Highly selective and sensitive colorimetric detection of Hg (II) ions using green synthesized silver nanoparticles

M. Annadhasan and N. Rajendiran*

Department of Polymer Science, University of Madras, Guindy Campus, Chennai- 600 025, Tamil Nadu, India. E-mail: nrajendiar@yahoo.com (N. Rajendiran*); Tel: +91-44-22202822.

Materials and Methods

1.1. Materials

Silver nitrate (AgNO₃) was purchased from Sigma Aldrich and used as received. N-Cholyl-L-cysteine was synthesized by adopting our previous method [22]. Salts of the different cations studied (LiCl, NaCl, KCl, MgCl₂, CaCl₂, BaCl₂, ZnCl₂, CoCl₂, NiCl₂, CdCl₂, HgCl₂, CuSO₄, MnSO₄, Pb(NO₃)₂, and Cr(NO₃)₃) were obtained from SRL chemicals Pvt. Ltd. (India) and used as received without further purification. The glass containers were washed with aqua-regia (HCl: HNO₃ = 3:1 (v/v)) and rinsed with triple distilled water prior to use.

1.2. Instruments

UV-visible spectroscopy measurements were carried out on Techcomp UV-2301 spectrophotometer. Dynamic light scattering (DLS) measurements were performed for colloidal solutions using Nanotrac Ultra NPA 253 from Microtrac, USA. High resolution transmission electron microscopy (HRTEM) images were recorded using a JEOL JEM 2100 equipped with a Gatan Imaging Filter operating at 200 kV. X-ray photoelectron spectroscopy (XPS) measurements were performed with Omicron Nanotechnology; GmBH, Germany XM1000-monochromator with Al Ka radiation of 1483 eV operated at 300 W (20 mA emission current, 15 kV) and a base pressure of 5 ×10⁻⁵ mbar. Powder X-ray diffraction of all samples
were recorded on SMART Bruker D8 Advance diffractometer using Cu Kα Xradiation ($\lambda = 1.54056$ Å) at 40 kV and 30 mA. Diffraction patterns were collected over a 20 range of $5-80^\circ$ at a scan rate of $1^\circ \text{min}^{-1}$.

1.3. \textit{Colorimetric Assay procedure}

In a typical procedure, 2ml of AgNPs solution was mixed with various metal ions at a fixed concentration and equilibrated for five minutes at room temperature before the spectral measurements. For specific detection limit various concentration of Hg$^{2+}$ ions were added and recorded the responses. UV-Vis absorption spectra were recorded using Techcomp UV-2301 spectrophotometer equipped with a 1cm path length quartz cell.
Fig. S1a ¹H and ¹³C NMR spectra of N-cholyl-L-cysteine.

**N-Cholyl-L-cysteine**

¹H NMR (CDCl₃/DMSO-d6) δ = 4.79 (d, 1H, C*H), 3.97 (s, 1H, 12a-CH), 3.83 (s, 1H, 7a-CH), 3.45 (m, 1H, 3a-CH), 2.20–0.98 (m, 25H, aliphatic H), 1.01–0.68 (m, 9H, 3 CH₃), 0.88 (s, 3H, 19-CH₃), 0.68 (s, 3H, 18-CH₃). IR (KBr): 3412, 2941, 2874, 2496, 1728, 1683, 1582, 1509.
Fig. S1b  FT-IR spectra of pure NaCysC and NaCysC stabilized AgNPs.

Fig. S2  UV-visible spectra of synthesized AgNPs samples stability (fresh and after 6 months).
Fig. S3  Powder-XRD spectrum of NaCysC stabilized AgNPs.

Fig. S4  EDAX spectrum of NaCysC stabilized AgNPs.
Fig. S5  XPS survey spectrum (top left) and high resolution XPS spectra of components present in NaCysC stabilized AgNPs.
Fig. S6  Plot of Hg$^{2+}$ concentrations versus absorbance intensity of AgNPs solution.

Fig. S7a. Plot of Hg$^{2+}$ concentrations in real water samples versus absorbance intensity differences of AgNPs solution.
Fig. S7b. Linear plot for real water samples

Drinking water

$R^2 = 0.99666$

Tap Water

$R^2 = 0.99643$