedure

GNRs, with AR of 3.9 ± 0.5 (width = 7.5 ± 0.8 nm, length = 28.6 ± 3.5 nm) were synthesized by a seed-mediated method according to Nikoobakht et al. [3] and Ratto et al. [4] with slight modifications. After the synthesis, the structural and optical properties of GNRs were characterized by Transmission Electron Microscopy (TEM) and spectrophotometric analysis. TEM images were acquired on a JEOL 200FX with a tungsten filament operating at 200 kV. Optical extinction spectra were recorded using a Molecular Devices SpectraMax M2 plate reader.

Step 2: GNR separation from CTAB

The GNRs were separated from excess CTAB and then purified from the by-products through centrifugation with an Eppendorf centrifuge 5810R using 2 ml centrifuge tubes. During centrifugation, temperature was kept at 27 °C to avoid the crystallization of CTAB that may affect the separation.