

MASTER'S THESIS

Synthesis, characterization and capillary electrophoretic studies of thiolated [α]-cyclodextrin-capped gold nanoparticles

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Date of Award:
2009

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**Synthesis, Characterization and Capillary
Electrophoretic Studies of Thiolated
 α -Cyclodextrin-Capped Gold Nanoparticles**

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**A thesis submitted in partial fulfillment of the requirements
for the degree of
Master of Philosophy**

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August 2009

Abstract

This thesis mainly focuses on two parts. This first one is the synthesis and characterization of ultrasmall (< 2.0 nm) thiolated α -cyclodextrin-capped gold nanoparticles (α -CD-S-AuNPs). Per-6-thio- α -cyclodextrins were firstly synthesized and were employed to protect gold nanoparticles (AuNPs) from aggregation. The as-prepared products were characterized by different techniques. Since the average size of the resulting AuNPs is highly dependent of the initial mole ratio of thiol and gold in the reaction mixture, the effect of cyclodextrin-to-gold (α -CD-SH/Au) ratio on the size of α -CD-S-AuNPs has been studied. In contrast to previous studies, we observed that when the α -CD-SH/Au ratio is larger than 1, the size of AuNPs increases with α -CD-SH/Au ratio which is opposite to the trend obtained for α -CD-SH/Au ratio smaller than 1. It is postulated that the increase in particle size is attributed to the interhydrogen bond between the thiolated α -cyclodextrin (α -CD-SH) molecules at higher concentrations with a decrease in the availability of free α -CD-SH to stabilize the AuNP surface. The chemical compositions of such AuNPs were further determined by thermogravimetric analysis, mass spectrometry, and atomic absorption spectroscopy. Interestingly, the number of attached thiolated α -cyclodextrin ligand per AuNP increases with the decrease in core size of AuNP,

possibly attributing to the formation of staple motifs on the Au core.

The second part of the research explores the effect of tetraalkylammonium ions on the electrophoretic mobility and the peak shape of α -CD-S-AuNPs of different core sizes in capillary zone electrophoresis (CZE). The effect of various tetraalkylammonium ions on the peak width and electrophoretic mobility of the α -CD-S-AuNPs was studied in detail. Tetrabutylammonium (Bu_4N^+) ions provide good migration peak shape for α -CD-S-AuNPs in CZE. Bu_4N^+ ions possibly interact simultaneously with several hydrophobic cavities of the surface-attached α -CDs on AuNPs, therefore, a narrow solute band of α -CD-S-AuNPs was maintained. The optimal run buffer was 10 mM Bu_4N^+ in 30 mM phosphate buffer (pH 12). The applied voltage was 15 kV. In addition, a linear inverse relationship ($r^2 > 0.998$) exists between the electrophoretic mobility and size of α -CD-S-AuNPs over the core range of 1.4–4.1 nm. Our work shows that CZE is an efficient tool for characterizing the size of α -CD-S-AuNPs using Bu_4N^+ ions.

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