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Cysteine-Capped Silver Nanoparticles as a Probe for Colorimetric Sensing of 3-Monochloropropane-1,2-Diol

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Abstract

This study reports the synthesis and application of cysteine-capped silver nanoparticles (Cys-AgNPs) as a rapid colorimetric probe for the detection of 3-monochloropropane-1,2-diol (3-MCPD). The nanoparticles were successfully produced, as indicated by a distinct yellow coloration and a single surface plasmon resonance band at 420 nm, confirming the formation of small, monodisperse, and well-stabilized AgNPs. The sensing performance was strongly dependent on temperature, with rapid color development achieved at 100 °C, enabling efficient detection within minutes. Under optimized conditions (pH 9.3, ~100 °C), the sensor exhibited a linear response for concentrations of 1–5 μg·mL^{-1}, described by the regression equation $\Delta A = 0.00389C - 0.00090$ ($R^2 = 0.9777$). The method demonstrated good analytical sensitivity, with an LOD of 0.31 μg·mL^{-1} and an LOQ of 1.03 μg·mL^{-1}. These findings highlight the potential of Cys-AgNPs as a simple, rapid, and cost-effective nanosensing platform for preliminary 3-MCPD monitoring in food-related matrices.

Keywords: Cys-AgNPs; 3-MCPD; colorimetric sensor; silver nanoparticles; food safety.

Cite This Article

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INTRODUCTION

3-Monochloropropane-1,2-diol (3-MCPD) is a heat-induced food contaminant commonly found in refined edible oils and various processed foods This compound is formed during high-temperature processing in the presence of chloride ions and has been associated with several adverse health effects, including kidney and testicular damage. Due to its potential toxicity and widespread occurrence, the presence of 3-MCPD in foods has raised serious concerns regarding consumer safety, prompting regulatory bodies to establish strict limits for its allowable concentration. Conventional analytical methods such as gas chromatography–mass spectrometry (GC-MS) and liquid chromatography-based techniques remain the reference standards for 3-MCPD detection. GC-MS methods employing derivatization such as phenylboronic acid derivatization or AOAC Official Method 2000.01 offer excellent sensitivity, with detection limits as low as 0.001–0.005 mg/kg. Modified solvent extraction protocols, such as those derived from the DGF C-III 18 method, have further improved recovery efficiency at low concentrations. Other approaches, including electrochemical sensors using zero-valent iron-modified electrodes, fluorescence systems based on quenching mechanisms, Fe-Mn-MOF electrochemical platforms, and molecularly imprinted polymer (MIP)-based nanoanalytical systems, have been explored to provide portable, rapid, and cost-effective alternatives. While these methods provide notable advantages, many still require complex sample preparation, expensive equipment, or lack validation in real-world food matrices.

Colorimetric sensing has emerged as a promising strategy for detecting food contaminants due to its operational simplicity, low cost, and ability to provide rapid and visually interpretable results. Colorimetric assays produce a visible color change upon interaction with the target analyte, enabling detection by the naked eye or with simple optical devices. The incorporation of metal nanoparticles, particularly silver nanoparticles (AgNPs), has significantly enhanced the sensitivity of colorimetric sensors. This enhancement is attributed to the unique surface plasmon resonance (SPR) properties of AgNPs, which respond strongly to changes in the local chemical environment and particle aggregation.

Cysteine-capped silver nanoparticles (Cys-AgNPs) offer additional benefits for colorimetric applications. The thiol group of cysteine binds strongly to the silver surface, improving nanoparticle stability in aqueous media, while its amine and carboxyl functional groups provide specific binding sites for target analytes. Interaction between cysteine and analytes such as heavy metals or organic contaminants can induce changes in the SPR band of the AgNPs, leading to distinct and measurable color shifts. These properties have enabled the use of Cys-AgNPs in detecting a variety of contaminants with high sensitivity and selectivity.

Despite these advancements, several research gaps remain. A comprehensive comparison of available analytical methods is still needed to determine suitability across diverse food matrices. Additionally, the long-term stability and reproducibility of nanoparticle-based sensors require further investigation, particularly under varying storage and operational conditions. Matrix effects and potential interferences in complex food samples also pose significant challenges that must be addressed to ensure reliability. Finally, opportunities exist for integrating colorimetric sensors with smart technologies to enhance real-time monitoring and traceability in food safety systems.

Therefore, the present study aims to develop and evaluate a cysteine-capped silver nanoparticle-based colorimetric sensor for the rapid detection of 3-MCPD. This work focuses on elucidating the interaction mechanism, assessing the sensor's sensitivity and selectivity, and demonstrating its applicability for on-site screening of 3-MCPD in edible oil matrices. The proposed method is expected to contribute to more accessible and efficient monitoring tools for ensuring food safety.

METHODS

The study was conducted in the Final Project Laboratory, Faculty of Science and Technology, Universitas Jambi. Chemicals used included silver nitrate ($AgNO_3$), L-cysteine, trisodium citrate, 3-MCPD standard, hexane–acetone (1:1 v/v), and distilled water. Interference studies employed 2-MCPD, glycidol, propylene glycol, ethylene glycol, and glycerol. Commercial palm cooking oil was used as the sample matrix.

Research Design

This study employs an experimental laboratory design to develop and evaluate a colorimetric sensing system based on cysteine-capped silver nanoparticles (Cys-AgNPs) for detecting 3-monochloropropane-1,2-diol (3-MCPD). The research includes synthesis of nanomaterials, characterization of their optical properties, optimization of sensing conditions, and performance evaluation using standard analytical parameters.

Research Target/Subject

The targets of this study include Cysteine-capped silver nanoparticles (Cys-AgNPs) as sensing probes. 3-MCPD standard solutions prepared at various concentrations for calibration.

Research Procedure

Synthesis of Cysteine-Capped Silver Nanoparticles (Cys-AgNPs)

Cys-AgNPs were synthesized using a modified Turkevich method. A 100 mL $AgNO_3$ solution (100 ppm) was heated to 100 °C under constant stirring, followed by addition of 6 mL L-cysteine (100 µM) and stirring for 5 min. Subsequently, 15 mL trisodium citrate (0.25% w/v) was added as a reducing agent, and the mixture was stirred for 60 min. Formation of AgNPs was indicated by a color change from pale yellow to brownish-grey. The product was characterized using UV–Vis spectroscopy (350–800 nm), FTIR, and PSA.

Optimization of 3-MCPD Detection

A mixture of 1 mL 3-MCPD standard (0.25 µg/mL) and 50 µL Cys-AgNPs was vortexed and heated at 25–125 °C. Absorbance at ~450 nm was used to determine the optimal temperature.

Using the optimal temperature, reaction times of 1–15 min were evaluated. The condition giving the highest absorbance change was selected as optimal.

Colorimetric Detection of 3-MCPD

For detection, 1 mL 3-MCPD standard or sample extract was mixed with 50 µL Cys-AgNPs, vortexed for 2 min, and heated at 100 °C for 5 min. Color change and absorbance (~450 nm) indicated nanoparticle aggregation. A calibration curve was constructed using 0–3 µg/mL 3-MCPD. The response was calculated as $\Delta A = (A_0 - A_i)$, where A_0 is the absorbance of blank Cys-AgNPs and A_i is absorbance after reaction

Instruments, and Data Collection Techniques

Instruments used include UV–Vis spectrophotometer for absorbance measurement, Glassware and pH meter. Data collected consist of Absorbance spectra (400–800 nm), Calibration data from standard 3-MCPD solutions.

Data analysis technique

The data obtained in this study were analyzed through several integrated approaches. Spectral data from UV–Vis measurements were examined by observing shifts in the λ_max and changes in absorbance intensity after the addition of 3-MCPD, which reflect the degree of nanoparticle aggregation. Image-based data derived from smartphone photographs were processed by converting the RGB values into quantitative analytical responses. To reduce variation due to illumination and camera settings, the RGB signals were normalized prior to further analysis.

Analytical performance was evaluated by constructing a calibration curve using linear regression, followed by calculating the limit of detection (LOD) and limit of quantification (LOQ) based on the standard deviation of the blank and the slope of the regression line. Method precision was determined through the percentage of relative standard deviation (%RSD) across replicate measurements, while accuracy was assessed using recovery tests from spiked samples.

All results were then interpreted to establish the relationship between nanoparticle aggregation behavior and the observed colorimetric response. The analytical capabilities of the developed method were further compared with previously reported studies and existing regulatory detection limits to contextualize the relevance and applicability of the proposed sensing system.

RESULTS AND DISCUSSION

The successful synthesis of cysteine-capped silver nanoparticles (Cys-AgNPs) was evidenced by a distinct change in solution color from nearly colorless to yellow, accompanied by the appearance of a well-defined surface plasmon resonance (SPR) band centered at 420 nm in the UV–Vis spectrum. The presence of a single, peak reflects the formation of predominantly spherical and uniformly dispersed nanoparticles, indicating that the particles exhibit a narrow size distribution with minimal aggregation. This spectral feature aligns with earlier reports showing that monodisperse, spherical AgNPs typically display a single SPR maximum within this region.

The lack of peak broadening or multiple absorption bands as seen in figure 1 suggests that cysteine effectively stabilizes the nanoparticle surface, preventing uncontrolled growth or agglomeration during synthesis. Since increases in particle size generally lead to red-shifts and the expansion of the SPR band, the intense and symmetrical peak observed in this study supports the formation of small, stable nanoparticles. The role of cysteine as both a capping and stabilizing ligand contributes to enhanced particle uniformity, consistent with previous findings that thiol-containing ligands promote tighter control over nanoparticle size and dispersion.

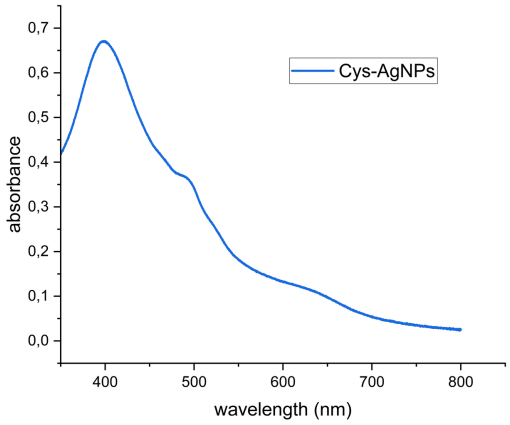

Figure 1. UV-Vis spectrum of cys-AgNPs

Optimum conditions for 3-MCPD detection

To determine the optimal conditions for 3-MCPD detection using the Cys-AgNPs system, the effect of temperature was evaluated at three levels: room temperature (28 °C), 50°C, 75 °C, and 100 °C, using a 0.25 μg·mL^{-1} 3-MCPD solution. The results as seen in figure 2 clearly show that the color response is strongly influenced by temperature. At room temperature, a noticeable color change appeared only after approximately five days, whereas heating the mixture to 80 °C reduced the reaction time to about 20 minutes. A much faster response was observed at 100 °C, where the solution turned pink within just five minutes.

These observations confirm that the interaction between 3-MCPD and Cys-AgNPs is highly temperature-dependent. Given the significantly accelerated color development at 100 °C, this temperature was selected for subsequent experiments to enable rapid and efficient detection.

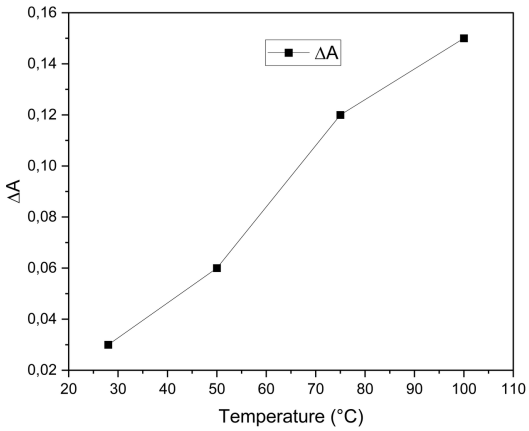

Figure 2. Effect of temperature to absorbance intensity change

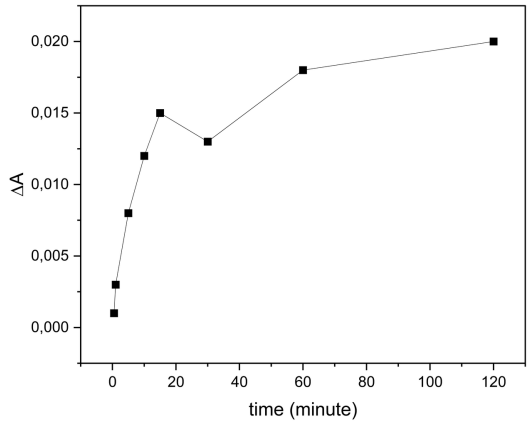

Figure 3. Effect of incibation time to absorbance intensity change

Analytical Performance of Cys-AgNPs foR 3-MCPD detection

A series of 3-MCPD concentrations ranging from 1 to 5 μg·mL^{-1} was introduced into the Cys-AgNP system to characterize its analytical response under optimized conditions (pH 9.3, ~100 °C, 120 min). As shown in Fig. 3B, the initially yellow nanoparticle suspension gradually shifted to a pink hue upon exposure to 3-MCPD. At most concentrations, this transformation occurred within the five-minute heating interval, whereas the lowest level (0.1 μg·mL^{-1}) required slightly more time before the color change became noticeable.

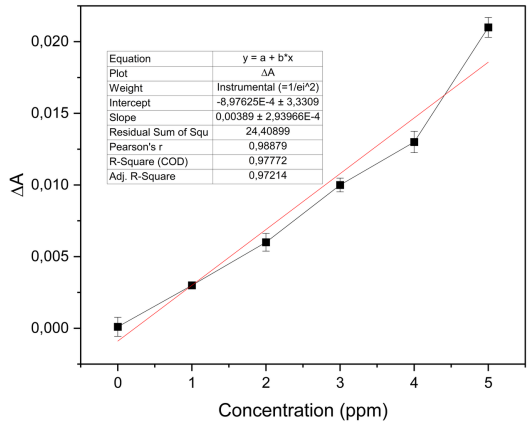

Figure. 4 effect of concentration to intensity change

For quantitative evaluation, the absorbance change (ΔA) was calculated as the difference between the initial Cys-AgNPs absorbance (A_0) and the absorbance after interaction with 3-MCPD (A_i). A linear calibration curve was obtained within the range of 1–5 μg·mL^{-1}, yielding the regression equation:

$$\Delta A = 0.00389C - 0.00090$$

with a correlation coefficient of R^2 = 0.9777, indicating good linearity across the tested range.

The standard deviation obtained from replicate measurements of the blank (0 μg·mL^{-1}), estimated from the error bars in the calibration graph, was s ≈ 0.00040. Using the standard criteria LOD = 3s/S and LOQ = 10s/S, the calculated performance metrics were

LOD=0.31 ppm and LOQ=1.03 ppm. These values demonstrate that the Cys-AgNPs probe exhibits good analytical sensitivity, with sufficient capability to detect 3-MCPD at low $\mu g \cdot mL^{-1}$ levels.

CONCLUSIONS

Cysteine-capped silver nanoparticles were successfully synthesized and applied as a rapid colorimetric probe for 3-MCPD detection. The system exhibited a clear temperature-dependent response, with 100 °C enabling efficient and accelerated color development. Under optimized conditions, the sensor produced a linear analytical response within 1–5 $\mu g \cdot mL^{-1}$, with an LOD of 0.31 $\mu g \cdot mL^{-1}$ and an LOQ of 1.03 $\mu g \cdot mL^{-1}$. These results demonstrate that Cys-AgNPs offer a sensitive and straightforward approach for preliminary 3-MCPD screening, supporting their potential use in rapid food safety assessments..

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AUTHOR CONTRIBUTIONS

Conceptualization, Martina Asti Rahayu and Dhian Eka Wijaya; Methodology, Martina Asti Rahayu; Software, Muhammad Irhash Shalihin; Validation, Martina Asti Rahayu, Dhian Eka Wijaya, and Ayu Nadila Safitri; Formal Analysis, Martina Asti Rahayu and Ayu Nadila Safitri; Investigation, Martina Asti Rahayu and Ayu Nadila Safitri; Resources, Dhian Eka Wijaya; Data Curation, Martina Asti Rahayu and Ayu Nadila Safitri; Writing – Original Draft Preparation, Martina Asti Rahayu; Writing – Review & Editing, Martina Asti Rahayu and Ayu Nadila Safitri; Visualization, Muhammad Irhash Shalihin; Supervision, Dhian Eka Wijaya; Project Administration, Dhian Eka Wijaya; Funding Acquisition, Dhian Eka Wijaya.

CONFLICT OF INTEREST

The authors declare that they have no known financial or personal conflicts of interest that could have appeared to influence the work reported in this study.

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