

Sintesis Nanopartikel Perak Tertiolasi dalam Matriks Kitosan sebagai Sensor untuk Deteksi Ion Merkuri

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INTISARI

Sintesis nanopartikel perak tertiolasi dalam matriks kitosan (AgNPs-SH@Cts) sebagai sensor merkuri dikembangkan melalui pendekatan kimia hijau berbantuan ultrasonik. Penelitian ini bertujuan mengatasi keterbatasan stabilitas AgNPs akibat agregasi dan oksidasi serta mengevaluasi pengaruh ligan ditizon, sistein, dan glutathione terhadap karakteristik fisikokimia, sensitivitas, selektivitas, dan stabilitas sensor elektrokimia untuk ion Hg^{2+} , sekaligus menentukan sistem modifikasi yang sesuai untuk analisis merkuri pada sampel air.

AgNPs disintesis menggunakan ekstrak *Hydrocotyle umbellata* dan *Centella asiatica* pada variasi waktu dan temperatur sonikasi, ditandai oleh puncak LSPR 400–500 nm. Citra TEM menunjukkan partikel berbentuk bulat dengan diameter rata-rata 7,39; 5,43; dan 4,49 nm untuk AgNPs yang distabilkan ditizon, sistein, dan glutathione dalam matriks kitosan. Keberhasilan sintesis dikonfirmasi melalui FTIR, XRD, analisis morfologi, serta EDX yang menunjukkan keberadaan Ag, S, dan N.

Sensor difabrikasi pada *screen-printed electrodes* (SPEs) dengan metode penetasan. Pengujian *cyclic voltammetry* menggunakan $[\text{Fe}(\text{CN})_6]^{3-/4-}$ 0,005 M menunjukkan peningkatan kinerja yang berkorelasi dengan luas permukaan aktif elektrokimia. Tiolasi meningkatkan I_{pa} sebesar 423%–515% dan mengubah I_{pc} sebesar 331%–502%.

SPEs-AgNPs-SH@Cts menunjukkan stabilitas pada sepuluh pengulangan dan selektivitas tinggi terhadap Hg^{2+} di hadapan berbagai ion pengganggu. Sensor memberikan linearitas rata-rata 0,9979, sensitivitas $1,20 \pm 0,03$ – $2,12 \pm 0,07$ $\mu\text{A/ppb}$, dan limit deteksi 0,15–0,20 ppb. Analisis sampel air menghasilkan RSD 2,27%–8,12% dan perolehan kembali $(82,93 \pm 0,02$ – $102,35 \pm 0,09)\%$. Sensor dapat digunakan kembali serta mendukung analisis secara cepat, sederhana, akurat, dan ramah lingkungan.

Kata kunci: nanopartikel perak tertiolasi; kitosan; *screen-printed electrode*; sensor elektrokimia; ion merkuri; *square-wave anodic stripping voltammetry*

Synthesis of Thiolated Silver Nanoparticles in Chitosan Matrix as Sensor for Mercury Ion Detection

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ABSTRACT

Thiolated silver nanoparticles embedded in a chitosan matrix (AgNPs-SH@Cts) were developed as an electrochemical sensor for mercury detection using an ultrasound-assisted green synthesis approach. This study aimed to improve the stability of AgNPs against aggregation and oxidation and to evaluate the effects of dithizone, cysteine, and glutathione on their physicochemical characteristics and sensing performance toward Hg^{2+} .

AgNPs were synthesized using *Hydrocotyle umbellata* and *Centella asiatica* extracts under varied sonication times and temperatures, as indicated by localized surface plasmon resonance peaks at 400–500 nm. TEM images showed spherical particles with average diameters of 7.39, 5.43, and 4.49 nm for AgNPs stabilized with dithizone, cysteine, and glutathione, respectively. Successful synthesis was confirmed by FTIR, XRD, morphological characterization, and EDX analysis, which identified Ag, S, and N.

The sensors were fabricated on screen-printed electrodes (SPEs) using a drop-casting method. Cyclic voltammetry using 0.005 M $[\text{Fe}(\text{CN})_6]^{3-/4-}$ demonstrated enhanced electrochemical performance associated with an increased electrochemically active surface area. Thiolation increased the anodic peak current by 423%–515% and altered the cathodic peak current by 331%–502%.

The modified SPEs exhibited good stability over ten repeated measurements and high selectivity toward Hg^{2+} in the presence of various interfering ions. The sensors showed an average linearity of 0.9979, sensitivities of 1.20 ± 0.03 – 2.12 ± 0.07 $\mu\text{A/ppb}$, and detection limits of 0.15–0.20 ppb. Water sample analysis yielded relative standard deviations of 2.27%–8.12% and recoveries of $(82.93 \pm 0.02$ – $102.35 \pm 0.09)\%$. The reusable AgNPs-SH@Cts-modified SPEs provide a sensitive, selective, stable, and environmentally friendly platform for mercury detection.

Keywords: thiolated silver nanoparticles; chitosan; screen-printed electrodes; electrochemical sensor; mercury ions; square-wave anodic stripping voltammetry